

SONICATION PRETREATMENT OF SLUDGE: PRELIMINARY STUDY OF SLUDGE CONDITIONS

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Received: 16 August 2014, Accepted for publication: 1 October 2015

ABSTRACT

The objective of this work was to investigate effects of sludge conditions serving sludge ultrasonication (*US*). First, specific energy input (*ES*) plays a key role in sludge disintegration (*DD_{COD}*). An optimal total solid concentration of 28 g/L was found to be convenient for sludge *US* pretreatment. Besides, secondary sludge showed the highest disintegration, followed by digested and mixed sludge regardless of temperature (*T*) control during *US*. Additionally, pH adjustment -addition of low NaOH dose (22 - 40 mg_{NaOH}/g_{TS}) could be useful, that significantly improved *COD* release under subsequent *US* while resulting in a suitable pH value for anaerobic digestion afterwards.

Keywords: alkali pretreatment, sludge disintegration, waste activated sludge, ultrasonic pretreatment.

1. INTRODUCTION

Incineration, ocean discharge, land application, and composting are no longer sustainable for sludge disposal due to high costs and/or negative impacts on the environment. Therefore, anaerobic digestion (*AD*) of sludge has been applied as an efficient and sustainable technology for sludge treatment. However, hydrolysis, the first stage of *AD*, is known as the rate-limiting step of microbial conversion. Therefore, some popular techniques have been applied in sludge pretreatment to rupture the cell wall and facilitate the release of intracellular matter into the aqueous phase to improve biodegradability and enhance *AD*: biological, mechanical, thermal, chemical methods, and intense electric fields [1 - 5].

In their review, Pilli *et al.* [6] claimed *ultrasonic irradiation (US)* to be a feasible and promising mechanical disruption technique for sludge pretreatment due to efficient sludge disintegration [6], improvement in biodegradability and bio-solid quality [7], increase in biogas/methane production [7 - 9], no need for chemical additives [10], less sludge retention time [11], and sludge reduction [8]. 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: gas and particulate matter, solvent, field type (standing or

progressive wave), types of *US* cavitation, attenuation, temperature, external pressure, and sample preparation, etc. [6, 12, 13].

The objective of this preliminary study was to separately or simultaneously investigate the effects of some key conditions of sludge serving *US* process: total solid content of sludge *TS* (12 - 36 g/L), sludge type (mixed, secondary, and digested sludge), and prior sludge alkalization (using 22 - 77 mg_{NaOH}/g_{TS}). The selected values are expected to serve for subsequent optimization of sludge *US* pretreatment efficacy.

2. MATERIALS AND METHODS

2.1. Sludge samples

Sludge samples (Table 1, 2) were collected from Ginestous wastewater treatment plants (Toulouse, France) at different periods in relation with the changes in *US* equipment along this work: mixed sludge (solid form, after centrifugation), secondary sludge (liquid form), and digested sludge (liquid form, after *AD* process of the secondary sludge).

Table 1. Characteristics of sludge samples from 1st collection.

Parameter		Value				
		a		b		c
<i>Synthetic sludge samples</i>		Defrosted mixed sludge (1 st -sampling)		Fresh secondary sludge (1 st -sampling)		Fresh digested sludge (1 st -sampling)
Total solids (<i>TS</i>)	g/L	28.0	14.0	28.0	14.0	14.0
Mean <i>SCOD</i> ₀	g/L	2.7	1.5	4.5	1.9	0.4
<i>SCOD</i> _{NaOH 0.5M}	g/L	18.5	11.3	22.9	14.0	11.0
Total <i>COD</i> (<i>TCOD</i>)	g/L	36.5	18.3	38.2	19.1	15.0
<i>SCOD</i> _{NaOH} / <i>TCOD</i>	%	50.7	61.7*	59.9	73.3*	73.3

(*Higher ratios *SCOD*_{NaOH}/*TCOD* at low *TS* might result from higher *NaOH*/*TS* ratios as same amount of *NaOH* was used).

Table 2. Characteristics of sludge samples from 2nd, 3rd, and 4th collections.

