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ACOUSTIC SONICATION EFFECTS ON WASTE ACTIVATED SLUDGE DISINTEGRATION

Ngoc Tuan Le[1, 2, *], Carine JULCOUR-LEBIGUE[2], Henri DELMAS[2]

[1]University of Science, Vietnam National University Ho Chi Minh City, Vietnam
[2]Université de Toulouse, Laboratoire de Génie Chimique, INP-ENSIACET, Toulouse, France

[*]Email: lntuan@hcmus.edu.vn

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ABSTRACT

This work aimed at investigating the effect of audible frequency (F_S = 12 kHz) on sludge pretreatment by sonication (*US*) under pressure for the first time. The main *US* parameters (power -P_{US}, intensity -I_{US}) were also looked into. The higher P_{US}, the higher sludge disintegration (DD_{COD}) was achieved due to the increase in cavitation intensity, *e.g.* at 7000 kJ/kg$_{TS}$, DD_{COD} improvement was about 74 % when increasing P_{US} from 50 to 360 W (I_{US} of 5.2 - 37.4 W/cm^2). In addition, about 16 % of DD_{COD} improvement was achieved when switching from 50 W-*SP* to 360 W-*BP* at same I_{US} (about 37.5 W/cm^2). Besides, sludge disintegration was significantly improved by low frequency *US* (12 *vs.* 20 kHz) due to more violent cavitation: by 64 % at *ES* of 7000 kJ/kg$_{TS}$. Positive effect of pressure associated with high P_{US} and low F_S was also found. This work provided general information and trends related to sonication process to be used or checked in other potential applications of physical effects of acoustic cavitation.

Keywords: acoustic sonication, audible frequency, particle size reduction, sludge disintegration, sonication pretreatment, waste activated sludge.

1. INTRODUCTION

Acoustic cavitation is a phenomenon that is mainly related to the sound pressure amplitude, its frequency, through the bubble size variations [1]. 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 [2]. The critical size range increases with the increase in acoustic pressure amplitude and the decrease in frequency.

Sound frequency (F_S) has a significant effect on the cavitation process because it alters the critical size of the cavitation bubble [3]. In general, the increase in F_S leads to the decrease in cavitation physical effects [4 - 5] due to the decrease in radius range that will provide cavitation [1], the too short finite-time of the rarefaction cycle for a bubble to grow and collapse [6], and the too short time for the compression cycle to collapse the bubble (if any) [3]. On the other hand, at higher F_S, although cavitation is less violent, there are more cavitation events and thus more radicals to be produced and consequently a promotion of chemical reactions [4]. Meanwhile, lower F_S have stronger shock waves and favor mechanical effects [7]. This more violent collapse at low F_S is due to the resonance bubble size being inversely proportional to the F_S [8]. The optimum frequency is system-specific and depends on whether intense temperatures and pressures or single electron transfer reactions are looked for. The choice of F_S therefore depends on the expected type of ultrasound (*US*) effects: mechanical (due to shock waves and high local shear stresses) or chemical (connected to free radical formation) [9 - 17]. With regard to sludge pretreatment, sonication (*US*) mechanically disrupts the floc matrix and cell structure. Tiehm *et al.* [18] and Zhang *et al.* [7] found that the degree of sludge disintegration (DD_{COD}) decreased owing to the increase in F_S, indicating mechanical effects, instead of free radicals, to be responsible for the biodegradability enhancement. It is important to note that DD_{COD} in most works is the most significant at low F_S [19 - 21], but the.lowest investigated F_S have been restricted to around 20 - 25 kHz. Lower F_S could then be interesting in sludge disintegration and needs detailed investigation.

For hydrostatic pressure effect, its modification was proved to change the resonance condition of cavitation bubbles via their equilibrium radius, and then may drive the system toward resonance conditions [3], increase consequently the rate and yield of *US*-assisted reactions [22 - 24]. However, most *US* experiments have been carried out at atmospheric pressure, only a few studies have been focusing on how increasing pressure affects cavitation but almost concern sonoluminescence [25 - 27], superplastic flow [28 - 31], 304L stainless steel corrosion [10], yeast disintegration [32], or other related researches [33 - 37]. Thereby, effects of very low F_S on sludge sonication under pressure are expected to improve the pretreatment efficiency and need taking into consideration.

