

# SLUDGE PARTICLE SIZE REDUCTION UNDER ALKALINE, ULTRASONIC, THERMAL, AND COMBINED PRETREATMENTS

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## ABSTRACT

Sludge particle size reduction (*PSR*) via pretreatment stage has been believed to accelerate the hydrolysis of sludge anaerobic digestion and enhance the degradation of organic matters. This work aimed at investigating the evolution of *PSR* under individual and combined pretreatment methods, including ultrasound (*US*), thermal, and alkaline pretreatments. In addition, the relationship between sludge *PSR* and *COD* release in the aqueous phase was taken into detailed consideration for the first time.

*US* pretreatment showed the most predominant role in reducing sludge particle size compared to other methods. The reduction of the volume moment mean diameter of sludge (*D*[4,3]) were dependent on *US* specific energy input (*ES*) (68 – 77 %). Moreover, the *PSR* was slightly improved in adiabatic conditions and applied pressure (about 10 %). Alkaline addition prior to *US* also further accelerated the *PSR*. However, the strong *D*[4,3] reduction observed in the early stage of the process (low *ES*) was insufficient to affect *COD* solubilisation. It is therefore necessary combine with other factors (chemical, biological parameters), not only base on *PSR* for process optimization.

**Keywords:** alkaline pretreatment, particle size distribution, particle size reduction, sludge disintegration, thermal pretreatment, ultrasonic pretreatment.

## 1. INTRODUCTION

Anaerobic digestion (*AD*) utilizes the biological processes of many bacteria classes. For methane fermentation of solid organic materials (such as waste activated sludge), the methane yield is significantly affected by the mass transfer in each biological step. The first two stages of *AD* process (hydrolysis, acidogenesis) are considered as rate-limiting steps, thus sludge pretreatment is required. Cell lysis technologies can be used to accelerate sludge solubilization and particle size reduction, subsequently improve the *AD* efficiency [1]. There are many processes, some primarily base on individual physical, chemical, biological, and thermal lysis while others combine different mechanisms [2].

Apart from chemical and biological change-based parameters, particle size reduction (*PSR*) has been used to describe the effect of pretreatment on sludge [3]. For *AD*, more rapid processing of input material has been believed to significantly save both capital and operating costs. *PSR* increases the surface area exposed to the microorganisms, resulting in increased food availability to bacteria, consequently increases anaerobic biodegradability [4] and leads to more rapid digestion [5 - 7]. Particle size is therefore one of concerned important factors in *AD* [8] as well as sludge dewatering afterwards, an effective method to reduce the volume of sludge [5, 9].

Excess sludge from wastewater treatment plants (*WWTP*) commonly has a wide range of particle sizes. Different pretreatment techniques resulted in different degrees of sludge *PSR*. Most studies about sludge pretreatment have focussed on the evolution of particle size distribution (*PSD*) under the pretreatment effect. The relationship between sludge *PSR* and chemical oxygen demand (*COD*) release in the aqueous phase has not been detailed investigated.

This work aimed at detailed investigating the evolution of *PSD* under different pretreatment methods. The individual and combined methods, including ultrasound (*US*), thermal, and alkaline pretreatments, were respectively looked into under different ambient conditions: temperature and pressure. Moreover, the relationship between sludge *PSR* and *COD* release in the aqueous phase was taken into detailed consideration for the first time. With respect to pretreatment for *PSR*, determination of its trend was expected to contribute to the selection of a suitable technique (regarding mechanism, pretreatment duration) and to save energy input.

## 2. MATERIALS AND METHODS

### 2.1. Sludge samples

Three types of sludge were collected from Ginestous *WWTP* or INSA (Toulouse, France): mixed sludge (solid form, after centrifugation), secondary sludge (liquid form), and digested sludge (liquid form, after *AD* process of the secondary sludge) (Table 1). An optimum *TS* concentration of 28 g/L was used for sludge disintegration [10]. Mixed and most of secondary sludge samples were conditioned in 100 g and 1 L plastic bottles, respectively and preserved in a freezer [11]. Some experiments were also conducted with fresh secondary and digested sludge sampled in 1 L plastic bottles and preserved at 3-4°C (without any freezing).

