A swirling jet-induced cavitation to increase activated sludge solubilisation and aerobic sludge biodegradability

Giuseppe Mancuso[a*], Michela Langone[a], Gianni Andreottola[a]

[a] Department of Civil, Environmental and Mechanical Engineering, University of Trento, via Mesiano 77, 38123, Italy

* Corresponding authors. Tel. +39 0461 282681 fax: +39 0461 282672

E-mail addresses: giuseppe.mancuso@unitn.it (G. Mancuso), michela.langone@unitn.it (M. Langone), gianni.andreottola@unitn.it (G. Andreottola)

Abstract

In this work, a modified swirling jet induced hydrodynamic cavitation (HC) has been used for the pre-treatment of excess sludge. In order to both improve the HC treatment efficiencies and reduce the energy consumption, the effectiveness of the HC reactor on sludge disintegration and on aerobic biodegradability has been investigated at different operating conditions and parameters, such as temperature, inlet pressure, sludge total solid (TS) content and reactor geometry. The inlet pressure was related to the flow velocity and pressure drop. The best results in terms of sludge solubilisation were achieved after 2 h of HC treatment, treating a 50.0 gTS L^{-1} and using the three heads Ecowirl system, at 35.0°C and 4.0 bar. Chemical and respirometric tests proved that sludge solubilisation and aerobic biodegradability can be efficiently enhanced through HC pre-treatment technique.

At the optimum operating conditions, the specific supplied energy has been varied from 3,276 to 12,780 kJ kg TS^{-1} in the HC treatment, by increasing the treatment time from 2 to 8 hours, respectively. Low endogenous decay rates (b_H) were measured on the excess sludge at low specific supplied energy, revealing that only an alteration in floc structure was responsible for the sludge solubilisation. On the contrary, higher b_H values were measured at higher specific supplied energy, indicating that the sludge solubilisation was related to a decreasing biomass viability, as consequence of dead cells and/or disrupted cells (cell lysis).

Keywords

Energy efficient; Sludge solubilisation; Swirling jet-induced cavitation; Sludge disintegration; Aerobic biodegradability.

Abbreviations

AC	Acoustic cavitation	WWTPs	Wastewater treatment plants
HC	Hydrodynamic cavitation	DD	Disintegration degree
COD	Chemical oxygen demand	TS	Total solids
TCOD	Total COD	TSS	Total suspended solids
PCOD	Particulate COD	VS	Volatile solids
SCOD	Soluble COD	VSS	Volatile suspended solids
$SCOD_{bio}$	Biodegradable SCOD	TKN	Total Kjeldahl nitrogen
$\Delta SCOD$	SCOD increase	P_{TOT}	Total phosphate
NH_4^+-N	Ammonia nitrogen	SE	Specific supplied energy
DO	Dissolved oxygen	EE	Energy efficiency
OUR	Oxygen uptake rate	b_H	Endogenous decay rate
SOUR	Specific OUR		

1. Introduction

Activated sludge system is the most used biological process in industrial and municipal wastewater treatment plants (WWTPs). This process produces a high amount of excess sludge, which has a relevant impact on the operating costs in WWTPs. Thus, any improvements in reducing the quantity of the excess sludge are always beneficial.

Several technologies, based on destruction of bacterial cell walls and membranes have been developed with the aim to reduce the sludge production by enhancing (*i*) the sludge biodegradability and its reuse in other biological processes, and (*ii*) the sludge dewaterability and consequently the performance of separation technologies. With this purpose, several pre-treatment techniques such as biological [1,2], thermal hydrolysis [3,4], chemo - thermal [5,6], mechanical [7–9], chemo mechanical [10], chemical [11–13], and alkali processes [14,15] have been proposed and successfully applied in WWTPs.

Among mechanical treatments, the most successful method is cavitation, that is taking a more prominent role in the field of wastewater treatment, mainly due to the ease of operation, flexibility and capability to vary the required intensities of cavitational conditions [16]. Cavitation acts by destroying bacterial cell walls and membranes resulting in a release of intracellular and extracellular matter [17]. Many of the intracellular constituents, including cytoplasm and nuclei acids, are readily biodegradable, resulting either in acceleration of both aerobic and anaerobic digestion processes in the sludge treatment line or in promoting denitrification in the wastewater treatment process. Among other benefits, cavitation process may counteract the formation of activated sludge bulking and foam, resulting in improved sludge sedimentation properties [18].

Depending on the mode of its generation, cavitation can be defined such as acoustic, hydrodynamic, optic and particle cavitation. Among these, the most used are acoustic (AC) and hydrodynamic cavitation (HC).

Over the past decades, many works have been carried out on the application of the AC on activated sludge in order to increase the biogas production in the anaerobic digestion process [19], to enhance the microbial activity and the sludge dewaterability [20], to increase the soluble chemical oxygen demand (SCOD), proteins and nucleic acids concentrations [21], to improve the sludge settling [17,22,23], and to reduce the excess sludge from the activated sludge system [24].

On the contrary, HC has been studied to a lesser extent than AC, but published researches have shown promising outcomes in sludge pre-treatment [25], dyes removal [26], chemical compounds oxidation [27], bacteria removal [28]. Further, recent studies have proven its cost-effectiveness [7,29]. HC is generated by pressure variation in a flowing liquid caused by the velocity variation in the system [30–32]. When the static pressure at the mechanical constriction falls below the vapour pressure of the liquid, cavities are generated. At the downstream of the constriction, as the liquid jet expands, the pressure recovers and this results in the violent collapse of the cavities [31], giving rise to high pressure and temperature pulses. Thus, HC can be generated by introducing constrictions, such as orifice plate [33], Venturi [34,35] or throttling valve, in the flow.

Further, the development of new HC reactor configurations have attracted the attention of researchers and enterprises due to the technical feasibility of the scale-up. Recently, a high-pressure jet device, where cavitation occurs, has been studied for activated sludge reduction [7]. HC has also been generated using a stator and rotor assembly, which has been effectively applied for enhancement of the biogas production from activated sludge [36] and lignocellulosic biomass, like wheat straw [37].

While literature is available on anaerobic biodegradability enhancement, there is a paucity of literature on the effect of HC on the aerobic biodegradability.

