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Effect of process parameters on the energy requirement in ultrasonical treatment of waste sludge

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A B S T R A C T

Mechanical treatment methods are used as pre-treatment methods in order to enhance the efficiency of conventional sludge treatment processes and the sludge becomes more suitable for its complete treatment. The ultrasound is an alternative method among other methods, but because of its high energy requirement it should be optimized before utilization. This work gives the optimized parameters such as sonication time, sonication power (these parameters are the two factors which play part for energy calculations), type of sludge, cooling requirements and solid content in the sludge solution. Even if the previous researchers prefer to use the energy (specific energy usually), we have found out that both the sonication time and the sonication power have individual importance. For municipal sludge the main conclusion can be summarized as: "high power-short retention time" is more effective than "low power-long retention time". As this phenomenon may alter from sludge to sludge, various combinations of power and retention time should be tried while keeping the volume small and the concentration below a certain level. The process should be performed at moderate temperatures and the efficiency increases if the sludge is as homogeneous as possible.

1. Introduction

The sludge produced by wastewater treatment plants is currently being disposed off either by agricultural spreading or by incineration. However, as the environmental legislation is stricter now, development of alternative technologies must be emerged to reduce the very huge amount of sludge produced all over the world. Among these alternative technologies mechanical treatment of sludge plays an important role because it favours solubilization of particulate matters in liquid phase. Once the organics are solubilized, the sludge becomes more convenient for further treatment. The mechanical treatment methods which are used as pre-treatment techniques include high pressure homogenization [1], centrifugation [2] and sonication [3–6].

By these methods, the aim is to increase the availability of organic matters in liquid phase by disrupting the flocs and/or lysing the bacterial cells before recycling the liquid portion to the biological treatment unit and the solid portion to the sludge treatment. Among mechanical treatments, sonication leads to encouraging results but requires further optimization. Sonication is an innovative technique for organic matter disintegration which has been explained by various researchers [7–9].

When the system is made of a solid–liquid biphasic medium, catalysis is a consequence of the disruption of the solid by the jetting phenomenon associated with the collapse of cavitation bubbles. It is important to note that many of such effects are observed when the heterogeneous medium is irradiated with low frequency, or power ultrasound at the 20–100 kHz range [9].

There were some researchers who have worked on the ultrasonic sludge disintegration. For example, Gronroos et al. [10] have studied the increase in the soluble chemical oxygen demand (SCOD) in sewage sludge treatment and they have found out that ultrasonic power, dry solid content of sludge, sludge temperature and ultrasonic treatment time affect significantly on the sludge disintegration. Chu et al. [11] have discussed possible mechanisms of ultrasonic waste sludge treatment. They pointed out that the ultrasonic treatment consisted of several stages. At the first stage of sonication, the porous flocs could be readily deteriorated into compact flocculi, while the dewaterability of sludge was markedly deteriorated. In the second stage, although the floc size had remained almost unchanged, the sludge was disinfected and the SCOD value increased accompanied with the reduction in the microbial density levels [11].

High-power ultrasound (200 W) is generally performed at low frequencies (20 kHz) in order to get an effective sludge disruption. At low frequencies, the implosions of stable cavitation bubbles create high energy waves and micro-jetting effects together with the inside-bubble pyrolysis phenomenon, which explains the slight reduction in the mass of the dry solids. This complex matrix

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medium provides release of intracellular materials into the bulk liquid [12]. On the other hand, sonication is a high energy process and its efficient usage is very important by means of cost-effectiveness. There have been also some researchers who have tried to optimize the sonification process and improve the disintegration rate for sludge disruption [13–16].

In this study, we have investigated the pre-treatment of two different types of sludge: municipal and industrial. The following operational parameters were deeply studied from the energy consumption point of view: the ultrasonic power, exposure time, solid concentration, medium temperature and volume. The objective of this work is to investigate the effects of these parameters on the energy consumption during ultrasonic waste sludge treatment.

2. Materials and methods

Considering from an environmental point of view, the wastewater treatment plants can be considered in two groups: municipal and industrial. The municipal wastewater treatment plant receives wastewaters only from human activities and the industrial wastewater treatment plants accept wastewaters only from industrial processes and the sludge produced by two different plants have different characteristics (both physical and chemical). Therefore in this study, two representative sludge samples were examined thoroughly.