In relation to the effect of *US intensity* (I_{US}) -the quotient of *US* power (P_{US}) and the surface area of the probe, it was proved that higher mechanical shear forces produced at higher I_{US} rupture microorganism cell walls, leading to the increase in DD_{COD} [21, 38]. Most researches [39 - 43] have varied only P_{US}, meanwhile the magnitude of the effect of each factor needs further investigation in connection with scale-up purpose. Besides, the role of I_{US} on the efficiency of sludge pretreatment by acoustic frequency sonication under pressure has not been investigated.

This work was the first research on the relationship among I_{US}, very low F_S (down to audible range), and pressure as well as their integrated effects on sludge sonication pretreatment (low temperature), assessed by DD_{COD} and particle size distribution (*PSD*). The best condition found in this work is expected to enhance sludge disintegration, to save energy input, and to facilitate the anaerobic digestion (*AD*).

2. MATERIALS AND METHODS

2.1. Sludge samples

Waste activated sludge (*WAS*), given in Table 1, was collected from Ginestous wastewater treatment plants (Toulouse, France). Sludge was sampled in 1 L and 100 mL plastic boxes and preserved in a freezer according to Kidak *et al.* [44]. When performing experiments, sludge was defrosted and diluted with distilled water to prepare synthetic sludge samples (28 g_{TS}/L).

2.2. Sonication application

A cup-horn sonicator included in an autoclave reactor that was connected to a pressurized N_2 bottle was used (Fig. 1). The equipment includes two generators working at 12 and 20 kHz, and for each two associated probes of 13 and 35 mm diameter, labeled as *SP* and *BP*, respectively. Maximum P_{US} (transferred from the generator to the transducer) is 100 W and 400 W for *SP* and *BP*, respectively. A constant volume of synthetic sludge sample (0.5 L) was used for each experiment.

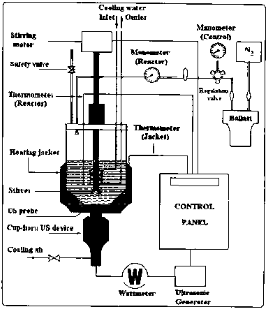

Figure 1. Autoclave sonicator set-up.

Table 1: Characteristics of the sludge samples.

Parameter		Value a	Value b
Raw sludge sample			
pH		6.3	6.3
Total solids (*TS*)	g/L	34.2	31.9
Volatile solids (*VS*)	g/L	30.2	26.4
VS/TS	%	88.3	82.8
Synthetic sludge sample			
Total solids (*TS*)	g/L	28.0	28.0
Mean $SCOD_0$	g/L	4.1	2.8
$SCOD_{NaOH\ 0.5M}$	g/L	22.1	22.7
TCOD	g/L	39.1	36.3
$SCOD_{NaOH}/TCOD$	%	56.5	62.5

Firstly, the effect of I_{US} in audible F_S sonication on sludge disintegration was examined in isothermal condition (T = 28 ± 2 °C) and at atmospheric pressure: vary P_{US} values or/and probe sizes. Different *US* durations corresponding to four values of *ES* (7000, 12000, 35000, and 50000 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}: power input (W), t: *US* duration (s), V: volume of sludge (L), and *TS*: total solid concentration (g/L). *Secondly*, pressures (1 - 4 bar, 0.25 bar of intervals) were applied to 12 kHz sonicator, *BP*, P_{US} (150 and 360 W), and isothermal mode (T = 28 ± 2 °C), to understand for the first time the combined effect of F_S and P_{US} on the optimum pressure. *Finally*, effects of audible F_S on sludge sonication under pressure were investigated at atmospheric and optimum pressure.

Experiments were duplicated and the coefficients of variation of DD_{COD} were about 5 %.

2.3. Analytical methods

Total and volatile solids contents (*TS* and *VS*) were measured according to APHA [45].

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): $DD_{COD} = (SCOD - SCOD_0)/(SCOD_{NaOH} - SCOD_0)*100$ **(%)** [46]. To measure the $SCOD_{NaOH}$ value, the sludge sample was mixed with 0.5 M NaOH at room temperature for 24 h [47]. Besides, total chemical oxygen demand (*TCOD*) was also measured by potassium dichromate oxidation method (standard AFNOR NFT 90-101). Prior to *SCOD* determination, the supernatant liquid was filtered under vacuum using a cellulose nitrate membrane with 0.2 μm pore size. Additionally, 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 ***particle size distribution*** (***PSD***) of sludge before and after treatment was determined by using a Malvern particle size analyzer (Mastersizer 2000, Malvern Inc.), a laser diffraction-based system (measuring range from 0.02 to 2000 μm) [48 - 50]. Since the primary result from laser diffraction is a volume distribution, the volume mean diameter *D[4,3]* (or de Brouckere mean diameter) was used to illustrate the mean particle size of sludge.

