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TESTS ON THE EFFECT OF ULTRASONIC TREATMENT ON TWO DIFFERENT ACTIVATED SLUDGE WASTE

The effect of ultrasonic treatment on two different waste activated sludges (WAS1 and WAS2) was compared by batch tests. WAS1 was taken from an industrial WWTP that treats pulp and paper wastewater, WAS2 was obtained from a traditional municipal WWTP. Physical and chemical characterization of sludges and supernatants was performed at various specific energy inputs. Upon increasing specific energy input, sludge structure changed, particle size decreased and organic and nitrogen release increased. A different release of organic matter and nitrogen compound related to the sludge origin was observed. The study is a preliminary assay of the potential of sonication treatment to enhance biogas production in anaerobic digestion.

1. INTRODUCTION

Due to great urban and industrial development, large amounts of waste activated sludge are produced in municipal and industrial wastewater treatment plants (WWTPs). Sludge disposal became one of the most crucial feature of a WWTP management. Both economical and environmental constraints must be taken into account. Up to 65% of the operating costs of a WWTP (including energy, man employment and ordinary management) are ascribed to sludge treatment, handling and disposal [1]. In according to the European Directive on the landfill of waste [2], traditional disposal methods will be gradually ban by promoting new methods with a greater environmental sustainability. Efforts must be done to minimize production of activated sludge and increase the efficiency of sludge treatment technology.

Anaerobic digestion is a well known technology used to stabilize organic matter and reduce sludge mass and pathogens. In order to enhance biodegradability of organ-

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ic solids, some physical and chemical pretreatments can be applied to sludge fed to anaerobic digesters [3, 4]. Among these handlings, ultrasonic treatment is reported as an efficient method to promote sludge deagglomeration and bacterial cell wall disruption [5–8]. Thus, extracellular matter, including organic debris, extra polymeric substances (such as polysaccharides, proteins, lipids, nucleic acids, etc.) and intracellular material are released into the aqueous phase.

Floc deagglomeration and microbial cell wall break-up are caused by formation, growth and collapse of cavitation microbubbles into the liquid system. The most significant disintegration of sludge occurs at the frequency range of 20–40 kHz [9]. Collapsing of the bubbles results in localized increase of temperature up to 5000 K and pressure up to 180 MPa [10]. As a result, hydromechanical shear forces are developed and radical species are generated. Although the contribution of radical species increases with the amount of the increasing ultrasonic density, the sludge disintegration is mainly due to hydro-mechanical shear forces at low frequencies [7, 11]. A higher availability of extracellular and intracellular compounds in liquid phase should facilitate the anaerobic microbial community to degrade the substrates with a higher kinetic degradation rate [12].

Although most of studies have focused on release of organic matter [5, 9, 13–15], ultrasonic treatment of sludge can also lead to leaching inorganic compounds and nitrogen species in the liquid phase [16–18]. In particular, given the potential inhibiting effect of ammonia nitrogen on anaerobic digestion and more stringent constraints for nutrient removal, it is very important to control nitrogen quantity and quality during sonication processes.

The aim of this work is to compare, at a bench-top scale, the ultrasonic treatment efficiency on two different sludges in order to enhance anaerobic digestion. Mechanisms of ultrasonic treatment were investigated by performing physical and chemical characterization of the two different sludges.

2. MATERIALS AND METHODS

Samples. Waste activated sludge WAS1 was collected from the return activated sludge stream of the industrial full-scale treatment plant of Tolmezzo (Udine, Italy). This plant has the capacity of 143 000 EP (equivalent persons) and treats pulp and paper wastewater using a contact tank followed by a conventional activated sludge process.

Waste activated sludge WAS2 was obtained from the return activated sludge stream of the municipal wastewater treatment plant of Udine (Italy). This plant, which has the capacity of 100 000 EP, operated with an optimized anoxic-oxic nitrogen removal process and an anaerobic process for sludge stabilization. Sludge characteristics are shown in Table 1.