Table 1. Characteristics of prepared samples.

| Parameter                  |       | Value                    |                        |                       |                          |                            |
|----------------------------|-------|--------------------------|------------------------|-----------------------|--------------------------|----------------------------|
|                            |       | a. 1st sludge collection |                        |                       | b. 2nd sludge collection |                            |
| Sludge samples             |       | Defrosted mixed sludge   | Fresh secondary sludge | Fresh digested sludge | Defrosted mixed sludge   | Defrosted secondary sludge |
| Total solids (TS)          | (g/L) | 28.0                     | 28.0                   | 14.0                  | 28.0                     | 28.0                       |
| Mean SCOD <sub>0</sub>     | (g/L) | 2.7                      | 4.5                    | 0.4                   | 3.4                      | 2.8                        |
| SCOD <sub>NaOH 0.5 M</sub> | (g/L) | 18.5                     | 22.9                   | 11.0                  | 19.6                     | 22.7                       |
| Total COD (TCOD)           | (g/L) | 36.5                     | 38.2                   | 15.0                  | 38.9                     | 36.3                       |
| SCOD <sub>NaOH</sub> /TCOD | (%)   | 50.7                     | 59.9                   | 73.3                  | 50.4                     | 62.5                       |

### 2.2. Pretreatment experiments

In terms of *US pretreatment*, ultrasonic irradiation was emitted by a cup-horn ultrasound unit (see Fig. 1) included in an autoclave reactor which was connected to a pressurized N<sub>2</sub> bottle.

The 20 kHz ultrasound system (35 mm diameter probe) had a maximum *US* power input ( $P_{US}$ ) of 158 W. For each experiment, 0.5 L of synthetic sludge sample was used. Different *US* durations corresponding to the *ES* range of 7000-75000 kJ/kg<sub>TS</sub> were tested:

$$ES = (P_{US} * t) / (V * TS)$$

where *ES*: specific energy input, energy per total solid weight (kJ/kg<sub>TS</sub>),  $P_{US}$ : *US* power input (W), *t*: sonication duration (s), *V*: volume of sludge (L), and *TS*: total solid concentration (g/L).

*Isothermal US pretreatment* at  $28 \pm 2$  °C was studied first, then *adiabatic US*. External pressures (1 - 16 bar) were also applied to understand their effects on the kinetics of *PSR*. For *thermal hydrolysis* effect, temperature of the solution was gradually increased (without *US*) corresponding to the profiles of *T* and treatment duration resulted from *adiabatic US* pretreatment. For *combined pretreatment*, experiments were conducted in conditions of varying alkaline dose (after 30 min of holding time [12]), *ES* range of 0-75000 kJ/kg<sub>TS</sub>, temperature profile, and pressure application. With regard to alkaline addition, NaOH was used for adjusting the pH of sludge [12 - 13]. Given amounts of NaOH, 22, 40, 47, and 77 mg<sub>NaOH</sub>/g<sub>TS</sub>, were added into a fixed volume of sludge for all experiments to obtain initial pH of approximately 9, 10, 11, and 12, labelled Sol.22, Sol.40, Sol.47, and Sol.77. Finally, for further comprehension of the *PSD* during the early stage of *US* and the degree of sludge disintegration (*DD<sub>COD</sub>*) afterwards, additional experiments with and without sludge pH modification prior to *US* were carried out.

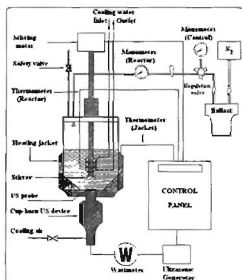


Figure 1. Ultrasonic apparatus.

## 2.3. Analytical methods

*TS* and *VS* were determined according to APHA [14]. The *degree of sludge disintegration* (*DD<sub>COD</sub>*) was calculated by determining the soluble chemical oxygen demand after strong alkaline disintegration of sludge (*SCOD<sub>NaOH</sub>*) and the chemical oxygen demand in the supernatant before and after treatment (*SCOD<sub>0</sub>* and *SCOD*, respectively):

$$DD_{COD} = (SCOD - SCOD_0) / (SCOD_{NaOH} - SCOD_0) * 100 (\%) [15 - 16].$$

The *particle size distribution (PSD)* of sludge before and after treatment was determined by using a Malvern particle size analyzer (Mastersizer 2000, Malvern Inc.), measuring range of 0.02 - 2000  $\mu\text{m}$  [17-19]. Since the primary result from laser diffraction is a volume distribution, the volume mean diameter  $D[4,3]$  was used to illustrate the mean particle size of sludge. In addition,  $d_{90}$ ,  $d_{50}$ , and  $d_{10}$  were analyzed and assessed. The Morphologi G3 particle characterization system from Malvern Instruments Ltd. (Malvern, UK) was used mainly to examine *sludge floc structures*: a few drops of sludge sample were placed on a carrying glass and covered with a lid before being examined with Morphologi G3 using 2.5 $\times$  to 50 $\times$  magnification.