Rheology is the study of flow and deformation of materials under applied forces and involves the measurement of shear stress τ in a fluid at various shear rates γ. The power law model is one of the most widely used to describe the relationship between the two for complex microstructure substances such as sludge and thus exhibit a non-Newtonian behavior, where $\tau = K \cdot \gamma^n$ and the apparent dynamic viscosity $\mu_{app} = \frac{\tau}{\gamma} = K \cdot \gamma^{n-1}$. K is the consistency coefficient of the fluid (the greater the value of K the more viscous the fluid); n is the flow behavior index - a measure of the degree of deviation from the Newtonian behavior: $n=1$ for Newtonian fluid, $n<1$ for pseudoplastic or shear-thinning material (effective viscosity decreases with shear rate), $n>1$ for dilatant or shear-thickening material. Note that the shear stress must exceed a critical value known as yield stress (τ_0) for the fluid to flow.

The measurements were performed using an AR 2000 Rheometer (TA Instruments®) equipped with a cone (6 cm, 2°) and plate geometry. 2 mL of sludge sample were placed on the horizontal plate controlled at 25 °C, and then the cone was rotated at a shear rate range of 0-1000 s^{-1}. Shear stress was measured and recorded corresponding to the investigated shear rates. The Herschel–Bulkley model (1926) was used to describe the rheological behavior of sludge with standard errors of less than 10%: $\tau = \tau_0 + K \cdot \gamma^n$

3. RESULTS AND DISCUSSION

3.1. Effect of P_{US} and I_{US} in audible frequency sonication on DD_{COD}

Effects of I_{US} on sludge disintegration were investigated at same P_{US} (50 W) by changing the probe: *SP* (I_{US} of 37.7 W/cm^2) *vs.* *BP* (I_{US} of 5.2 W/cm^2). These experiments were conducted at 12 kHz. Results are shown in Fig. 2 where additional experiments at I_{US} of 37.4 W/cm^2 but using a different combination of P_{US}-probe (360W-*BP*) are also reported for comparison of both effects.

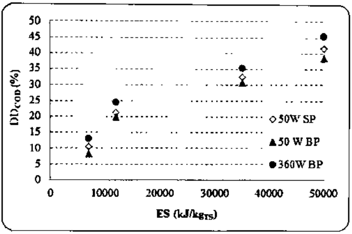

Figure 2. Comparison of I_{US} (same P_{US} of 50W) and P_{US} (same probe) effects on DD_{COD} at different *ES*: synthetic secondary sludge (Table 1a), 12 kHz, and atmospheric pressure.

First, for the *BP*, the higher P_{US}, the higher DD_{COD} was achieved as a function of *ES*, indicating more sludge disintegration following the increase in I_{US} due to the increase in the maximum pressures and temperatures within a transient collapse [6], *e.g.* at the same energy consumption of 7000 kJ/kg$_{TS}$, DD_{COD} improvement was about 74 % when increasing P_{US} from 50 to 360 W (I_{US} of 5.2 - 37.4 W/cm^2).

When comparing the two probe sizes, experiments at the same P_{US} of 50 W showed little improvements of DD_{COD} (about 13 %) when increasing I_{US} from 5.2 to 37.7 W/cm^2. Series at same I_{US} (about 37.5 W/cm^2) with different P_{US} showed a better effect of P_{US} than I_{US}, *e.g.* about 16 % of DD_{COD} improvement was achieved when switching from 50W-*SP* to 360W-*BP*.

Therefore, under isothermal mode (low *T*) and atmospheric pressure, high P_{US} - short *US* time should be preferred for sludge disintegration.

