Sludge pretreatment by sonication: Effect of temperature

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ABSTRACT

Effects of temperature (T) rise in conditions of isothermal, adiabatic sonication -US (the same power input -P_{US}), and sole thermal hydrolysis, then its effects associated with P_{US} for the same specific energy input (ES) and the same treatment duration were investigated. The main results were that the evolution of sludge T depended on P_{US}. In cases of the same ES (different P_{US} then different US duration), for the small probe, high P_{US} were still beneficial for sludge disintegration. However, for the big probe, a low disintegration efficiency was achieved at high P_{US} due to the high sludge T which leads to a significant damp of cavitation intensity. In cases of the same ES and treatment time, the sludge disintegration still benefited from high P_{US} if enough time was let for subsequent thermal hydrolysis. Therefore, the combined effect should be taken into account in optimization of US process: cavitation acts mainly during the early stage of the adiabatic US, then US being progressively damped by the increasing T, thermal hydrolysis takes over, being "boosted" by the initial work of US.

Keywords: *Adiabatic sonication, Sludge disintegration, Temperature effect, Waste activated sludge, Ultrasonic pretreatment.*

INTRODUCTION

The wastewater treatment via the activated sludge process produces a large amount of biomass, of which improper disposal generates a significant threat to ecosystems. Thus anaerobic digestion *(AD)* has been widely applied as a feasible method for the sludge treatment. However, the low rate of microbial conversion of hydrolysis stage requires a pretreatment of sludge. *Ultrasonic irradiation (US)* is proved as a feasible and promising mechanical disruption technique for the sludge pretreatment according to the treatment time and power, equating to specific energy input *(ES):* efficient sludge disintegration, improvement in biodegradability and bio-solid quality, increase in biogas/methane production, no need for chemical additives, less sludge retention time, and the sludge reduction [1].

Theory-based, increasing temperature (*T*) will decrease the surface tension and raise the equilibrium vapour pressure of the medium**,** leading to easier bubble formation. However, these kinds of cavitation bubbles contain more vapors that reduce the *US* energy produced by the cavitation, thus reduce the amount of free radicals and also mechanical effects. Besides, great numbers of cavitation bubbles generating simultaneously will provoke the attenuation or dampening effect on the propagation of *US* energy from the emitter through the system [2].

Nevertheless, in terms of sludge disintegration, the sludge ultrasonic pretreatment efficacy increases following an increase in the bulk *T* as *T* alone favors *COD* release. *US* treatment was proved to have two simultaneous effects: (i) vigorous agitation caused by the formation and explosion of tiny bubbles, and (ii) the increase in the bulk temperature. The higher the *T* of solution, the more efficient the *US* disintegration was [3-6]. This is opposite to most power *US* applications as cavitation intensity is higher at low *T*.

This work aims at investigating effects of *T* rise under "adiabatic" sonication then its effects associated with P_{US} (varying P_{US} and probe size) for the same *ES* as well as the treatment duration. The best condition found in this work is expected to enhance the sludge disintegration, to save energy input, and to contribute to the the optimization of sludge *US* pretreatment.

MATERIALS AND METHODS

Sludge samples

Waste activated sludge (Table 1) was collected from Ginestous wastewater treatment plants (Toulouse, France) then sampled in 1 L plastic bottles and stored in a freezer [6]. The sludge was defrosted and diluted with distilled water before experiments to make synthetic sludge samples with 28 g/L of *TS* [7].

Table 1. Characteristics of the sludge sample

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 $SCOD_0$	g/L	2.7	2.8	4.1
$SCOD_{NaOH\ 0.5\ M}$	g/L	18.5	22.7	22.1
Total COD (TCOD)	g/L	36.5	36.3	39.1
$SCOD_{NaOH}/TCOD$	%	50.7	62.5	56.5

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 (Fig. 1). The reactor, made of 316 L 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 *T* of the solution at 28±2 °C during *US*. The solution was stirred by a Rushton type turbine of 32 mm diameter at 500 rpm [7]. 0.5 L of synthetic sludge sample was used for each experiment. The *US* equipment, supplied by Sinaptec, includes a 20 kHz generator associated with 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. Note that a power ratio of 360/50 was applied between *BP* and *SP* as it corresponds to the surface ratio of the probes, allowing comparison at the same I_{US}.

