

Effect of External Pressure on the Efficacy of Ultrasonic Pretreatment of Sludge

Ngoc Tuan LE ⁺, Berthe RATSIMBA, Carine JULCOUR-LEBIGUE and Henri DELMAS

Universit éde Toulouse, Laboratoire de Génie Chimique, INP-ENSIACET, 31030 Toulouse, France

Abstract. The objective of this work was to optimize the *ultrasonic (US) pretreatment of sludge*, which is known to enhance sludge disintegration and to facilitate anaerobic digestion. Several main parameters related to the processing efficiency were varied for this purpose: total solids content of sludge (TS), thermal operating conditions (adiabatic vs. isothermal), specific energy input (ES), and for the first time external pressure. Five different TS concentrations of sludge (12-36 g/L) were tested with different values of ES (7000-75000 kJ/kg_{TS}) and 28 g/L was found as the optimum value according to the soluble chemical oxygen demand in the liquid phase (SCOD). The effect of external pressure was investigated in the range of 1-16 bar under controlled temperature (28 °C): an optimum pressure of 2 bar was found at all the ES values, which improved sludge disintegration efficacy (DD_{COD}) by 22 to 67% compared to that at atmospheric pressure. Mean particle diameter (D[4,3]) decreased from 408 µm to about 95-100 µm regardless of the conditions, and this size reduction was much faster than COD extraction. Finally, different thermal operating conditions, with and without temperature control, proved adiabatic US to be more efficient than isothermal US with again an optimal pressure of 2 bar.

Keywords: Mixed sludge, Ultrasonic pretreatment, External pressure, Disintegration of sludge

1. Introduction

Incineration, ocean discharge, land spreading and composting are the most common sludge disposal options used over the years, but they are no longer reliable due to economic reasons or negative impacts on environment. Therefore, anaerobic digestion (AD) of sludge has been applied as an efficient and sustainable technology for sludge treatment. However, hydrolysis is known as the rate-limiting step of microbial conversion, requiring a pretreatment of sludge which ruptures the cell wall and facilitates the release of intracellular matters into the aqueous phase to enhance the AD.

Pilli *et al.* [1] reported that ultrasound (US) irradiation is a feasible and promising mechanical disruption technique for sludge disintegration and microorganisms lyses because of improvement in biodegradability and bio-solids quality [2], increase in methane production [2,3,4], no chemical additives [5], less sludge retention time [6], and reduction of sludge volume [4].

Changing the hydrostatic pressure will modify the resonance conditions of cavitation bubbles through their equilibrium radius [7]. Operating at resonance conditions increases the rate and yield of reactions [8, 9, 10]. In short, increasing the external pressure leads to both an increase in the cavitation threshold and the intensity of cavity collapse [11]. Nevertheless, most US experiments have been carried out at atmospheric pressure, and only a few studies have been focusing on how increasing static pressure affects cavitation. Moreover, to our knowledge, the effect of pressure on sludge pretreatment has hardly been investigated.

Most works on pressure effects concern sonoluminescence. The findings by Finch (1955) cited by Chendke and Fogler [12] indicated that the greatest sonoluminescence intensity was observed in water at a static pressure of about 1.5 atm when varying in the range of 1-8 atm. Chendke and Fogler [12]

⁺ Corresponding author. Tel. : +33 (0)5 34 32 36 96; Fax: +33 (0)5 34 32 36 97.
E-mail address: ngoctuan.le@ensiacet.fr (LE Ngoc Tuan)

recommended a value of 6 atm to promote sonoluminescence in nitrogen-saturated water. In aqueous carbon tetrachloride solutions, the intensity of the sonoluminescence did not show any monotonous behavior over the range of 1-20 atm: it first rose up to 6 atm, then reached a minimum at 8 atm, got a new maximum value at 12 atm, and was finally almost inhibited above 18 atm [13]. Brett and Jellinek (1956) cited by Chendke and Fogler [12] reported the effects of pressure on cavitation bubbles; thereby cavitation bubbles could be visible for gas-applied pressure as high as 16 atm. Whillock and Harvey [14] investigated the effects of hydrostatic pressure on the corrosion of 304L stainless steel in an ultrasonic field. An increase in hydrostatic pressure up to 4 bar at constant temperature caused a strong increase in corrosion rate. Closer to the present subject, Neppiras and Hughes [15] investigated the influence of pressure (up to 5.8 atm) on the disintegration of yeast cells and found an optimum value of 4 atm.

Following these researches, static pressure seems to be an important parameter which has not been often investigated due to the requirement of more complex equipments. In case of sludge pretreatment, external pressure should be varied simultaneously with other related parameters, including total solids content of sludge (TS), specific energy input (ES) in order to compare, assess, and select optimal conditions for actual application. The effect of ultrasound will be presented in terms of disintegration degree (organic matters solubilization) and particle size reduction. The objective of this work is to optimize high-power low-frequency ultrasonic pretreatment of sludge, and especially to emphasize on static pressure, which is expected to enhance sludge disintegration and to facilitate the AD.