Table 1

Chemical characterization of untreated samples
(supernatants collected after sludge centrifugation and filtration)

Parameter	WAS1	WAS2
pH	7.50±0.05	5.64±0.05
ALK, mg $CaCO_3/dm^3$	326±29.34	196±15.68
TS, %	1.06±0.17	0.70±0.11
VS, $\%_{db}$	78.95±11.84	71.43±10.71
C, $\%_{db}$	40.78±0.12	34.37±0.06
N, $\%_{db}$	5.04±0.08	5.98±0.04
C/N	8.10	5.75
sCOD, mg/dm^3	111.90±16.78	25.30±3.03
DOC, mg/dm^3	29.71±2.67	17.18±1.37
TN, mg/dm^3	1.03±0.10	4.69±0.38
N-NH_4^+, mg/dm^3	0.18±0.03	0.5±0.07

Ultrasound apparatus. A 250 W ultrasonic cell disrupter (UIP250, Dr. Hielscher, Germany), operating at a fixed frequency of 24 kHz, was used to treat sludge samples. A titanium probe of 14 mm diameter and 132 W/cm^2 maximum sound power density was dipped 40 mm into the continuously mixed reactor containing the sample. For each test, a 200 cm^3 sludge sample was filled into a 250 cm^3 reactor equipped with a cooling mantel to maintain the temperature at 20±2 °C (Fig. 1).

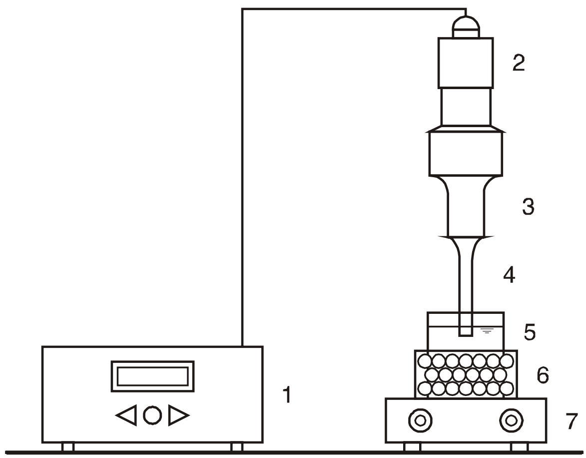

Fig. 1. Scheme of ultrasound apparatus
1 – ultrasound generator, 2 – transducer, 3 – booster, 4 – horn
5 – sample, 6 – ice-filled cooling vessel, 7 – magnetic stirrer

The apparatus was calibrated following the potassium iodide solution procedure [19]. The WAS1 and WAS2 samples were irradiated at various specific energy inputs.

Specific energy input (E_S, kJ/g TS) was defined as the product of power input (P, kW) and sonication time (t, s) divided by the product of sludge volume (V, dm^3) and total solids concentration (TS, g/dm^3):

$$E_S = \frac{Pt}{V\,\mathrm{TS}} \tag{1}$$

Analysis. Microscopic observations were performed by means of an optical microscope (Zeiss, Germany) equipped with a Panasonic F15HS system camera. A drop of sludge, spread onto a microscope slide, was observed and photographed at 125× magnification. A laser scattering particle size distribution analyzer (Horiba Partica LA-950) was used to carry out the particle size distribution. Results were expressed by d_{25}–d_{90} (μm) cut diameter. For the cut diameter d_{25} (μm) equal to 25, 25% of the particles has the diameter equal or lower than the value of 25 μm.

All other parameters were tested after centrifugation at 3000 rpm for 15 min and filtration through 0.45 μm pore size membrane filters of the supernatants. Treated and untreated sludges were analyzed for pH, alkalinity (ALK), total solids (TS), volatile solids (VS), soluble chemical oxygen demand (sCOD), dissolved organic carbon (DOC), ammonia nitrogen (N-NH_3) and total nitrogen (TN). All the analyses were performed in triplicate and in accordance with the standard methods [20].

