

Techno-economic evaluation of thermal treatment, ozonation and sonication for the reduction of wastewater biomass volume before aerobic or anaerobic digestion

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ABSTRACT

Aerobic and anaerobic digestions were compared with different sludge reduction processes such as ultrasonic, ozone, and thermal treatments. Each treatment was tested under the following conditions to improve batch aerobic or anaerobic digestion: ultrasound (200, 000 kJ kgTS₀⁻¹), thermal (40 °C, 60 °C, 90 °C for 90 min, 120 °C 15 min, 1 bar), and ozonation (0.1 gO₃ gTS₀⁻¹). The different pretreatments induced organic matter solubilisation and intrinsic sludge reduction (total suspended solids): ultrasound (47%), thermal 90 °C (16%), ozone (15%), thermal 60 °C (9%), thermal 40 °C (5%), autoclave (120 °C) (4.2%). TSS (and also VSS) solubilisation were found to be highly correlated to the pretreatment ability to break the flocs rather than to specific energy input. The total values of TSS reduction ranged from 57% to 71% under aerobic conditions and from 66% to 86% under anaerobic conditions. TSS solubilisation after pretreatment can be considered as a predictive parameter of sludge volume reduction enhancement after aerobic or anaerobic digestion while specific energy input did not show anything or negligible impact. In our experimental conditions, ultrasound and ozone led to the best TSS removal improvement after both aerobic (30% and 20%) and anaerobic digestion (20%). Ultrasonic and ozone pretreatments prior to aerobic or anaerobic digestion led to the best reduction of the specific energy required for removing 1 kg of TSS compared to the control. Anaerobic digestion was globally more effective (compare to aerobic digestion) in enhancing sludge production reduction.

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1. Introduction

The well-known biological process called “activated sludge process” is the most widely used process for biological wastewater treatment nowadays, but it results in the generation of a considerable amount of waste that has to be disposed of. This sludge contains high fractions of volatile solids (VS) and retains large amounts of water before a possible drying (>95% by weight), resulting in the production of extremely large volumes of residual solids, and significant disposal costs. Thereby, the conventional method leads to water pollution problem into solid waste disposal problem. The main alternative methods for sludge disposal are: landfilling, land application and incineration. Incineration is quite expensive and land application (or agricultural use) is subject to reservations from farmers and consumers.

There is therefore, a growing interest in developing technologies to reduce the wastewater sludge generation [1]. Aerobic or anaerobic

digestion of waste activated sludge (WAS) is often slow due to the rate limiting cell lysis step. Several systems combining biological and physico-chemical treatment have been studied in order to improve the aerobic and anaerobic biodegradation of solid wastes.

Flocs destruction and cells disruption can be achieved by various methods: ultrasonic disintegration [2,3], shear stress forces [4], alkaline pretreatment [5], thermal pretreatment [6], alkaline combined with thermal hydrolysis [7,8] as well as other oxidation processes (ozone, hydrogen peroxide) [9,10].

Possible applications of ultrasonic treatment have increased both in number and diversity of devices and ultrasonic treatment is recognized as a promising technology to reduce sludge production [11].

Solubilisation and release of organic components such as COD, proteins, nucleic acids, extracellular polysaccharides (EPS) [12–14], reduction of flocs size [12,15–17], and biodegradability improvement [12,18] are the main effects of sonication on sludge physico-chemical parameters.

Previous reports on the use of sonication before aerobic degradation (i.e. on the recycling loop) or anaerobic digestion led to significant reduction in the sludge production [1,3,19,20].

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Nomenclature

COD	chemical oxygen demand ($\text{mgO}_2 \text{L}^{-1}$)
TS	total solids (mgL^{-1})
TSS	total suspended solids (mgL^{-1})
VS	volatile solids (mgL^{-1})
VSS	volatile suspended solids (mgL^{-1})
S_X	solubilisation of parameter X (%)
P	power (W)
V	sample volume (L)
SE	specific supplied energy (kJ kgTS_0^{-1}) input during the pretreatment
E_T	total energy (pretreatment + digestion) (kWh)
EB	energetic balance ($\text{kWh kgTS}_{\text{removed}}^{-1}$)
WAS	waste activated sludge
t	sonication time (s)
Y	global yield of biogas production (mL BG gCOD_5^{-1})
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
BG	biogas

Sludge ozonation was shown to be one of the most cost effective technologies with the highest disintegration capability [21,22]. Ozone is a powerful oxidant which breaks the structure of natural organic matter and enhances the transformation of high molecular weight compounds into low molecular weight (MW) products, such as carboxylic acids, hydrophilic acids, carbohydrates, amino acids, etc. [23,24]. Sludge disintegration by ozone is well described by the sequential decomposition processes of floc disintegration, solubilisation and mineralization [25]. The feasibility of the activated sludge system coupled with an ozonation process was checked through the full scale plant operations without excess sludge production [26,27]. Ozonation was also considered as an attractive pretreatment procedure for solid hydrolysis prior to aerobic and anaerobic digestion [28].

Sludge heating could also be an interesting pretreatment approach to increase the methanogenic potential of the sludge [29] because it results in the breakdown of the gel structure of the sludge and the release of intracellularly bound water [30]. When sludge heating is to be performed in order to reduce sludge production, sludge solubilisation process would be necessary. For this purpose, two main temperature brackets are to be considered (from economic or efficiency point of view): temperatures either higher or lower than 150°C [31]. The necessary temperatures to obtain solubilised sludge would be around $160\text{--}200^\circ\text{C}$ [6,7]. Therefore, this treatment allows a high level of solubilisation, modification in sludge characteristics (increase in filterability and viscosity reduction) and reduction of pathogen microorganisms [32,33]. In fact, the temperature of treatment has more impact on sludge solubilisation than the time of treatment [6,34].

The association of a process for hydrolysis of organic matter and/or a process for biomass stress together with a biological process (either aerobic or anaerobic) was reported to improve sludge reduction. Different works have been achieved on the enhancement of anaerobic digestion [2,35–40]. On the contrary, literature on the effect of pretreatment on aerobic digestibility of activated sludges is scarcely available.

Few data are available in the literature concerning any comparison between aerobic and anaerobic digestion on sludge production improvement after pretreatment [41]. This is also the case for information related to the potential links between pretreatment

performances (such as solubilisation) and anaerobic or aerobic digestion performances.

The aim of this work was to compare the dynamic, technical and economical performances of aerobic and anaerobic digestion of sludge subjected to different pretreatments. In the first part, the effect of different sludge reduction processes such as ultrasonic treatment, ozone treatment, and thermal treatment on sludge solubilisation was assessed. In the second part, different combinations of pretreatment and digestion (aerobic/anaerobic) were evaluated in terms of (1) sludge production reduction improvement, (2) relation with initial solubilisation parameters or specific energy input and (3) energetic performances comparison. Then comparisons would provide valuable information in regard to selection of the best types of digestion and allow the evaluation of the best pretreatment for sludge reduction and cost saving.

2. Materials and methods

2.1. Waste activated sludge characteristics

The activated sludge was received from the municipal wastewater treatment plant of Limoges (France) (285,000 people equivalent). Samples of activated sludge were collected from the recirculation loop. Before pretreatment, activated sludge was concentrated up to 14.26 gL^{-1} of total solids (TS), with standard deviation ($\text{SD} = 2.18 \text{ gL}^{-1}$) and volatile solids (VS) content was 72.82% TS ($\text{SD} = 3.32\%$). Sludge was stored at 4°C .

Two series of samples were studied: ultrasound, autoclave, thermal treatment 90°C and compared to control 1, and thermal treatment 40°C , 60°C , ozone and compared to control 2, the main difference being the time when they were collected.

2.2. Pretreatment conditions

2.2.1. 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 continuous supplied power of 50 W. For each sonication experiment, 50 mL of sludge were filled in a stainless steel beaker and the ultrasonic probe was dipped into the sludge.

The specific energy applied to the sludge was ($200,000 \text{ kJ kgTS}^{-1}$).

