Sonication pretreatment of sludge: preliminary study of operation parameters

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(Bài nhận ngày26 tháng 08 năm 2014, nhận đăng ngày 02 tháng04 năm2015)

ABSTRACT

The objective of this work was to investigate effects of some key operation parameters serving sludge ultrasonication (US). First, a stirrer speed of 500 rpm was found to be convenient for sludge US pretreatment. A slight decrease in sludge US pretreatment efficacy due to the erosion of the probe surface was observed. Apart from DD_{COD} increase as a result of sludge floc disruption, apparent viscosity decreased as a function of ES during US pretreatment. Finally, sequential isothermal US showed significant improvement of sludge disintegration in some cases, especially at high US power and pressure.

Keywords: *Sequential sonication; Sludge disintegration; Ultrasonic pretreatment; Waste activated sludge.*

INTRODUCTION

Activated sludge processes for wastewater treatment produce large quantity of sludge, commonly treated by anaerobic digestion *(AD)* -a complex and slow process requiring high retention time to convert degradable organic compounds to CH_4 and CO_2 in the absence of oxygen, allowing mass reduction, odor removal, pathogen decrease, and energy recovery in the form of methane. However, hydrolysis -the first stage, is known as the rate-limiting step of microbial conversion. Therefore, biological, mechanical, thermal, and chemical methods, as well as intense electric fields, etc. have been applied in sludge pretreatment [1-5] to rupture the cell wall and facilitate the release of intracellular matter into the aqueous phase to improve biodegradability and enhance AD.

Ultrasonic irradiation (US) is claimed 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].

A part from sludge characteristics (such as pH, total solid concentration *(TS)*, sludge type, *etc.*), US parameters (frequency - F_{US}, intensity - I_{US}, density - D_{US}, power input - P_{US}, *etc.*) and external conditions (hydrostatic pressure, temperature - *T*, *etc.*) - playing a main role in the pretreatment efficiency [6,12-13], some other key operation parameters also need to be investigated: operation stirrer speed (250-1500 rpm), effect of the probe surface status, and operation modes (continuous vs. sequential treatment). The selected values of these key operation parameters are expected to serve for subsequent optimization of sludge *US* pretreatment efficacy.

MATERIALS AND METHODS

Sludge samples

Sludge samples (Table 1) 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) and secondary sludge (liquid form).

Table 1. Characteristics of sludge samples from 1st collection

Parameter		Value		
		a	b	c
Synthetic sludge samples		Defrosted mixed sludge	Defrosted secondary sludge	Defrosted secondary sludge
Total solids (TS)	g/L	28.0	28.0	28.0
Mean SCOD0	g/L	2.7	2.8	4.1
SCODNaOH 0.5 M	g/L	18.5	22.7	22.1
Total COD (TCOD)	g/L	36.5	36.3	39.1
SCODNaOH/TCOD	%	50.7	62.5	56.5

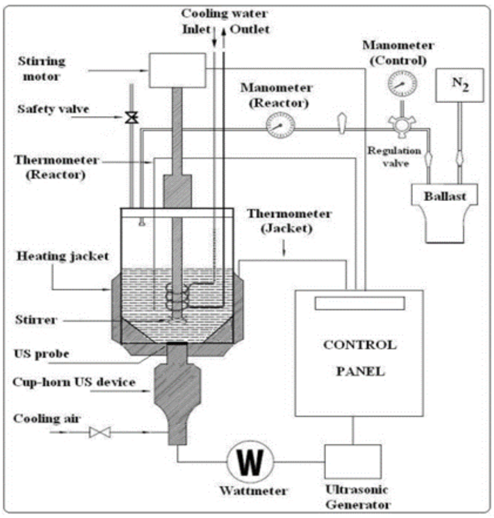

Fig. 1. Ultrasonic autoclave set-up

Sludge samples were preserved in a freezer. Kidak et al. [14] reported that this preliminary maintaining step might change some physical characteristics of the sludge, but it should not significantly affect COD solubilisation results. It was also confirmed in a first step of this work. When performing experiments, the required amount of sludge was defrosted and diluted with distilled water to prepare synthetic sludge samples with a given TS content.

