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Pre-treatment of activated sludge: Effect of sonication on aerobic and anaerobic digestibility

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ABSTRACT

Aerobic and anaerobic digestions were compared in reactors fed with sonicated activated sludge. Sonication treatment of activated sludge led to solubilisation of matter and especially of organic compounds. An important improvement of aerobic and anaerobic biodegradability was observed for a sonication treatment of 108,000 kJ kg TS^{-1} due to the increase of the instantaneous specific soluble COD uptake rate. Sonication led to an increase of biogas production due to the increase of available soluble COD. In this study, sludge sonication prior to aerobic digestion in the aim of enhancing sludge reduction was inconclusive. Under anaerobic conditions, the enhancement of sludge reduction due to sonication depended on the disintegration degree of the sludge. The combination of high disintegration degree of sonicated sludge prior to an anaerobic digestion led to very good results in term of sludge reduction (80%). Energy balance was also studied.

1. Introduction

Treatment and disposal of excess sludge represent a bottleneck of wastewater treatment plants, due to environmental, economic, social and legal factors. Many treatments such as dewatering, digestion, burning, land filling and use in agriculture have been carried out for the disposal of excess sludge. Because of the high cost of these treatments, interest for solutions allowing sludge volume and mass reduction is increasing [1].

Most mechanical and physico-chemical pre-treatment tested so far has targeted cell lysis: ultrasound disintegration [2,3], shear stress forces [4], alkaline pre-treatment [5,6], thermal pre-treatment [7], alkaline combined with thermal hydrolysis [8,9] as well as other oxidation processes (ozone, hydrogen peroxide, etc.) [10,11]. Ultrasonic treatment is one of the most promising recent technologies to reduce sludge production in wastewater treatment plants [12]. Ultrasonic treatment has positive effects: reliability of operation (high degree of research and development), no odour generation, no clogging problems, easiness to implement in a WWTP, good dewaterability of the final sludge but unfortunately negative aspects: erosion of the sonotrode, negative energy balance due to the high energy consumption of the equipment [13].

The major effects of sonication on physico-chemical characteristics of sludge are well known: solubilization and release of organic components measured as COD, proteins, nucleic acids, polysaccharides [14–16], reduction of flocs size [14,17–19], biodegradability improvement [14,20]. So, an ultrasonic pre-treatment of sludge could increase the extent of WAS biodegradability through enhanced hydrolysis. In order to reduce the global production of the sludge, the treated sludge could be recycled in the activated basin (that mean aerobically digested) or injected in a digester (anaerobic treatment) [1,3,21,22]. In both cases, the coupled process (pre-treatment plus digestion) leads to a global reduction of the quantity of effective sludge.

According to the above results, different works have been achieved on the optimization of sonication on waste activated sludge, and then on the enhancement of anaerobic digestion [3,23–28]. On the contrary, literature on the effect of sonication on aerobic digestibility of activated sludge is scarcely available and the effectiveness of aerobic digestion after sonication remains unclear [29]. For [30] the gain in sludge reduction is about 5% when comparing untreated and treated sludge while for [31] the gain of sludge reduction reaches 10%. Simplicity of process and lower capital costs are the advantages of aerobic digestion when compared to anaerobic process and because of these merits, aerobic digestion has been a popular option for the small scale WWTPs. However, high energy cost and lower pathogen inactivation could be the main disadvantages of aerobic digestion [32].

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Nomenclature

COD	chemical oxygen demand ($mgO_2\,L^{-1}$)
COD_{NaOH}	soluble COD after an alkaline hydrolysis ($mgO_2\,L^{-1}$)
DD	disintegration degree (%)
EB	energetic balance ($kJ\,kgTSS^{-1}_{removed}$)
Index 0	initial value
Index f	final value
Index S	parameter value in the soluble phase
Index T	total parameter value
Index P	parameter value in the particulate phase
P	power (W)
q_{COD}	Instantaneous specific soluble COD uptake rate ($mgCOD_S\,gVSS^{-1}\,d^{-1}$)
q_{BG}	instantaneous specific biogas production ($mLBG\,gVSS^{-1}\,d^{-1}$)
S	solubilisation (%)
SE	specific supplied energy ($kJ\,kgTS_0^{-1}$)
t	sonication time (s)
TN	total nitrogen ($mg\,N\,L^{-1}$)
TP	total phosphorus ($mg\,P\,L^{-1}$)
TS	total solids ($g\,L^{-1}$)
TSS	total suspended solids ($g\,L^{-1}$)
V	sample volume (L)
VS	volatile solids ($g\,L^{-1}$)
VSS	volatile suspended solids ($g\,L^{-1}$)
WAS	waste activated sludge
Y	global yield of biogas production ($L_{BG}\,gCOD_S^{-1}$)

The aim of this work is to understand and to compare performances and dynamics of aerobic and anaerobic digestion of sonicated activated sludge; this comparison should permit to choose the most suitable process in regard to sludge reduction.

In a first part, the effect of ultrasonic treatment on sludge solubilisation and membrane integrity was evaluated under different sonication energies. In a second part, the calculation of kinetic parameters, yields and removal efficiencies was done to assess the driving parameters of biodegradability and sludge reduction enhancement due to sonication. The real impact of sonication on aerobic and anaerobic digestion enhancement in term of sludge reduction and energetic balance was finally discussed.

2. Materials and methods

2.1. Waste activated sludge samples characteristics

The activated sludge came from the municipal wastewater treatment plant of Limoges (France) (advanced biological activated sludge treatment of 47,000 m^3 per day of influent composed by 85%, v/v of domestic and 15%, v/v of organic industrial wastewater). Samples of activated sludge were collected from the recirculation loop. Before sonication, activated sludge was concentrated. The characteristics of the sludge are reported in Table 1.