Specific energy (SE) is defined according to Eq. (1) with P the ultrasonic power (W), t the ultrasonic time (s), V the sample volume (L) and TS_0 the initial total solid concentration (gL^{-1}):

$$\text{SE} = \frac{P \times t}{V \times \text{TS}_0} \quad (1)$$

2.2.2. Ozone treatment

The ozonation device was composed of an ozone generator (TRAILIGAZ OZONE SAS), an ozone analyser (964 BT), an oxygen cylinder for oxygen supply, a contact column (1800 mm high water column), an ozone destructor (supplied by TRAILIGAZ), an air pump and a tail gas adsorption flask with potassium iodide inside. A pre-calibrated rotameter with a regulating valve for gas flow adjustment and an output control scale was mounted in front of the generator assembly. Pipes and valves were made of polypropylene (for water flow) and PTFE (for ozone flow), and the contact column was made of PVC (opaque in the base of column and transparent in the top of column). The power of the ozone generator and the oxygen flow and air pressure were set in all the tests at 180 W (from 50 W to 200 W) and 600 NL h^{-1} (from 300 NL h^{-1} to 800 NL h^{-1}), and 0.7 bar (from 0.0 atm to 1 atm), respectively, ensuring a constant supply of O_3 to the contact column. Ozone gen-

eration performances were determined by measuring the gas flow rate. Maximum ozone production was found to be 50 g/Nm³. Residual ozone was measured at the outlet of the contact column during the tests.

All the experiments were conducted at room temperature and in a semi-batch mode by ozone bubbling into the sludge sample. As sludge ozonation leads to pH decrease, pH was readjusted to 7.0–7.2 after ozonation by using NaOH (1N).

For each ozonation experiment, 700 ml of sludge was ozonated for 60 min in a cylindrical glass contactor (effective volume: 2L). Ozone consumption ratio was calculated from difference between the amount of ozone in the inlet and in the outlet of the ozone contactor per amount of initial ozonated sludge since dissolved ozone was not detected. The ozone dose applied was 0.1 gO₃ gTS⁻¹.

2.2.3. Thermal treatment

Two different types of thermal treatment were investigated: autoclave and thermostatic bath. Autoclave Reactor was a Précilave no. 942 (Autoclave, France), the temperature of treatment was 121 °C under 1 bar for 15 min and the sample volume was 0.7 L. Thermostatic bath reactor was an Isotemp 120. Three temperatures were applied to the sludge: 40 °C, 60 °C and 90 °C for 60 min. Five hundred milliliters of sludge were put in close bottles to avoid water loss by evaporation. The input power was set to 400 W, 600 W, 900 W and 6000 W for 40 °C, 60 °C, 90 °C and 121 °C (autoclave), respectively.

2.3. Aerobic and anaerobic reactors

Anaerobic and aerobic digestions were performed in eight stirred tank reactors. Four of them were dedicated to the aerobic digestion and the rest to the anaerobic digestion. The working volume of each reactor was 3 L. The reactors were initially filled with 500 mL of inoculum, collected from the aeration tank or in the digester of Limoges WWTP, and 2.5 L of pre-treated sludge (ultrasound, thermal, ozone) respectively. The digestions were carried out at room temperature for aerobic digestion and at 37 °C for the anaerobic digestion. The produced biogas was collected in calibrated glass cylinders. The cylinders were filled with deionised water acidified with HCl (pH close to 2) to avoid the solubilisation of CO₂ [2].

2.4. Sample analysis

The parameters 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 6000 × *g* for 20 min and filtration through a 1.2 μm membrane. All differences between soluble fraction (*S*) and total sludge (*T*) were related as particulate (*P*) ones.

2.4.1. Chemical and biochemical analysis

Chemical oxygen demand: COD_T, COD_S were measured following the micro-method HACH. The standard deviation for triplicates was 10% for soluble COD measurement and 15% for total COD measurement.

Total solids (TS) and volatile solids (VS): TS and VS were measured on total sludge and TSS and VSS on solids obtained after centrifugation (SORVALL T 6000 D) at 6000 × *g* for 20 min. TS, VS, TSS, VSS measurement were performed according to the normalised methods: 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.

Proteins measurements: Total and soluble proteins (proteins_T, proteins_S) were measured following the slightly modified technique [42] using BSA (bovine serum albumin) as a standard. The

standard deviation for triplicates was 5–8% for soluble proteins and 10–15% for total proteins.

Carbohydrates measurements: Total and soluble carbohydrates (carbohydrates_T, carbohydrates_S) concentrations were determined using the phenol sulphuric method proposed by [42].

2.5. Sludge solubilisation, removal efficiencies assessment

2.5.1. Activated sludge disintegration assessment

- COD (*S*_{COD}), proteins (*S*_{proteins}) and carbohydrates (*S*_{carbohydrates}) solubilisations were calculated by using the difference between soluble concentration (*X*_s) and initial soluble concentration (*X*_{s0}) divided by the initial particulate concentration (*X*_{p0}). *X* represents either COD, proteins or carbohydrates concentration (Eq. (2)):

$$S_X = \left[\frac{X_s - X_{s0}}{X_{p0}} \right] \times 100\% \quad (2)$$

- Total solids (*S*_{TS}) and volatile solids (*S*_{VS}) solubilisations were calculated according to Eqs. (3) and (4):

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

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

2.5.2. Performances assessment of aerobic and anaerobic digestion

- **Removal efficiencies:** TSS and VSS removal efficiencies were evaluated according to Eq. (5):

$$\begin{aligned} \text{Removal efficiency (\%)} \\ = \left(\frac{\text{Parameter value (t0)} - \text{Parameter value (tf)}}{\text{Parameter value (t0)}} \right) \times 100 \end{aligned} \quad (5)$$

2.6. Specific energy (SE) calculation

SE was determined for each treatment by using input power (*P*), treatment time (*t*), sample volume (*V*) and initial total solid concentration (*TS*₀) (Eq. (6)):

$$SE \text{ (kJ kgTS}^{-1}\text{)} = \frac{P_{(w)} \times t_{(s)}}{V_{(L)} \times TS_0 \text{ (g L}^{-1}\text{)}} \quad (6)$$

2.7. Energy balance

The specific energy required for removing one kilogram of TSS was calculated under aerobic and anaerobic conditions (Eqs. (7) and (8)).

2.7.1. *E*_T under aerobic condition (*E*_T)

For aerobic systems, total energy consumption (*E*_T) was considered as the sum of applied energy by air compressor during aerobic digestion (*E*_{AERATION}) and specific energy applied for solubilising samples during the pretreatment (*E*_{PRETREATMENT}) (Eq. (7)). The power of air compressor was 135 W.

$$E_T \text{ (kWh)} = E_{\text{AERATION}} \text{ (kWh)} + E_{\text{PRETREATMENT}} \text{ (kWh)} \quad (7)$$

The specific energy to remove one kilogram of TSS (EB aerobic) was calculated according to Eq. (8):

$$EB_{\text{aerobic}} \text{ (kWh kgTSS}_{\text{removed}}^{-1}\text{)} = \frac{E_T \text{ (kWh)}}{\text{TSS}_{\text{removed}} \text{ (kg L}^{-1}\text{)} \times V_{\text{reactor}} \text{ (L)}} \quad (8)$$

Table 1
Organic matter and sludge solubilisation after the different pretreatments.

	Ultrasound	Thermic (40 °C)	Thermic (60 °C)	Thermic (90 °C)	Autoclave (121 °C)	Ozone
SE (kJ kgTS ₀ ⁻¹)	200,000	144,000	216,000	558,620	665,024	46,285
S _{COD}	46	3.8	8	16.8	15.7	10
DBO ₅ /COD ₅	58	67	69	40	45	75
S _{proteins}	97.7	0.5	8.8	45	44	9
S _{carbohydrates}	33	0.3	4	37.6	33.6	7.1
S _{TSS}	46.5	5	8.8	15.8	4.2	15
S _{VSS}	55	6.5	11.7	21.2	4.8	19.2

2.7.2. E_{Total} under anaerobic condition (E_T)

For anaerobic system, two devices were used: agitator and heater. An agitator was used to mix the sludge during the digestion process. Heater was used to maintain the temperature of sludge at 35–37 °C. The duty cycle of heater was about 15%, the heater working time being only 15% of the digestion time. The powers of agitator and heater were 40 W and 640 W, respectively.