Ultrasound application

Ultrasonic irradiation was emitted by a cup-horn ultrasound unit included in an autoclave reactor which was connected to a pressurized N2 bottle (see Fig. 1). The reactor, made of 316L stainless steel, 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 by a Rushton type turbine of 32 mm diameter, with an adjustable speed up to 3000 rpm. 0.5 L of synthetic sludge sample was used for each experiment. The US equipment, supplied by Sinaptec, 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.

Different US durations (then ES) were tested: $\boldsymbol{ES = (P_{US} * t) / (V * TS)}$, 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).

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 – $SCOD_0$)/($SCOD_{NaOH}$ - $SCOD_0$)*100 (%) [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%.

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 $\tau = K.\gamma^n$ and the apparent dynamic viscosity $\mu_{app} = \tau/\gamma = K.\gamma^{n-1}$. 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%: $\tau = \tau_0 + K.\gamma^n$

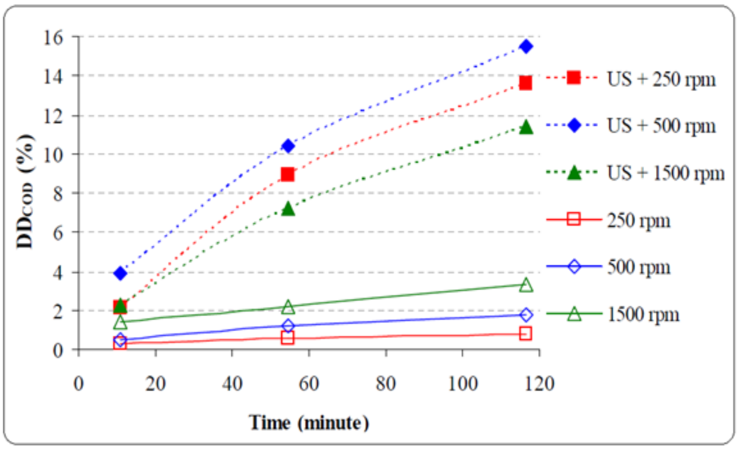

Fig. 2. Effect of stirrer speed on time-evolution of mixed sludge disintegration P_{US} = 150 W, BP, F_S = 20 kHz, TS = 28 g/L (Table 1.a), T = 28±2°C, and atmospheric pressure

Fig. 3. The surface of (a) the brand-new probe, (b) eroded probe, and (c) extremely eroded probe

RESULTS AND DISCUSSION

Effect of stirrer speed

The effect of stirrer speed on DDCOD was investigated and presented in Fig. 2. As expected, for blank experiments (without US), the faster the stirring was (250-1500rpm), the higher the sludge disintegration was (0.8- 3.3%). However, these DDCOD and the differences among the three corresponding series under US were rather low, indicating the main role of the stirrer to be to make a homogeneous dispersion, rather than to efficiently enhance the transfer of organic matters from solid to aqueous phase. Under US, DDCOD increased when raising the stirrer speed up to 500 rpm, but decreased at 1500 rpm. The reactor was not equipped with baffles, consequently, high rotation speed of the whole liquid could result in the centrifugation of particles, leading to less particles present in the central zone, then to a decrease of the sludge US pretreatment efficiency. In addition, aeration could occur and its main effect would be to severely damp the acoustic waves. Therefore, a stirrer speed of 500 rpm was applied in this work.

Effect of the probe surface status

The probe surface has been progressively eroded along the operation time (Fig. 3). Results, depicted in Fig. 4, show a slight decrease in sludge US pretreatment efficacy due to the erosion of the probe surface: about 10% at ES of 7000 kJ/kgTS but less than 5% at higher ES values, which could be ignored.

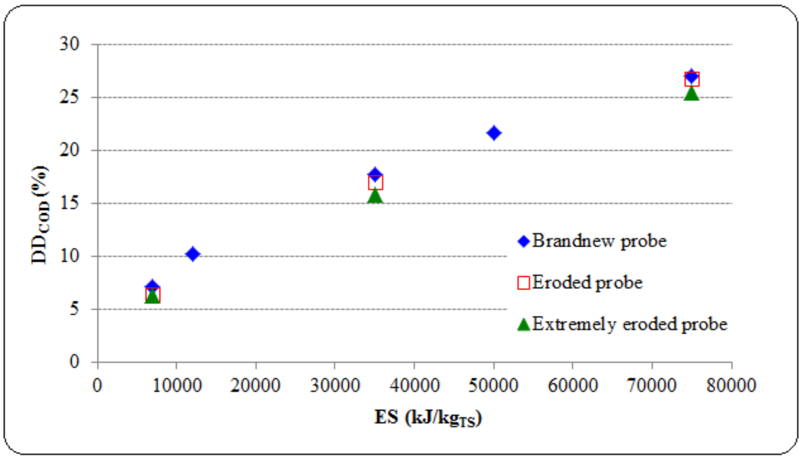

Fig. 4. Effect of the probe status on sludge US disintegration: P_{US} = 150 W, BP, F_S = 20 kHz, synthetic secondary sludge (Table 1.b), T = 28±2°C, and atmospheric pressure