Table 1
Characteristics of the concentrated activated sludge before sonication

pH	7.12
COD_{T0} $mgO_2\,L^{-1}$	18750
COD_{S0} $mgO_2\,L^{-1}$	920
COD_{P0} $mgO_2\,L^{-1}$	17830
COD_{S0}/COD_{T0} (%)	4.91
TS_0 $g\,L^{-1}$	17.81
VS_0 $g\,L^{-1}$	14.25
TSS_0 $g\,L^{-1}$	17.12
VSS_0 $g\,L^{-1}$	13.96
TN_{T0} $mg\,N\,L^{-1}$	2100
TP_{T0} $mgP\,L^{-1}$	1560

2.2. Ultrasonic treatment

The ultrasonic apparatus was a Sonopuls Ultrasonic Homogenisers (*BANDELIN – GM 70*). This apparatus was equipped with a probe and worked with an operating frequency of 20 kHz and a supplied power of 60 W. For each sonication experiment, 50 mL of sludge were filled in a stainless steel beaker and the ultrasonic probe was dipped 2 cm into the sludge. The range of the specific supplied energy varied from 0 to 108,000 $kJ\,kgTS_0^{-1}$. Three specific energies (SE) were investigated: 3600; 31,500; 108,000 $kJ\,kgTS_0^{-1}$. SE was determined by using ultrasonic power (P), ultrasonic time (t), sample volume (V) and initial total solid concentration (TS_0) according to the following equation [27]:

$$SE = \frac{P\,(W) \times t\,(s)}{V\,(L) \times TS_0 (g\,L^{-1})} \quad (1)$$

2.3. Aerobic and anaerobic reactors

The anaerobic and aerobic digestions were studied in eight stirred tank reactors (magnetic agitator (Fisher-Bioblock-France, P = 40 W at 10 rpm). Four of them were dedicated to the anaerobic digestion experiments and the others to aerobic digestion. In this last case air was supplied through a sparger and an air compressor (P = 135 W) to ensure a uniform concentration of 2 $mgO_2\,L^{-1}$. Each reactor had a working volume of 3 L. The reactors were initially filled with 500 mL of inoculum, collected respectively in the aeration tank or in the digestor of Limoges WWTP, and 2.5 L of sonicated sludge (SE respectively: 0; 3600; 31,500; 108,000 $kJ\,kgTS_0^{-1}$). The digestions were carried out at usual temperatures (room temperature for aerobic digestion and 37 °C for the anaerobic digestion (as in the plant)). The produced biogas was collected in calibrated glass cylinders. The cylinders were filled with deionised water acidified with HCL (pH is close to 2) to avoid the solubilization of CO_2 [3].

2.4. Analysis

2.4.1. Chemical analysis

2.4.1.1. Chemical oxygen demand (COD_T, COD_S), total nitrogen (TN_T, TN_S) and total phosphorus (TP_T, TP_S). COD, TN and TP were measured in the total sludge (T) and in the soluble fraction (S). The soluble fraction was evaluated after centrifugation (*SORVALL T 6000 D)* at 3600 × g's for 20 min and filtration through a 1.2-μm membrane. The difference between soluble fraction (S) and total sludge (T) was called particulate (P). COD, TN and TP were evaluated using the micro-method HACH.

2.4.1.2. Total solids (TS) and volatile solids (VS). TS and VS were measured on the total sludge and TSS and VSS on solids of centrifugation (*SORVALL T 6000 D)* at 3600 × g's for 20 min. TS, VS, TSS, VSS measurement were achieved according to normalised methods (ref APHA): samples were heated at 105 °C for 24 h (determination of the total dry matter concentration) and then heated at 550 °C for 2 h (determination of mineral matter). Organic matter concentration was then deduced.

2.4.2. Biological analysis: evaluation of membrane cells integrity

2.4.2.1. Sample preparation. In order to obtain a single cell suspension, sludge pre-treatment was performed according to the method described by [33] using a mechanical blender (IKA-Turrax-

T25; IKA Labortechnik, Germany). A 48-μm pore-size filtration was then done to avoid clogging of the cytometer nozzle. After break-up and filtration, samples were diluted with 0.22 μm filtered phosphate-buffered saline solution to give a final concentration of approximately 10^6 to 10^7 micro-organisms per mL before staining.

2.4.2.2. Cell staining. A SYTOX Green (Invitrogen) solution in dimethyl sulfoxide was delivered at a concentration of 5 mM. This commercial stock solution was stored at −20 °C. SYTOX Green was added to samples at a final concentration of 5 μM. Samples were then incubated during 5 min at room temperature as suggested by manufacturer before flow cytometric analyses.

2.4.2.3. Flow cytometric analyses. They were performed using a FACS Vantage cell sorter (Becton Dickinson, MD, USA) equipped with a 488 nm argon laser (excitation wave-length of SYTOX green). Two parameters were recorded: SSC (Side Scatter) related to cell structure and green fluorescence of SYTOX Green related to cell membrane integrity of bacteria. The results were analyzed with SSC versus green fluorescence cytograms. Green fluorescence was collected in four decades logarithmic scale whereas SSC was collected in linear scale. 10,000 cells were analyzed at a flow rate of 500 cells/s approximately.

2.5. Sludge solubilisation, specific rates and removal efficiencies assessment

2.5.1. Activated sludge disintegration assessment

- *COD (S_{COD}), Nitrogen (S_{TN}) and Phosphorus (S_{TP}) solubilisation* were calculated by using the difference between soluble concentration (Xs) and initial soluble concentration (Xs_0) divided by to the initial particulate concentration (Xp_0) as follows [27]; X representing either COD, nitrogen or phosphorus concentrations:

$$S_X = \left[\frac{Xs - Xs_0}{Xp_0}\right] \times 100\% \quad (2)$$

- *Total solids (S_{TS}) and volatile solids (S_{VS}) solubilisation* were calculated as follows [27]:

$$S_{TS} = \left[\frac{TS_0 - TS}{TS_0}\right] \times 100\% \quad (3)$$

$$S_{VS} = \left[\frac{VS_0 - VS}{VS_0}\right] \times 100\% \quad (4)$$

- *Degree of disintegration (DD)*: the degree of disintegration was defined by [34] as the comparison between ultrasonic process and the maximum soluble COD_{NaOH} obtained by alkaline hydrolysis (i.e. 24 h of incubation at 20 °C with NaOH 1 M)

$$DD_{COD} = \left[\frac{CODs - CODs_0}{COD_{NaOH} - CODs_0}\right] \times 100\% \quad (5)$$