The energy of methane production was also taken into account (E_{biogas}). Anaerobic digestion results in methane production which may be used to provide a fraction of required energy. E_{biogas} was calculated considering that CH₄ represents 65% (v/v) of the total biogas produced and that 1 mL of CH₄ corresponds to 35.95 J [43].

Total energy (E_T) is equal to the sum of energy due to the pre-treatment $E_{PRETREATMENT}$, agitation E_{MIXING} , and heater energies ($E_{HEATING}$) minus methane energy (E_{biogas}):

$$E_T \text{ (kWh)} = E_{PRETREATMENT} \text{ (kWh)} + E_{MIXING} \text{ (kWh)} + E_{HEATING} \text{ (kWh)} - E_{biogas} \text{ (kWh)} \quad (9)$$

The specific energy to remove one kilogram of TSS (EB aerobic) was calculated according to the equation (10).

$$EB_{anaerobic} \text{ (kWh kgTSS}_{removed}^{-1})} = \frac{ET \text{ (kWh)}}{TSS_{removed} \text{ (kg L}^{-1}) \times V_{reactor} \text{ (L)}} \quad (10)$$

3. Results and discussion

Different combinations of pretreatment/aerobic or anaerobic digestion were investigated in order to enhance sludge production reduction. Three types of pretreatments were evaluated: thermal (40 °C, 60 °C, 90 °C and autoclave), mechanical (ultrasonic treatment) and oxidative (ozone treatment) processes. Based on the earlier studies, the following conditions were adopted with regard to their effectiveness in terms of organic matter solubilisation: 40 °C, 60 °C, 90 °C for 90 min, 120 °C 1 bar 15 min, ultrasounds input power of 50 W ($SE = 200,000 \text{ kJ kgTS}_0^{-1}$) and $0.1 \text{ gO}_3 \text{ gTS}_0^{-1}$. The pre-treated sludge was aerobically and anaerobically digested for 50 days for evaluation of sludge reduction improvement and cost

effective analysis achieved for the different combinations (pretreatment + digestion).

3.1. Sludge solubilisation and reduction due to the different pretreatments

The effects of the different pretreatments on sludge solubilisation were first investigated (Table 1). Sludge solubilisation is highly dependent on the kind of treatment (mechanic, oxidative, thermic) rather than the specific energy input. Mechanical pretreatment (ultrasound) led to the best results in terms of TSS and VSS solubilisation (47% and 55%, respectively) but also to good organic matter solubilisation (S_{COD} : 46%, $S_{proteins}$: 98%, $S_{carbohydrates}$: 33%). Thermal treatment at 90 °C and ozonation process also led to noticeable results in terms of sludge solubilisation (16% and 15% of TSS solubilisation, respectively). However poor results were observed in terms of TSS or VSS solubilisation for other treatment (thermic: 40 °C, 60 °C, autoclave): 5%, 8% and 4.2%, respectively, for TSS solubilisation. In the case of autoclave organic matter solubilisation, the parameters S_{COD} , $S_{carbohydrates}$ and $S_{proteins}$ are not negligible as evidenced by the enhanced activity (15.7%, 33.6%, and 44%, respectively) due to the pretreatment.

Ultrasonic, thermal and ozone treatment of sludges induced *per se* sludge reduction due to solubilisation of total and volatile solids. The relative part of TSS removal specifically due to the pretreatment was evaluated (Table 2). The relative contributions of the pretreatment process to global sludge reduction vary largely according to the kind of treatment. Ultrasonic pretreatment contributed major role to sludge reduction improvement (61% under aerobic conditions and 54% under anaerobic conditions). This observation clearly indicates that the pretreatment can considerably reduce the digestion length to reach the TSS removal of the non-treated sludge. For thermal treatment the contribution in sludge reduction was as much important as the temperature is increasing: this removal ranged between 12% and 20% when the temperature varied from 40 °C to 90 °C. The contribution of ozone pretreatment (20%) to global sludge reduction was comparable to the one determined by thermal treatment (90 °C). For ozone treatment this result is found to be lower than the sludge reduction obtained by Park et al. [22]

Table 2
TSS removal yield under aerobic and anaerobic conditions, relative contributions of the pretreatment and of the digestion step in TSS reduction.

Pretreatment	Control 1	Ultrasound	Thermic (90 °C)	Autoclave	Control 2	Thermic 40 °C	Thermic (60 °C)	Ozone
Aerobic conditions								
TSS removal yield (%)	57	76	68	69	59	62.5	65	71
Removal yield improvement/reduction		1.3	1.2	1.2		1.06	1.1	1.2
Part of pretreatment (%)	0	61	23	5.5	0	8	13.5	21
Part of digestion (%)	100	39	77	94.5	100	92	86.5	79
Initial slope of TSS removal (gTSS L ⁻¹ d ⁻¹)	0.78	0.19	0.61	0.73	0.45	0.45	0.47	0.40
Anaerobic conditions								
TSS removal yield (%)	72	86.2	76.5	76.9	66	69.5	73	78.5
Removal yield improvement/reduction		1.2	1.06	1.07		1.05	1.1	1.2
Part of pretreatment (%)	0	53.5	19.8	4.4	0	7.2	12	19.1
Part of digestion (%)	100	46.5	80.2	95.6	100	92.8	88	80.9
Initial slope of TSS removal (gTSS L ⁻¹ d ⁻¹)	0.81	0.14	0.69	0.74	0.58	0.6	0.58	0.76

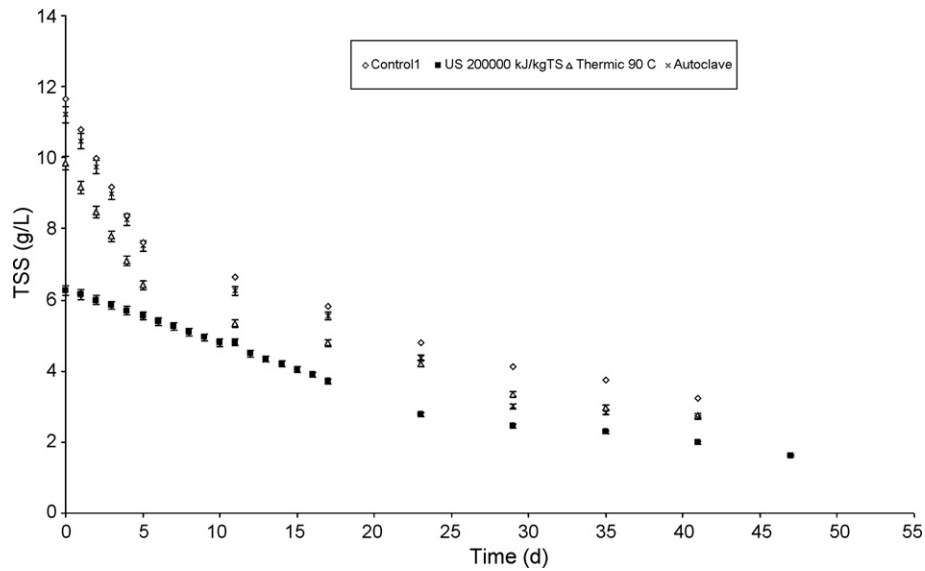


Fig. 1. Evolution of TSS concentration during anaerobic digestion (control 1, ultrasound 200,000 kJ kgTS⁻¹, thermic 90 °C, 121 °C).

(45%) for the same ozone dose. The autoclave pretreatment was found to be less important contribution to TSS global removal (only 4–5%).