Different *US* durations (then *ES*) 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).

First, the effect of *T* rise under "adiabatic" conditions was preliminarily investigated then its effect associated with P_{US} (varying P_{US} and probe size) for the same treatment duration. Experiments were duplicated and the coefficients of variation of DD_{COD} were about 5 %.

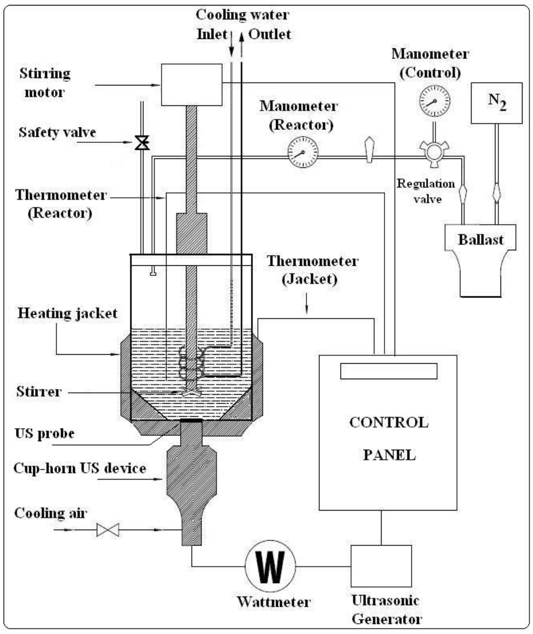


Fig. 1. Ultrasonic autoclave set-up

Analytical methods

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

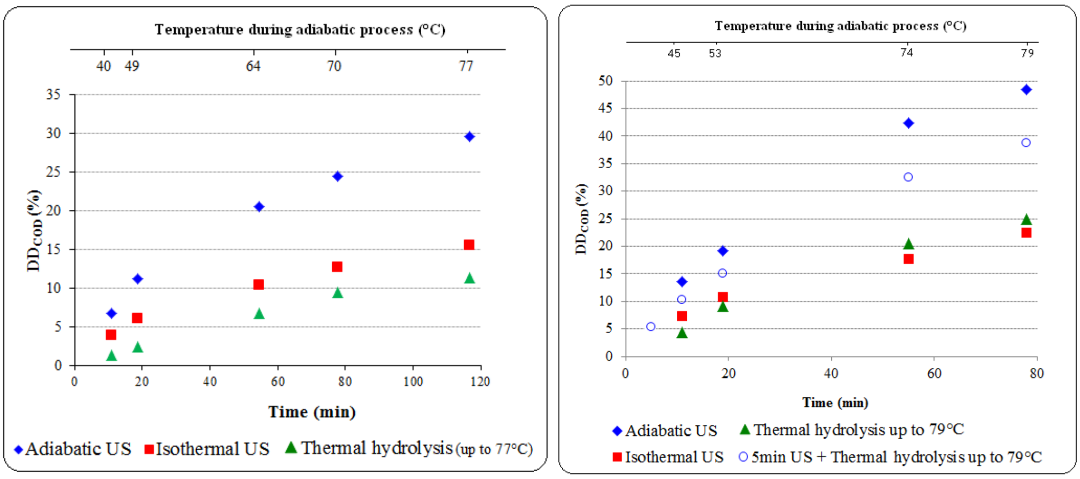
$$DD_{COD} = (SCOD - SCOD_0)/(SCOD_{NaOH} - SCOD_0)*100\ (\%) \qquad [8]$$

To measure the $SCOD_{NaOH}$ value, the sludge sample was mixed with 0.5 M NaOH at room *T* for 24 h [5]. Besides, total *COD* (*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. 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 that has been transferred from the cell content (disruption) and solid materials (solubilisation) into the external liquid phase of sludge [9-10].