### 3. RESULTS AND DISCUSSION

#### 3.1. Sludge particle size reduction under different pretreatment methods

##### 3.1.1. US pretreatment

Fig. 2 describes the  $D[4,3]$  reduction of mixed sludge samples as a function of  $ES$  for the three investigated  $P_{US}$  of 75, 100, and 150W at atmospheric pressure and  $28\pm 2^\circ\text{C}$ : 68-77% following the increase in  $ES$  of 7000-75000  $\text{kJ/kg}_{\text{TS}}$ . Gonze *et al.* [20] found that particle size was decreased gradually with the increase in  $US$  time and a reverse trend occurred after 10 min of  $US$  due to the re-flocculation of the particles. However, this phenomenon was not found in this work, probably due to higher  $P_{US}$ . In agreement with other works [21-22], main reduction of  $D[4,3]$  was observed within a short duration: after 10 to 20 min of sonication, a quasi-plateau was reached to about 100  $\mu\text{m}$  regardless of  $P_{US}$ .

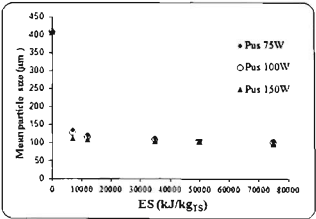


Figure 2 Mean particle size  $D[4,3]$  evolution of mixed sludge (Table 1a) during  $US$  pretreatment with different  $P_{US}$  values: isothermal mode ( $28 \pm 2^\circ\text{C}$ ) and atmospheric pressure.

In the  $ES$  range of 7000 – 75000  $\text{kJ/kg}_{\text{TS}}$ ,  $d_{90}$ ,  $d_{50}$ , and  $d_{10}$  values of mixed 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, also illustrated in Fig. 3 showing a very fast reduction of the class of large particles (about 1000 $\mu\text{m}$ ), is similar to conclusions in previous works [23 – 24].

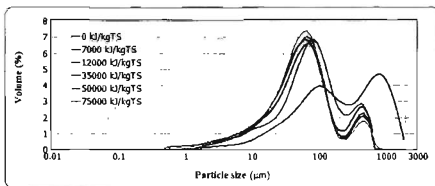
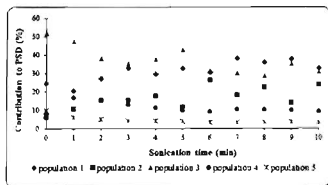


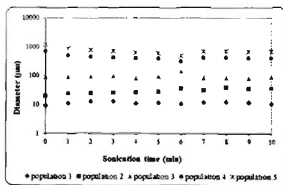
Figure 3. Evolution of particle size distribution of mixed sludge (Table 1.a) during US pretreatment:  $P_{US} = 150$  W, isothermal mode ( $28 \pm 2$  °C), and atmospheric pressure.

Following Gonze *et al.* [20], the PSD was deconvoluted into five populations, each following a lognormal distribution. The treatment was performed using OriginPro 8.6 (OriginLab). Figure 4 shows the evolution of each population contribution and mean diameter during the US treatment: two macro-floc populations (population 4 and 5 of 685  $\mu\text{m}$  and 1200  $\mu\text{m}$ , respectively) could be distinguished, both mean diameter and contribution significantly decreased during the first four minutes of US. Their diameter dropped to about 400  $\mu\text{m}$  and 650  $\mu\text{m}$  respectively while their contribution was divided by a factor 2.5 to 3. Conversely, the size of populations 1 to 3 (about 11  $\mu\text{m}$ , 28  $\mu\text{m}$  and 90  $\mu\text{m}$ , respectively) remained almost constant during short US. It seems thus that the decrease of the largest macro-flocs proceeded mainly according to erosion mechanism while population 3 was disrupted into micro-flocs (population 1).