3.2. Study of sludge reduction improvement during aerobic and anaerobic digestion

In the previous section it has been demonstrated that the pretreatment process led to intrinsic sludge reduction and to solubilisation. The aim of the pretreatment was also to enhance the sludge reduction production during aerobic or anaerobic digestion.

As the final sludge reduction was also due to the digestion step, the respective contributions in sludge reduction of pretreatment and aerobic/anaerobic digestions were assessed (Table 2). TSS removal was greatly at variance according to the kind of digestion (aerobic or anaerobic).

Sludge reduction production during digestion and after pretreatment can be due to:

- Uncoupling metabolism in which excess free energy would be directed away from anabolism so that the production of biomass can be reduced. Uncoupled metabolism was observed under some conditions such as: the presence of inhibitory compounds, heavy metals, excess energy source (high S/X ratios), abnormal temperatures and limitation of nutrients [44].
- Maintenance metabolism: microorganisms satisfy their maintenance energy requirements rather than producing additional biomass [44].

Sludge reduction was evaluated during 50 days in reactors fed with pre-treated (ultrasound, thermal treatment at 40 °C, 60 °C, 90 °C, autoclave and ozone) activated sludge under aerobic and anaerobic conditions and were compared with non pre-treated sludge (Table 2).

3.2.1. Sludge reduction improvement under anaerobic conditions

The different pretreatments led to TSS removal improvement under anaerobic conditions. Anaerobic conditions were observed

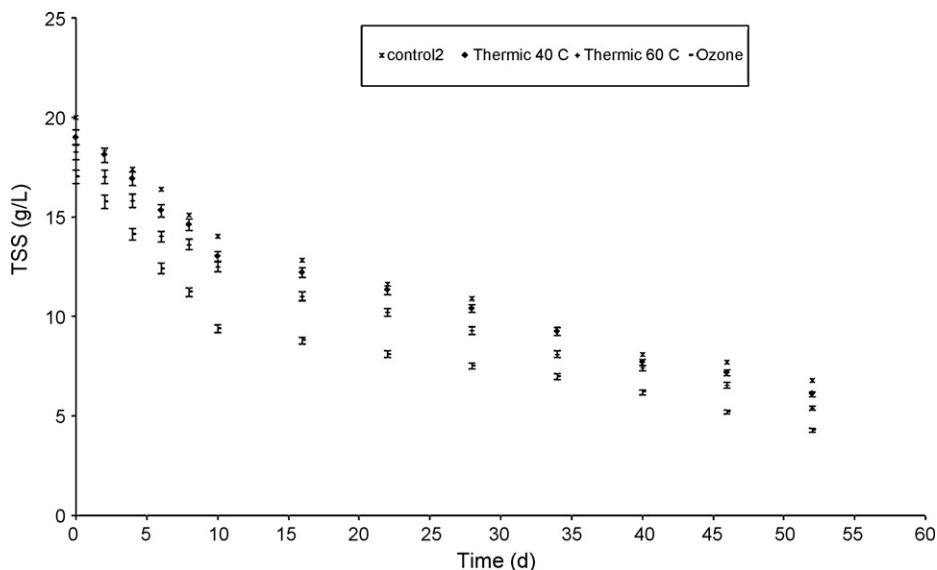


Fig. 2. Evolution of TSS concentration during anaerobic digestion (control 2, ozone, thermic 40 °C, 60 °C).

to be more favourable to TSS reduction than the aerobic conditions (Table 2). It has been observed that the global values of anaerobic TSS reduction ranged from 66% to 86% (Table 2). TSS anaerobic reduction improvement after the different pretreatment was evaluated: it showed that ultrasonically and ozonated pre-treated sludge led to 20% of TSS removal improvement. Sludge reduction improvements were comparable to the results found in the literature after anaerobic digestion. Kim et al. [45] obtained 89% and 56% of sludge reduction improvement respectively for an SRT of 7 after ultrasound or autoclave pretreatment. Bougrier et al. [39] reported a TSS removal improvement of 80% after a pretreatment at higher temperature (170 °C).

The thermal pretreatments at 40 °C, 90 °C, and autoclave led to poor levels of improvement of sludge removal (5%, 6% and 7% respectively) as found in Barjenbruch and Kopplow [46] (VSS degradation improvement: 80 °C, 4%; 90 °C, 5%; 121 °C, 6%).

For most of the pretreatment applied to the sludge, except ultrasound (47%), the digestion step represented the major part of sludge production reduction from 96% for autoclave to 80% for thermal treatment at 90 °C (Table 2).

The analysis of the dynamic of TSS removal during anaerobic digestion showed that sonicated sludge behaviour was specific when compared to other treatments (Figs. 1 and 2). As already mentioned in Salsabil et al. [47] TSS removal of sonicated sludge is linear as a function of time all along the process of anaerobic digestion, which means that the length of the digestion in accordance with the level of TSS removal expected can be easily calculated. For other treatments and controls the TSS removal is divided in two chronological parts: an important slope was noticed during the 5 (first series) or 10 (second series) first days whereas a slower one occurred until the end of the digestion. The calculation of the initial slope expressed in $\text{gTSS L}^{-1} \text{d}^{-1}$ (Table 2) showed that the kinetics of TSS removal of the treated sludges are always equal to or lower than the control except in the specific case of ozonation ($0.76 \text{ gTSS L}^{-1} \text{d}^{-1}$ compared to $0.58 \text{ gTSS L}^{-1} \text{d}^{-1}$ for the control). The sludge reduction improvement of the pretreatment cannot be related to enhance instantaneous removal rate.

The value of TSS concentration after 50 days for the non pretreated sludges (control) can be reached only 20 days after the pretreatment. It means that the length of the digestion can be considerably reduced (half) by sludge pretreatment to reach the same level of sludge reduction.

The best pretreatments in term of TSS removal improvement were ultrasound and ozonation under anaerobic conditions.

3.2.2. Sludge reduction under aerobic conditions

Sludge reduction was enhanced under aerobic conditions by the different pretreatments of the sludge. Global TSS reduction for pretreated and digested sludge varied between 62.5% and 76%. For most of the pretreatments applied to the sludge except ultrasound, the digestion step represented the most important part of sludge production reduction from 95% for autoclave to 77% for thermal treatment at 90 °C (Table 2).

The relative sludge removal improvement was more important under aerobic conditions than under anaerobic. The maximal TSS removal improvement was 33% for ultrasonically pre-treated sludges, 20% for ozonated and thermally pre-treated (90 °C and autoclave) sludges, then 10% for thermally treated at 60 °C. Low thermal treatments (40 °C and 60 °C) led to poor sludge removal enhancement (respectively 6% and 10%) after aerobic digestion.

Ultrasonic, thermal at 90 °C and autoclave treatment of sludge prior to aerobic digestion led to the highest TSS elimination efficiencies (76% for sonication, 68% for 90 °C and 69% for autoclave against 57% for control sample). Ozonation of samples prior to aerobic digestion led to an elimination efficiency of 63% after 40 days of digestion. This value reached 71% after approximately 50 days. This value was superior to the control sample one that was 57% for the same period of time.

Ultrasonic irradiation acceleration of sludge aerobic digestion may be due to the satisfaction of suitable aerobic condition for the microorganism like enzymatic activities enhancement and release of extracellular proteins and polysaccharides [48,49]. Ding et al. [48] and Yu et al. [49] demonstrated that for ultrasonic pre-treated sludge at lower specific energies (respectively $\text{SE} = 9500 \text{ kJ kgTS}^{-1}$ and $112,500 \text{ kJ kgTS}^{-1}$), TSS removal improvement could reach 40% and 48% after an aerobic digestion.

Ozone treatment prior to aerobic represented one of the best pretreatments in terms of TSS removal improvement and energy consumption under aerobic conditions. This result confirmed the interest of the use of ozone pretreatment [22,28,31,50]. The values of TSS removal improvement (71% and 78.5%) are above the values proposed by Paul et al. [31] (30%) to economically justify a process of sludge reduction furthermore above the results of Sievers et al. [50] on full scale application who reached 20–35% and 19% after aerobic or anaerobic stabilisation and ozone treatment of $0.05 \text{ gO}_3 \text{ gTSS}^{-1}$. Deleris et al. [51] obtained comparable results (70% of reduction of sludge production) with lower ozone dose (ozonation on the recycling loop $0.05 \text{ gO}_3 \text{ gVSS}^{-1}$).