Parameter		Value		
		a	b	c
<i>Synthetic sludge sample</i>		Defrosted mixed sludge (2 nd -sampling)	Defrosted secondary sludge (3 rd -sampling)	Defrosted secondary sludge (4 th -sampling)
Total solids (<i>TS</i>)	g/L	28.0	28.0	28.0
Mean <i>SCOD</i> ₀	g/L	3.4	2.8	4.1
<i>SCOD</i> _{NaOH 0.5M}	g/L	19.6	22.7	22.1
<i>TCOD</i>	g/L	38.9	36.3	39.1
<i>SCOD</i> _{NaOH} / <i>TCOD</i>	%	50.4	62.5	56.5

Mixed and most of secondary sludge samples were preserved in a freezer [14]. Digested sludge and some secondary sludge were preserved at 3 – 4 °C (without any freezing). When

performing experiments, the required amount of sludge was defrosted (for frozen sludge) and diluted with distilled water to prepare synthetic sludge samples with a given *TS* content.

2.2. Ultrasound application

Ultrasonic irradiation was emitted by a cup-horn ultrasound unit included in an autoclave reactor which was connected to a pressurized N_2 bottle (see Figure 1). The reactor had an internal diameter of 9 cm and the depth of 18 cm, for a usable capacity of 1 L. A cooling water stream was used to control temperature (T) of the solution at 28 ± 2 °C during *US*. The solution was stirred at 500 rpm. 0.5 L of synthetic sludge sample was used for each experiment. The *US* equipment includes two generators working at 12 and 20 kHz, probe size of 35 mm diameter, and maximum P_{US} (transferred from the generator to the transducer) is 400 W. Different *US* durations corresponding to five values of *ES* (7000 - 75000 kJ/kg_{TS}) were tested: $ES = (P_{US} * t) / (V * TS)$, where *ES*: specific energy input, energy per total solid weight (kJ/kg_{TS}), P_{US} : *US* power input (W), t : *US* duration (s), V : sludge volume (L), and *TS*: total solid concentration (g/L).

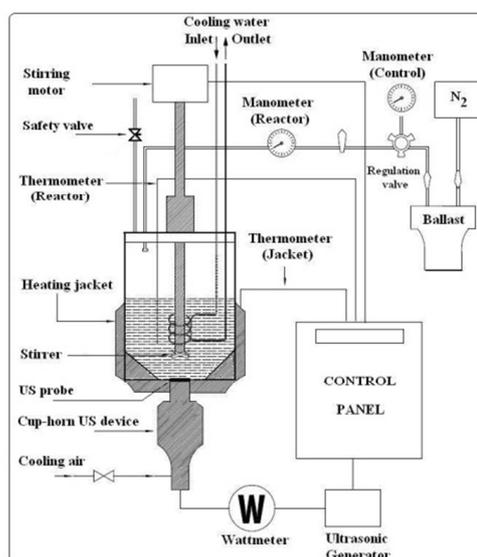


Figure 1. Ultrasonic autoclave set-up.

2.3. Analytical methods

Total and volatile solids (*TS* and *VS*, respectively) were measured according to APHA [15]. The **degree of sludge disintegration** (DD_{COD}) was calculated by determining the soluble chemical oxygen demand after strong alkaline disintegration of sludge ($SCOD_{NaOH}$) and the chemical oxygen demand in the supernatant before and after treatment ($SCOD_0$ and $SCOD$ respectively):

$$DD_{COD} = (SCOD - SCOD_0) / (SCOD_{NaOH} - SCOD_0) \times 100 (\%) \quad [16].$$

$SCOD_{NaOH}$ were measured according to Li *et al* [17]. Besides, total *COD* (*TCOD*) was also measured by potassium dichromate oxidation method (standard AFNOR NFT 90-101). For *SCOD*, the supernatant liquid was filtered under vacuum using a cellulose

nitrate membrane with 0.2 μm pore size. Colloidal *COD* fraction -between 0.2 and 1 μm - was also measured in some cases. 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 cell content and solid materials into the external liquid phase of sludge [18 - 19]. The errors in *COD* measurement were less than 5 %.