2.1. Materials

Industrial sludge, which we named STVULBAS2007 had been collected after the biological treatment unit of an industrial wastewater treatment plant in St Vulbas, France. The municipal sludge, named GINESTOUS2007, had been collected in Ginestous wastewater treatment plant (capacity 565,000 inhabitants). A probe type sonicator (Vibracell) was used as an ultrasonic homogenizer (Fig. 1). This apparatus works with an operating frequency of 20 kHz and a maximum applied power of ca. 200 W. Batch experiments were carried out in glass reactors of 0.3 and 0.5 L with temperature regulation (cooling). In some experiments the temperature was not regulated in order to see the effect of cooling on the obtained results. The solutions of domestic and industrial sludge with concentrations of 4–24 g/L were subjected to ultrasonic irradiation. The total duration of irradiation was 60–90 min.

2.2. Analysis methods

Several parameters were measured including solubilized chemical oxygen demand (COD) (Hach spectrophotometric method), solubilized total organic carbon (TOC) (Shimadzu TOC analyser), dp_{50} (average particle size diameter) using a Malvern Laser Diffraction Particle Size Analyzer (model 2000, Malvern Instruments Ltd.). The particle size analysis enables us to evaluate the reduction in the mean particle diameter and the change in the percentile volume of the particles. The TOC analysis is beneficial to explore the transfer of organic matters from the solid to the liquid phase and also we are able to observe the reduction of solid phase TOC. COD is a global parameter which indicates the required oxygen concentration for the oxidation of all oxidizable materials by a certain oxidant in the presence of a catalyst. The change in the soluble COD indirectly shows us the quantity of organic materials which have been transferred into the liquid phase of the solution from the solid phase.

2.3. Experimental methods

Sludge was sampled in gross amount to ensure that all the experiments could be performed with solutions prepared by the similar initial sludge characteristics. As the characteristics of the incoming wastewater to the treatment plant vary from time to time, the characteristics of the resulting sludge change drastically. For this reason, large amount of stock sludge was collected and by this way the samples which were used throughout the study did not show large differences among them.

The industrial sludge had an initial concentration of suspended matter of about 10 g/L. After a homogeneous mixing of the collected solids, each sample has been divided into pill-boxes of 5 g.

The municipal sludge was collected in concentrated form a wastewater treatment plant and it has been transferred directly into pill-boxes in 10 g lots, after manual mixing for a rough homogenization. Both of the sludge types were preserved in a freezer. This preliminary stocking step may change some physical characteristics of the sludge but it must be mentioned that performing experiments by using either non-frozen sludge or defrosted sludge did not have significant effects on the COD results. For example, after 60 min at 25 W sonication of defrosted sample, the final COD was 345 mg/L, whereas for the non-frozen sample, it was 329 mg/L. As the sludge does not have a homogeneous texture, this difference is within the acceptable limits.

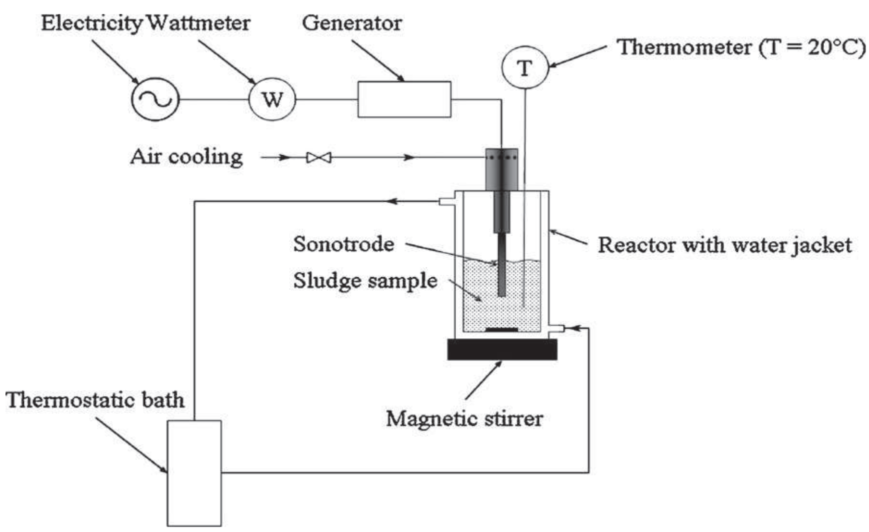

Fig. 1. Ultrasonic set-up.