3. RESULTS AND DISCUSSION

Microscopic observations and particle size distribution have been adopted for basic physical evaluation of effectiveness of ultrasonic treatment. As well, organic and nitrogen release have been adopted for chemical measurements of releasing matter during the ultrasound application.

3.1. MICROSCOPIC OBSERVATIONS

The structure of both sludge changed after ultrasonic treatment as shown in Figs. 2 and 3. Although sludge flocs were similarly disrupted and the bacteria were dispersed into the aqueous phase when specific energy input increases, a different disintegration change was noted for the flocs.

In untreated WAS1 many filamentous bacteria and *testate amoebae* appeared, and some not biological material can be observed among the flocs. By increasing specific energy input from 50 kJ/g TS to 100 kJ/g TS, these protozoa were disrupted. Even if a few material release from activated sludge flocs was noted when specific energy input increased, flocs did not clearly reduced their sizes and their structure until the energy went up to 200 kJ/g TS.

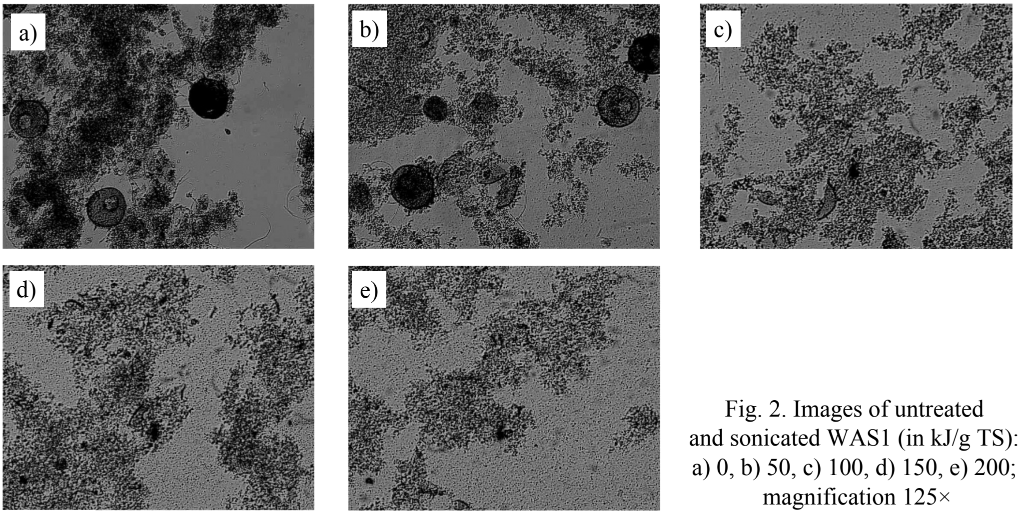

Fig. 2. Images of untreated and sonicated WAS1 (in kJ/g TS): a) 0, b) 50, c) 100, d) 150, e) 200; magnification 125×