When US experiments were conducted under pressure (Fig. 5a), especially at optimum pressure of 2 bar in terms of sludge disintegration [10], although the kinetics of disruption was slightly faster, the difference in final particle diameter was negligible (Fig. 5b), *e.g.*, the enhancement of PSR of mixed sludge dropped from 9.3% at 7000 kJ/kg<sub>TS</sub> to less than 1% at 35000-75000 kJ/kg<sub>TS</sub>. Besides, secondary and digested sludge, mainly composed of biological substances, exhibited higher prevalent sludge disintegration while mixed sludge contains many non-degradable materials from primary sludge that are not easily disrupted [23, 25].

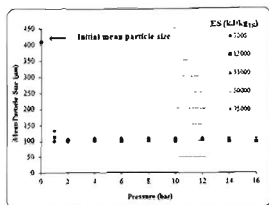


(a)

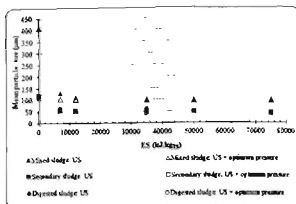


(b)

Figure 4. Evolution of PSD of mixed sludge (Table 1.b) during short US: (a) contribution of each population to PSD, (b) mean diameter of the populations ( $P_{US} = 150$  W,  $T = 28 \pm 2$  °C and atmospheric pressure).



(a)



(b)

Figure 5.  $D[4,3]$  evolution during US under pressure ( $P_{US} = 150$  W,  $T = 28 \pm 2$  °C): (a) different pressures, mixed sludge (Table 1a); (b) atmospheric and optimum pressure (2 bar), different sludge types (Table 1a).

### 3.1.2. Thermal hydrolysis

The US pretreatment has two main simultaneous effects: (i) extreme macro and micro agitation caused by the cavitation, and (ii) increase in the bulk  $T$ . To understand their individual effect on PSR, additional experiments were carried out: (1) isothermal US ( $P_{US} = 150$  W,  $28 \pm 2$  °C), (2) adiabatic US ( $P_{US} = 150$  W, no cooling), (3) without US + progressive increase of  $T$  to see the effect of thermal hydrolysis. Pretreatment duration and temperature profiles of case (3) were as same as those of case (2): 11, 55, and 117 min corresponding to 42, 70, and 78 °C (Fig. 6).

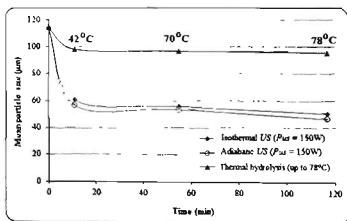


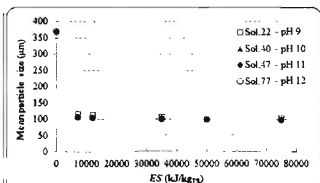
Figure 6. The  $D[4,3]$  evolution of secondary sludge (Table 1.a) under mild thermal hydrolysis ( $P_{US} = 0$ ) and sole US pretreatment ( $P_{US} = 150$  W,  $T = 28 \pm 2$  °C and adiabatic modes, atmospheric pressure).

Figure 6 describes the effect of mild thermal hydrolysis on secondary sludge PSR compared to that of US pretreatment (isothermal and adiabatic modes at atmospheric pressure). At all observed points of time, PSR values under adiabatic US were the highest, followed by those obtained under low  $T$  sonication and mild thermal hydrolysis, indicating that mild thermal hydrolysis has a slight positive effect on sludge PSR and much less than that of US.

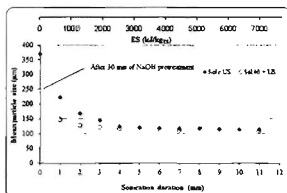
### 3.1.3. Alkaline and Combined pretreatments

For *alkaline pretreatment*, after 30 min under NaOH treatment,  $D[4,3]$  decreased from 370  $\mu\text{m}$  (untreated sludge) to 288, 247, 203, and 133  $\mu\text{m}$  for Sol.22, Sol.40, Sol.47, and Sol.77, respectively. Apart from causing the disintegration of floc structures and cell walls, hydroxyl anions also resulted in extensive swelling and subsequent solubilisation of gels in sludge [13].