Table 1
Dry solid content (DS) and total organic carbon (TOC) concentration determined after drying of sludge.

Sludge	STVULBAS2007	GINESTOUS2007
DS of raw sludge (% of mass)	12.1	24.3
TOC (% of DS)	38.5	43.1

The inlet concentrations for the sonication studies were always determined after the defrosting of the sludge and initial time ($t=0$) was always considered as the time when the sludge sample was totally defrosted. After defrosting, required amount of sludge was dispersed into 300 or 500 mL of distilled water.

The concentration was selected between 4 and 24 g dry solids/L. The ultrasonic power varied among 50,100 and 200 W. All the experiments were controlled by a blank study and were performed two times to avoid any experimental errors. Samples were withdrawn to investigate kinetics during the treatment.

The pre-treated solutions of sludge were immediately placed in 500 mL plastic bottles and frozen for their further oxidative treatment.

The power dissipated was about 80% of the electrical value. This parameter was measured by the calorimetric method which has been accepted as a standard method for the ultrasonic energy measurements [17].

3. Results

The ultrasonic pre-treatment implies mainly the physical changes on the sludge samples and it helps while breaking the bacterial cells, to solubilize the organic matters from the solid phase to the liquid phase of the sludge solution. It is an effective method but as was mentioned by the previous researchers, it is an expensive method comparing to other mechanical pre-treatment methods so system should be fully optimized [18].

The type of sludge has an important effect on sonication, because of their physical differences. The municipal sludge which was collected from a wastewater treatment plant in Ginestous (France) was composed of the sediments from the primary settling tank together with the waste activated sludge from the secondary settling tank. As it contains the non-bacterial solids, we have encountered a grand quantity of fiber particles. These fiber particles made it more difficult to filter comparing to the industrial sludge both before and after sonication.

On the other hand the industrial sludge has a more homogeneous structure as the industrial wastewater plant does not receive any wastewater other than the quantity produced in production.

Table 1 shows the dry solid content (DS, determined after an overnight drying in oven at 105 °C) and the total organic carbon (TOC) in the dried sludge. It may be noted that the carbon contents measured in municipal and industrial sludge were 43.1% and 38.5%, respectively.

3.1. Particle size distribution

Regarding the two sludge solutions, GINESTOUS2007 and STVULBAS2007, which initially have the same total solid concentrations, the municipal sludge has initially larger particles. In the municipal sludge the initial size varies in a larger range as there are big fiber particles found in its composition.

In both of the sludge types, the particle size has gradually changed during 90 min sonication. For example after sonication at 100 W, the average diameter decreased down to 70 μm from an initial of 1000 μm. The same tendency was valid for the industrial sludge (275–4.5 μm). This reduction in the particle size imposes some solubilization of solid material. The difference between the mass of dry solids in the raw sludge and that of the pre-treated sample is around 10%. The effect of power and the type of sludge are not very significant on the solubilization amount.

3.2. Solubilization of organic materials

When we consider the increase of organic matters in the liquid phase, the COD measurements show us that sonication provides a transfer of the organics to the liquid phase of the solution. When the sludge was mechanically stirred to obtain a homogeneous solution, the COD was increased until 65 mg/L, but then it stayed constant.

In Fig. 2, it was obvious that the elevated applied ultrasonic power (from 50 to 200 W) has a positive effect for better solubilization of organics that contribute to COD measurements.

The COD analysis does not let us measure the COD in the solids directly. A hydrolysis step is required to measure the COD in the solids. Eliminating this hydrolysis step, we have performed TOC analysis, by which the TOC both in the liquid phase and the solid phase can be measured, to calculate the total conversion (oxidation) of organic matters. As may be concluded from Fig. 3, the total TOC concentration stays almost constant, while the organics only change their state. The increase in the TOC of the liquid phase is in agreement with our results obtained from the COD analysis. Accordingly, at 200 W, while for the municipal sludge the organics are solubilized up to 29% of their total value in the solution (from TOC_l = 233 to 792 mg/L), it is only 12% for the industrial sludge (from TOC_l = 137 to 338 mg/L).