2. Materials and Methods

2.1. Sludge Samples

Mixed sludge was collected from Ginestous wastewater treatment plants (Toulouse, France) with a sufficient amount for conducting all experiments in this study. Its properties, given in Table 1, were evaluated according to standard analytical methods (see § 2.3). It was sampled in 100 g plastic boxes, and preserved in a freezer. This preliminary maintaining step might change some physical characteristics of the sludge, but it would not significantly affect COD solubilization results [16]. When performing experiments, the required amount of sludge was defrosted and diluted with distilled water to prepare synthetic sludge samples with TS contents of 12, 24, 28, 32, and 36 g/L, respectively.

Table 1: Characteristics of the initial sludge sample

Parameter	Value
Total solids (TS) (%)	28.5
Volatile solids (VS) (%)	23.8
VS/TS (%)	83.5

2.2. Ultrasound Application

Ultrasonication was emitted by a cup-horn ultrasound unit included in an autoclave reactor which was connected to a pressurized N₂ bottle. The ultrasound system had a fixed frequency of 20 kHz and a maximum total power of 200 W corresponding to an ultrasonic power (P_{US}) of 158 W. For each TS concentration, a constant volume of synthetic sludge sample (500 mL) was poured into the stainless steel reactor. US tests were performed at constant P_{US} of 150 W, close to the maximum, as it was verified by Kidak *et al.* [16] that high power and short time should be preferred for a given ES. Five different sonication time ranges corresponding to five values of ES (7000, 12000, 35000, 50000, and 75000 kJ/kg_{TS}) were tested. A cooling water stream (15 °C) was continuously circulated in an internal coil to maintain a constant temperature (T) of the solution at 28±2 °C during sonication. In some experiments, adiabatic behavior was also checked. Conjugated effects of ultrasound and temperature (which was increased due to US) on the release of organic matters in solution were then investigated.

During the experimental procedure, the effect of TS content (12-36 g/L) was looked into first; subsequently, the effect of temperature was examined. Blank experiments (without US) were also conducted to understand effects of stirring and of temperature. Finally, the effect of external pressure (1-16 bar) on

ultrasonic pretreatment of sludge was evaluated in conditions of optimum value of TS, controlled T, P_{US} of 150 W, and ES in the range of 0-75000 kJ/kg_{TS}.

2.3. Analytical Methods

First *total and volatile solids contents* (TS and VS respectively) were measured according to the following procedure (APHA, 2005). A well-mixed sample was evaporated in a weighed dish and dried to a constant weight in an oven at 105 °C. The TS was the increase in weight over that of the empty dish. The residue was followed by incineration to a constant weight at 550 °C. The remaining solids represented the fixed solids (FS) while the difference between the TS and FS represented the volatile solids (VS).

The *degree of sludge disintegration* (DD_{COD}) was calculated by determining the soluble chemical oxygen demand after alkaline disintegration of sludge ($SCOD_{NaOH}$) and the chemical oxygen demand in the supernatant before and after treatment ($SCOD_0$ and $SCOD$ respectively):

$$DD_{COD} = (SCOD - SCOD_0) / (SCOD_{NaOH} - SCOD_0) * 100 (\%) [17];$$

To measure the $SCOD_{NaOH}$ value, used as the reference to evaluate the efficiency of organic matters solubilization under US, the sludge sample was mixed with 0.5M NaOH at room temperature for 24 h [18].

Prior to SCOD determination, the supernatant liquid obtained after sedimentation was filtered under vacuum using a 0.2 µm pore-size cellulose nitrate membrane. The filtered liquid was subjected to COD analysis as per Hach spectrophotometric method. The change in the SCOD indirectly represents the quantity of organic carbon which has been transferred from the solid phase into the liquid phase of the solution.

The *particle size distribution* of sludge before and after US was determined using a Malvern particle size analyzer (Mastersizer 2000, Malvern Inc.).

3. Results and Discussion

3.1. Effect of TS Concentration on DDCOD

Five synthetic sludge samples, S12, S24, S28, S32, and S36 corresponding to 12, 24, 28, 32, and 36 g/L of TS, respectively, were treated under P_{US} of 150 W (0.3 W/mL ultrasonic density) without any temperature control. The respective ES was varied (7000, 12000, 35000, 50000, and 75000 kJ/kg_{TS}) via the sonication time. The effect of TS concentration on sludge disintegration was derived from the release of intercellular substances measured as SCOD and DD_{COD} . The results are presented in Fig. 1.