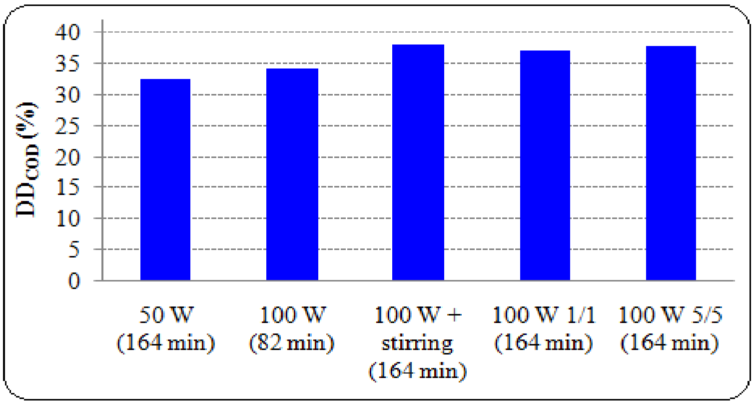

Fig. 5. Effect of isothermal sequential sonication on *DD$_{COD}$*: synthetic secondary sludge (Table 1.c), *SP*, *ES* = 35000 kJ/kg$_{TS}$, 12 kHz, T = 28±2°C, and 1 bar

Effect of sequential isothermal sonication

This part aims at investigating the performance of sequential US which could improve the efficiency of sludge disintegration as in other reported US applications [20-21]. The following conditions were compared for the SP (Fig.5):

(i) 50 W continuous sonication (164 min);

(ii) 100 W continuous sonication (82 min);

(iii) 82 min of 100 W continuous sonication, as in (ii), but followed by stirring up to 164 min, to get the same treatment time as in (i) (marked as 100W + stirring);

(iv) sequence made of 1 min sonication at 100 W followed by 1 min stirring (no sonication) and pursued for a total duration of 164 min (marked as 100W-1/1);

(v) sequence made of 5 min sonication at 100 W followed by 5 min stirring (no sonication) and pursued up to 164 min of treatment (marked as 100W-5/5).

Note that the US pulses of 1 min and 5 min were selected as particle size reduction was mainly achieved within these periods (data was not shown).

For continous sonication, a highest efficiency of the high PUS – short time US mode was observed. When compared at same PUS (100 W), ES (35000 kJ/kgTS) and treatment time (164 min), there is no improvement in DDCOD by using sequential sonication in these conditions. However, it is important to note that after sonication the process of disintegration goes on, slowly but significantly. So in other conditions, alternative sonication and silent periods might be beneficial.

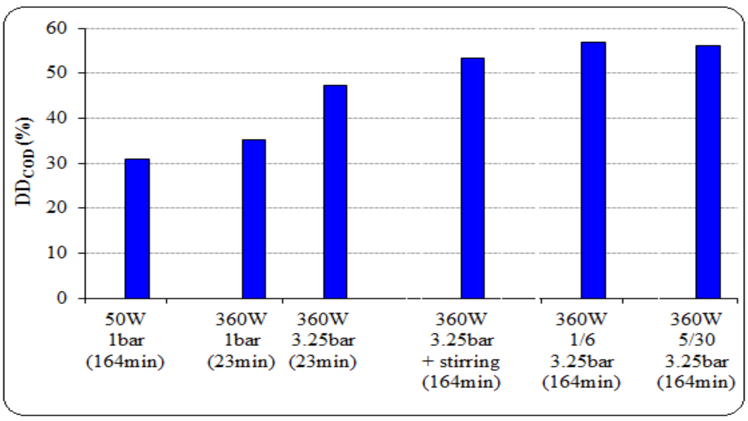

Fig. 6. Effect of isothermal sequential sonication on sludge disintegration: synthetic secondary sludge (Table 1.c), BP, ES = 35000 kJ/kg$_{TS}$, 12 kHz, T = 28±2°C, 1 and 3.25 bar