2.5.2. Performances assessment of aerobic and anaerobic digestion

- *Removal efficiencies*: total COD, and VSS removal efficiencies were evaluated according to the following equation:

$$\text{removalefficiency}(\%) = \left(\frac{\text{parametervalue}(t_0) - \text{parametervalue}(t_f)}{\text{parametervalue}(t_0)}\right) \times 100 \quad (6)$$

2.5.3. Rates and yield during aerobic and anaerobic digestion

- Instantaneous soluble COD uptake rate r_{COD}, and instantaneous biogas production rate r_{BG} were calculated according to the following equations:

$$r_{COD} = \frac{\Delta COD_{St_1t_2}}{t_2 - t_1} \quad \text{in mgO}_2\ \text{L}^{-1}\,\text{d}^{-1} \quad (7)$$

$$r_{BG} = \frac{\Delta VBiogas_{St_1t_2}}{t_2 - t_1} \quad \text{in mLBG d}^{-1} \quad (8)$$

- Instantaneous specific soluble COD uptake rate q_{COD} ($\text{mgCOD}_S\,\text{gVSS}^{-1}\,\text{d}^{-1}$) and instantaneous specific biogas production rate q_{BG} ($\text{mLBG}\,\text{gVSS}^{-1}\,\text{d}^{-1}$) were calculated according to the following equations:

$$q_{COD} = \frac{r_{COD}}{\overline{VSS_{t_1t_2}}} \quad \text{mgCOD}_S\ \text{gVSS}^{-1}\,\text{d}^{-1} \quad (9)$$

$$q_{BG} = \frac{r_{BG}}{\overline{VSS_{t_1t_2}}} \quad \text{mLBG gVSS}^{-1}\,\text{d}^{-1} \quad (10)$$

- Global yields of biogas production Y ($\text{L}_{BG}\,\text{gDCO}_S{}^{-1}$) was calculated according to Eq. (11), Vbiogaz represents the total amount of biogas produced between t_0 and t_f:

$$Y = \frac{VBiogas}{(COD_{St_0} - COD_{St_f})V_{reactor}} \quad (11)$$

2.6. Energy balance (EB)

The specific energy to remove 1 kg of TSS was calculated under aerobic and anaerobic conditions.

$$\text{EB aerobic}(\text{kJ kgTSS}^{-1}_{removed}) = \frac{(E_{ultrasound}(\text{kJ}) + E_{air\ compressor}(\text{kJ}))}{([TSS_{removed}(\text{kg L}^{-1})V_{reactor}(\text{L}))} \quad (12)$$

$$\text{EBanaerobic}(\text{kJ kgTSS}^{-1}_{removed}) = \frac{E_{ultrasound}(\text{kJ}) + E_{mixing}(\text{kJ}) - E_{biogas}(\text{kJ})}{TSS_{removed}(\text{kg L}^{-1})V_{reactor}(\text{L})} \quad (13)$$

E_{biogas} was calculated considering that CH_4 represents 55% (v/v) of the total biogas produced and that 1 mL of CH_4 corresponds to 35.95 J.

3. Results and discussion

3.1. Ultrasonic sludge disintegration

The effects of ultrasonic treatment on sludge disintegration were studied in order to evaluate: the preferential pre-treatment conditions in terms of organic matter solubilisation and then the possible improvement of digestion, the source of organic matter solubilisation.

3.1.1. COD, N and P solubilisation

3.1.1.1. COD solubilisation. COD solubilisation increased linearly with increasing specific energy (Fig. 1) for a constant total COD in each experiment. The maximal percentage of COD solubilisation was less than 10% for a specific energy of 108,000 kJ kgTS_0^{-1}. The low solubilisation level can be explained by sludge composition: the mineral matter represented an important part (20%), and the organic matter was mainly particulate (97.9%). Only a little fraction of the particulate might be hydrolysable with a mechanic attack probably be due to the presence of refractory organic compounds (coming from industrial wastewater). Higher power supplied could lead to higher solubilisation level [14,26].

If the COD solubilisation calculation permits the evaluation of the effectiveness of an ultrasonic treatment [29], the disintegration

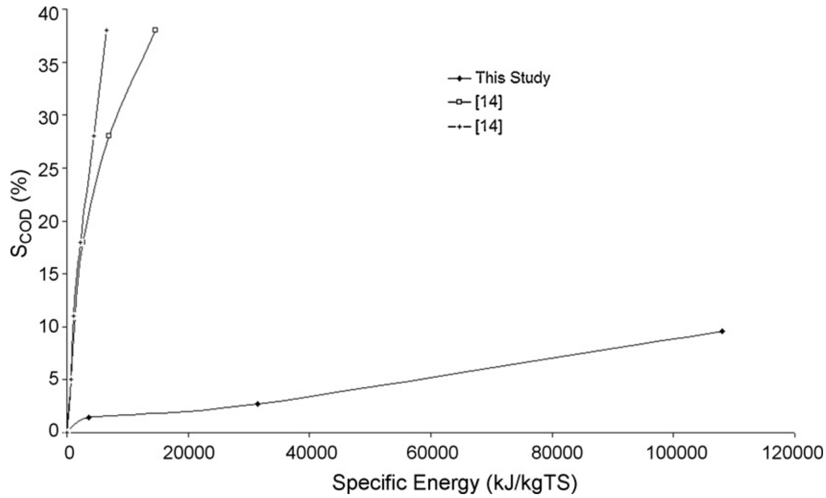

Fig. 1. Comparison of COD solubilisation with different ultrasonic treatments.

degree permits to evaluate the maximum level of sludge solubilisation. The disintegration degree of sonicated sludge increased significantly with increasing specific energy (Fig. 2). It was low for specific supplied energies under 3600 kJ kg TS^{-1} (7.8%), but for supplied energies over this value, the disintegration degree increased strongly. A disintegration degree of 47% was obtained for the maximal specific supplied energy (108,000 kJ $kgTS_0^{-1}$). This result was comparable to the results of [14] at a lower specific energy but better than the results of [18] (less than 20%) for comparable specific energy and lower total solids concentration (Fig. 2).