RESULTS AND DISCUSSION

Effect of temperature rise under "adiabatic" conditions (without cooling)

To evaluate the individual contribution of extreme macro and micro mixing caused by cavitation and increase in the bulk *T*, different operating procedures were carried out for mixed (Fig. 2A) and secondary sludge (Fig. 2B): (1) *US* under isothermal conditions (cooling at 28±2 °C), (2) *US* under "adiabatic" conditions, (3) thermal hydrolysis: without *US* and with progressive increase of *T* as recorded in (2), and (4) 5 min of *US* and progressive increase of *T* afterwards (this series was conducted only on the secondary sludge).



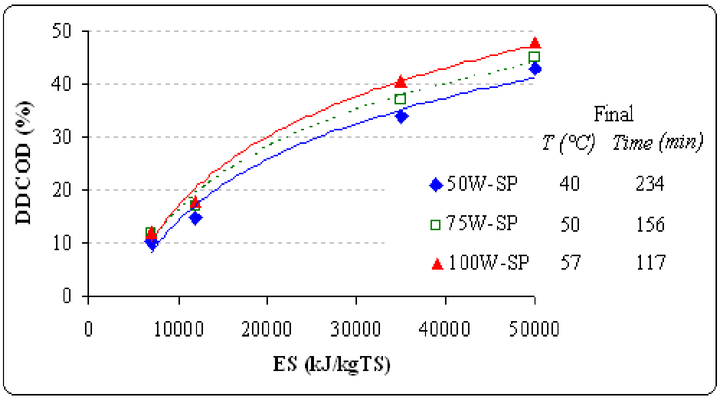
A B

Fig 2. Effect of T profile* on time-evolution of sludge disintegration (DD_{COD}): P_{US} = 150 W, BP, F_S = 20 kHz, TS = 28 g/L, and atmospheric pressure.
(A) Mixed sludge (Table 1A), (B) Secondary sludge (Table 1B)
*The upper x-axis indicates the evolution of T during adiabatic US and thermal hydrolysis (note that higher T at th same ES was achieved with the new equipment)

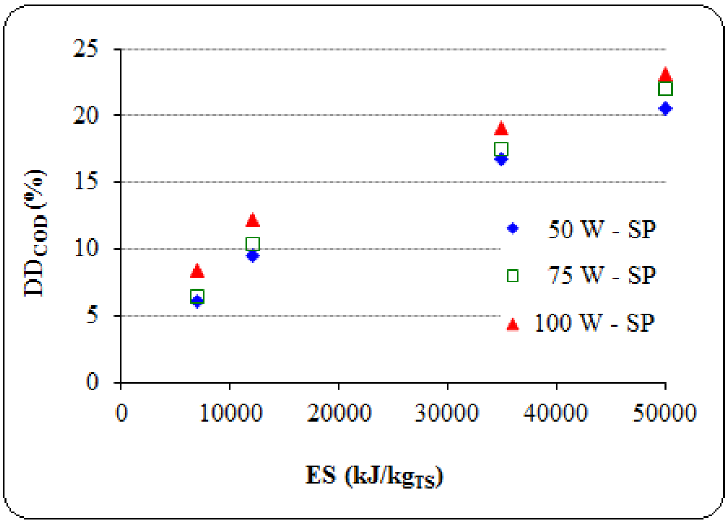
Fig. 2 shows that DD_{COD} values under the adiabatic *US* were the highest, followed by those under the short time *US* + thermal hydrolysis, then under the low *T* sonication and the thermal hydrolysis only. DD_{COD} of sonicated samples under cooling (28 °C) were about half of those obtained under the adiabatic *US*. It could be seen that (i) cavitation and thermal hydrolysis seem to show almost additional effects during the adiabatic *US*, (ii) thermal hydrolysis of early disrupted sludge is faster than that of raw sludge (Fig. 2B); therefore the combined effect is actually more complex: cavitation acts mainly during the early stage of the adiabatic *US*, then the *US* being progressively damped by the increasing *T*, thermal hydrolysis takes over, being "boosted" by the initial work of *US*. The resulting positive effect of combining *US* and *T* for the sludge disintegration is in agreement with Chu *et al.* [8], Kidak *et al.* [6] and Li *et al.* [10] but opposite to most power *US* applications in which *T* only damps cavitation.