To compare our samples with the scientific research done by other researchers before, the DD_{COD} (degree of disintegration based

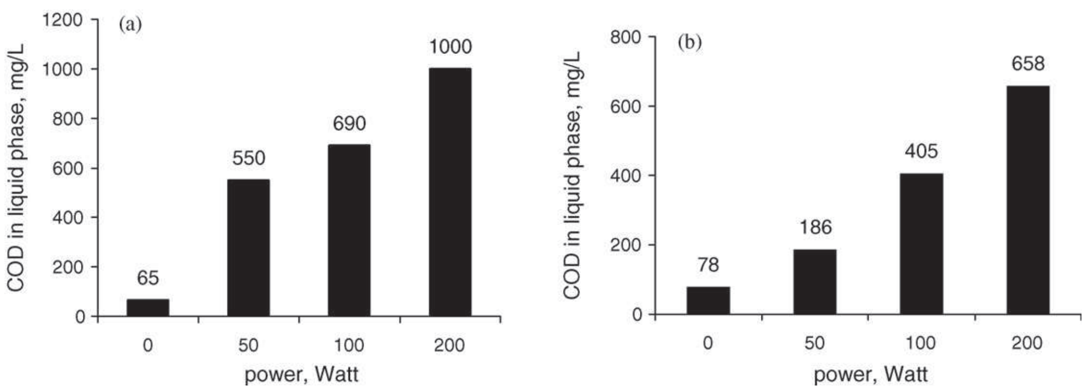

Fig. 2. Evolution of COD in liquid phase of (a) municipal sludge (GINETOUS2007) solution and (b) industrial sludge (STVULBAS2007) solution. The process time was 10 min, and the concentration was 8 g/L.

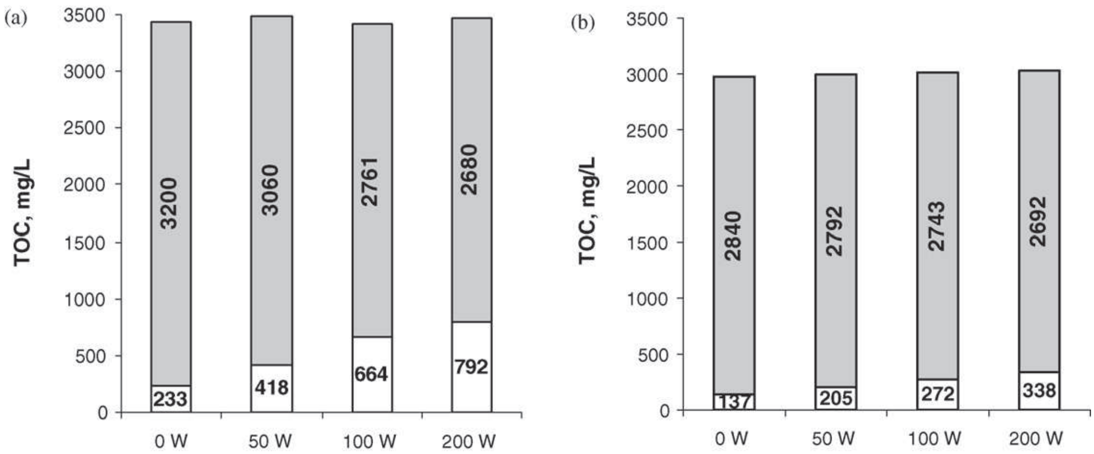

Fig. 3. Change of TOC in (a) municipal sludge and (b) industrial sludge. (■: TOC in the solid phase, □: TOC in the liquid phase) versus ultrasonic power. The process time was 90 min, and the concentration was 8 g/L.

on COD) was calculated for our samples [19]:

$$DD_{COD} = \frac{COD_t - COD_0}{COD_{NaOH} - COD_0} \times 100\% \quad (1)$$

where DD_{COD}: degree of disintegration based on COD (%); COD_t: COD soluble at time t (g O_2/L); COD_0: COD soluble at time zero (g O_2/L); COD_{NaOH}: COD soluble in NaOH solution: total estimated disintegration (g O_2/L).