The discrepancy between the low values of COD solubilisation (10%) and high value of disintegration degree (47%), could be attributed to the nature and composition of the sludge: higher TS concentration led to higher disintegration degree [29].

3.1.1.2. Nitrogen and phosphorus solubilisation. During the experiments, total nitrogen concentration remained constant in the sludges (soluble + particulate concentrations). The total nitrogen solubilisation increased with increasing specific energy (Fig. 3). For specific energies above 3600 kJ $kgTS_0^{-1}$, the increase was linear. For a specific energy of 108,000 kJ $kgTS_0^{-1}$ the solubilisation of nitrogen reached 19.6%, value less important than the solubilisation reached by [14] (40%) with the use of a higher power supplied. The behaviour of phosphorus forms after sonication, rarely described in literature, is reported in Fig. 3. Total phosphorus solubilisation increased with increasing sonication energy up to 31,500 kJ $kgTS_0^{-1}$. The solubilisation reached 12%. For supplied energy above 31,500 kJ $kgTS_0^{-1}$, total phosphorus solubilisation remained constant.

3.1.2. Origins of the solubilised organic matter

The membrane integrity has been investigated by using flow cytometry. Fig. 4 shows cell structure (SSC) versus membrane integrity (FL1) of cells obtained from untreated (A), heat treated (B) and sonicated (C1, C2, C3) activated sludge samples stained with SYTOX green. A heat treated sample (where bacteria were in majority permeabilized) was used as positive control for SYTOX green efficiency. The R1 population corresponded to stained cells, e.g. permeabilized cells. After thermal treatment (60 min at 80 °C), the cells exhibited an increase of green fluorescence due to the loss of membrane integrity. But after sonication (C1, C2 and C3), no membrane integrity loss was exhibited. In all samples, the total cell concentration (permeabilised and intact) remained constant at approximately 7×10^9 cells/ml (data not shown). Intact cells concentration remained approximately constant during sonication whatever the specific energy applied. The increase of soluble forms of carbon, nitrogen and phosphorus cannot be attributed in the

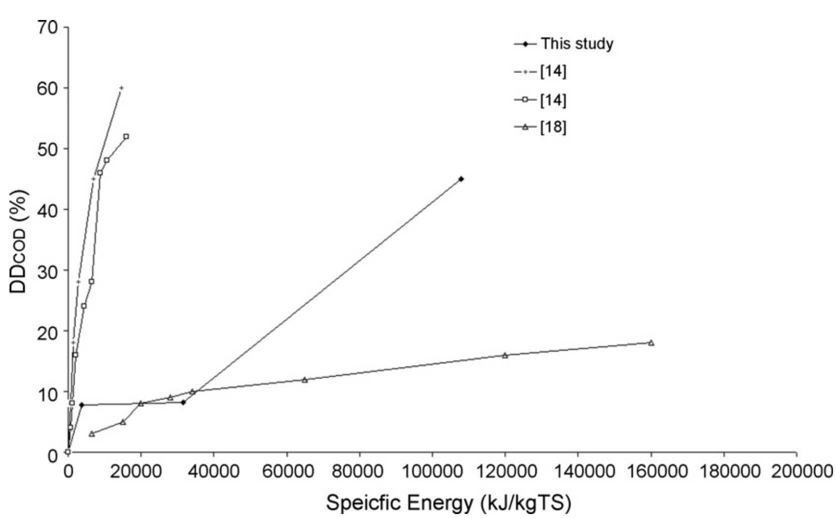

Fig. 2. Comparison of the degree of disintegration for different ultrasonic treatments.

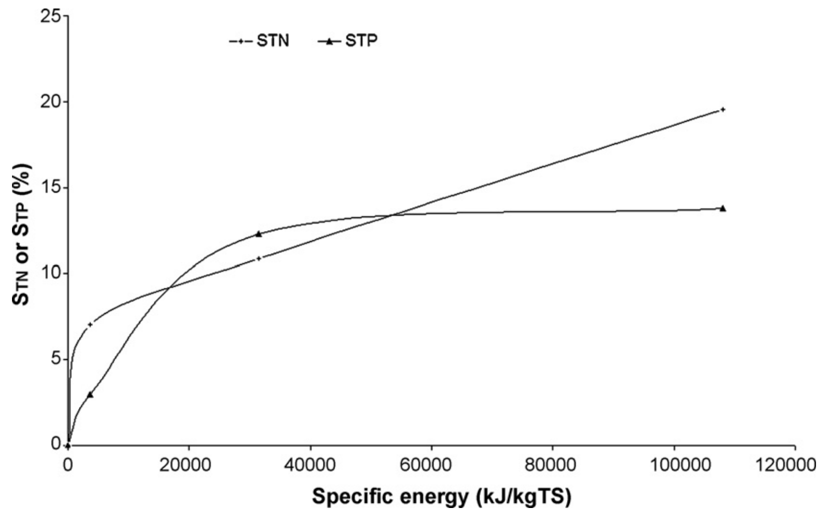

Fig. 3. Total Nitrogen and Phosphorus solubilisation.

conditions of the study to cell lysis but more probably to floc disintegration and especially to EPS destructuration promoting the shift of extracellular proteins and polysaccharides from inner layer to outer, resulting in increasing soluble COD with increasing sonication power supply [31].