In this study, a modified swirling jet-induced reactor, named Ecowirl reactor [38], has been used in order to generate HC, by creating a vacuum-core vortex. The main aim of the present work was to prove the effectiveness of the Ecowirl reactor on sludge solubilisation exploring the potential for reducing the energy consumption and examining the influences of the pre-treatment on aerobic biodegradability. With this purpose, the effects of different factors such as medium temperature, inlet pressure (and consequently flow rate), solid concentration, reactor geometry, HC treatment time on sludge solubilisation, disintegration degree and microbial activity have been investigated.

2. Materials and methods

2.1 Sludge characterization

Excess activated sludge of a nitrification/denitrification process, collected at Trento municipal WWTP, Italy, was used for the experimental investigations. In order to get sludge with a high total solid (TS) concentration, sludge was collected after the dynamic thickening process. Thickened sludge was then diluted with tap water to obtain the desired TS concentration. The physical and chemical characteristics of the thickened sludge used were: pH 6.8 ± 0.2, TS = 33.4 ± 0.5 g L^{-1}, volatile solids (VS) = 27.9 ± 0.4 g L^{-1}, total chemical oxygen demand (TCOD) = 38,015 ± 321.0 mg L^{-1}, soluble COD (SCOD) = 318.6 ± 5.0 mg L^{-1}, total Kjeldahl nitrogen (TKN) = 2,856 ± 3.0 mg L^{-1}, ammonia nitrogen (NH_4^+-N) = 33.7 ± 1.0 mg L^{-1}, total phosphate (P_{TOT}) = 1,062 ± 56.0 mg L^{-1}.

2.2 HC system

Fig. 1 shows a schematic representation of the experimental setup for the pre-treatment using HC process. It consists of a closed loop circuit designed to treat 50.0 L of sludge from a feed tank, then taking it into a flow channel internally accommodating the HC reactor (Ecowirl reactor) and then discharging the treated sludge back to the main tank by means of a Mohno pump (3.0 kW, nominal power, Netzsch Pumps & Systems GmbH Germany). A solid geometry was created using Autodesk Inventor Professional software (Fig. 2). Ecowirl reactor is a modified swirling jet reactor, in which cavitation is generated by using a multi-dimensional vortices generator, consisting of a frustum-conical pre-swirling chamber (Fig. 2, (2)) preceded by another chamber (Fig. 2, (1)) where is located an orifice plate with injection slots through which the flow enters and a vacuum-core vortex is created, and a double cone chamber (Fig. 2, (3)) where a collision plate is present to fast recover the pressure. In the present work, the sum of the regions (1) and (2) is called “Ecowirl head”. Detailed information of experimental setup and Ecowirl reactor are reported in Mancuso et al. [26]. In order to measure the pressure changes in the Ecowirl reactor, pressure gauges were located as shown in Fig. 2. The static (relative) pressure has been measured.

2.3 Methods

2.3.1 HC tests

The feed tank was filled with 50.0 L of sludge. The sludge was recirculated in the loop by using the by-pass line for about 15 min in order to homogenise its content. At this stage, cavitation did not occur. In the meantime, the temperature of the sludge was adjusted at the desired temperature by using a heating and cooling system with steel coils. Temperature was kept constant throughout the experiments (with a variation of ± 3.0°C). During the experiments, the required value of inlet pressure at cavitation system was achieved by adjusting the frequency of the pump inverter. Consequently, the flow rate and the flow velocity varied.

HC was first optimized in terms of different operating parameters. As reported in Table 1, effects of temperature (20.0, 25.0, 30.0 and 35.0 °C), inlet pressure (2.0, 3.0, 4.0 bar), TS content (7.0, 12.0, 23.0 and 50.0 g L^{-1}) on sludge solubilisation and disintegration degree have been investigated using one Ecowirl heads with the standard configuration (six injection slots, upstream diameter 10 mm - downstream diameter 8 mm). In this study, the inlet pressure, instead of flow rate and flow velocity, was taken into account because it is an easy operational parameter to set. Then the geometry of the Ecowirl reactor has been studied, varying the number and the diameter of the injection slots present in the orifice plate and the number of Ecowirl heads in series (Table 1). Each test was conducted for 2 h.

Following the optimization of HC parameters, an 8h-experiment was carried out at 50.0 gTS L^{-1}, 4.0 bar, 35.0°C and using 3 standard Ecowirl heads in series in order to evaluate the HC efficiencies as function of specific energy applied (SE) to the sludge.

Sludge samples were taken from the main tank through the sampling port at the initial and at the end of experiments and analysed. In 8h-experiments sludge samples were collected at 0, 2, 4 and 8 h. pH in the main tank was continuously monitored.

2.3.2 Respirometric technique

Fig. 3 shows a schematic representation of the respirometric experimental setup used to evaluate the HC effect on aerobic degradability and microbial activity. Details are as follows: (1) a respirometric reactor that consists in a glass cylinder filled with 1.2 L of sludge; (2) a magnetic agitator to mix and homogenise the solution; (3) an air compressor; (4) an air diffusor; (5) probes to measure DO, pH and temperature of the solution; (6) a thermostatic bath to kept constant the temperature of the solution at 20.0°C; (7) a computer used for storing and monitoring all data connected to the DO probe through a RS-232 port. The DO depletion in the vessel, due to the substrate utilization, was monitored over the time. First, the Oxygen Uptake Rate (OUR), expressed as $mgO_2\ L^{-1}\ h^{-1}$, was determined by the slope of the plot of DO concentration versus time after stopping for few minutes the air flow inlet [39]. Then, the specific OUR (SOUR), expressed as $mgO_2\ gTSS^{-1}\ h^{-1}$, was obtained dividing the OUR by the TSS concentration in the assays.

2.3.2.1 Aerobic biodegradability

A series of respirometric tests have been carried out on filtered sludge samples from the optimized HC experiment in order to evaluate the effect due to the HC process on aerobic biodegradability as a function of the applied energy. The activated sludge from the oxidation tank of the municipal WWTP of Trento was used as inoculum. TSS content of the inoculums was 3.0 ± 0.1 g L^{-1}. Samples at 0, 2, 4 and 8 h from the optimized HC experiment have been analysed.

The soluble biodegradable COD ($SCOD_{bio}$) of the untreated and treated sludge has been determined according to Andreottola et al. [39], and Wentzel et al. [40].