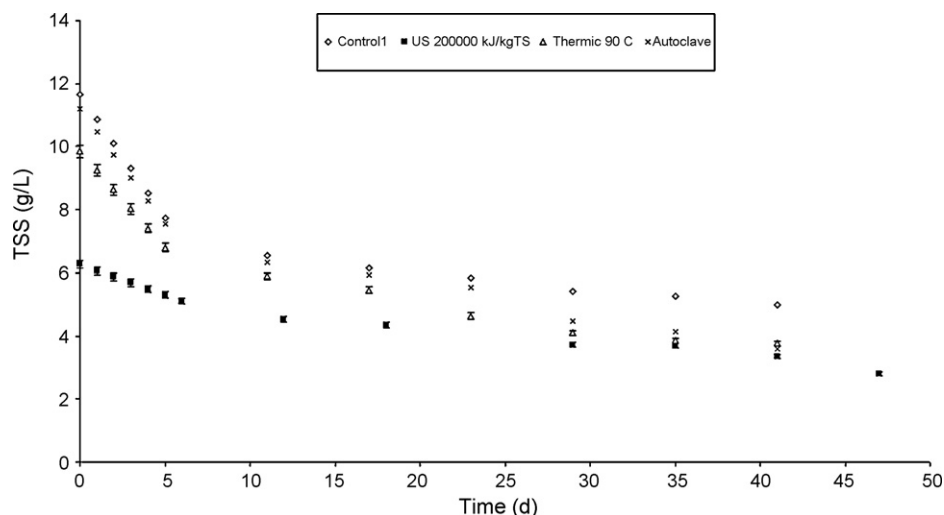


Fig. 3. Evolution of TSS concentration during aerobic digestion (control 1, ultrasound 200,000 kJ kgTS_0^{-1} , thermic 90 °C, autoclave).

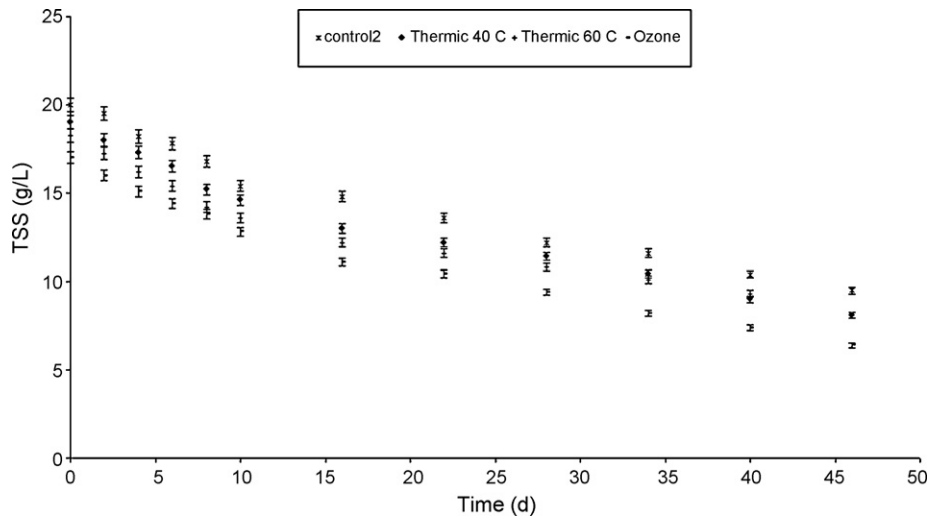


Fig. 4. Evolution of TSS concentration during aerobic digestion (control 2, ozone, thermic 40 °C, 60 °C).

Different processes could explain the good results of thermal treatment (90 °C and autoclave) before aerobic digestion: the important release of organics, the immediate and reversible biological inactivation associated with additional maintenance energy requirements and the potential inert production [52].

The analysis of the dynamic of TSS removal during aerobic digestion can be compared to what was observed under anaerobic conditions (Figs. 3 and 4): there is also a specific behaviour of sonicated sludge compared to other treatments. Aerobic TSS removal of sonicated sludge is linear all along the process of aerobic digestion while for other treatments and controls the TSS removal is divided in two parts: an important slope during the 5 (first series) or 10 (second series) first days and then a more slowly until the end of the digestion. The calculation of the initial slope expressed in $gTSSL^{-1} d^{-1}$ (Table 2) showed that the kinetics of TSS removal of the treated sludges were always equal to or lower than the control. The sludge reduction improvement of the pretreatment cannot be related to enhance instantaneous removal rate.

As already mentioned under anaerobic conditions: pretreatment prior to aerobic digestion can considerably reduce the digestion length. The values of TSS concentration after 50 days in the control can be reached after 25 days with a pretreatment of

the sludge which means a reduction by half of the duration of the process.

Under aerobic conditions the pretreatment process leading to the best combination in regard to sludge production reduction improvement was ultrasound.

3.2.3. Driving parameters of sludge reduction under aerobic and anaerobic conditions

The research of rapid parameters which can predict pretreatment efficiency would be of interest for setting up of the processes of sludge reduction.

Correlations between TSS removal improvement and solubilisation parameters of organic matter (S_{COD} , $S_{proteins}$, $S_{carbohydrates}$) after pretreatment were investigated under both aerobic and anaerobic conditions (Figs. 5 and 6). Under anaerobic conditions, no linear correlation was noticed between solubilisation parameters of organic matter and sludge reduction enhancement (Fig. 6). This is probably due to the discrepancy of the different mechanisms studied.

On the contrary, TSS removal under aerobic conditions was linearly correlated to the solubilisation parameters ($S_{carbohydrates}$ and $S_{proteins}$) (Fig. 5) obtained after the pretreatment: TSS removal

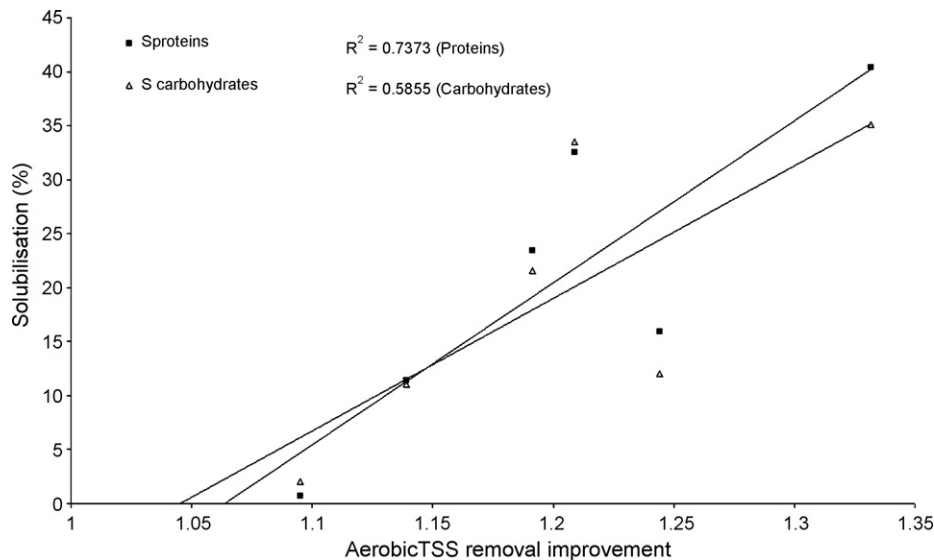


Fig. 5. Proteins, COD and carbohydrates solubilisation as a function of aerobic TSS removal.