3.1.3. Sludge reduction due to the pre-treatment (sonication)

Ultrasonic treatment induced per se sludge reduction due to the solubilisation of total and volatile solids (Fig. 8). The contribution of the ultrasonic pre-treatment to sludge reduction was 7.1, 17.6 and 22.3% for TSS and 13.5, 24.6 and 29.7% for VSS, respectively, for specific energy values of 0; 3600; 31,500 and 108,000 kJ $kgTS_0^{-1}$.

For a constant total matter quantity (in this study, using ultrasound did not mineralize the organic matter), soluble matter increased with SE, whereas particulate matter concentration decreased. The increase of solubilisation of total solids (S_{TS}) versus increasing specific energies between 3600 and 108,000 kJ $kgTS_0^{-1}$ (Fig. 5) is linear. Total solids solubilisation reach 14.65% for the maximal specific energy input. As it is shown Fig. 5, the volatil solids solubilisation (S_{VS}) rapidly increased for specific energies varying between 0 to 31,500 kJ $kgTS_0^{-1}$ and reached 15.8%. For higher specific supplied energies volatile solids solubilisation was slower. S_{VS} reached 23% for the maximal energy input. Volatil solids solubilisation was proportionally more important than total solids solubilisation.

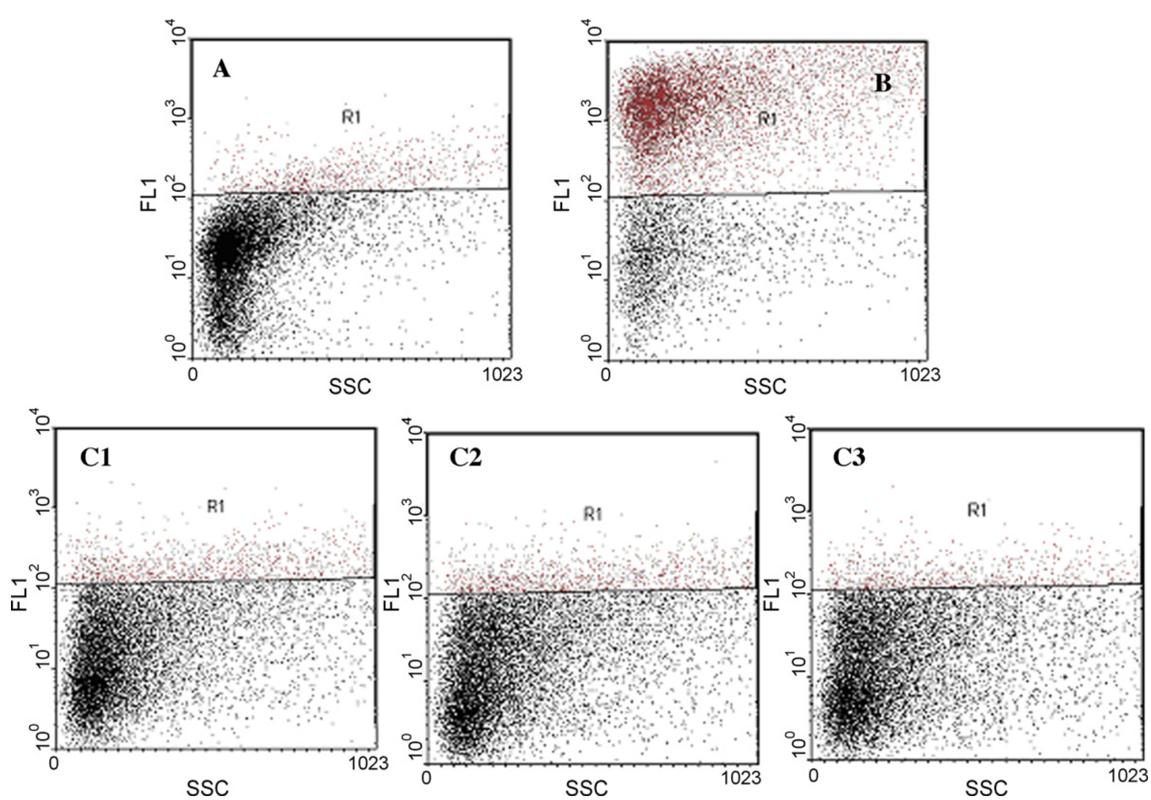

Fig. 4. Cell structure (SSC) versus membrane integrity (FL1) cytogram for untreated (A), heat treated (B) (positive control), and sonicated (C1, C2, C3) activated sludge cells stained with SYTOX green. SE are 3600, 31500 and 108,000 kJ $kgTS_0^{-1}$ for C1, C2 and C3, respectively.

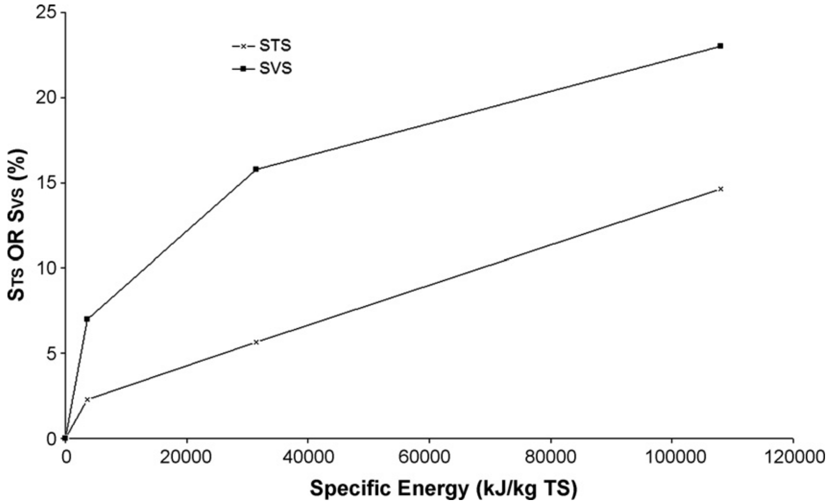

Fig. 5. Total solids and volatile solids solubilisation.