Low values of substrate and biomass concentrations (F/M : food to microorganism) were maintained during the test, approximately 0.05 mg COD mg^{-1} VSS. For all the experiments, thiourea (20.0 mg L^{-1}) was added to inhibit the nitrification process.

The recorded data of the oxygen uptake rate (OUR) were plotted as a function of time. A primary curve segment, which is characterized by a higher slope, represented substrate consumption, operated by biomass; on the contrary, the final segment, characterized by a lower slope, represents the DO consumption under endogenous conditions. The amount of exogenous oxygen consumption (ΔO_2) has been determined by calculating the area under the OUR curve and subtracting the contribution of the endogenous respiration. In order to evaluate the $SCOD_{bio}$, the exogenous oxygen consumption was then converted to equivalent COD using the expression based on the COD mass balance [41].

2.3.2.2 Microbial activity

A series of respirometric tests have been carried out on sludge samples from the 8h-optimized experiment in order to evaluate the effect of HC on microbial activity as a function of the applied energy. The change of sludge microbial activity was evaluated by determining the endogenous decay rate (b_H) of heterotrophic biomass, through the "single batch test" procedure [42]. For all the experiments, thiourea (20.0 mg L^{-1}) was added to inhibit the nitrification process. Samples at 0, 2, 4 and 8 h from the optimized HC experiment have been analysed. Sludge samples were diluted by adding purified water in order to obtain a TSS content of 3.5 ± 0.5 g L^{-1}.

2.4 Calculations

The process efficiency was evaluated by measuring the improvement of solubilisation of sludge in terms of the SCOD-increase (Eq. 1), [43], the ratio of change in soluble chemical oxygen demand (SCOD) after cavitation to particulate chemical oxygen demand ($PCOD_0$ = TCOD - $SCOD_0$) (Eq. 2), [44], and the ratio of change in ammonia after cavitation to initial organic nitrogen content ($Norg_0 = TKN_0 - NH_4^+\text{-}N_{,0}$) (Eq. 3):

$$\Delta SCOD(\%) = SCOD_{cav} - SCOD_0 \qquad \text{Eq. (1)}$$

$$DD_{PCOD}(\%) = \frac{SCOD_{cav} - SCOD_0}{PCOD_0} \times 100 = \frac{SCOD_{cav} - SCOD_0}{TCOD - SCOD_0} \times 100 \qquad \text{Eq. (2)}$$

$$DD_N(\%) = \frac{NH_4^+ - N_{cav} - NH_4^+ - N_0}{\mathrm{Norg}_0} \times 100 = \frac{NH_4^+ - N_{cav} - NH_4^+ - N_0}{\mathrm{TKN}_0 - NH_4^+ - N_0} \times 100 \qquad \text{Eq. (3)}$$

where $SCOD_{cav}$ is the soluble COD of the treated sludge by using HC [mg L^{-1}] at the time t, $SCOD_0$ is the soluble COD of the untreated sludge [mg L^{-1}], TCOD is the total COD of the untreated sludge [mg L^{-1}], NH_4^+-N_{cav} is the ammonia content of the treated sludge by using HC [mg L^{-1}] at the time t, NH_4^+-N_0 and TKN are the ammonia and total Kjeldahl nitrogen content of the untreated sludge [mg L^{-1}].

Other index of importance in sludge disintegration includes the sludge disintegration degree calculated as the ratio of SCOD increase by cavitation to the SCOD increase by the chemical disintegration (Eq. 4), [20]:

$$DD_{COD\ NaOH}(\%) = \frac{SCOD_{cav} - SCOD_0}{SCOD_{NaOH} - SCOD_0} \times 100 \qquad \text{Eq. (4)}$$

where $SCOD_{NaOH}$ [mg L^{-1}] is the soluble COD of the reference sample obtained with a strong alkaline disintegration (NaOH digestion).

In order to compare results, the specific supplied energy (SE) has been determined by using Eq. (5):

$$SE\left(\frac{kJ}{kgTS}\right) = \frac{P_{abs} \times t}{V \times TS} \qquad \text{Eq. (5)}$$

where P_{abs} is the pump absorbed power [W], t is the treatment time [sec], V is the volume of the treated sludge [L] and TS is the solids content [g L^{-1}].

Finally, according to Zhang et al. [43], the energy efficiency (EE), expressed as mg DSCOD kJ $^{-1}$, has been calculated as the mg of SCOD-increase per unit of energy supplied (Eq. 6). Higher EE values correspond to higher removal efficiencies.

$$EE\left(\frac{mg\Delta SCOD}{kJ}\right) = \frac{V \times \Delta SCOD}{P_{abs} \times t} \times 1000 \qquad \text{Eq. (6)}$$

2.5 Analytical methods

At the beginning and at the end of each HC test, sludge samples were collected from the bottom of the feed tank through the sampling port (Fig. 1) and stored at 4.0°C for subsequent analysis. TS, VS, TSS, TCOD, SCOD, TKN, NH_4^+-N were calculated according to the standard methods [45]. Prior to SCOD and NH_4^+-N determinations the sludge samples were centrifuged at 5000 × g and the supernatant liquid was filtered using cellulose nitrate membrane of pore size 0.45 micron by compression. The filtrates were further used in aerobic biodegradation tests. pH was monitored by using a Crison 25 portable pH-meter. All the analyses were performed in duplicate and the results were expressed as average of the values. A reference sample was defined as the soluble COD obtained by chemical sludge disintegration in 1.0 mol L^{-1} sodium hydroxide for 24 h at 20.0°C [46].

3. Results and discussion

Objective of all the experiments was to understand the effect of different operating parameters and arrive at a set of parameters that will give the maximum disintegration degree for an activated sludge. Operative parameters, operating conditions and efficiencies measured at the end of each test are summarized in Table 1. The results have been described and discussed with more details in the following paragraphs. The experimental errors were within 2 - 3% of the reported value of the extent of degradation. Finally, the effect of the HC treatment on the aerobic biodegradability and microbial activity is discussed.

3.1 Effect of temperature

Heating alone up to 60°C can increase the sludge solubilisation [47,48], however larger increases in SCOD can be obtained with AC than with heating alone [48].

Although the effect of medium temperature on sludge disintegration has been studied in AC systems, the influence of temperature on sludge disintegration in HC process has not been investigated. A recent study only reports the effect of temperature on hydrodynamic cavitation intensity [49].