In fact, SCOD gradually increased with sonication time (0-150 min), but not linearly. In addition, the relation between SCOD and TS content was not simple as the best DD_{COD} was not found at maximum TS, which complies with Zhang *et al.* [19]. For example, with an ES value of 7000 kJ/kg_{TS}, when TS of sludge increased from 12, 24 to 36g/L, SCOD changed by 2.57 and 3.44 folds, respectively.

As shown in Fig. 1, the higher the concentration of TS was in the range of 12-28 g/L, the higher DD_{COD} was at a given ES. When TS was increased out of this range (32 and 36 g/L), DD_{COD} gradually deteriorated. In agreement with other researchers [1, 5, 16, 19-21], an optimum value of TS for efficient disintegration of sludge by ultrasound was therefore observed. In this work, the corresponding value was 28 g/L.

This could be explained by opposite effects. The increase in TS provides more cells and aggregates to be in contact with cavitation bubbles; thereby, the US power input, which is required to generate cavitation (threshold), is more efficiently consumed. Nevertheless, at higher sludge loading, the acoustic pressure field will decrease faster from the emitter due to the degraded propagation of the ultrasonic wave in a denser suspension. Consequently, acoustic cavitation intensity will be reduced. These two opposite effects lead to an optimum TS concentration which could slightly depend on sludge characteristics, reactor design, and US power. In other words, in case TS concentration exceeds the optimum value, the ultrasonic wave cannot be evenly propagated into the medium, and its efficiency is only kept in a small volume near the emitter. Subsequently, DD_{COD} goes down because of the attenuation effect [1, 5, 16].

3.2. Effect of Temperature on DDCOD

The ultrasonic pretreatment has two simultaneous effects: (i) extreme macro and micro agitation caused by the cavitation, and (ii) increase in the bulk temperature. To separately evaluate their effects, four operating procedures were carried out: (1) without US + controlled T (28°C), (2) US + controlled T (28°C), (3) US + uncontrolled T (no cooling), (4) without US + progressive increase of T up to 77°C (with same T rise profile as found with US in (3) to see the effect of thermal hydrolysis). Results are illustrated in Fig. 2.

After about 2 hours of stirring at a constant temperature and without US, DD_{COD} in this sample was very low (0.3%), which indicated that the stirrer played a main role in making a homogeneous solution, but did not significantly affect the release of COD. At all observed time ranges, DD_{COD} values under adiabatic sonication were the highest, followed by those obtained under low temperature sonication and thermal hydrolysis. DD_{COD} values of sonicated samples under controlled T (28 °C) were about half of those obtained without cooling.

In accordance with recent works [16, 18, 22], the higher the temperature of sludge samples was, the higher the ultrasonic disintegration efficiency was. This is opposite to most power US applications as cavitation intensity is higher at low temperature. It is then clear that ultrasonic disintegration of sludge is the result of two different effects: the specific cavitation effect and the thermal effect. Despite lower performances, next experiments were conducted under controlled T condition in order to have a clear understanding of US effect under different values of static pressure.

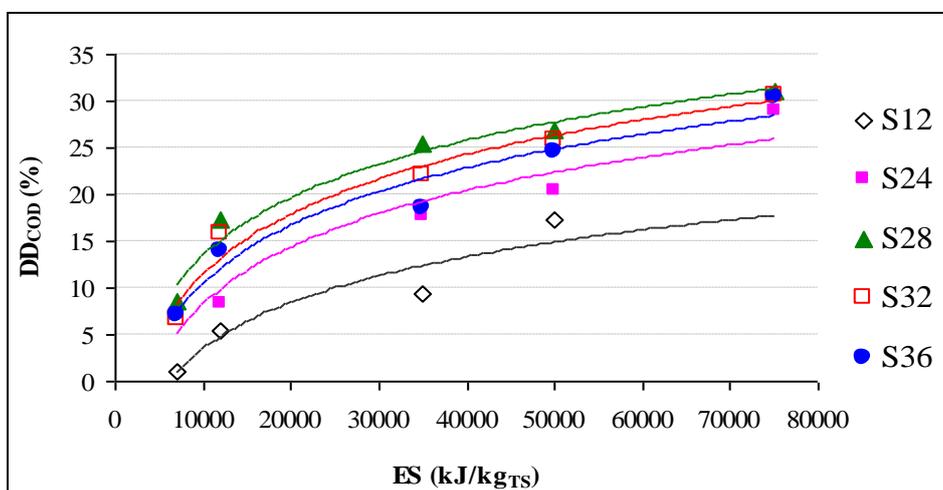


Fig. 1: Effect of TS content on sludge disintegration (DD_{COD}): P_{US} of 150 W, V of 500 mL, uncontrolled T, atmospheric pressure