For *alkaline-US pretreatment*, Fig. 7a shows the remarkable *PSR* of all samples within short alkaline-US duration. Compared with sole *US* pretreatment, this combination further accelerated the size reduction (Fig. 7b), but the final  $D[4,3]$  values were almost the same, about 100  $\mu\text{m}$ , regardless NaOH doses (Fig. 7a), indicating the predominant effect of *US* on sludge *PSR*. After 30 min of NaOH pretreatment (Sol.40), the diameters of population 1 and 4 were reduced about 20 % as compared to raw mixed sludge and the contributions of populations 4 and 5 were reduced by a factor 1.3 and 1.8, respectively (in favour of populations 2 and 3). However, their evolution under subsequent *US*, described in Fig. 8, remained similar as without NaOH addition (Fig. 4). In this condition, mean diameter of population 4 and 5 dropped to 400 and 600  $\mu\text{m}$ , respectively while that of populations 1 to 3 kept almost unchanged.

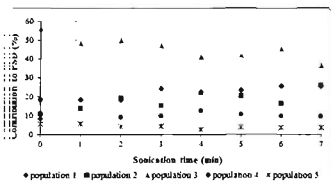


(a)

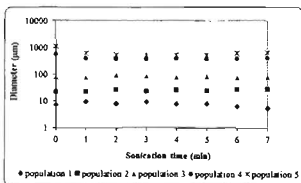


(b)

Figure 7. The  $D[4,3]$  evolution of mixed sludge (Table 1.b) during (a) alkali-US pretreatment and (b) the early stage of *US* and alkali-US pretreatments:  $P_{US} = 150$  W,  $T = 28 \pm 2$  °C, and atmospheric pressure.



(a)



(b)

Figure 8. Evolution of *PSD* of mixed sludge (Table 1.b) during short sonication after NaOH addition (40  $\text{mg}_{\text{NaOH}}/\text{g}_{\text{TS}}$ ): (a) contribution of each population to *PSD*, (b) mean diameter of the populations ( $P_{US} = 150$  W,  $T = 28 \pm 2$  °C, and atmospheric pressure).

The effect of alkaline, thermal, and ultrasonic under pressure pretreatment on mixed sludge *PSR* was investigated and presented in Fig. 9. Chu *et al.* [22] showed that extracellular polymeric substances (*EPS*) and gels surrounding cells limit the efficiency of *US* treatment on sludge disintegration. Adjustment pH of sludge to alkaline medium promotes the *EPS* hydrolysis and gel solubilisation. After that, cell walls cannot maintain an appropriate turgor pressure [12] and easily disrupts. Therefore, the combination of alkaline and *US* pretreatment under pressure, based on different mechanisms of sludge disintegration takes advantage of both and gives a better efficiency of sludge *PSR* compared to sole *US* pretreatment.

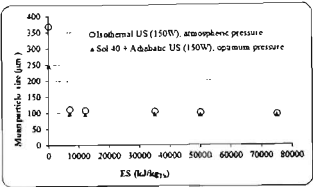


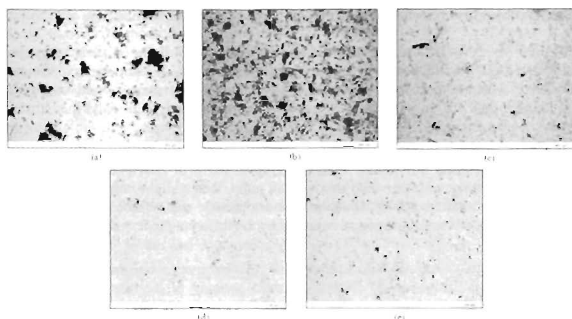
Figure 9 Effect of alkaline, thermal, and *US* pretreatments on *PSR* of mixed sludge (Table 1b):  $P_{US} = 150\text{ W}$ , atmospheric and optimum pressure of 2 bar, NaOH dose = 40 mg/g<sub>TS</sub>.

### 3.2. Analysis of sludge particle images

Apart from analysis of Mastersizer 2000 *PSD*, effects of *US* and temperature rise on particle size and morphological parameters were examined by image processing of secondary sludge (Table 1.b) photographs from Morphologi G3 (Fig. 10). Sludge particles, especially large ones, exhibit irregular shape, therefore the volume moment mean CE diameter (diameter of the circle of equivalent area to the 2D object), but also volume moment mean length and width ( $L[4,3]$  and  $W[4,3]$ , respectively) are given in Table 2.