3.2. Performances assessment of aerobic and anaerobic digestion of sonicated activated sludge

The ability of ultrasonic pre-treatment to solubilize particulate organic matter having been demonstrated, the analysis and the comparison of the quality of released organic matter (in terms of its aerobic and anaerobic biodegradability) was required for the future choice of process (both pre-treatment coupled to the digestion step).

3.2.1. Quantitative and qualitative aspects of biogas production of sonicated sludge under anaerobic conditions

In a first time, the anaerobic fermentation was investigated. The total volume of produced biogas was 3.7, 3.9, 5.1 and 6.8 L for non-treated sludge and sonicated sludge respectively with increasing specific energies (Table 2). The amount of produced biogas was as much important as the specific energy was high. This result confirms previous studies on the subject [14,24].

The calculation of instantaneous specific rates of biogas production (q_{BG}) allowed a more accurate understanding of the mechanisms of biogas production (Fig. 6) which revealed a bell-like behaviour comparable whatever the power supplied during ultrasonic treatment. They increased until day 20 and then decreased to 0. Moreover, specific rates of gas production were greatly improved by ultrasonic pre-treatment. The q_{BG} values increased with increasing specific energies. The maximum values were: 9.3, 13, 22.6, and 43 mL biogas gVSS^{-1} d^{-1} respectively for specific energy of 0; 3600; 31,500; 108,000 kJ kgTS$_0^{-1}$.

According to [14,26], the fact that there was a linear correlation between the soluble organic matter to degrade and the volume of produced biogas, means that sonication did not have an impact on the kinetic parameters of conversion of soluble COD to biogas (i.e. the quality of released biodegradable matter is the same whatever the pre-treatment and the specific energy). The biogas to soluble COD yield (Y) was assessed for the different specific energies (Table 2). After 50 days of digestion *Y* values were not significantly influenced by increasing specific energies. So, the increase in biogas production could be attributed to the increase of available soluble COD due to sonication and not to an improvement of Y. This result is in accordance with the work of [25]. A possible explanation of *Y* evolution in this study could be that the important length of the digestion time (50 days) provides enough time for non sonicated sludge to achieve a more complete hydrolysis of particulate matter non-easily biodegradable.

Table 2
Aerobic and anaerobic digestions efficiencies after 50 days

Aerobic digestion

Energy of sonication $E_{ultrasound}$		COD_T removal efficiency (%)	Global VSS removal efficiency (%)	Global TSS removal efficiency (%)	Global energetic balance EB (kJ kgTSS$_{removed}^{-1}$)
kJ kgTS$_0^{-1}$	kJ				
Non-treated (control)	0	79.7	39.3	23.1	56,650,00
3,600	160	80.1	45.4	28.2	46,500,000
31,500	1400	80.7	49.3	31.6	41,550,000
108,000	4806	89.9	61.3	45.3	29,150,000

Anaerobic digestion

Energy of sonication $E_{ultrasound}$		V_{TBG} (mL)	E_{biogas} (kJ)	Y (L_{BG} gCOD$_s^{-1}$)	COD_T removal efficiency (%)	Global VSS removal efficiency (%)	Global TSS removal efficiency (%)	Global energetic balance EB (kJ kgTSS$_{removed}^{-1}$)
kJ kgTS$_0^{-1}$	kJ							
Non treated (Control)	0	3725	74	1.98	86.2	44	38.5	8,400,000
3,600	160	3975	79	1.61	88.3	53.8	54.5	5,930,000
31,500	1400	5095	101	1.92	88.7	66.1	59.6	5,480,000
108,000	4806	6835	135	1.34	92.1	80.7	73.6	4,540,000

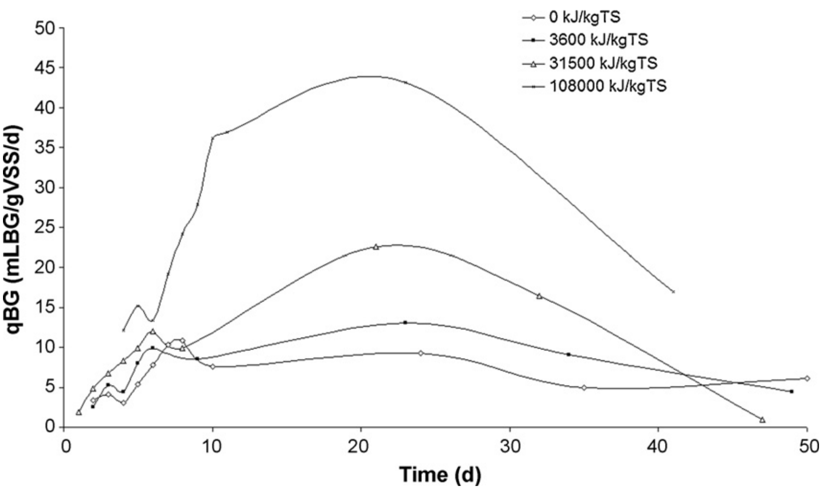

Fig. 6. Instantaneous specific biogas production rate q_{BG}.