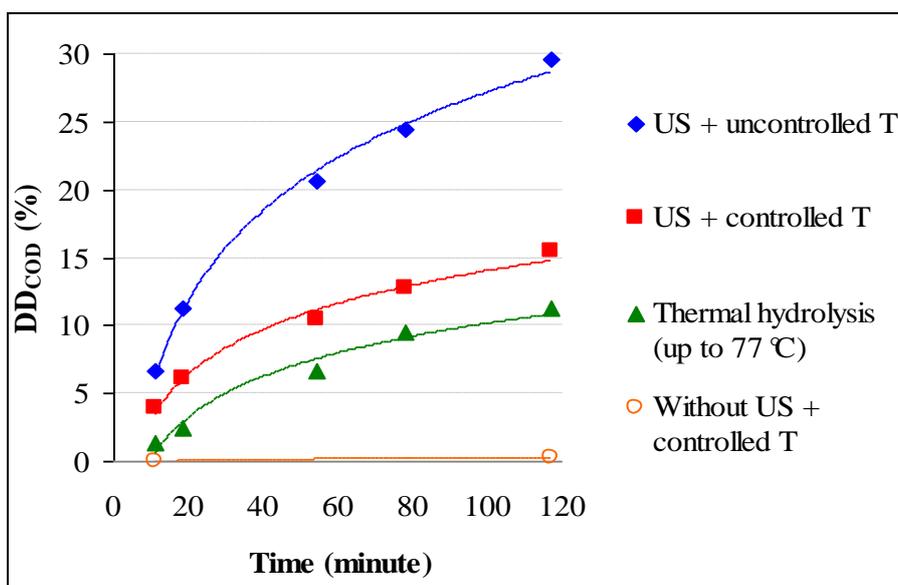


Fig. 2: Effect of temperature control on sludge disintegration (DD_{COD}): P_{US} of 150 W (when under US), V of 500 mL, TS content of 28 g/L, atmospheric pressure

3.3. Effect of External Pressure on DDCOD

Experiments used to assess the effect of the external pressure (1-16 bar) on the efficacy of ultrasonic pretreatment of sludge were deployed for the following conditions: optimum TS of 28 g/L, volume of 500 mL, controlled T, P_{US} of 150W, and ES in the range of 0-75000 kJ/kg_{TS}. Results are demonstrated in Fig. 3.

The curves corresponding to the five different ES values show the same trends in terms of DD_{COD} . All maximum values of DD_{COD} were observed at an external pressure of 2 bar. As aforementioned, changing the external pressure would change the resonance conditions of cavitation bubbles [7] and increase the cavitation threshold. At higher static pressure, the acoustic pressure must then be increased to obtain cavitation; and cavitation intensity might be higher at that time. However, at a given US intensity, too high static pressure prevents bubble formations, cavitation, and then sludge ultrasonic disintegration.

Compared with experiments carried out at atmospheric pressure, sludge disintegration efficacy was significantly improved when conducted at 2 bar, the optimum pressure. The corresponding increase in DD_{COD} was by 67%, 36%, 27%, 23%, and 22% with ES values of 7000, 12000, 35000, 50000, and 75000 kJ/kg_{TS}, respectively. This approach might lead to energy savings in sludge pretreatment applications with ultrasound. For instance, when operating at the optimum pressure, DD_{COD} obtained with ES of 7000, 35000, and 50000 kJ/kg_{TS} were higher than those at atmospheric pressure with ES of 12000, 50000, and 75000 kJ/kg_{TS}, respectively.

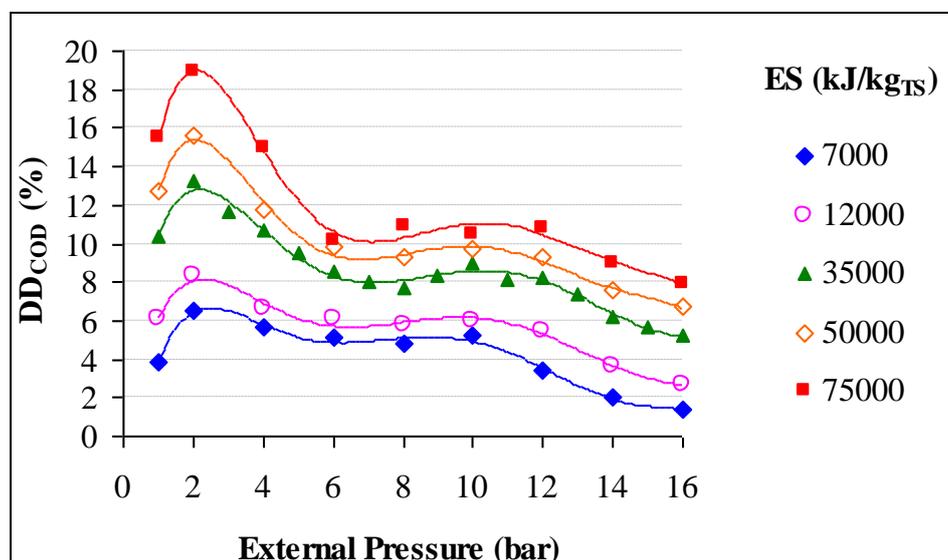


Fig. 3: Effect of external pressure on sludge disintegration (DD_{COD}): P_{US} of 150W, V of 500mL, controlled T (28 °C), TS content of 28 g/L