Thus, in this study, for the first time, the effect of temperature in the range of 20.0 - 35.0°C on sludge disintegration was examined. Experiments were conducted using the sludge containing 50.0 gTS L^{-1}, working with an inlet pressure of 2.0 bar. Without temperature control, the bulk solution gradually raised in temperature (data not showed). Thus, for each test, temperature was kept constant for the entire duration of the experiment by using a heating and cooling system.

From Fig. 4 it is evident that the higher the temperature, the more efficient the HC efficiency, in terms of sludge solubilisation, was. The DSCOD increased from 649 to 1397 mg L^{-1} with temperatures ranged from 20.0 to 35.0°C at 2 h. At 35.0°C, DD_{PCOD} and $DD_{COD\ NaOH}$ reached values of 3.6 and 5.4%, respectively.

The effects of temperature on HC are complex, which have positive and negative effects on HC treatment efficiency. The increase of temperature implies a decrease of the surface tension, leading to an easier bubble formation [32]. However, these generated bubbles are richer in vapour content, which cushioning their implosion, reducing the intensity of bubbles collapse [32]. Recently, Sarc et al. [49] demonstrated that the magnitude of pressure oscillations, which occur due to cavitation bubble collapses, increases for temperature of 40°C, but then drops significantly for higher temperatures.

In this study, as a result from the obtained data, it was possible to observe the positive effects of temperature (in the range of 20.0 - 35.0°C) on sludge solubilisation in HC treatment, resulting in an increase of SCOD for increasing temperatures.

Our results agree with previous studies on the effect of temperature on sludge disintegration by using AC. Indeed, Chu et al. [47], found that both ultrasonic vibration and bulk temperature rise, from 20.0 to 55.0°C, contribute to the AC treatment efficiency. The same effect was observed by Grönroos et al. [48]. Huan et al. [20] showed that the temperature of sludge samples would increase with ultrasonic energy input and the rise of temperature helps to ultrasonic disintegration. Xu et al. [50] also proved that an increase in temperature involved an intensification of the sludge solubilisation in an ultrasound combined with ozone treatment.

3.2 Effect of inlet pressure: flow velocity and pressure drop

Effect of the inlet pressure on solubilisation was evaluated with the activated sludge containing 50.0 gTS L^{-1}, at 25.0°C. The pressure measured at the inlet of the Ecowirl head was varied from 2.0 to 4.0 bar in steps of 1.0 bar. As shown in Table 1, increasing the inlet pressure from 2.0 to 4.0 bar, an increase in flow rate was observed from 4.6 to 6.8 $m^3 h^{-1}$, respectively. In this study, the velocity profile along the HC device has not been calculated due to the complex geometry of the Ecowirl reactor. However, the velocity of the flow through the narrowest reduced area of the orifice plate has been evaluated. Table 1 shows that being equal the geometry, increasing the flow rate an increase of flow velocity has been measured.

The results shown in Fig. 5 demonstrated that, after 2h HC treatment, higher SCOD concentrations were measured at higher inlet pressures, and thus at higher flow velocities. The DSCOD increased from 617 to 1259 mg L^{-1} with inlet pressures increased from 2.0 to 4.0 bar, respectively.

The reasons behind this are the increased turbulence level and higher local pressure oscillations due to the higher flow velocity. Recently, Sarc et al. [49] proved the importance of flow velocity, which increased with the flow rate and the inlet pressure. Authors verified that by the increase of the flow velocity the amplitude of pressure waves generally increases.

Further, in this study, higher inlet pressures produced lower pressures in the vacuum zone and thus higher pressure drops (Fig. 6), which means higher shear forces that break down bacterial cell walls and release the intracellular substances into aqueous phase resulting in an increase in the sludge solubilisation. The role of the intense turbulent shear zone behind the orifice plate in enhancing cavitational activity has been qualitatively established by Kumar et al. [51].

As shown in the pressure profile (Fig. 6), the static (relative) pressure downstream the Ecowirl reactor decreased sharply as the flow passed the Ecowirl reactor, reaching negative values. Beyond Ecowirl reactor, static pressure started to recover but it never got to the upstream value, and a pressure drop can be measured.

The pressure measured in the vacuum zone in this study was -0.65±0.13, -0.79±0.15 and -0.83±0.15 bar, at 2.0, 3.0 and 4.0 bar, respectively, which was higher than the vapour pressure of the water equal to -0.96 bar at 25.0°C. The development of cavitation for pressure higher than the vapour pressure of water was also reported theoretically by Kumar and Pandit [52] and experimentally Kim et al. [34]. The presence of dissolved gas and suspended solids improved the cavitation development due to the formation of weak spots, which might play a crucial role in creating cavitation when the pressure in the vacuum zone was higher than the vapour pressure.

The importance of the pressure drop seen in this study is in accordance with others reports on HC. Kim et al. [34] reported a very low DSCOD increase (20 mg L^{-1}) after 2 h of HC treatment with an activated sludge containing 18.0 gTS L^{-1}, by using a Venturi system with an inlet pressure of -0.07 bar. This Venturi system produced a pressure drop of only 0.42 bar between the inlet and the throat section. On the contrary, Lee and Han [33] reported a 23% disintegration degree (DD_{PCOD}) after 20 min of HC treatment of activated sludge (TS = 9.8 g L^{-1}) working with an orifice plate and at an inlet pressure of 7.0 bar.

During our HC tests, it was necessary to progressively reduce the frequency of the pump inverter in order to keep constant the inlet pressure value, and thus the flow rate and flow velocity. This was due to a progressive alteration of the rheology of the sludge resulting in a decrease of the viscosity of the sludge during the HC treatment, as consequence of the disruption of cell or microbial flocs [34].

3.3 Effect of sludge solid concentration

Fig. 7 shows the effects of solid concentration on the sludge disintegration. Four sludge concentrations were tested, 7.0, 12.0, 23.0 and 50.0 g L^{-1}. The higher values of ΔSCOD and DD have been observed for the sludge with the highest TS content. For example, ΔSCOD for activated sludge with TS of 12.0, 23.0, 50.0 g L^{-1} were 2.35, 7.23 and 36.30 times higher than that for activated sludge with TS of 7.0 g L^{-1} after 2 h of HC treatment. A linear relationship between TS concentration and DD_{PCOD} was found (DD_{PCOD} = 0.0298 TS - 0.0111, R^2 = 0.989).