The COD_{NaOH} is hypothetically accepted as the maximum possible soluble COD concentration and the procedure to obtain this measure is given elsewhere [20]. To be able to find out the COD_{NaOH}, the sludge must be kept in highly concentrated NaOH solution for 24 h under continuous stirring. After disintegration experiments at various ultrasonic powers, according to the graph (Fig. 4), it was found that in 60 min we have been able to reach 56%, 64% and 80% DD_{COD} by applying 50, 100 and 200 W of sonication, respectively.

Even if the DD_{COD} is not able to tell the total solubilization of organics, it is helpful to show the percentage of solubilization which the actual practices are capable of. To have better understanding of this phenomenon, it must be kept in mind that the TOC analysis shows all the organic carbon in the solution (solid and liquid). On the other hand, performing the COD analysis, we need to apply a pre-digestion (hydrolysis) method in order to determine the total COD of the solution. Somehow, this hydrolysis step does not allow the solubilization of all the solid particles and also there are some refractory organics which are not oxidized by the oxidizing agent used in the COD analysis. The main issue about using COD tests is that; even if the COD tests may show very high solubilization, due to the gaps in this analysis method, the TOC tests give more accurate results.

Apart from the differences in the measurement techniques, COD may be useful because in real applications the solubilization techniques are also limited by the same reasons as in the analysis method. So the COD results may be used as indicators for exploring the solubilization process, if TOC cannot be measured.

3.3. *Investigation of DD_{COD} versus specific energy*

As it is well know that the reaction time and the applied sonic power are the major parameters influencing the process, the scientists have defined an energy parameter which is called the specific energy and it is obtained by dividing the energy given into the heterogeneous system by the mass of total dry solids initially present [21]:

$$E_s\,(\text{kJ/kg}) = \text{power}\,(\text{W}) \times \text{time}\,(\text{s}) \times \frac{1\,\text{kJ}/1000\,\text{J}}{\text{mass of dry solid (kg)}} \quad (2)$$

It is possible to have identical E_s values by changing each of the physical measures. To investigate these phenomena in more detail, we performed various experiments by the following ways:

- Keeping the time and the mass of dry solid constant, changing the power.

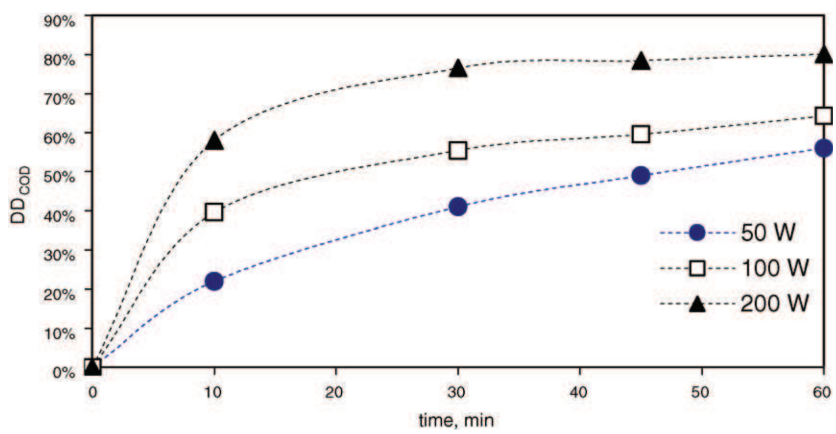

Fig. 4. Evolution of DD_{COD} (degree of disintegration on the basis of COD) in liquid phase of municipal sludge solution, with respect to exposure time. The concentration was 8 g/L at 20 °C.