Further experiments were performed to examine the effect of pressure along with temperature during ultrasonic duration. Experimental conditions were TS content of 28 g/L, P_{US} of 150W, ES of 35000 kJ/kg_{TS}, pressure in the range of 1-16 bar, and with and without temperature control. The results shown in Fig. 4 once again confirmed that the optimum pressure found in this work was about 2 bar regardless of temperature conditions.

However, beyond these maximum values, DD_{COD} decreased subsequently. Noticeably, DD_{COD} significantly deteriorated at pressures over 4 bar (Fig. 3 and 4). This phenomenon which is much more important at ambient temperature (controlled T) could be explained by a lower vapour pressure and then a higher cavitation threshold.

Fig. 5 compares the efficiency of different ultrasonic pretreatment conditions of sludge. All curves show an increase of DD_{COD} with sonication duration. Different conditions brought rather different effectiveness of sludge disintegration which was in the following order: (i) US + uncontrolled T + optimum pressure of 2 bar (DD_{COD} of 7.9-32.8%) > (ii) US + uncontrolled T + atmospheric pressure (DD_{COD} of 6.7-29.6%) > (iii) US + controlled T (28 °C) + optimum pressure of 2 bar (DD_{COD} of 6.5-18.9%) > (iv) US + controlled T + atmospheric pressure (DD_{COD} of 3.9-15.5%). These conditions (ii) and (iii) showed that the effect of sole

pressure was less than that of sole adiabatic condition in association with US. The best condition of ultrasonic pretreatment of sludge (i) was found to be the combination of high P_{US} , low frequency, optimum TS content, adiabatic, and optimum pressure of 2 bar.

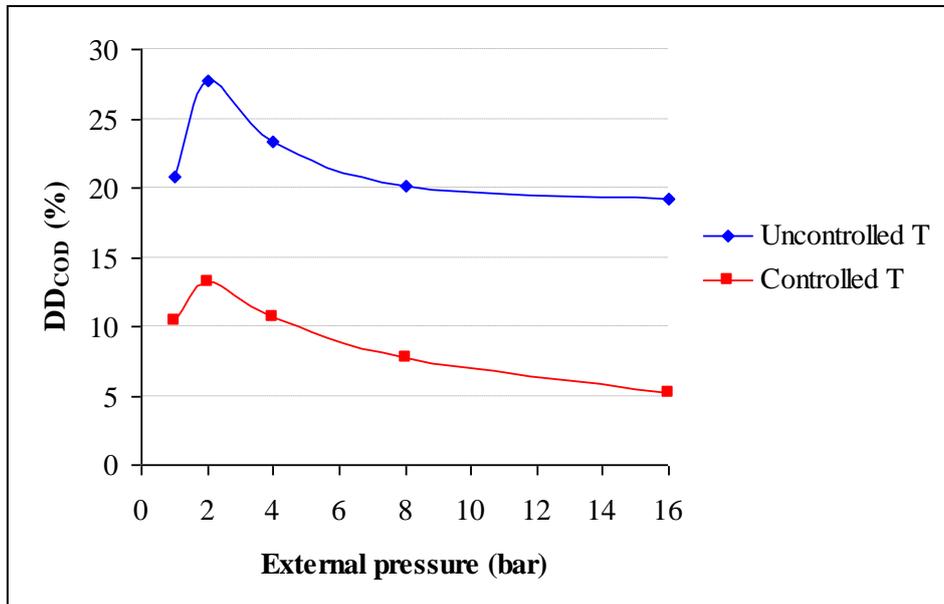


Fig. 4: Effect of external pressure on sludge disintegration (DD_{COD}) under different temperature conditions: P_{US} of 150W, V of 500 mL, TS content of 28 g/L, ES of 35000 kJ/kg_{TS}.