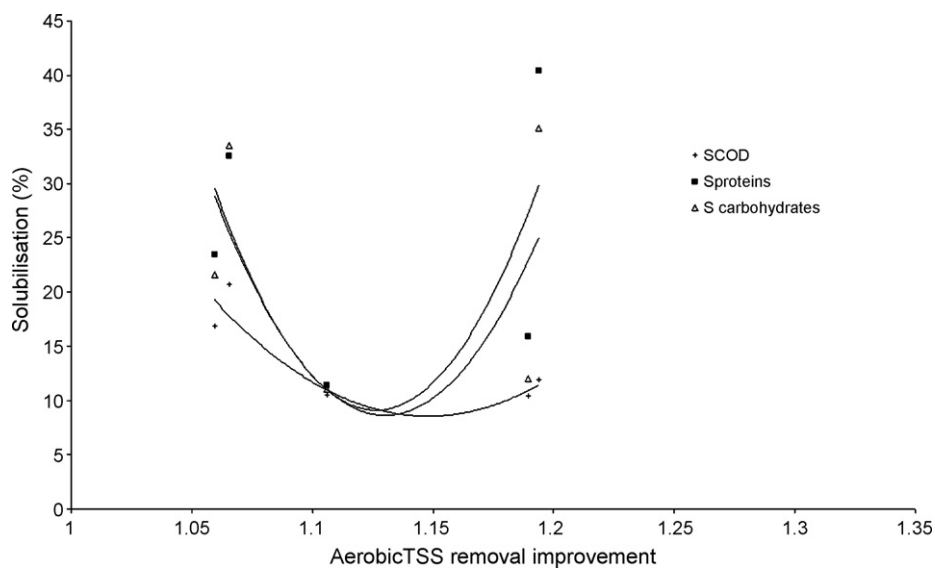


Fig. 6. Proteins, COD and carbohydrates solubilisation as a function of anaerobic TSS removal.

increased with increasing solubilisation level whatever the pretreatment.

Some authors mentioned that improvement of VSS removal after ozonation and anaerobic stabilisation can be related to VSS solubilisation during the pretreatment [28]. Specific energy input during the pretreatment has already been mentioned as a parameter of interest for the processes of sludge reduction [47]. Correlations between global TSS removal improvement (after pretreatment and digestion) and TSS solubilisation (Fig. 7) (or specific energy input (Fig. 8) during the pretreatment were investigated under both aerobic and anaerobic conditions.

The global TSS removal improvement (after pretreatment+digestion) increased with increasing TSS solubilisation (after pretreatment only) whatever the kind of treatment under both aerobic and anaerobic conditions (Fig. 7). TSS solubilisation is thus an interesting parameter to predict sludge reduction production improvement. The use of the parameter “specific energy” to predict the efficiency of a pretreatment varies according to the kind of digestion. Under anaerobic conditions, TSS removal improvement was slightly decreasing with increasing

specific energy while under aerobic conditions there was no impact of input specific energy during the pretreatment on sludge reduction production. It can be concluded that the amount of energy provided to the system before digestion was not as much important as the ability of the pretreatment by itself to solubilise TSS.

3.3. Energetic cost comparison of the different combination pretreatments and aerobic or anaerobic procedures

Economic efficiency and energy balance calculations are important tools for performing the cost-benefit analysis of a disintegration process. An energetic study was achieved in order to compare each combination pretreatment/aerobic or anaerobic digestion. The energetic calculations were based on the total energy requirement of each method and the TSS removal yield achieved by that method. The total specific energy required to remove 1 kg of TSS ($EB_{\text{aerobic}} : \text{kWh kgTSS}_{\text{removed}}^{-1}$) was calculated for each pretreatment method (solubilisation if applicable and digestion) under aerobic and anaerobic condition (Table 3).

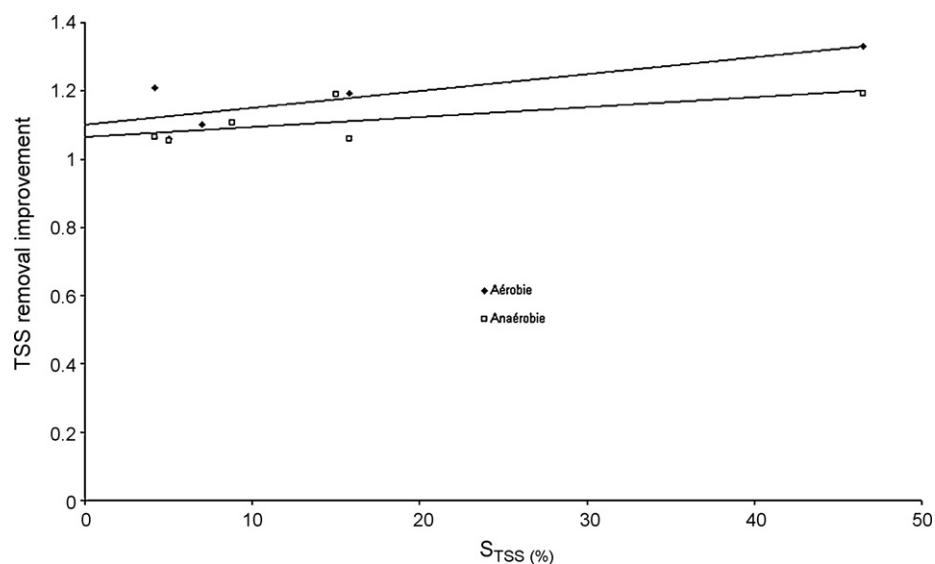


Fig. 7. TSS removal improvement as a function of TSS solubilisation.

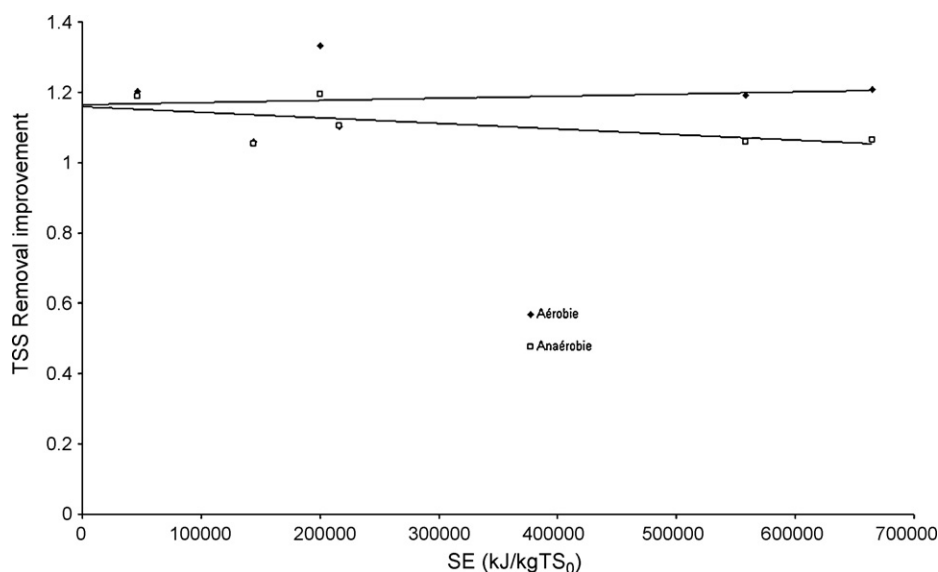


Fig. 8. TSS removal improvement as a function of specific energy input.

Table 3

Economic balance calculation of different pretreatments applied before aerobic and anaerobic digestions.

	Control 1	Ultrasound	Thermal (90 °C)	Autoclave	Control 2	Ozonation	Thermal (40 °C)	Thermal (60 °C)
Aerobic								
$E_{\text{pretreatment}}/E_T$ (%)	–	0.17	2.13	3.57	–	0.32	0.66	1.06
EB (kWh kgTSS ⁻¹ _{removed})	3153	1647	2137	2085	2197	1602	1945	1747
Cost reduction (%)	–	48	32	34	–	27	11	20.5
Anaerobic								
$E_{\text{pretreatment}}/E_T$ (%)	–	0.10	1.10	2	–	0.18	0.39	0.61
EB (kWh kgTSS ⁻¹ _{removed})	4655	2601	3665	3381	3212	2496	2958	2682
Cost reduction %	–	44	21	27	–	22	8	16.5

The energy consumed during the solubilisation step (or pretreatment step) (E_S) was found to be negligible when compared to total applied energy (E_T) during both pretreatment and digestion steps (Table 3). Under aerobic and anaerobic conditions, the minimum E_S/E_T ratio was 0.1% (for ultrasonically) pre-treated sludge and the maximum ratio was 3.6% (for autoclave pre-treated sludge).