Apparent viscosity and rheological behavior

Fig. 7 depicts the evolution of apparent viscosity vs. shear rate before and after isothermal US in standard conditions: the sonicated sludge curves are lower than that of raw sludge, indicating a decrease in apparent sludge viscosity μ_{app} (for a given shear rate) as a function of ES (7000-50000 kJ/kgTS). Sludge viscosity is probably controlled by sludge floc structure and interaction [22]; consequently, the disintegration of sludge flocs led to the decrease in viscosity of sonicated sludge [22-24]. As shown in Table 2, the consistency coefficient K which serves as a viscosity index of the system thus decreased. However, yield stress τ_0 and flow index n of sonicated sludge only showed relatively small changes with respect to raw sample, indicating *US* under cooling decreased the apparent viscosity but did not significantly affect the sludge rheological behavior.

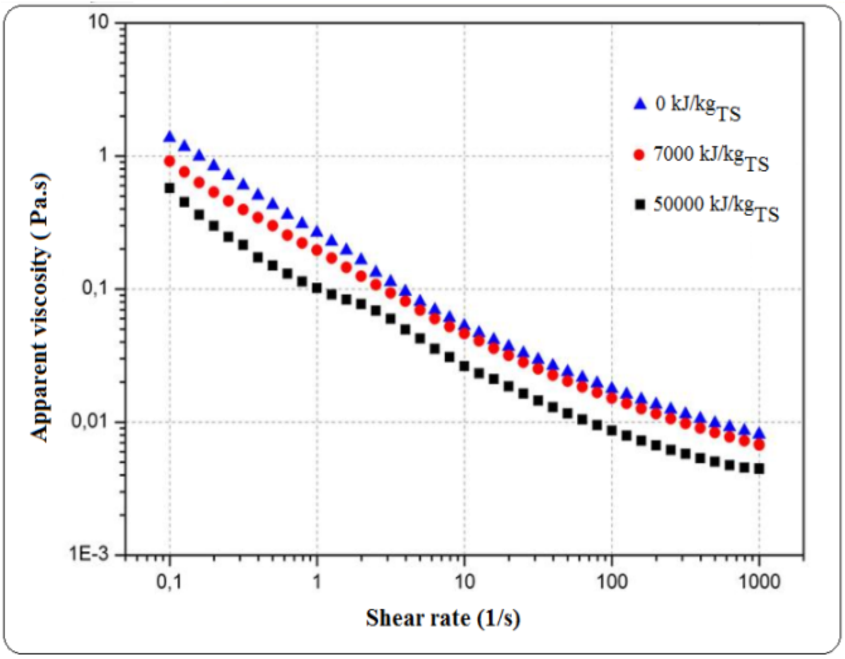

Fig. 7. Apparent viscosity versus shear rate curves for raw and sonicated secondary sludge: P_{US} = 360 W, *BP*, F_S = 20 kHz, *TS* = 28 g/L (Table 1.c), *T* = 28±2°C, and atmospheric pressure

For the effects of temperature and sequential mode, three sludge samples (TS of 28 g/L, Table 1.c) were prepared:

(S1) *Raw sludge*;

(S2) *Sequential sonicated sludge* (sequential 5 min 360 W US-on/30 min US-off pretreatment, ES = 35000 kJ/kg$_{TS}$, 12 kHz, P_h = 3.25 bar, and adiabatic mode), and

(S3) *Shortly sonicated sludge* (ES of 7000 kJ/kgTS at 360 W and 12 kHz + stirring up to 164 min, Ph = 3.25 bar, and adiabatic mode).

That isothermal *US* (T = 28°C) at 20 kHz and 1 bar did not significantly affect the sludge rheological behavior can be generalized to other pressures or frequencies accounting for the discrepancies in raw samples (Table 2). A larger reduction of yield stress may be however attributed to the 12 kHz treatment. In addition, sludge viscosity reduction by mechanical effect of US is enhanced thanks to the effect of temperature (Fig. 8), e.g. μ_{app} at γ = 1 s^{-1} is divided by 4.0 and 7.5 as compared to raw sludge for isothermal and adiabatic *US* (360 W, 35000 kJ/kg$_{TS}$ 12 kHz, 3.25 bar), respectively. In this condition, the flow index comes close to 1, but the yield stress is still significant.