3. RESULTS AND DISCUSSION

3.1. DD_{COD} evolution

3.1.1. Effect of *TS* concentration

Five synthetic mixed sludge samples (Table 1.a) (S12, S24, S28, S32, and S36 corresponding to 12, 24, 28, 32, and 36 g/L of *TS*, respectively) were treated at atmospheric pressure, under adiabatic condition. The stirrer speed was set at 500 rpm. The results, presented in Figure 2, showed a gradual increase of *SCOD* with sonication time (0-150 min), but less and less. The relation between *SCOD* and *TS* content is not simple because the best DD_{COD} was not found at the maximum *TS*.

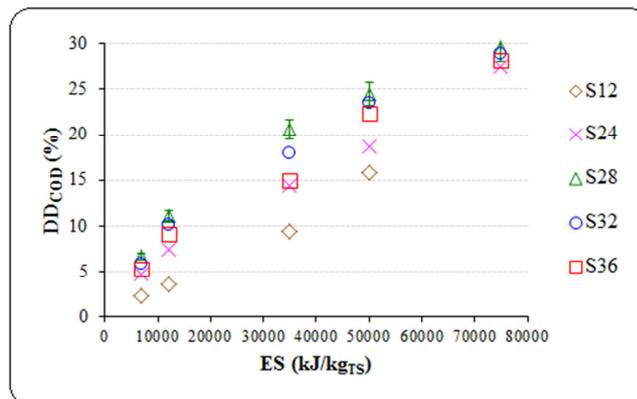


Figure 2. Effect of *TS* on mixed sludge disintegration (DD_{COD}) vs. *ES*: $P_{US} = 150$ W, *BP*, $F_S = 20$ kHz, mixed sludge (Table 1.a), adiabatic conditions, and atmospheric pressure.

Figure 2 actually exhibits a *TS* optimal value of 28 g/L in terms of DD_{COD} over the whole *ES* range. This behavior is in agreement with other studies [6, 10, 14, 20 - 22], and can be explained by opposite effects. The more *TS*, the more cells and aggregates are in contact with cavitation bubbles, thus the more efficiently P_{US} is consumed. On the other hand, the acoustic pressure field decreases faster from the emitter, then the reduction of acoustic cavitation intensity, due to the poor propagation of the *US* wave in a higher *TS* suspension. These two opposite effects lead to an optimum *TS* concentration that could slightly depend on sludge characteristics, operating conditions, and reactor design, *etc.* Some additional isothermal experiments on secondary sludge (Table 2.c) at higher pressure and P_{US} , presented in Figure 3, seem to confirm this *TS* optimum not to depend on sludge type, P_{US} , nor pressure (P_h). It is interesting to note that this “optimum” sludge concentration is close to the actual concentration of secondary sludge which could be treated directly.