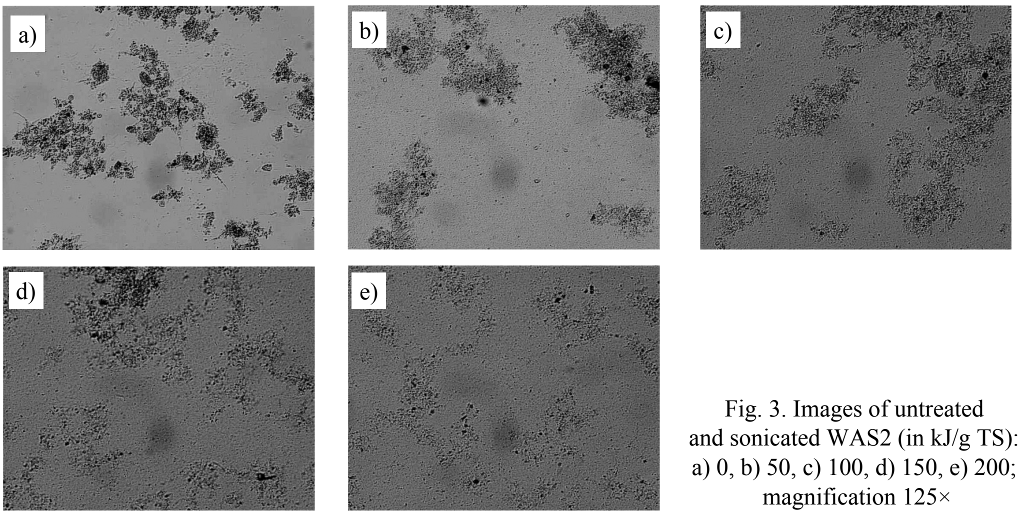

Fig. 3. Images of untreated and sonicated WAS2 (in kJ/g TS): a) 0, b) 50, c) 100, d) 150, e) 200; magnification 125×

Flocs of untreated WAS2 were small, rounded and compact, filamentous bacteria were not clearly noticeable. When a 50 kJ/g TS specific energy input was applied, the flocs maintained mainly a rather compacted structure. Rising up to 100 kJ/g TS and 150 kJ/g TS specific energy input, flocs were mostly disrupted. Main decomposition occurred at 200 kJ/g TS specific energy when the floc structure was almost completely disintegrated without sizeable visual differences between flocs and dispersed material in mixed liquor. In these observations, because of a light microscope was used, no information about cellular dimension can be provided.

3.2. PARTICLE SIZE DISTRIBUTION

The particle size distributions of WAS1 and WAS2 are shown in Figs. 4 and 5, respectively. The d_{25}–d_{90} cut diameters are reported in Table 2. The particle size of untreated WAS1 ranged from 4.5 to 265 μm. When the maximum 200 kJ/g TS specific energy was applied during the test, particle size distribution resulted in a range from 3.4 to 39.2 μm. The d_{50} cut diameter of WAS1 untreated sludge was 48.49 μm and decreased from 15.51 to 11.96 μm by increasing specific energy input.

A similar reduction in particle size distribution was noted when WAS2 samples were sonicated increasing specific energy inputs. The particle size of untreated WAS2 ranged from 5.15 to 262 μm with a maximum at 38.9 μm. A 200 kJ/g TS specific supplied sonication energy led to particle size distribution ranging from 3.4 to 34.25 μm with a maimum at 11.55 μm.

Table 2

Cut diameter of WAS1 and WAS2
at specific supplied energy increases [μm]

E_s [kJ/g TS)	0	50	100	150	200
WAS1					
d_{25}	25.31	11.82	10.52	10.12	10.02
d_{50}	48.49	15.51	13.53	13.19	11.96
d_{75}	71.48	26.34	16.98	16.56	14.18
d_{90}	93.30	58.95	22.92	20.76	17.48
WAS2					
d_{25}	24.90	11.69	8.99	11.04	8.82
d_{50}	38.88	15.49	12.33	13.26	11.55
d_{75}	54.64	22.07	15.88	13.51	14.32
d_{90}	77.64	29.92	20.77	16.75	16.06

As shown in Table 2, the 50 kJ/g TS specific energy input produced the strongest decrease of all cut diameters on both sludges. In fact, the d_{50} cut diameter of WAS1 decreased from 48.49 μm of the control sample to 15.51 μm (68.01% reduction) when the sludge was treated at 50 kJ/g TS, other uncrease of energy input from 50 to 200 kJ/g TS gave only 22.88% further reduction. Similarly, the d_{50} (μm) cut diameter of WAS2 reduced the value from 38.88 μm of the control sample to 15.49 μm (60.15% reduction) after the treatment at 50 kJ/g TS, for higher specific energy inputs cut diameters decreased more gradually.