Effect of *T* associated with P_{US} for the same treatment duration

First, the effect of *T* associated with P_{US} (varying P_{US} and probe size) on DD_{COD} was investigated at the same *ES*: 50-100 W for *SP* and 50-360 W for *BP*. Results are given in Figs. 3 and 4. As expected, the evolution of the sludge *T* was found to depend on the P_{US}: the higher P_{US} resulted in a more rapid increase of *T* and yielded a higher final value at given *ES* as the reactor was not fully insulated.



A



B

Fig 3. Effect of ES and P_{US} on DD_{COD} (SP, F_S = 20 kHz, secondary sludge TS = 28 g/L – Table 1B, atmospheric pressure): (A) adiabatic US and (B) isothermal US (28 °C). Final temperatures of adiabatic US are also given.

Fig. 3, corresponding to *SP*, shows the positive effect of P_{US} in the adiabatic mode to be not better than in the isothermal mode, *e.g.* at *ES* of 50000 kJ/kg_{TS}, DD_{COD} increased by 12 % and 13 % from 50 to 100 W for adiabatic (Fig. 3A) and isothermal *US* (Fig. 3B), respectively. That meant there was no positive effect of the slight *T* gain at 100 W as compared to 50 W (up to 17 °C) despite the *T* level reached was still moderate.

Conversely, the 50 W-*US* could have benefit from the *T* increase when switching from *SP* to *BP*, as in the latter case higher DD_{COD} were reached despite lower I_{US} (Fig. 4). With *BP*, the high power was only efficient in adiabatic conditions for *ES* lower than 20000 kJ/kg_{TS} (when the increase in sludge *T* and *US* duration were still small). The apparently surprising reverse trend at higher *ES*, then higher *T*, might be explained by the result of lower *US* efficiency at higher *T*. So in this range, the beneficial effect of *T* through thermal hydrolysis should be overpassed by the detrimental effect on the cavitation efficiency.

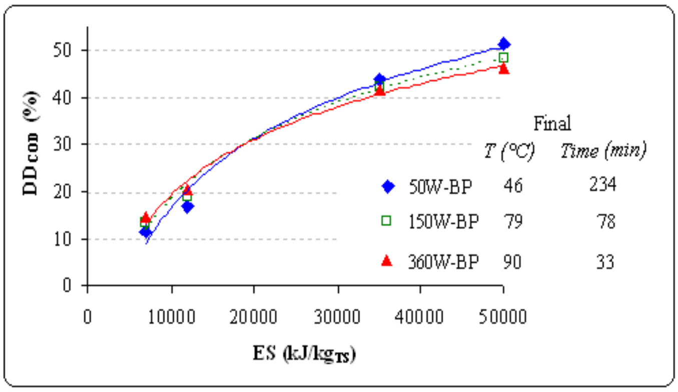


Fig 4. Effect of ES and P_{US} on DD_{COD} under adiabatic sonication (BP, F_S = 20 kHz, secondary sludge TS = 28 g/L – Table 1B, atmospheric pressure). Final temperatures of adiabatic US are also given.

To further understand the effect of *T* on the cavitation efficiency, additional experiments were conducted on another secondary sludge (Table 1C) at 150 W, atmospheric pressure, and isothermal conditions at constant *T* of 28, 55, 80 °C. Results, given in Fig. 5, showed an increase in DD_{COD} when increasing *T* from 28 to 55 °C but a decrease at *T* of 80 °C. Moreover, there was only small differences in DD_{COD} between the isothermal *US* and the sole thermal hydrolysis at the same *T* of 80 °C. It then clear that cavitation intensity significantly dampened at a too high *T* sonication and had much less effect than the thermal hydrolysis.