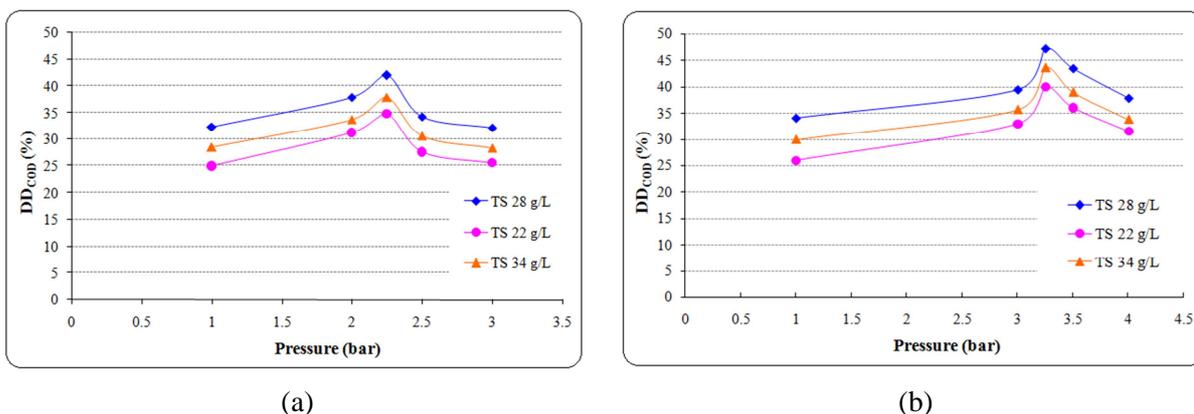


Figure 3. Effect of P_h , TS , and P_{US} on sludge US pretreatment: BP , $ES = 35000 \text{ kJ/kg}_{TS}$, 12 kHz , secondary sludge (Table 2.c), and $T = 28 \pm 2 \text{ }^\circ\text{C}$. (a) $P_{US} = 150 \text{ W}$, (b) $P_{US} = 360 \text{ W}$.

3.1.2. Effect of sludge type

The disintegration of different sludge types by both isothermal and adiabatic sonication was investigated with TS of 14 g/L (as digested sludge was not available at 28 g/L). Figures 4-5 showed higher disintegration degrees (either based on $TCOD$ or $SCOD_{NaOH}$) for secondary sludge, followed by digested sludge and mixed sludge in all conditions. AD might have therefore consumed a fraction of COD which was the most readily solubilized. They also indicate the predominance of adiabatic US as compared to isothermal US in terms of sludge disintegration [23].

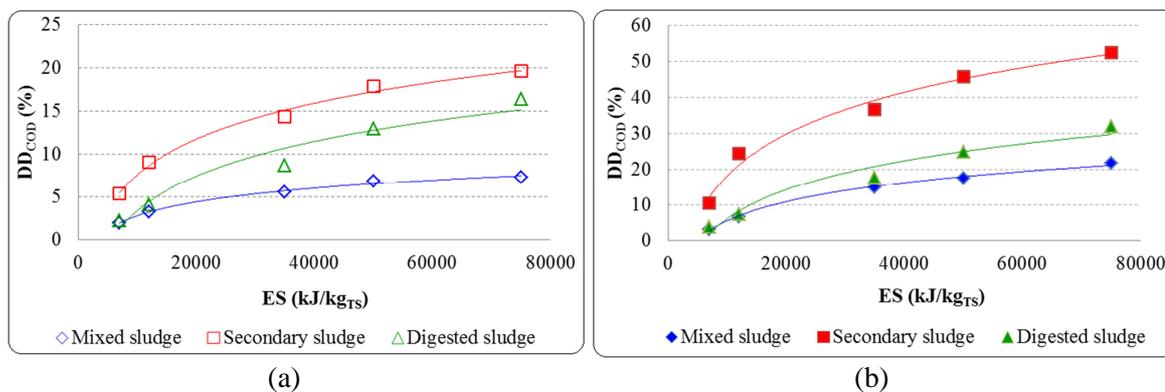


Figure 4. Effect of ES on US pretreatment efficacy of different sludge types (DD_{COD} based on $SCOD_{NaOH}$): $P_{US} = 150 \text{ W}$, BP , $F_S = 20 \text{ kHz}$, $TS = 14 \text{ g/L}$ (Table 1), and atmospheric pressure. (a) $T = 28 \pm 2 \text{ }^\circ\text{C}$ and (b) adiabatic conditions.

As mentioned, different sludge collections were conducted following the variations of US equipment along this work. This part compares these different samples for the same sludge type (e.g. secondary sludge) when treated at the same US conditions. Results, depicted in Figure 6, showed that these secondary sludge samples collected at different times of the work resulted in different US disintegration degrees. Obviously, comparisons to assess sludge US efficiency were then always done on the same substrate.