The increase in TS content provides more cells and aggregates and thus a higher viscosity of the sludge, due to the inter- and intra- particle interactions. Both growth and collapse of bubbles are slowed down by viscosity in hydrodynamic cavitation [53]. On the other hand, the increase in TS enhances the collisions between sludge flocs and cavitation bubbles, allowing the subsequent increase in sludge disintegration. Thus, the negative effect of high viscosity of the sludge with high TS was negligible compared to the positive effect of TS concentration on the sludge disintegration.

Kim et al. [34] discussed the effect of solid concentration (0.5, 1.5, 3.0 and 4.0%) on sludge disintegration by using a Venturi system (HC). In accordance with the present work, they observed that ΔSCOD was greater for sludge with higher solids content. They also observed that viscosity significantly decreased during the first 30 min of HC treatment, as consequence of the disruption of cell or microbial flocs.

The same linear relationship has not been reported in studies on AC systems, where an increase in TS content has opposite effects. The increase in TS content provides more cells to be in contact with cavitation bubbles, but can also impede the propagation of ultrasonic pressure waves and reduces the power of ultrasonic waves reached to sludge flocs. Indeed, Le et al. [54], using five synthetic mixed sludge samples (12.0, 24.0, 28.0, 32.0 and 36.0 g L^{-1}), observed a gradual increase in ΔSCOD by increasing the TS content, but the best DD was not found at the maximum TS. This is in accordance with the studies reported by Kidak et al. [55], Sahinkaya [56] and Zhang et al. [57]. On the other hand, Xu et al. [50] observed a decrease of ΔSCOD with the increase of initial sludge concentration. This result was also confirmed by Huan et al. [20], that founded that sludge with low TS concentration was easier to disintegrate by AC.

3.4 Effect of geometry

3.4.1 Number and diameter of injection slots of Ecowirl reactor

Different Ecowirl heads with different number and diameter of injection slots have been considered. Experiment were carried out with sludge at 50.0 gTS L^{-1}, at 25.0°C. The inlet pressure was set at 4.0 bar in order to reduce the possible blocking of the holes.

The standard configuration (6 injection slots, upstream diameter 10 mm - downstream diameter 8 mm) was first compared with the configuration A (6 injection slots, upstream diameter 5 mm - downstream diameter 3 mm) in order to evaluate the effect of diameter of injection slots on sludge disintegration. Then, the effect of the number of the injection slots has been investigated. Thus, the configuration A was compared with configurations B (9 injection slots, upstream diameter 5 mm - downstream diameter 3 mm) and C (12 injection slots, upstream diameter 5 mm - downstream diameter 3 mm).

The number of injection slots being equal, reducing the diameter of injection slots reduced the flow rate of the sludge in the HC system. Flow rates of the standard configuration and the configuration A were 6.8 and 2.3 $m^3\ h^{-1}$, respectively. However, the flow velocity through the reduced area of the orifice of the configuration A was higher than that calculated in the standard configuration, due to the lower flow passage area of the orifice of the configuration A (Table 1). Making a comparison between the standard configuration and the configuration A, it was possible to observe that a decrease in diameters of injection slots caused an increase in intensity of the cavitation, due to the higher flow velocity. DD_{PCOD} and DD_{NaOH} increased from 3.8%, and 5.7% using the standard configuration to 5.9%, and 8.2% using the Configuration A, respectively (Table 1).

The diameter of injection slots being equal, decreasing the number of injection slots the flow rate of the sludge in the HC system decreased, but the flow velocity increased (Table 1). The results are shown in Fig. 8. Lower number of injection slots caused high sludge disintegration degrees, due to the higher flow velocity.

According to Sarc et al. [49], all those results proved the influence of the flow velocity on the effectiveness of hydrodynamic cavitation.

3.4.2 Number of Ecowirl heads

Effects of the number of Ecowirl heads in series were evaluated for the single-, two-, and three-heads systems with activated sludge containing 50.0 g TS L^{-1}, at 25.0°C and 2.0 bar. Fig. 9 shows a schematic representation of Ecowirl reactor consisting of three -heads in series.

Increasing the number of Ecowirl heads the flow rate decreased. In addition, the calculated flow velocity through the reduced area of the orifice of the first head decreased. In spite of this observation, increasing the number of Ecowirl heads, the sludge degradation efficiency increased.

Results on sludge disintegration are reported in Fig. 10. After 2 h of treatment, the DSCOD value of the two-heads system was almost equal to that of the single-head system. On the contrary, the DSCOD value of the three -heads system was 2.1 time higher than that of the single-head system. Combing three Ecowirl heads in series, an increase in DD_{PCOD} and DD_{NaOH} from 1.4%, and 2.1% using the single-head system to 3.7%, and 4.5% using the three -heads system, respectively, was observed. The results obtained in this study are consistent with previous findings. Kim et al. [34] reported that a better HC treatment efficiency with a two-Venturi system was expected since DSCOD would be proportional to the volume of the cavitation zone. However, the number of the heads connected in series would be limited since there must be some energy dissipation during the course of cavitation development and collapse.

3.5 Comparison of energy efficiency

The variation of each operating condition in the HC treatment implied a variation of both the absorbed power and the COD disintegration degree, here expressed as the SCOD-increase. Fig. 11 reports the energy efficiency (EE) vs the specific supplied energy (SE). As can been seen from the graphs, not always an increase in SE corresponds to an increase in EE. Observing the diagrams, the main findings of the investigation reported in this study can be summarized as follows:

- increasing the flow velocity by decreasing the injection slots number while keeping constant the inlet pressure, the EE increased with the increase of SE (Fig.11.a);
- increasing the number of Ecowirl heads while keeping constant the inlet pressure, the EE increased with the increase of SE (Fig.11.b);
- using the same geometric configuration of Ecowirl reactor and increasing the inlet pressure up to 3.0 bar, the EE increased with the SE (Fig.11.c). However, no significant differences were detected, in terms of EE, working at a higher inlet pressure of 4.0 bar;
- increasing the flow velocity by decreasing the diameters of the injection slots while keeping constant the inlet pressure, the EE decreased with the increase of SE (Fig.11.d). The maximum EE was obtained by using a geometry of Ecowirl reactor with the smallest diameters of the injection slots, in correspondence of which the lowest SE was applied;
- the maximum EE was obtained working at the higher temperature and the higher solid concentration tested while keeping constant the inlet pressure, in correspondence of which the lowest SE was applied (Fig.11.e,f).