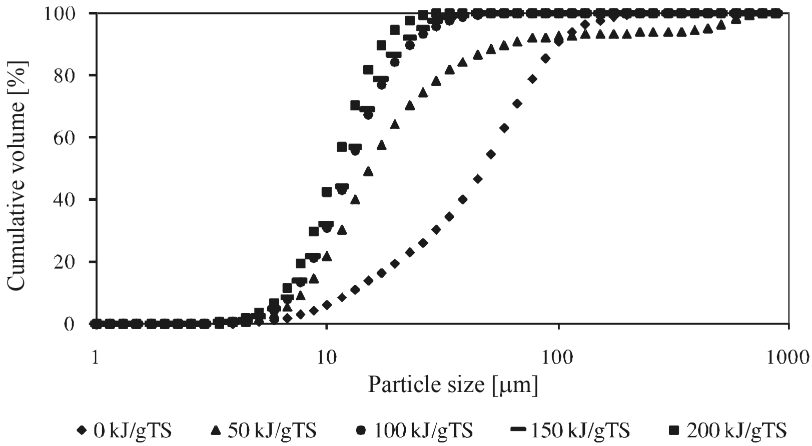

Fig. 4. WAS1 particle size distribution

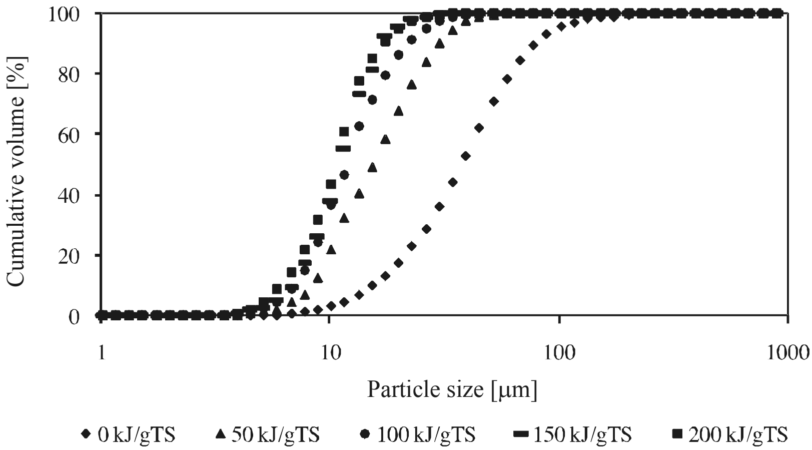

Fig. 5. WAS2 particle size distribution

In conclusion, we can note that WAS1 sample resisted better to the particle size smoothing and rounding during sonication. This effect could be due to lack of homogeneity of the crude sample in which biomass embed many other not biological fragments, on the other hand the more compact WAS2 sample was disintegrated to a more uniform particle size distribution.

3.3. ORGANIC RELEASE

Parameters such as sCOD and DOC were chosen to measure decomposition of the soluble organic matter in sludge supernatants. The sCOD and DOC profiles are shown in Fig. 6. The sCOD and DOC concentrations increased linearly with specific energy input in both sludge treated. Generally, the higher the specific energy input, the more intensive the release of soluble organics from sludge is.