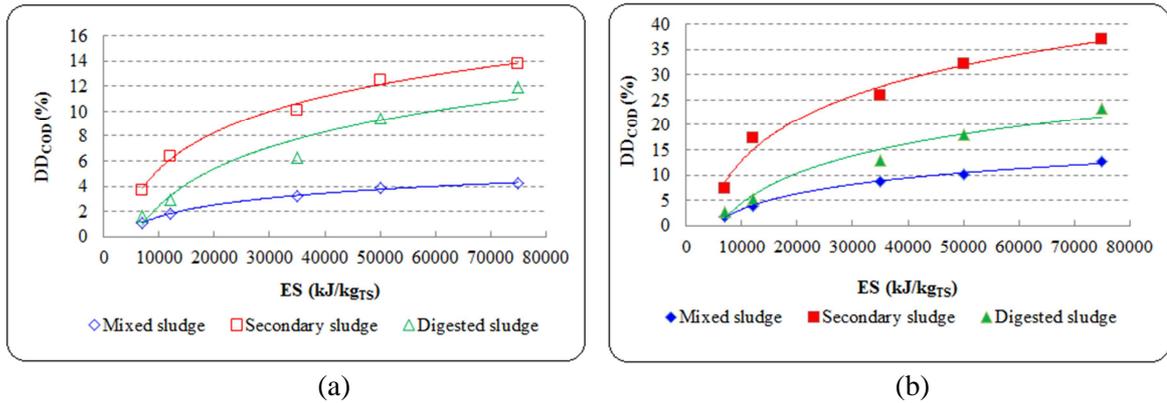


Figure 5. Effect of ES on US pretreatment efficacy of different sludge types (DD_{COD} based on $TCOD^*$): $P_{US} = 150$ W, BP , $F_S = 20$ kHz, $TS = 14$ g/L (Table 1), and atmospheric pressure. (a) $T = 28 \pm 2^\circ C$ and (b) adiabatic conditions. $*$ = $(SCOD_{US} - SCOD_0)/(TCOD - SCOD_0) * 100$

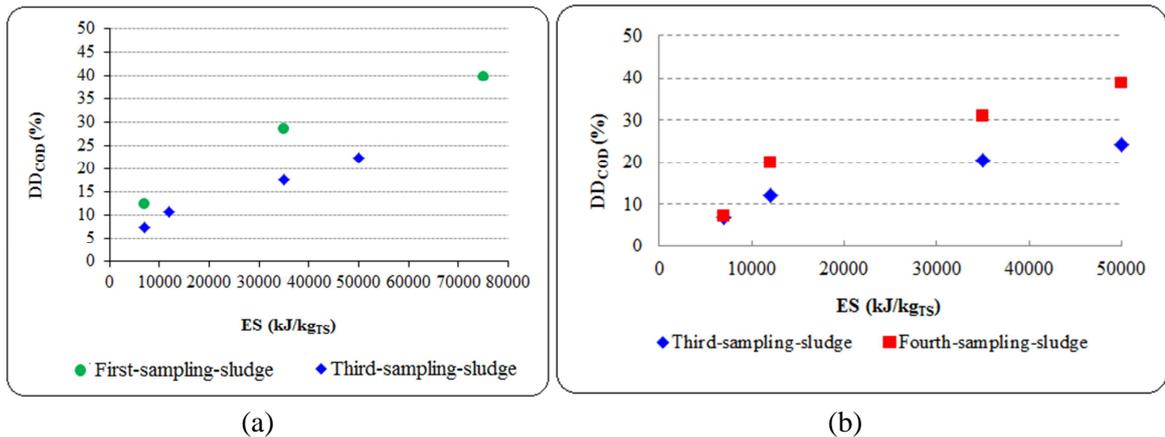


Figure 6. Disintegration degree (DD_{COD} vs. ES) of different secondary sludge samples. (a): $P_{US} = 150$ W, BP , $F_S = 20$ kHz, $T = 28 \pm 2^\circ C$, atmospheric pressure, and $TS = 28$ g/L (Table 1b and 2b) (b): $P_{US} = 50$ W, BP , $F_S = 12$ kHz, $T = 28 \pm 2^\circ C$, atmospheric pressure, and $TS = 28$ g/L (Table 2b and 2c).

3.1.3. Effect of alkaline addition prior to sonication

According to previous studies [24 - 25], “alkalisation (using NaOH) followed by US pretreatment” procedure was chosen for alkaline- US experiments. A given amount of NaOH was added into the fixed volume of mixed sludge to ensure the same condition of chemical application.