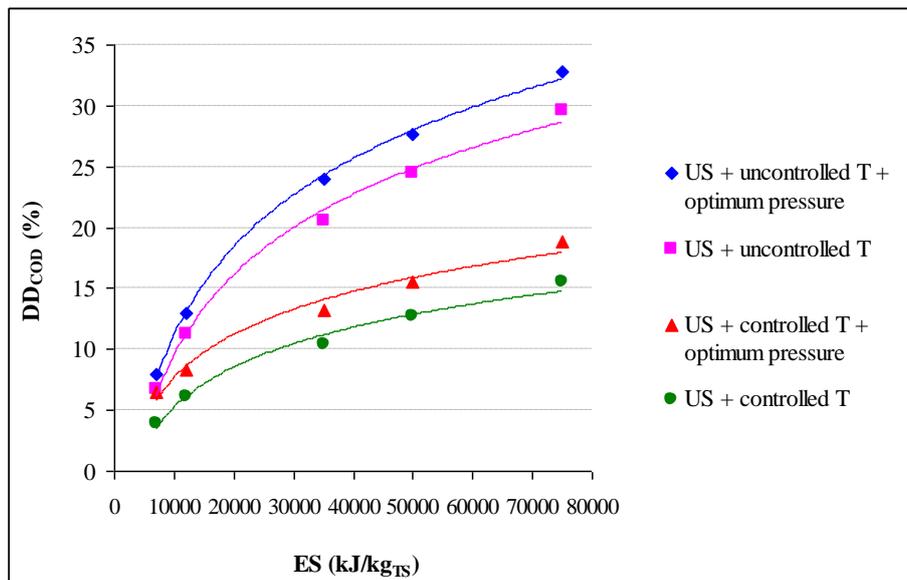


Fig. 5: Comparison of different ultrasonic pretreatment conditions on sludge disintegration (DD_{COD}): P_{US} of 150W, V of 500mL, TS content of 28 g/L

3.4. Particle Size Reduction

Ultrasonic pretreatment is also very effective in reducing the particle size and then sludge volume, which is sometimes used to assess the degree of sludge disintegration and commonly analyzed by laser diffraction. The reduction in particle size accelerates the hydrolysis stage of sludge AD and enhances degradation of organic matters. However, the findings by Muller *et al.* (2004) cited by Dogan [23] indicated that this parameter is not convenient for process optimization.

At given values of TS and at ambient temperature and pressure, the particle size reduction depended on ES through the sonication time. Gonze *et al.* (2003) cited by Pilli *et al.* [1] found that particle size was decreased gradually with the increase in sonication time and a reverse trend occurred after a sonication

period of 10 min due to re-flocculation of the particles. However, this phenomenon was not found in this work. The particle size distribution (PSD) and D[4,3] mean diameter (volume mean diameter) of sonicated sludges are shown in Fig. 6 and Fig. 7, respectively. D[4,3] was reduced from 408 μm to 130, 111, 100, 98 and 95 μm after respective sonication time ranges of 11, 19, 55, 78, and 117 min. In other words, compared with the untreated sludge, particle size was reduced by 68%, 73%, 75%, 76%, and 77% following the subsequent increase in ES: 7000, 12000, 35000, 50000, and 75000 kJ/kg_{TS} , respectively. In agreement with recent works [21, 22], these data indicated that the process of sludge particle size reduction happened very fast and much faster than COD release in the aqueous phase.

In the ES range of 7000 – 75000 kJ/kg_{TS} , d_{90} , d_{50} , and d_{10} values of sludge decreased by 74%, 70% and 58%, respectively. This indicated that different particle sizes had slightly different reduction extents, in which large particles were disrupted more effectively by US than smaller ones due to their larger surface exposed to sonication or to different consistency. This point is similar to conclusions in previous works [1, 21, 24].