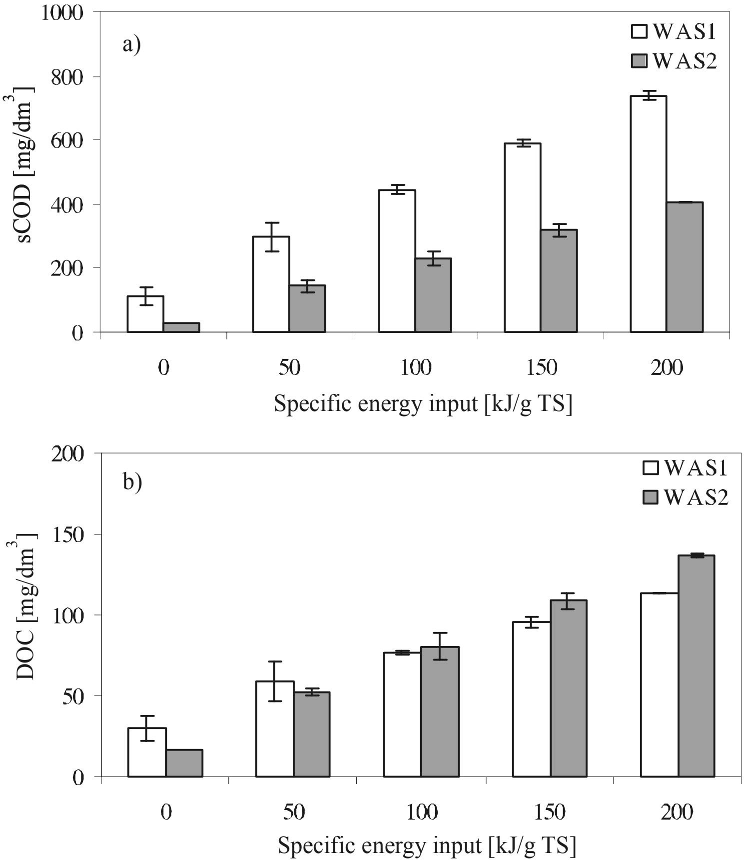

Fig. 6. Effect of specific energy on sCOD and DOC concentrations

In WAS1, the sCOD and DOC concentrations increased rapidly whenthe 50 kJ/g TS specific energy input was applied, then increased less reaching a maximum concentration of 546.73 mg/dm^3 and 92.5 mg/ dm^3 for sCOD and DOC, respectively. A maximum release in sCOD and DOC occurred when a 200 kJ/g TS specific energy input was applied.

In WAS2, a fast increases of the sCOD and DOC concentrations occurred when specific energy inputs ranged from 50 kJ/g TS to 100 kJ/g TS, therefore over the 150 kJ/g TS specific energy input, both parameters did not increased significantly. At the highest specific energy input of 200 kJ/g TS, the maximum release values of 426.86 mg/dm^3 and 142.95 mg/dm^3 in sCOD and DOC were observed, respectively.

For the same specific energy inputs, the rate of organic matter release due to ultrasound application was different for the two sludges. Comparing the sCOD and DOC values for 150 kJ/g TS specific energy input samples to the control ones (0 kJ/g TS), the organic matter release was 2.5 times higher in WAS2 than in WAS1.

It can be emphasized that to obtain the same results in terms of released organic matter, a higher specific energy input must be applied in WAS1 than in WAS2 ultrasonic treatment. It was probably related to the different source of the sludges. WAS1 is an industrial sludge characterized by a high content of lignin-refractory components, whereas WAS2 is a municipal sludge, with a predominantly fecal component, which makes sludge disintegration easier.

3.4. NITROGEN RELEASE

The release of ammonium ion and other nitrogen compounds at various specific energy inputs was also studied. Profiles of ammonium ion and total nitrogen are shown in Fig. 7. The relationship between concentration of ammonium nitrogen and specific energy input in WAS1 supernatants was almost linear, while the total nitrogen concentration increased linearly for low specific energy inputs until reaching a maximum value of about 25 mg/dm^3 for specific energy inputs higher than 150 kJ/g TS.

The percent increase of concentration of ammonium ion with specific energy input was asymptotic and characterized by an initial rapid growth in WAS2. Moreover, concentration of WAS2 total nitrogen increased linearly with specific energy input reaching a maximum of 41 mg/dm^3 for the highest specific energy input against a concentration of 25.46 mg/dm^3 in WAS1.

Other authors [14, 18] reported that during sonication of sludge, the oxidative effects are negligible and the concentration of nitrate and nitrite in the supernatant can be assumed much smaller than that of ammonium ions, further the total nitrogen increase is mainly related to the presence of organic nitrogen that was transformed to a soluble form.

As many studies report, nitrogen release is mainly due to the cell break-up produced by ultrasonic treatment. As a result of disintegration, intracellular organic nitrogen was released into the liquid phase. Ammonium nitrogen increase may be linked to a partial ammonification of organic nitrogen. Nevertheless, release of the ammonium nitrogen can also be originated by disintegration of organic nitrogen from not-biological debris.