NaOH doses of 22, 40, 47, and 77 mg_{NaOH}/g_{TS} were added to the mixed sludge solution (Table 2.a), labelled sol. 22, sol. 40, sol. 47, and sol. 77, respectively, and let under stirring at room temperature for different time periods up to 2 h. The evolutions of pH and DD_{COD} of the samples as a function of time are shown in Table 3. According to Kim *et al.* [26], alkaline pretreatment usually acts faster than other methods. Indeed, in all cases, alkaline treatment resulted in a fast solubilisation of COD , more than 50% of the maximal observed yield being achieved within 10 min, followed by a quasi-plateau after 30 min. Therefore, a holding time of

30 min was selected for subsequent experiments combined with *US*. During this period, the pH of the sludge samples dropped about one unit as shown in Table 3.

Table 3. Alkaline pretreatment of mixed sludge (Table 2a) at room temperature.

		Holding time (min)					
		0.5	10	20	30	40	117
		<i>DD_{COD}</i> (%)					
Sol. 22	(<i>pH</i> 9.6)	6.4	7.3	9.5	(<i>pH</i> 8.6)	10.7	12.3
Sol. 40	(<i>pH</i> 10.2)	11.5	13.3	17.0	(<i>pH</i> 9.4)	18.3	21.0
Sol. 47	(<i>pH</i> 11.1)	13.0	15.8	19.3	(<i>pH</i> 10.1)	21.0	22.5
Sol. 77	(<i>pH</i> 12.2)	24.4	26.3	29.0	(<i>pH</i> 11.0)	30.4	33.1

Subsequently, effect of *NaOH* addition prior to *US* was looked into. As expected, Figure 7 showed alkali-*US* pretreatment to be the most effective technique for sludge disintegration, and the resulting efficacy was nearly the sum of individual pretreatments when sol. 22 or sol. 40 were kept under isothermal *US* (28 °C). Jin *et al.* [25] also observed such a result. Alkalisiation also significantly reduced the differences observed between isothermal and adiabatic *US*. It is also worth noting that under *US*, the differences resulting from the addition of high *NaOH* amounts tended to vanish. As shown in Table. 3, the higher the pH, the more easily the processes of natural shape losing of proteins, saponification of lipid, and hydrolysis of *RNA* occur [1, 27]. However, for overall process economy, *NaOH* addition should be limited. Moreover, high concentration of Na^+ was reported to cause the inhibition of *AD* [1] - requiring a narrow pH range between 6.5 and 8 [24]. According to pH of alkali-sonicated mixed sludge solutions shown in Figure 7, addition of a small *NaOH* dose (as per sol. 22 or sol. 40) could be indeed suitable for the whole process. Nevertheless, no real synergy effect was observed as the best performance of the combined treatment was the sum of the individual ones.

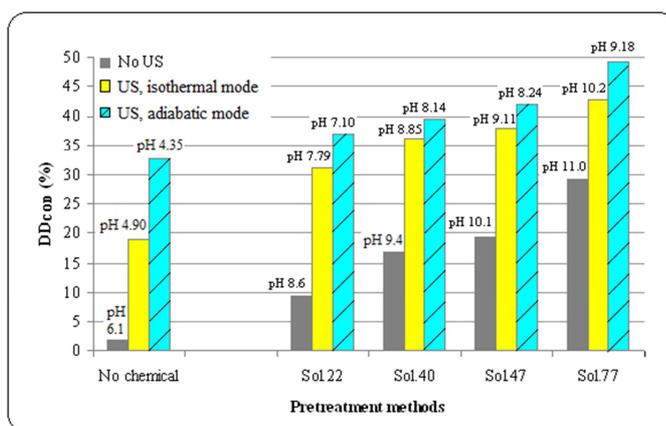


Figure 7. Comparison of different methods for mixed sludge disintegration ($T_S = 28$ g/L, Table 2.a): $F_S = 20$ kHz, $P_{US} = 150$ W, BP , *US* duration = 117 min, NaOH dose = 0-77 $\text{mg}_{\text{NaOH}}/\text{g}_{\text{TS}}$ (holding time = 30 min), and atmospheric pressure. Final pH value after treatment is also indicated on top of each corresponding bar.