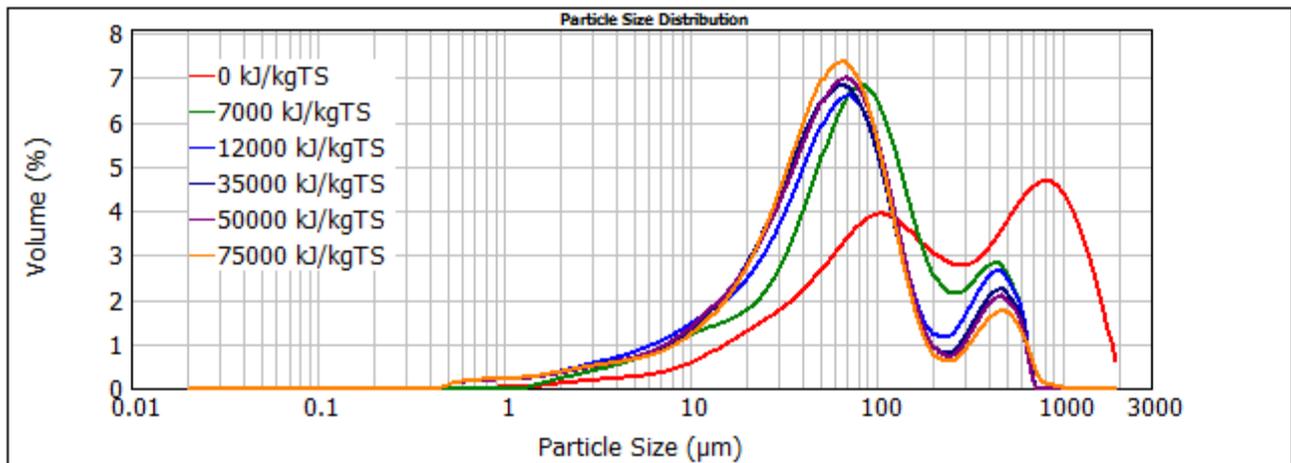


Fig. 6: Evolution of particle size distribution during US pretreatment: $P_{\text{US}}=150 \text{ W}$, $V=500 \text{ mL}$, controlled T ($28 \text{ }^\circ\text{C}$), TS content= 28 g/L , atmospheric pressure.

When US experiments were conducted at optimum pressure, although the kinetics of disruption was faster, the difference in final particle diameter compared to that at atmospheric pressure was negligible. The enhancement of particle size reduction dropped from 9.3% at 7000 kJ/kg_{TS} to less than 1% at 35000-75000 kJ/kg_{TS} .

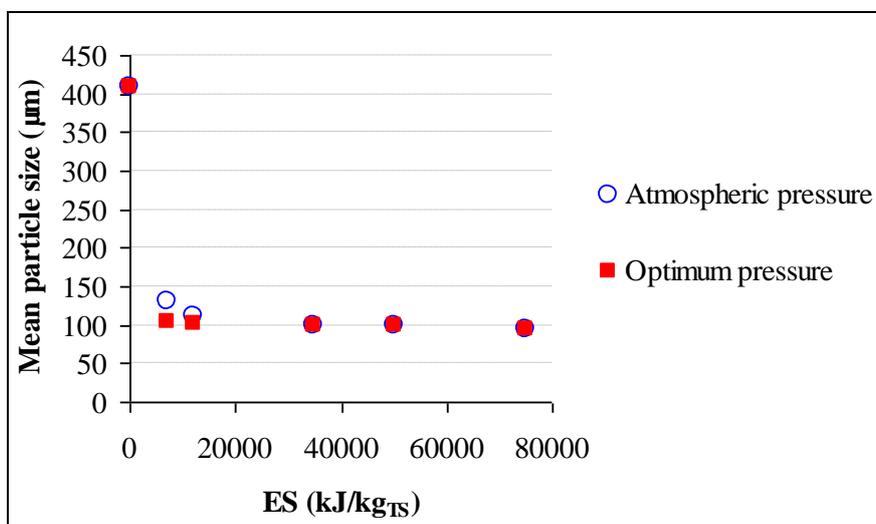


Fig. 7: Mean particle size evolution during US pretreatment (based on D[4,3]): P_{US} of 150 W , V of 500 mL , controlled T ($28 \text{ }^\circ\text{C}$), TS content of 28 g/L .

4. Conclusion

Sludge samples with different TS contents were treated with various sonication time ranges. An initial value of 28 g/L yielded the highest COD release in the aqueous phase and was selected for subsequent experiments.

Different US/temperature combinations were then investigated to evaluate the effect of US. At all observed time ranges, DD_{COD} values were the highest under adiabatic sonication, followed by those obtained under sonication with cooling, and then thermal hydrolysis ones. The effect of US was clearly more important than that of sole thermal hydrolysis. Effect of external pressure in association with US was studied for the first time using pressurized nitrogen in the range of 1-16 bar. DD_{COD} exhibited an optimum with respect to applied pressure of about 2 bar for all sonication time ranges. When conducted at the optimum pressure, sludge disintegration efficacy was improved up to 67% compared to atmospheric pressure in the investigated ES range (7000-75000 kJ/kg_{TS}).

Compared with the untreated sludge, particle size was decreased by 68 to 77% and particles were almost entirely disrupted in the initial period of the ultrasonic process. At 2 bar, the final size was nearly obtained at the first sampling time.

All these data suggest that the best energy efficiency would correspond to very short US exposure at the optimal pressure and under adiabatic condition. The under pressure ultrasonic pretreatment of sludge might offer a potential of energy savings in sludge pretreatment applications with ultrasound.

5. Acknowledgment

The authors acknowledge the financial support from the Ministry of Education and Training of Vietnam and Institut National Polytechnique of Toulouse (France). They also thank Alexandrine BARTHE (Ginestous), Ignace COGHE, Jean-Louis LABAT, Jean-Louis NADALIN (LGC), and Christine REY-ROUCH (SAP, LGC) for technical and analytical support.