The total cost of consumed energy during the pretreatment step (ultrasound, temperature and ozonation) and the biological digestion step for different pretreatment methods and control sample (only biological digestion) was investigated (considering that the price of electricity was 0.11€ per kWh for France in 2008). A comparison of results is reproduced in Table 3.

In fact, a wide variety of parameters may interfere with economic efficiency. Some of the examples considered may be investment costs, personnel costs, energy costs of disintegration process, operating and maintenance costs, dewatering, disposal, etc. However, most of the above mentioned parameters were not considered in this study because we focused on the exploitation costs. In this regard, it must be pointed out that these results are obtained with the laboratory scale devices with low energetic performances with respect to the full scale module applications.

The introduction of a pretreatment before aerobic and anaerobic digestion always led to the cost reduction compare to the control. Sonication led to the best cost reduction: 48% and 44%, respectively, under aerobic or anaerobic conditions. High thermal treatment (90 °C and autoclave) and ozonation led to interesting cost reduction: about 30% under aerobic conditions and about 25% under anaerobic conditions. Even low thermal treatment led to non-negligible cost reduction: respectively 11% and 8% under aerobic and anaerobic conditions.

Simplicity of process and lower capital costs are the main 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.

4. Conclusion

The effect of different pretreatment like ultrasonic treatment (200,000 kJ kgTS⁻¹), ozone treatment (0.1 gO₃ gTS⁻¹) and thermal treatment (40 °C, 60 °C and 90 °C for 60 min and 121 °C under 1 bar for 20 min) on sludge solubilisation were assessed. Pretreatments led to TSS solubilisation quite efficiently and thus to intrinsic sludge reduction which varied from 47% to 4%. Ultrasound process led to the best TSS reduction. In regard to our results, solubilisation can be related to the pretreatment ability to break the flocs (mechanical or chemical effect) rather than to the specific energy input.

The pre-treated sludge was aerobically and anaerobically digested and sludge reduction production efficiency after aerobic or anaerobic conditions was secondly investigated. TSS removal was globally improved by sludge pretreatment. The global values of TSS reduction ranged from 57% to 71% under aerobic conditions and from 66% to 86% under anaerobic conditions respectively. Anaerobic conditions are globally more favourable to TSS reduction than aerobic conditions. The best sludge reduction improvement can be attributed to ozone and ultrasonic pretreatment (20% of improvement) under both aerobic and anaerobic conditions. For thermal treatments (40 °C, 60 °C, 90 °C and autoclave) the results are less spectacular. The digestion step represented the main part of sludge

production reduction (i.e. in the case of ozonation, the digestion step represents 80% of the total sludge reduction improvement and the pretreatment step 20%). In the case of ultrasonic treatment the respective implication in sludge reduction improvement of pretreatment and anaerobic digestion was equal (50%).

TSS solubilisation during the pretreatment was a good indicator of TSS removal improvement (under aerobic or anaerobic conditions). On the contrary the specific energy input into the system was not sufficient to guarantee good TSS removal improvement. COD, proteins and carbohydrates solubilisation can be used to predict sludge reduction improvement under aerobic conditions but not under anaerobic conditions.

An energetic balance and the determination of the energy required for removing 1 kg of TSS led to the conclusion that ultrasonic and ozonation pretreatment prior to aerobic or anaerobic digestion always led to cost reduction compare to the control.

Ozone and ultrasonic treatment before anaerobic digestion led to the best improvement of TSS removal: ultrasonic treatment is energetically costly but the digestion time can be reduced, ozone treatment is less costly but the length of the digestion largely contributes to sludge reduction.