3.2.2. Biodegradability of sonicated activated sludge: comparison between aerobic and anaerobic conditions

As it was shown before, sonication induced solubilisation of organic matter and consequently, it is supposed to improve the global digestion of the matter. In fact, aerobic or anaerobic digestions could be different in regard to the rate of matter biodegradation and by extension, to the quality of the released matter. Whatever the conditions (aerobic or anaerobic), total COD removal efficiencies (and thus biodegradability) were strongly influenced by the level of solubilisation of organic matter and thus by the level of the specific energy: as expected, high specific energy supplied led to higher removal efficiency (Table 2). For specific energies (SE) lower than 31,500 kJ $kgTS_0^{-1}$ the removal efficiency of total COD was not improved under either aerobic or anaerobic conditions. Nevertheless, an interesting improvement of total COD removal was observed for a specific energy of 108,000 kJ $kgTS_0^{-1}$. After 50 days the removal efficiencies were respectively 89 and 92% under aerobic and anaerobic conditions compare to 80 and 88% for the control. The calculation of instantaneous specific rates q_{COD} aimed to a better understanding of soluble COD removal efficiencies (Fig. 7(a) and (b)). The specific instantaneous soluble COD uptake rate increased with increasing specific energy until day 15 either under aerobic and anaerobic conditions. After this period, q_{COD} drastically decreased to reach a very low value (close to 0). The maximal q_{COD} values obtained after 15 days for a specific energy of 108,000 kJ $kgTS_0^{-1}$ were respectively 10.2 and 7.8 $mgCOD_S$ $gVSS^{-1}$ d^{-1} under aerobic and anaerobic conditions. The increase of soluble COD due to sonication positively influenced the soluble COD removal of specific removal rates. Ultrasonic pretreatment might enhanced enzymatic activities and promoted the shift of extracellular proteins, polysaccharides and enzymes from inner layers of sludge flocs to outer layers resulting in improved efficiency of digestion [31].

3.2.3. Assessment of sludge reduction under aerobic and anaerobic conditions

3.2.3.1. Quantitative estimation of sludge reduction. The relative contribution of pre-treatment and digestion step on sludge reduction were investigated (Fig. 8(a) and (b)). Ultrasonic treatment of sludge induced *per se* sludge reduction (3.1.3). The specific contribution of aerobic and anaerobic digestions on VSS reduction were, respectively, after 50 days: 39, 32, 25 and 32%, and 44, 40, 41 and 51%. Under aerobic conditions VSS removal was not improved by activated sludge sonication and a slight decrease of VSS removal in reactor fed with sonicated sludge was observed. Moreover, VSS removal remained constant after about 35 days whatever the conditions. Other authors demonstrated on the contrary VSS removal improvement after sonication and aerobic digestion [29–31]. However, the enhancement of sludge reduction under aerobic conditions could be greatly influenced by the density of the power supplied. In this study, it was 60 W, corresponding to a density of 1200 W L^{-1}, value 2 times lower than the density applied by [31]. Sludge reduction under anaerobic conditions was slightly improved by sonication pre-treatments under certain conditions: VSS removal efficiency was improved by 14% when the reactor was fed with sludge treated with the maximal sonication energy of 108,000 kJ $kgTS_0^{-1}$.

The global VSS removal including the pre-treatment and the digestion for increasing specific energies were: 39; 45, 49 and 61% and 44, 53, 66 and 80%, respectively, under aerobic and anaerobic conditions (Table 2). The improvement of VSS removal under aerobic conditions was only due to the pre-treatment itself; on the

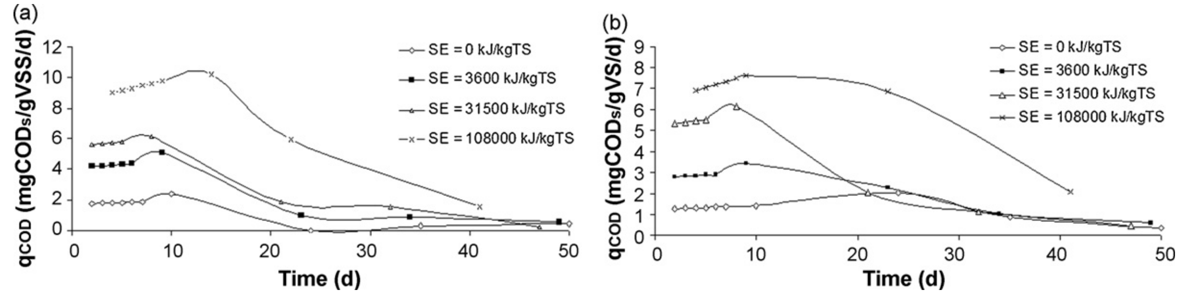

Fig. 7. Instantaneous specific soluble COD uptake rate q_{COD} during aerobic (a) and anaerobic (b) digestions.

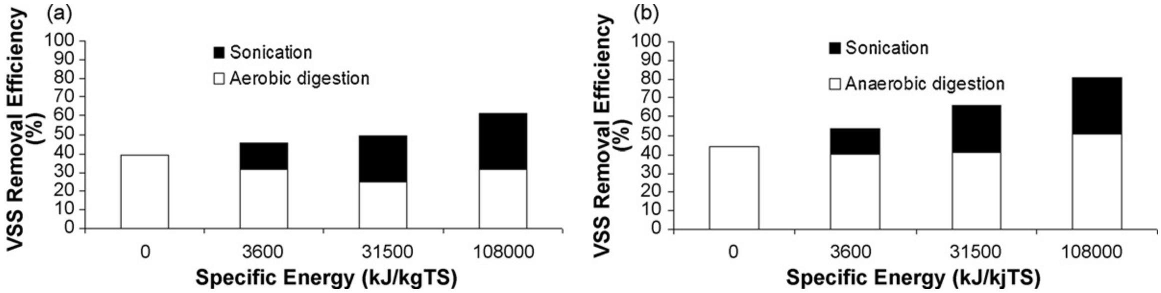

Fig. 8. Comparison of the relative contribution of ultrasonic pre-treatment (black stick) and aerobic (a) or anaerobic (b) digestion (white stick) to sludge reduction for different specific energies.