Table 2. Size parameters of raw and pretreated sludge samples (see legend of Fig. 10).

| Sample<br>(magnification) | D[4,3] based on CE<br>diameter (µm) | L[4,3] (µm) | W[4,3] (µm) |
|---------------------------|-------------------------------------|-------------|-------------|
| (a) (10×)                 | 185.6                               | 297.3       | 201.8       |
| (b) (10×)                 | 145.1                               | 342.3       | 182.5       |
| (c) (10×)                 | 51.1                                | 100.8       | 69.3        |
| (d) (50×)                 | 8.8                                 | 25.4        | 11.2        |
| (e) (50×)                 | 3.3                                 | 6.3         | 3.3         |



**Figure 10.** Photographs of raw and pretreated secondary sludge (Table 1.b, 1 bar): (a) Raw sludge after defrosting (2.5x), (b) after 78 min of thermal hydrolysis up to 80°C (2.5x), (c) after 5 min of *US* (150 W) + 73 min of thermal hydrolysis up to 80°C (2.5x), (d) after 78 min of adiabatic *US* (150 W) + 162 min of stirring (10x), (e) after 117 min of isothermal *US* 150 W (10x).

As previously found from laser granulometry, the size of the flocs is marginally affected by thermal hydrolysis (Fig. 10b and entry (b) of Table 2). Conversely, *US* provokes a significant floc disruption. Their structural integrity is almost broken down after a short time of *US* (Fig. 10c) and a longer treatment seems to lead to a further reduction in size (entry (d-e) of Table 2).

The effect of *US* on sludge flocs can be also observed by the analysis of other morphological parameter variations, *e.g.* elongation, convexity, solidity, and circularity. Table 3 exhibits the volume median values of these parameters after the different treatments. Very low solidity (< 0.4) is found for filamentous structures, while large flocs (> 100 µm) formed by agglomerates exhibit values between 0.4 and 0.75 and particles under 10 µm have a median solidity of 0.9. These small particles are also very smooth with a median convexity of 0.9. Table 3 confirms that the irregular and fluffy macroflocs are disrupted into smaller, smoother, and more compact structures by *US* which corresponds to an increase of median convexity and solidity (to values very close to 1), as well as of circularity (as elongation is conversely not much changed) with an increase of sonication time (or *ES*).

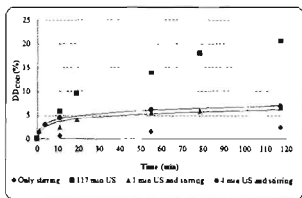
**Table 3.** Morphological parameters of raw and pretreated sludge samples (see legend of Fig. 10).

| Sample<br>(magnification) | Vol. median<br>circularity | Vol. median<br>convexity | Vol. median<br>elongation | Vol. median<br>solidity |
|---------------------------|----------------------------|--------------------------|---------------------------|-------------------------|
| (a) (10x)                 | 0.40                       | 0.53                     | 0.32                      | 0.71                    |
| (b) (10x)                 | 0.31                       | 0.45                     | 0.35                      | 0.65                    |
| (c) (10x)                 | 0.39                       | 0.54                     | 0.33                      | 0.69                    |
| (d) (50x)                 | 0.67                       | 0.88                     | 0.37                      | 0.84                    |
| (e) (50x)                 | 0.83                       | 0.95                     | 0.30                      | 0.94                    |

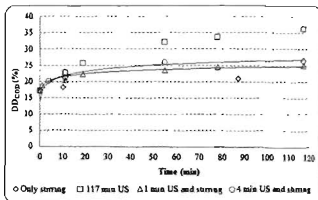
### 3.3. The relationship between $PSR$ and $DD_{COD}$

Finally, for a further comprehension of the relationship between mean particle size reduction and  $COD$  solubilisation (or sludge disintegration  $DD_{COD}$ ), additional experiments for mixed sludge with and without pH adjustment ( $40 \text{ mg}_{\text{NaOH}}/\text{g}_{\text{TS}}$ ) were carried out.  $US$  were applied during the first minute or the first four minutes, and then only the stirrer was continuously operated under cooling afterwards (Fig. 11).

Despite these two sonication durations resulted in distinct  $D[4,3]$ , especially under natural pH (Fig. 8), no significant differences were observed in terms of  $DD_{COD}$  afterwards (Fig. 11). These short  $US$  pretreatments provided first a small jump, then a slight improvement of  $DD_{COD}$ . Therefore, it proves that the strong reduction of mean particle size observed at low  $ES$  was not sufficient to affect  $COD$  solubilisation as expected by the different process dynamics.