3.2. Effect of frequency on the efficacy of sludge sonication (at atmospheric pressure)

As mentioned, even though most applications using mechanical effects of *US* are improved when reducing F_S, nearly no information is available under 20 kHz - the usual limit of commercial equipment corresponding also to the limit of human hearing. Therefore, effects of audible F_S on the efficacy of sludge pretreatment were investigated for the first time using *BP*, P_{US} of 360 W, and assessed by DD_{COD}. Results are shown in Fig. 3.

Figure 3 shows that the lower the F_S, the more the sludge was disintegrated due to more violent cavitation. DD_{COD} were significantly improved at 12 kHz *US* as compared to 20 kHz *US*, by 64 % at *ES* of 7000 kJ/kg$_{TS}$. According to Laborde *et al.* [8], Thompson and Doraiswamy [3], Zhang *et al.*[41], Pham *et al.* [19], Carrère *et al.* [20] and Pilli *et al.* [21], the lower F_S, the stronger shock waves and mechanical effects are favored due to the resonance bubble size being inversely proportional to the acoustic F_S. However, noting that at low F_S the maximum collapse time and the maximum size of the expanded cavity are increased, the optimum cavitation effect should occur at higher P_{US} [10].

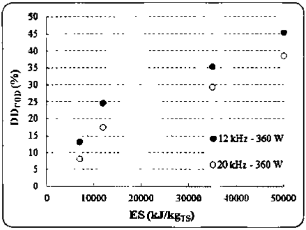

Figure 3. Effect of *ES* and F_S on DD_{COD}: synthetic secondary sludge (Table 1a), *BP*, and atmospheric pressure.

Evolution of colloidal *COD* fraction during *US* at different F_S (along with corresponding soluble *COD* fraction) was also measured and presented in Fig. 4. Unlike *SCOD/TCOD* which gradually increased following an increase in *ES*, *CCOD/TCOD* increased quickly up to *ES* of 12000 kJ/kg$_{TS}$, then slowed down, and almost reached a plateau with *ES* more than 35000 kJ/kg$_{TS}$. Regardless of F_S, *CCOD/TCOD* were much higher than *SCOD/TCOD* in the investigated *ES* range. In addition, both soluble and colloidal fractions were increased under lower F_S sonication (12 *vs*. 20 kHz) although the improvements were rather low.

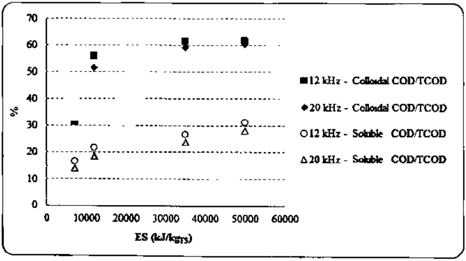

Figure 4. Effect of frequency on *SCOD/TCOD* and *CCOD/TCOD* during *US*: synthetic secondary sludge (Table 1a), *BP*, P_{US} = 360 W, and atmospheric pressure.

Besides, the lower the F_S, the faster the sludge particle size was reduced, especially within the first two minutes. However, the differences in size thereafter were insignificant as depicted in Fig. 5.

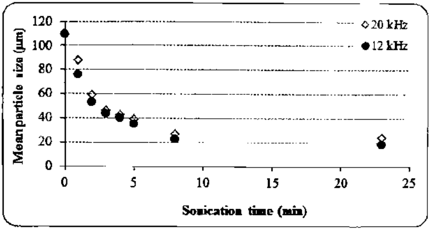

Figure 5. Mean particle size reduction under *US* at different F_S: synthetic secondary sludge (Table 1a), P_{US} = 360 W, *BP*, and atmospheric pressure

3.3. Effect of audible frequency on the optimum pressure and subsequent DD_{COD}

Pressure (1 - 4 bar, 0.25 bar of intervals) was applied to the 12 kHz sonicator, using the *BP* with P_{US} of 150 and 360 W at the same *ES* of 35000 kJ/kg$_{TS}$. Additional experiments were conducted on another secondary sludge (Table 1b) at 50000 kJ/kg$_{TS}$ and 20 kHz to further understand the effect of F_S on the optimum pressure. Results are presented in Fig. 6.

In both cases of F_S, the optimum pressure shifts when increasing P_{US} (or I_{US}). Besides, the location of this optimum seems to be independent from *ES* and sound frequency in the restricted investigated range: 2 bar at 150 W and 3.5 bar at 360 W (using 0.5 bar intervals) for 20 kHz as compared to 2.25 bar at 150 W and 3.25 bar at 360 W (0.25 bar intervals) for 12 kHz sonicator.