Thus, working at temperatures of 35.0 °C, solid concentrations of 50.0 g L^{-1} and small diameters of injection slots of the orifice plate in the Ecowirl reactor, the highest HC treatment efficiencies has been achieved with the lowest energy consumptions. In this study, the increase in the inlet pressure contributed to an increase in the EE only up to a pressure value of 3.0 bar.

3.6 Effect of supplied energy

An 8-hours HC experiment was performed considering a combination of optimal parameters values as defined above, in order to evaluate the HC efficiency as function of the specific supplied energy. Thus, a HC test was carried out with activated sludge containing 50.0 gTS L^{-1}, using the three-heads system at 35.0°C and 4.0 bar. Table 2 summarizes the results obtained from the HC 8h-experiment.

The disintegration degree and the values of SCOD before and after HC treatment are shown in Fig. 12. The increase in treatment time enhanced the amount of organic substance solubilised, starting from an initial mean value of SCOD of 244 mg L^{-1} ($SCOD_0$) to a final value of 4,578 mg L^{-1} ($SCOD_{8h}$). At 4 h and 8 h of treatment, the increase of soluble chemical oxygen demand ($SCOD_t$ - $SCOD_0$) was 1.7 and 2.9 time higher than the value measured after 2 h of treatment.

SCOD increased with the treatment time and, thus, with the specific supplied energy. For example, when HC time was 2, 4 and 8 h, the specific supplied energy was 3,276, 6,444 and 12,780 kJ $kgTS^{-1}$, while DD_{PCOD} values were 6.5, 10.8 and 19.2%, respectively.

Organic matter disintegration degree versus duration of HC treatment had a pseudo-linear trend. A similar trend also emerged by looking at DD_N. During the HC experiment, also ammonia concentrations increased. Nitrogen is mainly in proteins or amino acids. The increase in ammonia concentrations showed that proteins were made soluble and degraded, which confirms, that HC has a permanent effect on the sludge, while the SCOD indicates the level of disintegration, which is important for biodegradability evaluations. Interestingly, the Ecowirl reactor caused an increase of the pH in the bulk solution (data not showed), mainly due to the CO_2 stripping as consequence of high temperature and high turbulence. The increase in pH shifted the equilibrium towards ammonia rather than ammonium that may volatilize. However, further researches are needed to study the fate of nitrogen solubilisation, investigating the nitrogen mineralization and ammonia volatilization.

Absorbed power rating of the pump decreased with the treatment time, starting from an initial value of 1,400 W at the beginning of the HC test to a final value of 1,100 W after 8 h of HC treatment. This result was obtained by decreasing the frequency of the pump inverter in order to keep constant the inlet pressure value. Indeed, increasing the treatment time, a tendency of inlet pressure to increase was observed. This could be explained by a progressive alteration of the rheology of the sludge resulting in a decrease of the viscosity of the sludge during the HC treatment, as consequence of the disruption of cell or microbial flocs.

The increase in HC efficiency with the specific supplied energy found in this study agrees with other works on AC and HC. In particular, working with specific supplied energy lower than 15,000 kJ $kgTS^{-1}$, similar sludge disintegration degree were obtained by using a venturi tube [34], a high-pressure jet device [7] and sonolysis [9,23].

Using AC, Delmas et al. [58] proved that it was possible to achieve greater efficiencies in terms of DD (about from 5% to 25%), but their acoustic cavitation system required much more energy supplied (about from 5,000 to 50,000 kJ $kgTS^{-1}$) than a conventional HC system. This is in accordance with the studies by Le et al. [54], where the authors measured the higher DD (30%) at 75,000 kJ $kgTS^{-1}$. Similarly, Kidak et al. [55] reached the maximum AC efficiency in terms of DD applying a specific powers of about 80,000 kJ $kgTS^{-1}$.

Though the highest specific supplied energy (SE) had the greatest SCOD increase, the energy efficiency (EE) did not improve with the power input. In order to consider both sludge characteristics and lysis efficiency, the EE has been calculated during the 8-hours experiment. From Fig. 13, it can be seen that the higher EE corresponded to the lower SE. This may be related to the fact that the HC sludge treatment might be divided into two stages of disintegration, the first stage requiring lower energy than the second one: in the first stage of HC the structure of sludge flocs would be disintegrated and organic matter contained in the flocs can be dissolved, increasing the SCOD, while in the subsequent stage, cells can be damaged by HC cavitation, realising intracellular organic matter, but requiring a higher energy consumption than the first stage.

3.7 Effect of HC on biodegradability of treated sludge

HC induces shear forces that break down bacterial cell walls and release the intracellular substances into aqueous phase resulting in an increase in the sludge solubilisation. It is thus supposed to occur an improvement of the biodegradability of the treated sludge. In this study, for the first time, respirometric tests were carried out in order to prove the increase in aerobic biodegradability due to the HC treatment. Respirometric tests were performed for HC 8h-experiment. Fig. 14 shows four respirograms obtained from the filtrate of the samples collected at 0, 2, 4 and 8 h, where the sample at t = 0 h denotes the untreated sludge.

As can be seen from Fig. 14, HC treatment leads to an increase in the value of the area under the OUR curve, resulting in a progressive increase of the $SCOD_{bio,}$ with the elapsed time and, thus, with the increase of the specific supplied energy. Results are summarized in Table 2. These results agree with those on SCOD (Table 2). The $SCOD_{bio}$ / SCOD ratio was always in the range of 50 - 75%. Due to the increase in aerobic biodegradability, HC system could applied to excess activated sludge in order to provide an organic carbon source for denitrification process in conventional activated sludge systems.

3.8 Effect of HC on microbial activity

Cell inactivation was evaluated estimating the decay rate of aerobic heterotrophic bacteria. Respirometric tests were carried out on the untreated and treated sludge collected by HC tests after 0, 2, 4 and 8 h. By plotting the OUR curve vs time, two phases could be distinguished in the graphs. The phase 1 was characterized by a rapid OUR decrease that lasts for about 10 hours using the untreated sludge and for more than 20 h using the HC treated sludge, due to the produced biodegradable compounds. The phase 2, that shows for 40 h a true exponential decrease of the endogenous respiration rate, directly followed it.