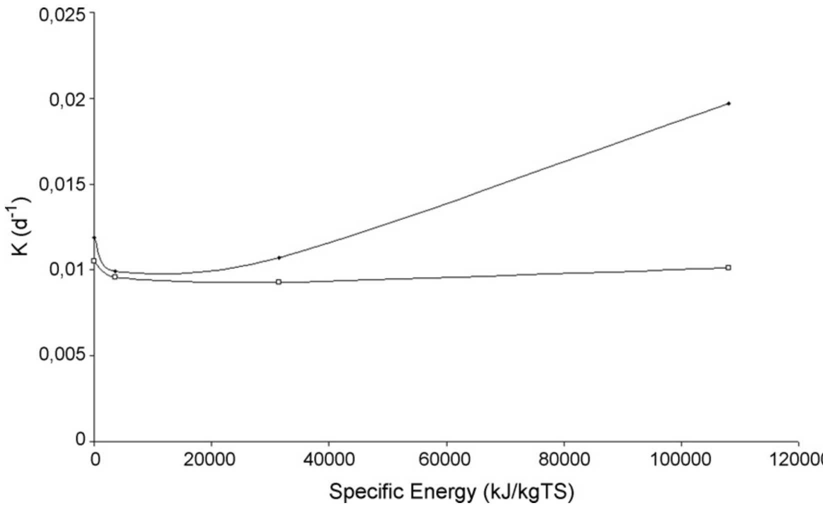

Fig. 9. Evolution of *K* (constant of reaction rate, VSS consumption rate) as a function of the specific energy applied to the sludge.

contrary, under anaerobic conditions, sonication positively influenced VSS removal especially when a sufficient specific energy is supplied to the sludge. The best result in term of VSS removal (80%) was obtained for anaerobic digestion of sonicated sludge with a specific energy of 108,000 kJ $kgTS_0^{-1}$ (degree of disintegration equal to 47%), in other ways, an improvement of 82% of sludge reduction was noticed when compared to the control. The proportional improvement of digestion is less important than the contribution of the pre-treatment itself.

3.2.3.2. Qualitative assessment of sludge reduction. The determination of kinetics of sludge reduction enhancement due to sonication under aerobic and anaerobic conditions was investigated. The initial slope of VSS decrease could be described with a first order reaction dynamics with *K* the constant of reaction rate in d^{-1}:

$$\frac{d[VSS]}{dt} = -K[VSS] \quad (14)$$

K (calculated in each aerobic and anaerobic reactors containing pre-treated sludge) has been plotted against specific energy both in aerobic and anaerobic conditions (Fig. 9): sonication of sludge before aerobic treatment did not improve significantly the kinetic of sludge reduction. On the contrary, *K* and thus sludge reduction were improved during anaerobic digestion when the sludge was previously sonicated. So, anaerobic digestion was as much efficient in sludge reduction, when the disintegration degree of sludge was important.

3.2.3.3. Energy balance of sludge reduction. The energy required by sonication ($E_{ultrasound}$ (kJ)) and the energy recovered by biogas production (E_{biogas} (kJ)) are compared in Table 2. Without any optimisation of energy balance, the energy input required by sonication was not offset by the recovery of energy due to the greater gas production. Nevertheless, the global specific energy used to remove TSS was decreasing with increasing specific energy and anaerobic conditions were more favourable to sludge reduction. The global energy balance confirmed the interest of using high ultrasonic treatment (108,000 kJ $kgTS_0^{-1}$) followed by an anaerobic digestion.

4. Conclusions

This work aimed to compare the performances (biodegradability and sludge reduction) of aerobic and anaerobic reactors fed with sonicated activated sludge.

- Ultrasonic treatment of sludge at different specific energies: 3600, 31,500, 108,000 kJ $kgTS_0^{-1}$ led to solubilisation (disintegration) of matter. TS, VS, Total Nitrogen, and COD solubilisation increased with increasing specific energy supplied. COD solubilisation was proven not to be a relevant parameter of sonication efficiency. The disintegration degree gave more interesting information as it included the maximal potential of sludge disintegration. Poor solubilisation results (10% in this study) could correspond to good disintegration degree (47% in this study). In the conditions of the study (f = 20 Hz, power supply = 60 W, TS: 17.8 g/L), flow cytometry experiments showed that organic matter solubilisation was not due to cell membrane breakage but more probably to floc breakage.

- The biodegradability of the sonicated sludge was comparable under aerobic and anaerobic conditions. The improvement of aerobic and anaerobic performances in term of total COD removal was noticeable only for high specific energy (108,000 kJ $kgTS_0^{-1}$) or disintegration degree (47%) and was due to higher instantaneous specific rates.
- Biogas production was greatly improved by sonication of WAS. Nevertheless, it was proven that the yield of biogas to soluble COD conversion remained constant.
- Ultrasonic pre-treatment of activated sludge did not improve the total sludge reduction under aerobic conditions. The improvement of global sludge reduction production of sonicated sludge under aerobic conditions is only due to the ultrasonic treatment itself.
- Ultrasonic pre-treatment improved sludge reduction under anaerobic condition to certain extends. This improvement is closely conditioned to the effectiveness of ultrasonic treatment in term of degree of disintegration or solubilisation. The improvement of global sludge reduction production of sonicated sludge under anaerobic conditions was due to both ultrasonic treatment and digestion.
- Sludge digestion was found to be enhanced by ultrasonic pre-treatment under anaerobic conditions which was not the case under aerobic conditions. This enhancement is greatly conditioned to the quality of the ultrasonic treatment. The disintegration degree was found to be a good parameter for the evaluation of the effectiveness of an ultrasonic treatment.
- The global VSS removal (including the pre-treatment and the digestion) was improved by 82 and 36% respectively after 50 days of anaerobic or aerobic digestion for a specific energy supplied of 108,000 kJ $kgTS_0^{-1}$. This improvement was mainly due to the pre-treatment itself.
- The energy input required by sonication was not offset by a greater gas production but the energy balance was largely in favour of highly ultrasonically pre-treated sludge (108,000 kJ $kgTS_0^{-1}$) followed by an anaerobic digestion.

In these experimental conditions, sludge sonication prior to aerobic digestion in the aim of enhancing sludge reduction was inconclusive. On the contrary, the combination of high disintegration degree of sonicated sludge prior to an anaerobic digestion led to very good results in term of sludge reduction (80%). The combination of these two processes appears to be the most suitable with moreover energy save and certainly financial recovery.

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