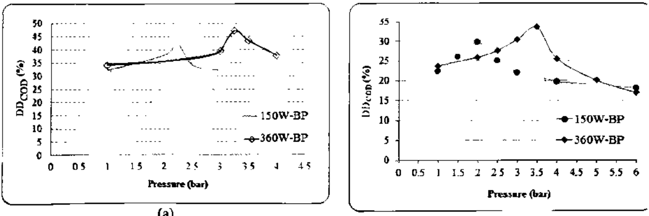

Figure 6. Effect of pressure on DD_{COD} for different P_{US} and F_S: (a) 12 kHz, *ES* = 35000 kJ/kg$_{TS}$, synthetic sludge (Table 1a), (b) 20 kHz, *ES* = 50000 kJ/kg$_{TS}$, synthetic sludge (Table 1b).

Figure 7 describes the effect of F_S on sludge sonication under optimum pressure. It again indicates that the lower the F_S, the more the sludge is disintegrated, which generalizes the results of Tiehm *et al.* [18], Zhang *et al.* [7], Pham T.D. *et al.*[19], and Carrère *et al.* [20] to audible F_S.

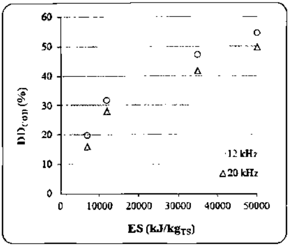

Figure 7. Effect of *ES* and frequency on secondary sludge disintegration under optimum pressure (3.25 bar): synthetic secondary sludge (Table 1a), P_{US} = 360 W, and *BP*.

Table 2. Apparent viscosity and parameters of Herschel-Bulkley model for different sonicated secondary sludge samples (P_{US} = 360 W).

	Yield stress τ_0 (Pa)	Consistency K (Pa.s^n)	Flow index n (-)	Apparent viscosity μ_{app} (Pa.s)	
				γ = 1 (s^{-1})	γ = 100 (s^{-1})
Isothermal US (28°C) at 20 kHz and 1 bar					
0 kJ/kg$_{TS}$	0.124	0.072	0.680	0.266	0.018
7000 kJ/kg$_{TS}$	0.093	0.066	0.667	0.196	0.015
Isothermal US (28°C) at 20 kHz and 3.25 bar					
0 kJ/kg$_{TS}$	0.124	0.072	0.680	0.266	0.018
7000 kJ/kg$_{TS}$	0.109	0.041	0.712	0.138	0.012
Isothermal US (28°C) at 12 kHz and 1 bar					
0 kJ/kg$_{TS}$	0.246	0.057	0.731	0.399	0.019
7000 kJ/kg$_{TS}$	0.123	0.053	0.684	0.196	0.014
Isothermal US (28°C) at 12 kHz and 3.25 bar					
0 kJ/kg$_{TS}$	0.246	0.057	0.731	0.399	0.019
7000 kJ/kg$_{TS}$	0.087	0.051	0.683	0.163	0.013
35000 kJ/kg$_{TS}$	0.079	0.029	0.724	0.099	0.009

According to Lorimer and Mason [6], increasing hydrostatic pressure leads to an increase in both the cavitation threshold and the intensity of cavity collapse. Therefore, the amplitude of acoustic pressure (P_A directly depending on I_{US}) should be in excess as compared to hydrostatic pressure (P_h) for cavitation bubbles to be generated: it can be qualitatively assumed that if $P_h - P_A > 0$, there is no resultant negative pressure and cavitation cannot occur. These combined effects explain why different I_{US} (or P_{US}) lead to different optimum pressures (Fig. 6). In short, an optimum of pressure is achieved due to opposite effects of hydrostatic pressure: a reduction of the number of cavitation bubbles due to a higher cavitation threshold, but a more violent bubble collapse. This optimum pressure is dependent on P_{US} (or I_{US}), not on F_S or *ES*.

Apart from DD_{COD} and *PSD*, apparent viscosity and Herschel-Bulkley parameters of sludge samples treated at 28 °C, in different conditions of pressure (1/3.25 bar) and frequency (12/20 kHz) were also taken into account. Results are given in Table 2. It was found that isothermal *US* (T = 28 °C) at 20 kHz and atmospheric pressure did not significantly affect the rheological behavior of sludge. This result can be generalized to other pressures or frequencies accounting for the discrepancies in between raw samples. A larger reduction of yield stress may be however attributed to the 12 kHz treatment.