The decay rate has been estimated as the slope of the exponential interpolation function in the phase 2 (Fig. 15). The quality of the experiments are mostly expressed in terms of R^2 with respect to the fitted data. Thereby it is common practice to consider acceptable R^2 values higher than 0.90.

The obtained value of the untreated sludge ($b_H = 0.31d^{-1}$) was in the wide range reported in other studies for heterotrophic biomass (0.059 – 0.500 d^{-1}) [59]. Treating the activated sludge by HC treatment, the b_H values increased from 0.34 d^{-1} to 0.91 d^{-1} passing from 2 h to 8 h, respectively, thus indicating a decreasing biomass viability. Applying a specific supplied energy of 3,276 kJ $kgTS^{-1}$ (2 h of HC treatment), the b_H was almost equal to that of the untreated sludge, thus showing that low specific supplied energy could only change the floc structure and may release extracellular polymeric substances (EPS), while microorganisms were not destroyed.

On the contrary, at 6,444 and 12,780 kJ $kgTS^{-1}$ (4 h and 8 h, respectively), the respirometric functions of the heterotrophic biomass were compromised, as consequence of dead cells (reduced activity) and/or disrupted cells (cell lysis). Some authors reported that in an AC system working at SE values higher than 26,000 kJ $kgTS^{-1}$, [60], the disruption of microorganisms and the consequent lysis caused an increase in the concentration of soluble COD in sludge and the enhancement of biodegradability.

4. Conclusions

The present study provided interesting results about the effect of HC treatment on sludge disintegration and aerobic biodegradability. The main findings of the investigation can be summarized as follows:

- The higher the temperature of the HC process (up to 35.0 °C), the more efficient the HC treatment, in terms of sludge solubilisation, was.
- Increasing the inlet pressure an acceleration in sludge disintegration was observed. Indeed, higher inlet pressures involved higher flow rate and flow velocity, increasing turbulence level and local pressure oscillations. Further, higher inlet pressures involved higher pressure drops through the HC device, resulting in higher shear forces that break down bacterial cell walls and release the intracellular substances into aqueous phase.
- Comparing different sludge solid concentrations, higher values of ΔSCOD and DD have been observed for the highest TS content tested, 50.0 g L^{-1}.
- In order to maximize the efficiency of HC process, different geometric configurations of the modified swirling jet induced HC reactor (Ecowirl reactor) have been investigated. It was possible to observe that the geometry of the cavitating device can deeply influence the intensity of the cavitation and then the disintegration of the sludge in terms of COD and Nitrogen solubilisation. For Ecowirl reactor configurations in which the injection slots diameters have been decreased, keeping constant both the injection slots number and the inlet pressure values, it was possible to observe an increase in flow rate that caused an increase in intensity of the cavitation. The same result has been observed reducing the injection slots number, keeping constant both the injection slots number and the inlet pressure value. Moreover, an increase in efficiency of the HC process was also achieved combing three Ecowirl heads in series.
- A series of respirometric showed that the sludge pre-treatment by using the HC treatment process might be divided into two stages: sludge flocs were changed and disintegrated firstly, and then the exposed cells were disrupted or damaged. At the first stage, organic matter contained in the flocs was dissolved and SCOD increased. At the second stage, some cells were damaged/disrupted by HC cavitation, requiring a higher energy consumption than the first stage in order to damage the cell membrane and completely disrupt it. The intracellular organic matter was released, which resulted in a further increase of SCOD and a decrease of microbial activity.

Among possible applications, the HC experiments conducted in this study indicate that the excess activated sludge can be available as carbon source for biological processes in WWTP, such as denitrification process. Alternatively, HC could be applied as a pre-treatment for anaerobic digestion, in order to increase the biogas production. However, further studies on the anaerobic biodegradability are needed. The HC treatment could be applied either at high SE as a side-stream treatment or at low SE as an in-line treatment in biological tanks. Moreover, although the effectiveness of Ecowirl reactor has already experimentally been proved in this study, the authors are developing a mathematical model in order to both understand the fluid dynamics inside the modified swirling jet induced HC reactor and optimize the geometry of this device with the main goal of enhancing the HC efficiencies, resulting in a decrease of the energy consumption.

Supporting material

- *SCOD-increase:*

$$\Delta\boldsymbol{SCOD}(\%) = SCOD_{cav} - SCOD_0 \qquad \text{Eq. (1)}$$

$SCOD_0$ – 251.0 mg L^{-1}

$SCOD_{cav}$ – 900.0 mg L^{-1}

$$\Delta\boldsymbol{SCOD}(\%) = 900.0 - 251.0 = 649 \text{ mg L}^{-1}$$

- *Disintegration degree (DD_{PCOD}):*

Reaction volume – 50.0 L

TS content – 50.0 mg L^{-1}

Inlet pressure – 2.0 bar

Temperature – 20.0°C

$$\boldsymbol{DD_{PCOD}}(\%) = \frac{SCOD_{cav} - SCOD_0}{PCOD_0} \times 100 = \frac{SCOD_{cav} - SCOD_0}{TCOD - SCOD_0} \times 100 \qquad \text{Eq. (2)}$$

$SCOD_0$ – 251.0 mg L^{-1}

$SCOD_{cav}$ – 900.0 mg L^{-1}

TCOD – 46,423 mg L^{-1}

$$\boldsymbol{DD_{PCOD}}(\%) = \frac{900.0 \text{ mg L}^{-1} - 251.0 \text{ mg L}^{-1}}{46{,}423 \text{ mg L}^{-1} - 251.0 \text{ mg L}^{-1}} \times 100 = 1.4\%$$

- *Disintegration degree (DD_N):*

$$\boldsymbol{DD_N}(\%) = \frac{NH_4^+ - N_{,cav} - NH_4^+ - N_{,0}}{\text{TKN}_0 - NH_4^+ - N_{,0}} \times 100 \qquad \text{Eq. (3)}$$

NH_4^+-$N_{,0}$ – 26.8 mg L^{-1}

NH_4^+-$N_{,cav}$ – 86.0 mg L^{-1}

TKN_0 – 3,009 mg L^{-1}

$$\boldsymbol{DD_N}(\%) = \frac{86.0 - 26.8}{3{,}009 - 26.8} \times 100 = 2.0\%$$