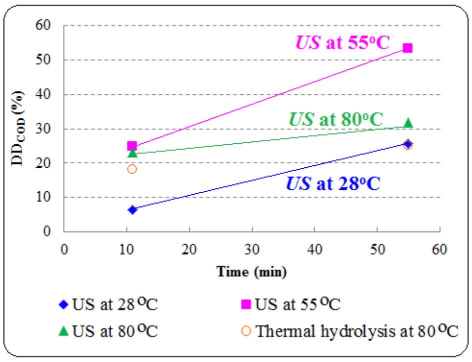


Fig. 5. Effect of temperature on DD_{COD} by isothermal US (20 kHz, P_{US} = 150 W, BP, secondary sludge solutions with TS = 28 g/L – Table 1c, and atmospheric pressure) and thermal hydrolysis.

It should be mentioned that previous results presented in Fig. 4 were achieved on samples rapidly cooled at the end of *US*. In this case, the beneficial effect of high *T* for hydrolysis could not be fully recovered during the shortest treatments as the thermal hydrolysis is a slower process than the *US* solubilisation. Another comparison (using *BP*) could then be made based on both the same *ES* and treatment time, including *US* plus maturation under stirring only. At 50 W, adiabatic *US* was applied in the *ES* range of 7000-50000 kJ/kg_{TS} and the solutions were then cooled down immediately to 28 °C. At 150 W and 360 W, *US* was turned off after the same *ES* values were reached, but the stirrer was still working (no cooling) until the whole durations equaled those of 50 W experiments. Temperature evolutions corresponding to experiments at 50000 kJ/kg_{TS} are depicted in Fig. 6. Results of DD_{COD}, given in Fig. 7, showed that at an atmospheric pressure, the sludge disintegration still benefited from the high P_{US} if enough time was let for the thermal hydrolysis induced by *US* heating to operate. Besides, the positive effect of *high* P_{US} *– short time US* at the atmospheric pressure was found, thanks to the thermal hydrolysis after the *US* disintegration. Of course thermal insulation of our equipment would provide even better results by keeping higher *T*, then saved the *US* energy for the same result in terms of DD_{COD}.

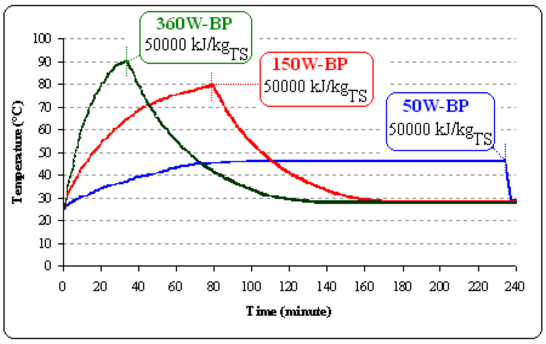


Fig 6. Temperature evolutions for experiments with BP using "adiabatic" US at ES = 50000 kJ/kg$_{TS}$ and stirring afterwards up to 240 min: F_S = 20 kHz, secondary sludge solutions with TS = 28 g/L (Table 1B), atmospheric pressure.

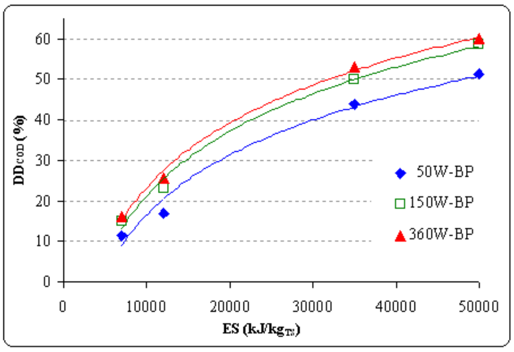


Fig 7. Effect of ES and PUS on DDCOD under adiabatic US followed by stirring up to 240 min (same conditions as in Fig. 6).

To sum up, effects of *T* induced by sonication were investigated in details and the important results are as follows: (i) cavitation and thermal hydrolysis seem to show almost additional effects during adiabatic *US*; (ii) high P_{US} results in a more rapid increase of *T*; (iii) cavitation acts mainly during the early stage of the adiabatic *US*, then *US* being progressively damped by the increasing *T*, thermal hydrolysis takes over, being "boosted" by the initial work of *US*; (iv) sludge disintegration still benefits from high P_{US} then high *T* if enough time is let for the thermal hydrolysis induced by the *US* heating to operate

It was also noted that *US* at high P_{US} resulted in too high sludge *T*, more than 80 °C (Fig. 4), out of the safety range recommended by the manufacturer, which might harm the transducer, lead to unstable P_{US} during *US*, and are not convenient to provide intense cavitation. In agreement with Kidak *et al.* [6], it could be suggested for any scale up operation, the *US* system should be controlled at the possible highest *T* in order to both take advantage of *US* (cavitation and *T* effects) and to maintain the system. Sequential *US* therefore should be investigated to limit the *T* increase and possibly improve the process.