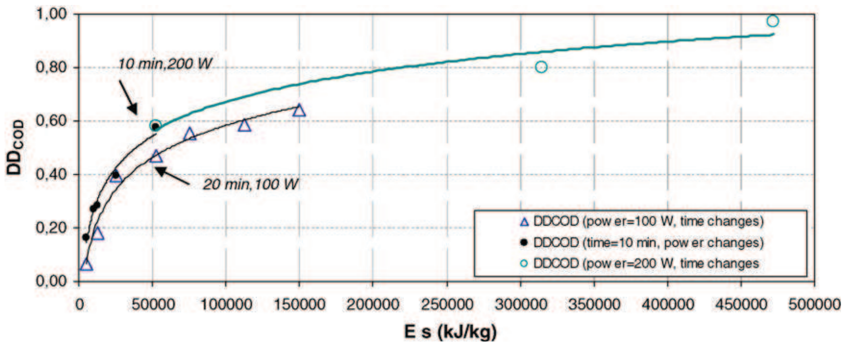

Fig. 5. Evolution of DD_{COD} (degree of disintegration on the basis of COD) in liquid phase of municipal sludge solution, with respect to specific energy. The specific energy was calculated by two different ways. (1) By keeping the ultrasonic power constant at 100 W and exposing it at different durations. (2) By application of different powers at constant process times. The dark coloured curve is DD_{COD} achieved at extreme conditions (200 W and high temperature, 70 °C) of the system. The concentration was 8 g/L.

- Keeping the power and the mass of dry solid constant, changing the time.
- Keeping the power constant, changing the time and the mass of the dry solids.

In Fig. 5, the degree of disintegration was plotted against the E_S values which were obtained under different process conditions. Even if the specific energies are the same, the results of DD_{COD} are not the same for different power and time combinations. Some small displacement of the curve was observed.

These results lead us to conclude that: below 200 W, treating the sludge at lower powers for long process times, is more effective than treating it at high-power ultrasound during short period of times. For example if we consider Table 2, for lower powers (<200 W), it is effective to keep the solution under irradiation for comparatively long times but at 200 W, which was the maximum power limit of the ultrasonic generator, 10 min at 200 W is more effective than 20 min at 100 W and also 20 min at 200 W is more effective than 40 min at 100 W. So, the power of ultrasound is the major parameter in disintegration of waste activated sludge. The practical results should always be reported with their parameters in detail.

To investigate the limits of the apparatus, the system was operated at its maximum (200 W) and at the end of 90 min, nearly 97% DD_{COD} of was achievable.

Another curve for the STVULBAS2007 sludge has been plotted in Fig. 6 and the maximum power (200 W) results were compared with other powers. Looking at Fig. 6, when we consider one common point between two curves (E_s = 225,000 kg/kJ), we observe that the DD_{COD} is higher when we operate at 100 W for 90 min, instead of at 200 W for 45 min.

Both from Figs. 5 and 6 it can be concluded that for municipal sludge “high power-short retention time” combination is more effective than “low power-long retention time” combination. But on the contrary for industrial sludge this phenomenon is valid on the vice versa way. This difference comes from the difference between two types of sludge. The municipal sludge contains particles which are resistant to ultrasonic disruption (the fibrous particles coming from toilet papers), so increase of power (stronger micro-jets) is required to break these particles. On the other hand, industrial sludge is composed mainly of the settled bacteria which are broken to soluble materials even at lower powers, so as the retention time increases more solubilization can be achieved.

3.4. Effect of solid concentration

In the experiments, the improvement of degree of disintegration was observed by augmentation in the concentration of total solids until 12 g/L. This phenomenon could be explained by the statistical manners: when the solids are more in the vicinity of the sonic probe, they are better disintegrated, because the tip of the probe forms an active zone just around it. Even more, the effect of particle crush is simultaneously more possible because they can find the other particles more easily around. On the other hand, when the total solids content is increased beyond a certain point, this effect turns the opposite. The ultrasonic waves cannot be evenly distributed into the solution and the degree of disintegration of sludge is severely diminished. For example in Fig. 7, 24 g/L curve is lower than the 12 g/L curve.

When the statistical curve fitting was applied, the following equation obeyed with very high regression coefficient at all the solid concentrations:

$$DD_{COD} = DD_{COD\text{-}max}(1 - \exp[-SC \times 10^{-5} \times 0.33 \times E_S]) \quad (3)$$

where DD_{COD}: degree of disintegration (%); $DD_{COD\text{-}max}$: maximum achievable degree of disintegration in the current conditions (%); SC: solid concentration (g/L); E_S: specific energy (kJ/kg).