(a)



(b)

Figure 11. Effect of short  $US$  time on  $DD_{COD}$  of mixed sludge (Table 1.b):  $P_{US} = 150 \text{ W}$ ,  $T = 28 \pm 2 \text{ }^{\circ}\text{C}$ , and atmospheric pressure. (a) Without  $\text{NaOH}$  and (b) with addition of  $40 \text{ mg}_{\text{NaOH}}/\text{g}_{\text{TS}}$  (30 min of holding time).

In short,  $US$  pretreatment significantly decreased the particle size of sludge, especially in the very short time of sonication. The predominant effect of  $US$  in terms of  $PSR$  could be explained that the mechanical effects of sonication led to sludge floc disintegration, forming small particles, and microorganism lyses [26]. Increase in  $ES$  (or sonication time) resulted in the breakup of cell walls, disintegration of the sludge solids, and release of dissolved organic compounds [15, 27]. However, there was a critical  $ES$  value (or sonication duration) for significant sludge  $PSR$ ; beyond this value, sludge solubilisation might still increase but no significant  $PSR$  could be observed which was in agreement with Chu *et al.* [22].

## 4. CONCLUSIONS

The particle size reduction of sludge has been believed to accelerate the hydrolysis stage of sludge  $AD$  and to enhance degradation of organic matters, thus has been used to describe sludge pretreatment efficiency. The effects of some pretreatment methods, including alkaline, thermal, and ultrasonic pretreatments, on sludge  $PSR$  were individually and simultaneously investigated in different conditions of temperature control and pressure application.

Ultrasonic pretreatment showed the most predominant role in reducing sludge particle size compared to other methods, especially in adiabatic conditions and applied optimum pressure. Moreover, the addition of alkaline (NaOH) prior to *US* further accelerated the *PSR* in the early stage (low *ES*) of the process. However, the relationship between *PSR* and *DD<sub>COD</sub>* indicated that the strong reduction of mean particle size observed at low *ES* was insufficient to affect *COD* solubilisation. For process optimization, it is therefore necessary combine with other factors (chemical, biological parameters), not only base on *PSR*.

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## TÓM TẮT

### HIỆU QUẢ GIẢM KÍCH THƯỚC HẠT Bùn THỎNG QUA TIỀN XỬ LÝ BẰNG KIỀM, SIÊU ÂM, NHIỆT VÀ KẾT HỢP

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Việc giảm kích thước hạt của bùn thải (*PSR*) thông qua tiền xử lý giúp đẩy nhanh giai đoạn thủy phân cũng như tăng cường sự phân hủy chất hữu cơ trong quá trình tiêu hóa kỵ khí. Nghiên cứu này nhằm đánh giá sự thay đổi *PSR* bởi tác động riêng lẻ và kết hợp của các phương pháp tiền xử lý bùn thải khác nhau, bao gồm siêu âm (*US*), nhiệt và kiềm. Bên cạnh đó, mối quan hệ giữa *PSR* và *COD* chuyển từ pha rắn sang pha lỏng lần đầu tiên được nghiên cứu chi tiết.

Kết quả cho thấy phương pháp tiền xử lý bùn thải bằng siêu âm mang lại hiệu quả *PSR* cao nhất. Sự thay đổi kích thước hạt trung bình ( $D[4,3]$ ) phụ thuộc vào năng lượng siêu âm (*ES*). Hiệu quả *PSR* còn được nâng cao trong điều kiện siêu âm đoạn nhiệt và áp suất ngoại tác thích hợp. Kiềm hóa bùn thải cũng giúp đẩy nhanh tốc độ *PSR* trong giai đoạn siêu âm. Tuy nhiên, việc giảm mạnh  $D[4,3]$  trong giai đoạn đầu của quá trình siêu âm (*ES* thấp) không ảnh hưởng đáng kể đến nồng độ *COD* được giải phóng. Do đó, để tối ưu hóa quá trình xử lý bùn thải, cần nghiên cứu kết hợp *PSR* với các thông số hóa học và sinh học khác.

*Từ khóa:* giảm kích thước hạt, phân bố cấp hạt, phân rã bùn thải, tiền xử lý bằng nhiệt, tiền xử lý bằng kiềm, tiền xử lý bằng siêu âm.