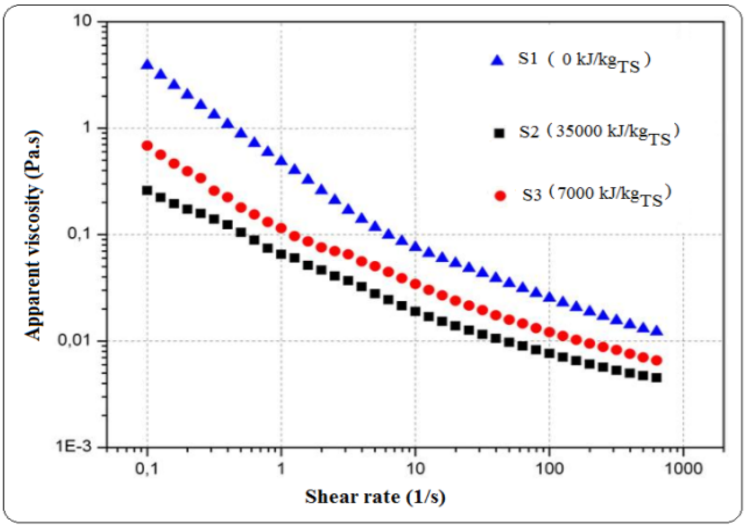

Fig. 8. Apparent viscosity versus shear rate of secondary sludge under *US* pretreatment: 360 W, 12 kHz, *TS* = 28 g/L (Table 1.c), adiabatic sonication, and 3.25 bar

Table 2. Apparent viscosity and parameters of Herschel-Bulkley model for different sonicated samples of secondary sludge (*TS* = 28 g/L, Table 2.c) (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
50000 kJ/kg$_{TS}$	0.089	0.023	0.757	0.102	0.009
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
Conditions of Fig. 8 (Sequential adiabatic US at 12 kHz and 3.25 bar)					
S1 (0 kJ/kg$_{TS}$)	0.312	0.113	0.646	0.486	0.025
S3 (7000 kJ/kg$_{TS}$)	0.117	0.017	0.853	0.115	0.012
S2 (35000 kJ/kg$_{TS}$)	0.069	0.007	0.947	0.065	0.008

CONCLUSIONS

First, it is confirmed specific energy input ES plays a key role in sludge US disintegration. Stirrer speed of 500 rpm was found to be convenient for US pretreatment of sludge. Besides, a slight decrease in sludge US pretreatment efficacy due to the erosion of the probe surface was observed but could be ignored at high ES. As a result of sludge floc disruption, apparent viscosity decreased as a function of ES during US pretreatment, especially under adiabatic US. Finally, sequential isothermal sonication was investigated, and due to consecutive disintegration after sonication, significant improvement of sludge disintegration was achieved in some cases. Such sequential mode should then be checked again when searching for the optimal non-isothermal conditions.

Ảnh hưởng của một số thông số hoạt động đến hiệu quả tiền xử lý bùn thải bằng siêu âm

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TÓM TẮT

Nghiên cứu nhằm mục tiêu đánh giá sơ bộ ảnh hưởng của một số thông số hoạt động đến hiệu quả tiền xử lý bùn thải bằng công nghệ siêu âm. Kết quả cho thấy tốc độ khuấy phù hợp cho hệ thống là 500 vòng/phút. Chất lượng bề mặt đầu dò -bị ăn mòn trong quá trình vận hành- ảnh hưởng nhất định đến hiệu quả tiền xử lý, nhưng không đáng kể khi xử lý với năng lượng siêu âm cao. Năng lượng siêu âm và nhiệt độ vận hành (siêu âm đoạn nhiệt) thúc đẩy sự phân rã bùn thải, theo đó là sự suy giảm độ nhớt của bùn. Ngoài ra, chế độ siêu âm tuần tự đẳng nhiệt (~28^{o}C), đặc biệt ở công suất và áp suất cao, cải thiện đáng kể hiệu quả tiền xử lý bùn thải. Chế độ xử lý này nên được tiếp tục nghiên cứu trong các trường hợp siêu âm đoạn nhiệt.

Keywords: *Bùn thải hoạt tính; Phân rã bùn thải; Tiền xử lý bùn thải bằng siêu âm; Thông số hoạt động*

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