6. References

- [1] Sridhar Pilli, Puspendu Bhunia, Song Yan, R.J. LeBlanc, R.D. Tyagi, R.Y. Surampalli, Ultrasonic pretreatment of sludge: A review, *Ultrasonics Sonochemistry* 18 (2011) 1–18
- [2] S.K. Khanal, D. Grewell, S. Sung, J. Van Leeuwen, Ultrasound applications in wastewater sludge pretreatment: A review, *Crit. Rev. Environ. Sci. Technol.* 37 (2007) 277–313.
- [3] W.P.Barber, The effects of ultrasound on sludge digestion, *J. Chart. Inst. Water Environ. Manage.* 19 (2005) 2–7.
- [4] T.I. Onyeche, O. Schlafer, H. Bormann, C. Schroder, M. Sievers, Ultrasonic cell disruption of stabilised sludge with subsequent anaerobic digestion, *Ultrasonics* 40 (2002) 31–35.
- [5] T. Mao, S.Y. Hong, K.Y. Show, J.H. Tay, D.J. Lee, A comparison of ultrasound treatment on primary and secondary sludges, *Water Sci. Technol.* 50 (2004) 91–97.
- [6] A. Tiehm, K. Nickel, U. Neis, The use of ultrasound to accelerate the anaerobic digestion of sewage sludge, *Water Sci. Technol.* 36 (1997) 121–128
- [7] L. H. Thompson and L. K. Doraiswamy, REVIEWS - Sonochemistry: Science and Engineering, *Ind. Eng. Chem. Res.* 38 (1999) 1215-1249
- [8] G. Cum, R. Gallo, and A. Spadaro, 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. II* (1988)
- [9] G. Cum, R. Gallo and A. Spadaro, Temperature Effects in Ultrasonically Activated Chemical Reactions, *Il Nuovo Cimento Vol. 12 D, N. 10*, (1990)
- [10] G. Cum, G. Galli, R. Gallo and A. Spadaro, Role of frequency in the ultrasonic activation of chemical reactions, *Ultrasonics Vol 30 No 4*, (1992)
- [11] John P. Lorimer and Timothy J. Mason, Sonochemistry: Part 1-The Physical Aspects, *Chem. Soc. Rev.* 16 (1987), 239-274

- [12] P. K. Chendke and H. S. Fogler, Effect of Static Pressure on the Intensity and Spectral Distribution of the Sonoluminescence of Water, *J. Phys. Chem.*, 87, (1983b) 1644-1648
- [13] P. K. Chendke and H. S. Fogler, Sonoluminescence and Sonochemical Reactions of Aqueous Carbon Tetrachloride Solutions, *J. Phys. Chem.* 87, (1983a) 1362-1369
- [14] G.O.H. Whillock, B.F. Harvey, Ultrasonically enhanced corrosion of 304L stainless steel I: The effect of temperature and hydrostatic pressure, *Ultrasonics Sonochemistry* 4 (1997) 23-31
- [15] E. A. Neppiras and D. E. Hughes, Some Experiments on the Disintegration of Yeast by High Intensity Ultrasound, *Biotechnology And Bioengineering VOL VI*, (1964) 247-270
- [16] Rana Kidak, Anne-Marie Wilhelm, Henri Delmas, Effect of process parameters on the energy requirement in ultrasonical treatment of waste sludge, *Chemical Engineering and Processing* 48 (2009) 1346–1352
- [17] U. Schmitz, C.R. Berger, H. Orth, Protein analysis as a simple method for the quantitative assessment of sewage sludge disintegration, *Water Res.* 34 (2000) 3682–3685.
- [18] L. Huan, J. Yiyang, R.B. Mahar, W. Zhiyu, N. Yongfeng, Effects of ultrasonic disintegration on sludge microbial activity and dewaterability, *J. Hazard. Mater.* 161 (2009) 1421–1426.
- [19] Guangming Zhang, Panyue Zhang, Jing Yang, Huanzhi Liu, Energy-efficient sludge sonication: Power and sludge characteristics, *Bioresource Technology* 99 (2008) 9029–9031
- [20] B. Akin, S.K. Khanal, S. Sung, D. Grewell, J. Van-Leeuwen, Ultrasound pre-treatment of waste activated sludge, *Water Sci. Technol.* 6 (2006) 35–42.
- [21] K.Y. Show, T. Mao, D.J. Lee, Optimization of sludge disruption by sonication, *Water Res.* 41 (2007) 4741–4747.
- [22] C.P. Chu, B.V. Chang, G.S. Liao, D.S. Jean, D.J. Lee, Observations on changes in ultrasonically treated waste-activated sludge, *Water Res.* 35 (2001) 1038–1046.
- [23] I. Dogan, Combination of Alkaline solubilisation with microwave digestion as a sludge disintegration method: effect on gas production and quantity and dewater-ability of anaerobically digested sludge, [MSc/MA Dissertation], 2008
- [24] S. Na, Y.U. Kim, J. Khim, Physiochemical properties of digested sewage sludge with ultrasonic treatment, *Ultrason. Sonochem.* 14 (2007) 281–285.