The combined effect of mild alkalization and hydrostatic pressure on mixed sludge adiabatic *US* pretreatment was also investigated and is presented in Figure 8. The same conclusions were pointed out regarding the effect of *T* and alkalisiation, but at 2 bar of

hydrostatic pressure, the overall process was still improved: up to about 46 % of DD_{COD} after 2 h of adiabatic *US* for sol. 40. The final pH of 7.6 was also suitable for *AD*.

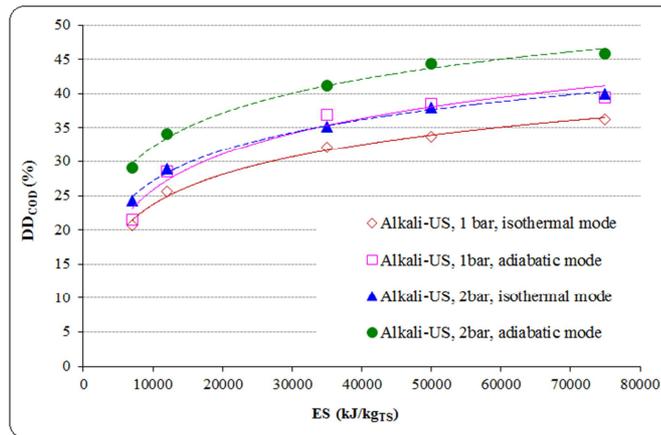


Figure 8. Mixed sludge disintegration under alkali-*US* pretreatment: $P_{US} = 150$ W, BP , 20 kHz, $TS = 28$ g/L - Table 2.a, NaOH dose = $40\text{mg}_{\text{NaOH}}/\text{g}_{\text{TS}}$, holding time of 30 min.

3.2. Solubilisation of organic fractions

Four secondary sludge samples (TS of 28 g/L, Table 1.b) were analyzed for proteins (organic *N* dosage) and sugars (anthrone test), *i.e.* total amounts and in solution after filtration on 1 μm pore size membrane:

(*Si*) Raw sludge;

(*Sii*) US_1 pretreated sludge (150 W, 75000 kJ/kg_{TS}, 20 kHz, 2 bar, and 28 ± 2 °C);

(*Siii*) US_2 pretreated sludge (150W, 75000 kJ/kg_{TS}, 20 kHz, 2 bar, and adiabatic conditions);

(*Siv*) Thermally pretreated sludge (constant 70 °C for 2 h, treatment resulting in almost same *SCOD* as that of *Sii*).

Results were given in Table 4. The total protein and sugar concentrations in these sludge samples were almost constant, suggesting negligible sonochemistry [28 - 29]. The release of organics is due to the disruption of chemical bonds in cell walls and membranes [29], the degradation of *EPS* (including saccharides, proteins, nucleic acids, and humic acids), and the release of intracellular matter from the cells where proteins are mainly located in [28].

Table 4. Solubilisation of organic fractions.

Sample	Solubilisation yield (%) [*]	
	Protein	Sugar
<i>Si</i>	0	0
<i>Sii</i>	61.3	28.0
<i>Siii</i>	70.0	34.3
<i>Siv</i>	42.0	29.1

* = (difference between the (soluble + colloidal) amounts in treated and raw samples) / (difference between the total amount and the initial (soluble + colloidal) amount) *100

In accordance with previous *COD* results, solubilisation of proteins and sugars was higher under adiabatic *US* (*Siii*) than under isothermal *US* (*Sii*) or thermal hydrolysis (*Siv*). Table 4 shows proteins to be released the most in all cases, but the different pretreatments resulted in different yields. For example, protein solubilisation of *Sii* was about 46 % higher than that of *Siv* although almost same *SCOD* and solubilisation of sugars. The low solubilized amount of proteins in *Siv* could be due to the fact that thermal treatment at temperatures lower than 95 °C mainly affects *EPS* which are embedded in the sludge floc matrix, leading to solubilisation of carbohydrates and few proteins [28]. Therefore, lower protein solubilisation of *Siv* compared to those of *Sii* and *Siii* indicated *US* pretreatment to be more effective than low temperature thermal hydrolysis (70 °C) in terms of floc disruption and cell lysis.