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References

- [1] S. Yoon, H. Kimb, S. Lee, Incorporation of ultrasonic cell disintegration into a membrane bioreactor for zero sludge production, *Process Biochemistry* 39 (2004) 1923–1929.
- [2] U. Neis, K. Nickel, A. Tiehm, Enhancement of anaerobic sludge digestion by ultrasonic disintegration, *Water Science and Technology* 42 (9) (2000) 73–80.
- [3] A. Tiehm, K. Nickel, M. Zellhorn, U. Neis, Ultrasonic waste activated sludge disintegration for improving anaerobic stabilization, *Water Research* 35 (8) (2001) 2003–2009.
- [4] C.J. Rivard, N.J. Nagle, Pretreatment technology for the beneficial biological reuse of municipal sewage sludges, *Applied Biochemistry and Biotechnology* 57–58 (1996) 983–991.
- [5] S. Mace, J. Costa, J. Mata-Alvarez, Sewage sludge pretreatments for enhancing its anaerobic biodegradability, *Bioprocessing of Solid Waste & Sludge* 1 (3) (2001) 3.
- [6] Y.Y. Li, T. Noike, Upgrading of anaerobic digestion of waste activated sludge by thermal pretreatment, *Journal of Water Science and Technology* 26 (3–4) (1992) 857–866.
- [7] S. Tanaka, T. Kobayashi, K.I. Kamiyama, L.N.S. Bildan, Effects of thermo-chemical pretreatment on the anaerobic digestion of waste activated sludge, *Water Science & Technology* 35 (8) (1997) 209–215.
- [8] M. Rocher, G. Goma, A.P. Begue, L. Louvel, J.L. Rols, Towards a reduction in excess sludge production in activated sludge processes: biomass physicochemical treatment and biodegradation, *Applied Microbiology and Biotechnology* 51 (1999) 883–890.
- [9] Y. Sakai, T. Fukase, H. Yasui, M. Shibata, An activated sludge process without excess sludge production, *Water Science and Technology* 36 (1997) 163–170.
- [10] A. Huysmans, M. Weemaes, P.A. Fonseca, W. Verstraete, Ozonation of activated sludge in the recycle stream, *Journal of Chemical Technology and Biotechnology* 76 (3) (2001) 321–324.
- [11] H. Odegaard, Sludge minimization technologies—an overview, *Journal of Water Science & Technology* 49 (10) (2004) 31–40.
- [12] C. Bougrier, H. Carrère, J.P. Delgenès, Solubilisation of waste-activated sludge by ultrasonic treatment, *Chemical Engineering Journal* 106 (2005) 163–169.
- [13] P. Zhang, G. Zhang, W. Wang, Ultrasonic treatment of biological sludge: floc disintegration, cell lysis and inactivation, *Bioresource Technology* 98 (2007) 207–210.
- [14] G. Zhang, P. Zhang, J. Yang, Y. Chena, Ultrasonic reduction of excess sludge from the activated sludge system, *Journal of Hazardous Materials* 145 (2007) 515–519.
- [15] I.W. Nah, Y.W. Kang, K.Y. Hwang, W.K. Song, Mechanical pretreatment of waste activated sludge for anaerobic digestion process, *Water Research* 34 (8) (2000) 2362–2368.
- [16] E. Gonze, S. Pillot, E. Valette, Y. Gonthier, A. Bernis, Ultrasonic treatment of an aerobic activated sludge in a batch reactor, *Journal of Chemical Engineering and Processing* 42 (12) (2003) 965–975.
- [17] R. Dewil, J. Baeyens, R. Goutvriind, Ultrasonic Treatment of Waste Activated Sludge, Published online in Wiley Inter Science (www.interscience.wiley.com), 2006, doi:10.1002/ep, p. 10130.
- [18] T.I. Onyeche, O. Schlaefer, C. Schroeder, H. Bormann, M. Sievers, Ultrasonic cell disruption of stabilised sludge with subsequent anaerobic digestion, in: *Proceedings of the 9th World Congress, Anaerobic Conversion for Sustainability*, 2001.
- [19] X.Q. Cao, J. Chen, Y.L. Cao, J.Y. Zhu, X.D. Hao, Experimental study on sludge reduction by ultrasound, *Water Science and Technology* 54 (9) (2006) 87–93.
- [20] T. Mao, K. Show, Performance of high-rate sludge digesters fed with sonicated sludge, *Water Science and Technology* 54 (9) (2006) 27–33.
- [21] J.A. Müller, Pre-treatment processes for the recycling and reuse of sewage sludge, *Water Science and Technology* 42 (9) (2000) 167–174.
- [22] K.Y. Park, K.H. Ahn, S.K. Maeng, J.H. Hwang, J.H. Kwon, Feasibility of sludge ozonation for stabilization and conditioning, *Ozone: Science & Engineering* 25 (1) (2003) 73–80.
- [23] A.A. Yavich, K.H. Lee, K.C. Chen, L. Pape, S.J. Masten, Evaluation of biodegradability of NOM after ozonation, *Water Research* 38 (2004) 2839–2846.
- [24] D.P. Saroj, A. Kumar, P. Bose, V. Tare, Y. Dhopavkar, Mineralization of some natural refractory organic compounds by biodegradation and ozonation, *Water Research* 39 (2005) 1921–1933.
- [25] K.H. Ahn, K.Y. Park, S.K. Maeng, J.H. Hwang, J.W. Lee, K.G. Song, S. Choi, Ozonation of wastewater and ozonation for recycling, *Water Science and Technology* 46 (10) (2002) 71–77.
- [26] H. Yasui, M. Shibata, An innovative approach to reduce excess sludge production in the activated sludge process, *Water Science and Technology* 30 (1994) 11–20.
- [27] H. Yasui, K. Nakamura, S. Sakuma, M. Iwasaki, Y. Sakai, A full-scale operation of a novel activated sludge process without excess sludge production, *Water Science and Technology* 34 (3–4) (1996) 395–404.
- [28] R. Goel, T. Tokutomi, H. Yasui, Anaerobic digestion of excess activated sludge with ozone pretreatment, *Water Science and Technology* 47 (12) (2003) 207–214.
- [29] S. Graja, J. Chauzy, P. Fernandes, L. Patria, D. Cretenot, Reduction of sludge production from WWTP using thermal pretreatment and enhanced anaerobic methanisation, *Water Science & Technology* 52 (1–2) (2005) 267–273.
- [30] M.P.J. Weemaes, W.H. Verstraete, Evaluation of current wet sludge disintegration techniques, *Journal of Chemical Technology and Biotechnology* 73 (8) (1998) 83–92.
- [31] E. Paul, P. Camacho, D. Lefebvre, P. Ginestet, Organic matter release in low temperature thermal treatment of biological sludge for reduction of excess sludge production, *Water Science & Technology*, IWA Publishing 54 (5) (2006) 59–68.
- [32] A. Valo, H. Carrère, J.P. Delgenès, Thermal, chemical and thermo-chemical pretreatment of waste activated sludge for anaerobic digestion, *Journal of Chemical Technology and Biotechnology* 79 (2004) 1197–1203.
- [33] H. Odegaard, B. Paulsrud, I. Karlson, Wastewater sludge as a resource—sludge disposal strategies and corresponding treatment technologies aimed at sustainable handling of wastewater sludge, *Journal of Water Science and Technology* 46 (10) (2002) 295–303.
- [34] J. Barlindhaug, H. Odegaard, Thermal hydrolysis as carbon source for denitrification, *Water Science & Technology* 33 (12) (1996) 99–108.
- [35] Y.C. Chiu, C.N. Chang, L.G. Lin, S.J. Huang, Alkaline and ultrasonic pretreatment of sludge before anaerobic digestion, *Water Science & Technology* 36 (11) (1997) 155–162.
- [36] J. Wang, C.P. Huang, H.E. Allen, I. Poesponegoro, H. Poesponegoro, L.R. Takiyama, Effects of dissolved organic matter and pH on heavy metal uptake by sludge particulates exemplified by copper(ii) and nickel(ii): three-variable model, *Water Environment Research* 71 (2) (1999) 139–147.
- [37] A. Tiehm, K. Nickel, U. Neis, The use of ultrasound to accelerate the anaerobic digestion of sewage sludge, *Journal of Water Science and Technology* 36 (11) (1997) 121–128.
- [38] A. Grönroos, H. Kyllönen, K. Korpijärvi, P. Pirkonen, T. Paavola, J. Jokela, J. Rintala, Ultrasound assisted method to increase soluble chemical oxygen demand (COD_s) of sewage sludge for digestion, *Ultrasonic Sonochemistry* 12 (2005) 115–120.
- [39] C. Bougrier, C. Albasi, J.P. Delgenes, H. Carrere, Effect of ultrasonic, thermal and ozone pretreatments on waste activated sludge solubilisation and anaerobic biodegradability, *Chemical Engineering and Processing* 45 (8) (2006) 711–718.
- [40] S. Lafitte-Trouqué, C.F. Forster, The use of ultrasound and g-irradiation as pretreatments for the anaerobic digestion of waste activated sludge at mesophilic and thermophilic temperatures, *Bioresource Technology* 84 (2002) 113–118.
- [41] S.K. Khanal, D. Grewell, S. Sung, J.H. Van Leeuwen, Ultrasound applications in wastewater sludge pretreatment, *Critical Reviews in Environmental Science and Technology* 37 (2007) 277–313.
- [42] R. Kamra, H.-J. Thorkild, N. Per. Halkjre, Measurement of pools of protein, carbohydrate and lipid in domestic wastewater, *Water Research* 28 (2) (1994) 251–262.
- [43] C. Bougrier, Optimisation du procédé de méthanisation par mise en place d'un co-traitement physico-chimique: Application au gisement de biogas représenté par les boues d'épuration des eaux usées, PhD thesis, Université de Montpellier II, 2005.
- [44] Y. Wei, R.T. Van Houten, A.R. Borger, D.H. Eikelboom, Y. Fan, Minimization of excess sludge production for biological wastewater treatment, *Journal of Water Research* 37 (18) (2003) 4453–4467.
- [45] J. Kim, C. Park, T.H. Kim, M. Lee, S. Kim, S.W. Kim, J. Lee, Effects of various pretreatments for enhanced anaerobic digestion with waste activated sludge, *Journal of Bioscience and Bioengineering* 95 (3) (2003) 271–275.

- [46] M. Barjenbruch, O. Kopplow, Enzymatic, mechanical and thermal pretreatment of surplus sludge, *Advances in Environmental Research* 7 (2003) 715–720.
- [47] M.R. Salsabil, A. Prorot, M. Casellas, C. Dagot, Pretreatment of activated sludge: effect of sonication on aerobic and anaerobic digestibility, *Chemical Engineering Journal*, in press.
- [48] W.C. Ding, D.X. Li, X.L. Zeng, T.R. Long, Enhancing excess sludge aerobic digestion with low intensity ultrasound, *Journal of Central South University Technology* 13 (4) (2006) 408–411.
- [49] G.H. Yu, P.J. He, L.M. Shao, Y.S. Zhu, Extracellular proteins, polysaccharides and enzymes impact on sludge aerobic digestion after ultrasonic pretreatment, *Water Research* 42 (8–9) (2008) 1925–1934.
- [50] M. Sievers, A. Ried, R. Koll, Sludge treatment by ozonation—evaluation of full scale results, *Water Science and Technology* 49 (4) (2004) 247–253.
- [51] S. Déléris, V. Geaugey, P. Camacho, H. Debellefontaine, E. Paul, Minimization of sludge production in biological processes: an alternative solution for the problem of sludge disposal, *Water Science & Technology* 46 (10) (2002) 63–70.
- [52] P. Camacho, P. Ginestet, J.-M. Audic, Understanding the mechanisms of thermal disintegrating treatment in the reduction of sludge production, *Water Science and Technology* 52 (10–11) (2005) 235–245.