CONCLUSION

Effects of *T* were investigated in conditions of isothermal, adiabatic *US (*same P_{US}*)*, and sole thermal hydrolysis. Subsequently, its effects were looked into associated with P_{US} (varying P_{US} and probe size) for the same *ES* and then the same treatment duration.

Cavitation and thermal hydrolysis seem to show almost additional effects during adiabatic *US*. Besides, the thermal hydrolysis of early disrupted sludge by *US* is faster than that of the raw sludge. Increasing P_{US} leads to an increase in the sludge *T*, thereby affecting the cavitation intensity, and then sludge disintegration efficiency. For the *SP*, increasing P_{US} resulted in a slight *T* increase, and then the positive effect to sludge disintegration was still observed. For the *BP*, a significant increase in temperature following an increase in P_{US} caused significant decrease in the cavitation intensity, and then low sludge disintegration efficiency. In cases of the same *ES* and treatment time, the sludge disintegration still benefits from high P_{US} if enough time is let for thermal hydrolysis to operate. The combined effect is thus proved:

cavitation acts mainly during the early stage of the adiabatic *US*, then *US* being progressively damped by the increasing *T*, thermal hydrolysis takes over, being "boosted" by the initial work of *US*.

It was also noted that *US* at high P_{US} resulted in too high sludge *T* which might harm the transducer and is not convenient to provide the intense cavitation. Sequential *US* thus should be investigated to limit the *T* increase and possibly improve the process.

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Ảnh hưởng của nhiệt độ đối với hiệu quả tiền xử lý bùn thải bằng công nghệ siêu âm

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TÓM TẮT

Ảnh hưởng của nhiệt độ trong điều kiện siêu âm đẳng nhiệt, siêu âm đoạn nhiệt (cùng công suất – PUS), thủy phân nhiệt (thermal hydrolysis), tác động kết hợp với PUS (thay đổi giá trị PUS và kích thước đầu dò siêu âm) ở cùng giá trị năng lượng siêu âm (ES) và thời gian xử lý được nghiên cứu, đánh giá. Kết quả cho thấy nhiệt độ bùn thải biến đổi phụ thuộc vào giá trị PUS. Ở cùng giá trị ES (các tổ hợp PUS và thời gian siêu âm khác nhau), đối với đầu dò kích thước nhỏ, giá trị PUS cao vẫn mang lại hiệu quả phân rã bùn thải. Tuy nhiên, đối với đầu dò kích thước lớn, hiệu quả phân rã bùn thải tương đối thấp ở những PUS cao do sự gia tăng nhiệt độ dẫn đến việc ức chế đáng kể cường độ cavitation. Trường hợp cùng ES và thời gian xử lý, bùn thải vẫn phân rã tích cực ở PUS cao nếu thời gian lưu bùn trong lò phản ứng đủ lâu cho quá trình thủy phân nhiệt. Do vậy, ảnh hưởng tổng hợp của cavitation và nhiệt độ nên được xem xét khi tối ưu hóa quy trình siêu âm: cavitation hoạt động chủ yếu trong giai đoạn đầu của quá trình siêu âm đoạn nhiệt, bị ức chế dần dần khi nhiệt độ tăng cao, lúc này thủy phân nhiệt đảm nhận vai trò phân rã bùn thải (và được tăng cường hơn nhờ tác động ban đầu của siêu âm).

Từ khóa: *siêu âm đoạn nhiệt, phân rã bùn thải, ảnh hưởng của nhiệt độ, bùn thải hoạt tính, tiền xử lý bằng siêu âm.*

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