- *Disintegration degree ($DD_{COD\ NaOH}$):*

$$\boldsymbol{DD_{COD\ NaOH}}(\%) = \frac{SCOD_{cav} - SCOD_0}{SCOD_{NaOH} - SCOD_0} \times 100 \qquad \text{Eq. (4)}$$

$SCOD_0$ – 251.0 mg L^{-1}

$SCOD_{cav}$ – 900.0 mg L^{-1}

$SCOD_{NaOH}$ – 29,350 mg L^{-1}

$$DD_{COD\ NaOH}(\%) = \frac{900.0\ \mathrm{mg\ L^{-1}} - 251.0\ \mathrm{mg\ L^{-1}}}{29{,}350\ \mathrm{mg\ L^{-1}} - 251.0\ \mathrm{mg\ L^{-1}}} \times 100 = 2.2\%$$

- *Specific supplied energy (SE):*

Inlet pressure – 4.0 bar

Temperature – 35.0°C

$$\boldsymbol{SE}\left(\frac{kJ}{kgTS}\right) = \frac{P_{abs} \times t}{V \times TS} \qquad \text{Eq. (5)}$$

TS content – 50.0 g L^{-1}

Absorbed Power rating of pump – 1,137.5 W

Reaction volume – 50.0 L

Treatment time – 120 min (7,200 sec)

$$\boldsymbol{SE}\left(\frac{kJ}{kgTS}\right) = \frac{1{,}137.5\ \times\ 7{,}200}{50.0\ \times\ 50.0} = 3{,}276\ \frac{kJ}{kgTS}$$

- *Energy efficiency (EE):*

Inlet pressure – 4.0 bar

Temperature – 35.0°C

$$\boldsymbol{EE}\left(\frac{mg\Delta SCOD}{kJ}\right) = \frac{V \times \Delta SCOD}{P_{abs} \times t} \times 1000 \qquad \text{Eq. (6)}$$

Reaction volume – 50.0 L

ΔSCOD – 1,475 mg L^{-1}

Absorbed Power rating of pump – 1,137.5 W

Treatment time – 120 min (7,200 sec)

$$\boldsymbol{EE}\left(\frac{mg\Delta SCOD}{kJ}\right) = \frac{50 \times 1{,}475}{1{,}137.5\ \times\ 7{,}200} \times 1000 = 9.0\,\frac{mg\Delta SCOD}{kJ}$$

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Figure Captions

Fig. 1 - Schematic representation of the experimental setup.

Fig. 2 - Solid geometry of Ecowirl reactor.

Fig. 3 - Schematic representation of the respirometric experimental setup.

Fig. 4 - Effect of temperature on DD and SCOD (volume 50.0 L; TS content 50.0 g L^{-1}; initial pH 6.8; inlet pressure 2.0 bar; temperature 20.0 - 25.0 - 30.0 - 35.0 ± 3.0°C). Ecowirl reactor standard configuration.

Fig. 5 - Effect of inlet pressure on DD and SCOD (volume 50.0 L; TS content 50.0 g L^{-1}; initial pH 6.8; temperature 25.0± 3.0°C; inlet pressure 2.0 - 3.0 - 4.0 bar). Ecowirl reactor standard configuration.

Fig. 6 - Variations in pressure in Ecowirl reactor. $DP_{\text{Inlet - Vacuum zone}}$: DP_{2bar}~2.7 bar, DP_{3bar}~ 3.8 bar, DP_{4bar}~ 4.8 bar; $DP_{\text{Vacuum zone – 371 mm downstream}}$: DP_{2bar} ~1.0 bar, DP_{3bar} ~ 1.3 bar, DP_{4bar} ~ 1.5 bar.

Fig. 7 - Effect of TS content on DD and SCOD (volume 50.0 L; inlet pressure 2.0 bar; initial pH 6.8; temperature 25.0°C ± 3.0°C; TS content 7.0 - 12.0 - 23.0 - 50.0 g L^{-1}). Ecowirl reactor standard configuration.

Fig. 8 - Effect of different number of injection slots on DD and SCOD (volume 50.0 L; inlet pressure 4.0 bar; initial pH 6.8; temperature 25.0°C ± 3.0°C; TS content 50.0 g L^{-1}). Ecowirl reactor configuration A, B, and C.

Fig. 9 - Ecowirl reactor with three-heads in series.

Fig. 10 - Effect of Ecowirl heads in series on DD and SCOD (volume 50.0 L; inlet pressure 2.0 bar; initial pH 6.8; temperature 25.0 ± 3.0°C; TS content 50.0 g L^{-1}). Ecowirl reactor standard configuration.

Fig. 11 - Energy efficiency vs Specific supplied energy: (**a**) injection slots number; (**b**) Ecowirl heads number; (**c**) inlet pressure; (**d**) injection slots diameter; (**e**) temperature; (f) TS content.

Fig. 12 - HC 8h-experiment - Effect of applied energy on DD and SCOD (volume 50.0 L; initial pH 6.8; temperature 35.0 ± 1.0°C; TS content 50.0 g L^{-1}; inlet pressure 4.0 bar; 6 injection slots; 3 Ecowirl heads).

Fig. 13 - Energy efficiency vs Specific supplied energy.

Fig. 14 - Respirogram 1: filtered untreated sludge (time 0 h); **Respirogram 2**: filtered treated sludge after 2 h of HC; **Respirogram 3**: filtered treated sludge after 4 h of HC; **Respirogram 4**: filtered treated sludge after 8 h of HC; (volume 50.0 L; initial TS content 50.0 g L^{-1}; initial pH 6.8; inlet pressure 4.0 bar; initial temperature 35.0 ± 1.0°C; 3 Ecowirl heads in series).

Fig. 15 - OUR profiles. **1**: untreated sludge (time 0 h); **2**: treated sludge after 2 h of HC; **3**: treated sludge after 4 h of HC; **4**: treated sludge after 8 h of HC; (volume 50.0 L; initial TS content 50.0 g L^{-1}; initial pH 6.8; inlet pressure 4.0 bar; initial temperature 35.0 ± 1.0°C; 3 Ecowirl heads in series).

Table Captions

Table 1 - Operative parameters, operating conditions and efficiency measured at the end of each test.

Table 2 - HC 8h-optimized test.