3.5. Effect of volume

When working for the disintegration of GINESTOUS2007 sludge, two solutions with the same concentration were exposed to sonication in two different volumes. Even if the difference between the two volumes is not very big, there is a slight decrease of disintegration at high volume. The main reason for this effect may be the difficulties in providing homogeneous agitation given both by magnetic stirring and the sonication waves. When the volume gets larger, the solution cannot be mixed as effectively as in a smaller volume and just as in the case of solid concentration the particles cannot move to the most active sonication zone which is just at the tip of the sonication probe (Fig. 8).

Table 2
Evolution of degree of disintegration versus specific energy. The same specific energies were obtained by changing the sonication time or the applied electric power.

Concentration of solids (mg/L)	8	8	8	8	8	8	8	8	8	8
Time (min)	2	10	5	10	15	30	20	10	40	20
Power (W)	100	20	100	50	100	50	100	200	100	200
E_s (kJ/kg)	5000		12,500		37,500		50,000		100,000	
DD_{COD}	7.00%	16.40%	18.00%	28.20%	38.20%	39.10%	47.00%	57.90%	52.30%	71.20%

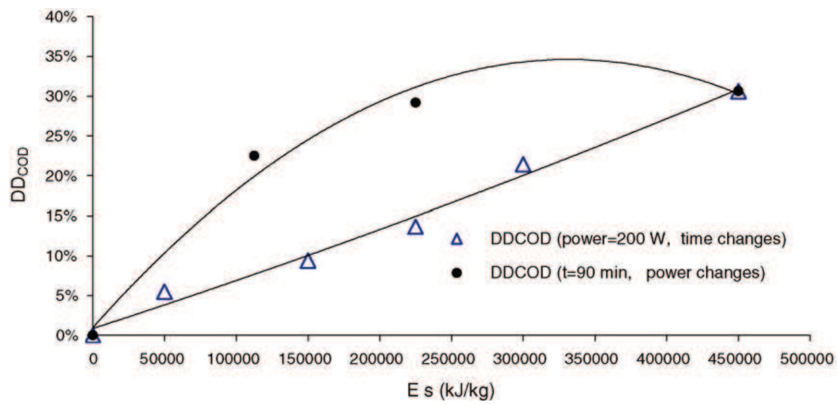

Fig. 6. Evolution of DD_{COD} in liquid phase of industrial sludge solution, with respect to specific energy. The specific energy was calculated by two different ways. (1) By keeping the ultrasonic power constant at 200 W and exposing it at different durations. (2) By application of different powers at constant process time (90 min). The concentration was 8 g/L.

3.6. Effect of cooling

All the experiments were run in a double envelope reactor which allows us to keep solutions at 20 °C. In a power ultrasound system the temperature reaches high values, if not cooled down. When we stop the cooling liquid that runs around the reactor, the system heats up to 70 °C at the end of 90 min. The DD_{COD} increases to 100% in 60 min.

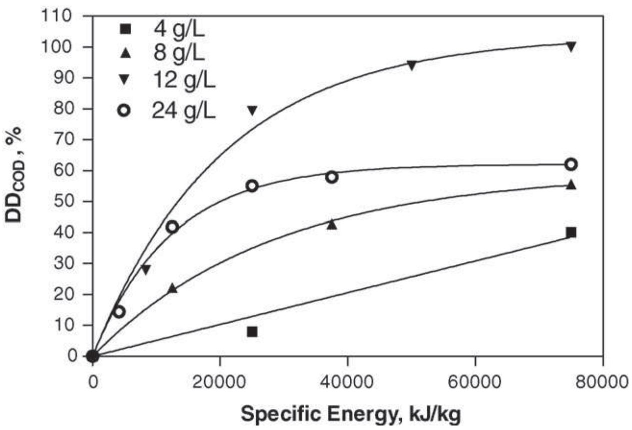

Fig. 7. Evolution of DD_{COD} in liquid phase of municipal sludge solution, with respect to total solid content. The applied power was 50 W.