4. CONCLUSIONS

This work aimed at investigating the effect of audible F_S sonication on sludge pretreatment for the first time. In addition, the main *US* parameters at same energy consumption (P_{US}, I_{US}, F_S) and operating condition, *i.e.* hydrostatic pressure, were also looked into in a systematic approach to obtain the best conditions for sludge disintegration, then to get general information and trends to be used or checked in other potential applications of physical effects of acoustic cavitation.

The *high P_{US}-short time sonication* procedure was the best option for sludge *US* pretreatment at atmospheric pressure due to the increase in cavitation intensity involving maximum pressures and temperatures. Besides, sludge disintegration was significantly improved by low F_S sonication due to more violent cavitation: up to 64 % of DD_{COD} from 12 kHz isothermal sonication as compared to 20 kHz at low *ES*. Sonication effects on reduction of mean particle size were somewhat improved at low F_S. Additionally, positive effect of hydrostatic pressure associated with high P_{US} and low F_S was also found. Future works should investigate lower F_S as there is no indication for any optimum F_S or for practical limitations at very low F_S.

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TÓM TẮT

TIỀN XỬ LÍ BÙN THẢI BẰNG SÓNG ÂM TẦN SỐ THẤP (12 kHz)

Lê Ngọc Tuấn[1, 2, *], Carine JULCOUR-LEBIGUE[1], Henri DELMAS[2]

[1]*Đại học Khoa học Tự nhiên, Đại học Quốc gia TpHCM, 227 Nguyễn Văn Cừ, TpHCM*

[2]*Université de Toulouse, Laboratoire de Génie Chimique, INP-ENSIACET, Toulouse, France*

[*]Email . *lntuan@hcmus.edu.vn ; ngoctuan.le@ensiacet.fr*

Tiền xử lí bùn thải bằng năng lượng âm thanh (*sound energy*, hay *sonication*), đặc biệt là siêu âm (*ultrasonication*) hiện đang được đầu tư nghiên cứu. Bài báo nhằm mục tiêu đánh giá hiệu quả *sonication* tần số thấp -là nghiên cứu đầu tiên trong lĩnh vực tiền xử lí bùn thải sử dụng

tần số nghe được (*audible frequency* - F_S = 12 kHz). Các thông số chính của *sonication* (công suất -P_{US}, cường độ - I_{US}) cũng được xem xét. Kết quả cho thấy P_{US} càng cao -cường độ *cavitation* càng lớn- thì độ phân rã bùn thải (DD_{COD}) càng đáng kể. Với cùng năng lượng đầu vào ES = 7.000 kJ/kg$_{TS}$, DD_{COD} trong trường hợp P_{US} = 360 W cao hơn trường hợp P_{US} = 50 W khoảng 74 % (I_{US} tương ứng là 37,4 và 5,2 W/cm^2). Hiệu quả của P_{US} cao còn được thể hiện khi so sánh DD_{COD} tại cùng một giá trị I_{US}: với I_{US} = 37,5 W/cm^2, DD_{COD} tăng khoảng 16 % khi chuyển đổi từ hệ thống *50 W - đầu dò đường kính 13 mm* sang hệ thống *360 W - đầu dò đường kính 35 mm*. Bên cạnh đó, mức độ phân rã bùn thải còn được cải thiện đáng kể khi xử lí ở tần số thấp (12 kHz so với 20 kHz) nhờ sự tăng cường *cavitation*: 64 % tại ES = 7000 kJ/kg$_{TS}$. Đáng lưu ý, tác động tích cực của áp suất khi kết hợp với P_{US} cao và F_S thấp lên độ phân rã bùn thải cũng được tìm thấy. Nghiên cứu này cung cấp những thông tin hữu dụng và các xu hướng liên quan đến quá trình *sonication* - là cơ sở để áp dụng hoặc tái kiểm tra trong các ứng dụng tiềm năng khác, đặc biệt là tác động vật lí của *cavitation*.

Từ khóa: bùn thải hoạt tính, năng lượng âm thanh, phân rã bùn thải, siêu âm, suy giảm kích thước hạt, tần số nghe được, tiền xử lí bùn thải.