Additional experiments were conducted to follow the effect of *ES* on the evolution of soluble (*SCOD*) and colloidal (*CCOD*) fractions of *COD*. Figure 9 depicts evolutions of *SCOD/TCOD* and *CCOD/TCOD* during *US*. While *SCOD/TCOD* gradually increased, *CCOD/TCOD* increased quickly with *ES* up to 12000 kJ/kg_{TS}, then slowed down, and almost reached a plateau afterwards. Colloidal fraction was also much higher than soluble one over the investigated *ES* range (2 to 3-fold).

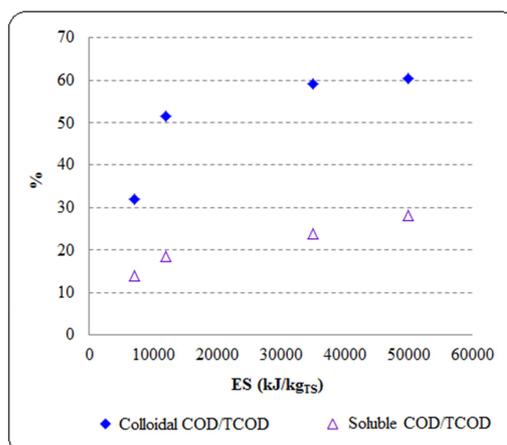


Figure 91. Effect of *ES* on *SCOD/TCOD* and *CCOD/TCOD* during *US*: $P_{US} = 360$ W, BP , $F_S = 20$ kHz, $TS = 28$ g/L (Table 2.c), $T = 28 \pm 2$ °C, and atmospheric pressure.

4. CONCLUSIONS

For given external and *US* parameters, sludge disintegration degree increases nonlinearly with *ES*. Mixed sludge samples with different *TS* contents were pretreated using various sonication durations, exhibiting an optimal concentration of 28 g/L to get the highest *COD* release in the aqueous phase. Besides, the highest disintegration was found with secondary sludge, followed by digested and mixed sludge regardless of temperature control during sonication. Additionally, pH adjustment -addition of low NaOH dose, between 22 and 40 mg_{NaOH}/g_{TS}- could be useful, that significantly improved *COD* release under subsequent *US* treatment while resulting in a final pH value suitable for subsequent *AD*.

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TÓM TẮT

BUƯỚC ĐẦU NGHIÊN CỨU ẢNH HƯỞNG CỦA CÁC ĐIỀU KIỆN Bùn THẢI ĐẾN HIỆU QUẢ TIỀN XỬ LÝ BẰNG SIÊU ÂM

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Nghiên cứu nhằm mục tiêu đánh giá sơ bộ ảnh hưởng của các điều kiện bùn thải đến hiệu quả tiền xử lý bằng siêu âm. Kết quả cho thấy năng lượng siêu âm đóng vai trò quan trọng trong việc phân rã bùn thải. Giá trị tối ưu của tổng hàm lượng chất rắn là 28 g/L. Trong cả hai trường hợp siêu âm đẳng nhiệt và đoạn nhiệt, bùn thứ cấp cho thấy mức độ phân rã cao hơn bùn tiêu hóa và bùn hỗn hợp (sơ cấp + thứ cấp). Ngoài ra, việc kiềm hóa bùn thải (bổ sung 22 - 40 mg_{NaOH}/g_{TS}) thúc đẩy mạnh mẽ sự giải phóng COD trong giai đoạn siêu âm cũng như đảm bảo độ pH thích hợp cho quá trình xử lý yếm khí bùn thải sau đó.

Từ khóa: bùn thải hoạt tính, phân rã bùn thải, tiền xử lý bùn thải bằng kiềm, tiền xử lý bùn thải bằng siêu âm.