Different sludge materials release ammonium but in a similar way, however they have different total nitrogen release potential. This may be due to different homogeneity and quality of the flocs.

It can be supposed that during treatment of the WAS1 sample, the disrupted biomass material quickly releases intracellular organic nitrogen and consequently a better release of ammonium ion may occur. Because of typical nitrogen poor industrial pulp and paper origin of WAS1, when specific energy inputs increased, TN decreased as a consequence of a scarce presence of not-biological debris in the biomass.

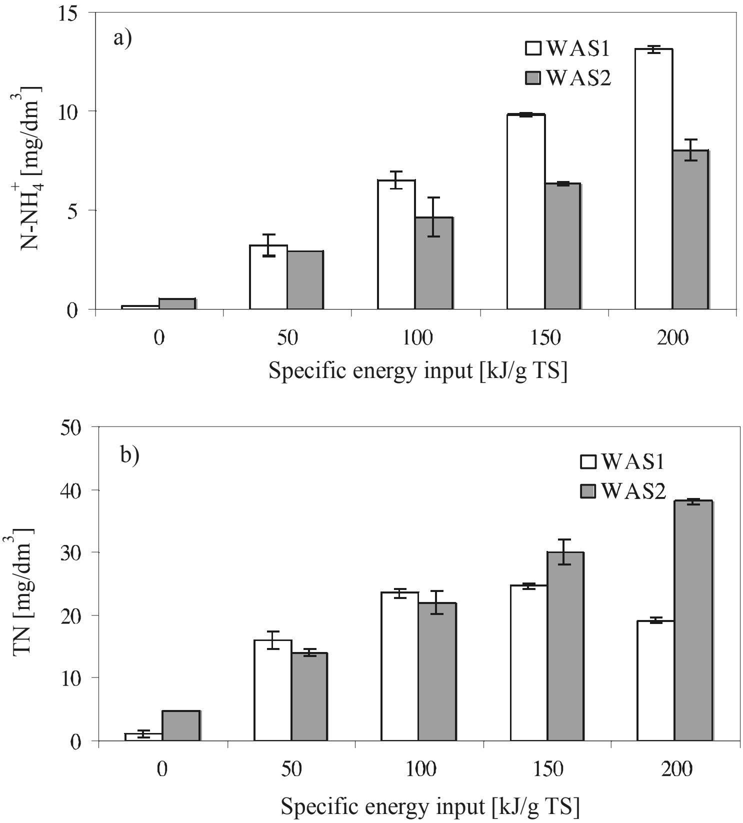

Fig. 7. Effect of specific energy on ammonium ions and total nitrogen concentrations

Otherwise, WAS2 sample, taken from municipal wastewater, had a characteristic high nitrogen content due to biomass and nitrogenous colloidal-not biological particulate fraction of the sludge, thus the release of ammonium ion and TN in the liquid phase increased both linearly with the sonication specific energy supplied.

4. CONCLUSIONS

In this study, the effect of low frequency ultrasonic treatment on two different sludges (from industrial treatment plant, WAS1, and from municipal WWTP, WAS2) was investigated by batch tests. The efficiency of sludge disintegration was strongly influenced by sludge characteristics such as sludge origin and their TS content.

As results from microscopic observations, the highest specific energy input for both sludges led to a complete (in WAS2) or partial floc disruption (WAS1).

Particle size distribution analysis has shown a gradual decrease of the particle diameter with the increase in specific energy input on both sludges.

Moreover, increase in sCOD, DOC, ammonium ion and total nitrogen concentrations at various specific energy inputs have been observed in both sludges. In WAS2, a higher organic matter release rate was observed and this supported different sludge origin. Lower ammonium nitrogen increase was found in WAS2 than in WAS1. This fact could be related to a higher ammonification rate in WAS1 than that in WAS2.

Ultrasounds seem to have a useful ability to enhance solubilization of organic matter and nitrogen compounds during sludge treatment.

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