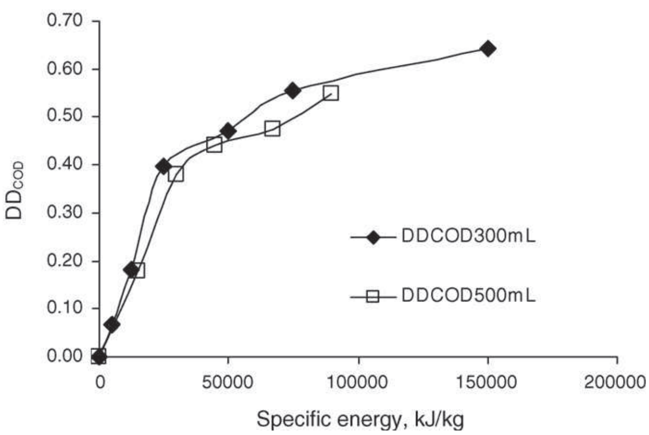

Fig. 8. Effect of solution volume on the degree of disintegration of GINESTOUS2007 sludge. The sludge concentration was 8 g/L and the applied power was 200 W.

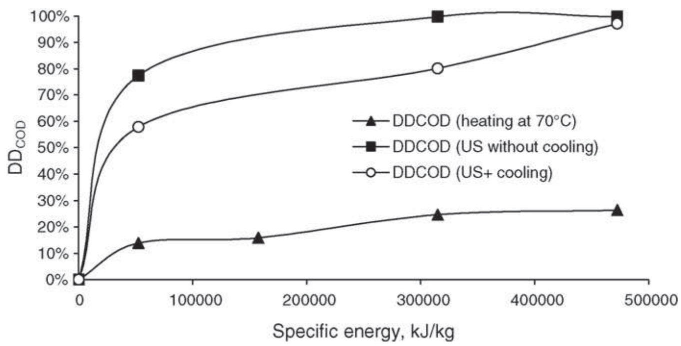

Fig. 9. Evolution of DD_{COD} in liquid phase of municipal sludge solution, with respect to specific energy, under different conditions of temperature control. The concentration was 8 g/L.

The curve obtained without cooling is better than the curve with cooling. To investigate the participation of heat, the sludge was exposed to heating at 70 °C for 90 min and the DD_{COD} was calculated. Fig. 9 clearly declares that the upper curve is a sum of the effect of ultrasonic process and the effect of heating process. What we can suggest is that for any scale up operation the process can (must) be performed without cooling and by the way the expenses for cooling will simultaneously be avoided. On the other hand very high temperatures my damage the mechanical equipments so extreme heating should be avoided.

4. Conclusion

Ultrasonic disintegration is an alternative for mechanical treatment of waste activated sludge. Even though it is a user-friendly technique and shortens the conventional long retention times, as it requires high energy usage, the operating parameters need to be optimized in order to get cost-efficient results. Low frequency ultrasound (20–100 kHz) should be used in this process because at this frequency the cavitation bubbles act like mechanical shredders by the formation of waters jets in the solution. Unlike the high frequencies, the free-radicals do not play any role for chemical oxidation. The ultrasonic power is the key parameter in sludge disintegration. Higher the power, higher is the efficiency of the process. When the particle size is considered, up to 100 W, the particles are divided into the smallest diameter which can be reached by ultrasonic disintegration and beyond this power no further decrease can be obtained. Besides, after the first 30 min the average particle size stays constant. On the other hand, the sludge type is very important to decide about the operational parameters. Even if the calculated energy consumption is equal, for heterogeneous sludge with resistant particles (for municipal sludge) “high power-short retention time” is more effective than “low power-long retention time”. For more homogeneous sludge types (industrial type) retention time should be kept longer but at lower powers. The degree of disintegration showed that at high specific energies (150,000 kJ/kg and higher), the sludge was disintegrated effectively (50–60% of DD_{COD}); while at lower powers the percentage of disintegration stays low. Another important aspect is the concentration of the sludge solution: up to a certain concentration the degree of disintegration increases with the increasing concentration, but in our case after “12 g/L” this effect turns the contrary. The volume of the solution should be kept as small as possible, depending on the affective volume of ultrasonic probe in order to keep the solution in homogeneous turbulence while the ultrasound is on. The heat which is produced in the system is an extra contribution for disintegration purposes, so we suggest that the system should be cooled down to the highest temperature which allows the effective usage of the mechanical equipments, avoiding very high temperatures at which they could be damaged.

Acknowledgment

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