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Effect of sewage sludge sonication parameters on the content of organic compounds in the supernatant liquid

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ABSTRACT

Propagation of ultrasound waves is one of the advanced methods of oxidation. Its disintegrating effect makes it possible to increase the biodegradability of the sonicated substrates. In the case of sewage sludge, it can accelerate the hydrolytic phase and thus shorten the methane fermentation process and increase the efficiency of biogas production. The aim of the study was to analyze the effect of industrial sewage sludge preparation with the ultrasonic field in which the disintegration potential of ultrasonic waves was determined based on the dependence of dissolved organic carbon and total organic carbon on the acoustic energy supplied. The following combinations based on variable amplitudes (A_1 = 24.4 μm, A_2 = 36.6 μm, A_3 = 48.8 μm, and A_4 = 61.0 μm) and sonication times (t_S = 180; 360; 540; 720; 900; 1,080; and 1,260 s) were used in the study, which corresponded to a wide range of the supplied acoustic energy (E), from 7 to 170 kJ. The dominant effect of the highest amplitude of A = 61.0 μm was observed. The layout of the measurement points for this series allowed for the determination of the optimum sonication time of 900 s. A further increase in the sonication time leads to an increase in the preparation energy, whereas no increase is observed in the generated concentrations of organic and total carbon. It should be emphasized that the determined values of the ratio of total carbon to acoustic energy were the highest for the highest amplitude of A = 61.0 μm. The other amplitudes do not have such a strong effect. For the amplitude of A = 48.8 μm, the experimentally determined best sonication time is 1,080 s. For the other two amplitudes, this time is 1,260 s. Increasing the ultrasound energy (through increasing the amplitude of vibrations and sonication times) causes a cyclic rise in concentrations of dissolved forms of carbon and nitrogen, which corresponds to an increase in the degree of disintegration of prepared substrates.

Keywords: Ultrasounds; Total dissolved carbon and organic carbon; Total dissolved nitrogen

1. Introduction

The low-frequency ultrasonic field (10–60 kHz) has been used in many areas for many years, it has great potential in wastewater treatment and sewage sludge processing technologies. As a result of ultrasonic wave propagation, sewage sludge breaks down, a series of mechanical and chemical phenomena occur. Ultrasonic disintegration is used to break down the floc structure and lys microorganisms. The cellular material released into the solution is subjected to further transformations during chemical reactions with H and OH radicals produced by ultrasonic wave propagation [1,2]. The effects of ultrasonic disintegration of the floc structure and lysis of microorganisms (sewage sludge) are generally expressed by transferring organic matter into dissolved form and referred to as the degree of disintegration (DD). Obtaining a high degree of distribution above 80% is possible by using high specific energy, E_S = 470,000 kJ/kg TS [3,4]. The use of excessive sludge sonication prior to fermentation can increase biogas production and increase the reduction of organic matter [5–8]. Sonication can also contribute to the minimization of the excess sludge by oxidation

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of disintegrated mass of microorganisms in the process of activated sludge [9,10]. The primary effect of low energy sonication is destruction of sludge floc structure and size reduction of biological agglomerates [11–13]. After ultrasonic decomposition of flocs, re-flocculation occurs, and this results in the formation of sludge agglomerates with different characteristics, including altered settleability [14,15]. Sonication can be also used as a method for sludge conditioning prior to dewatering. At low input energy, it is possible to improve the ability of sludge to release water from the floc structure. However, this requires careful selection of the sonication parameters [7]. The potential of low-energy sonication is also the subject of research on the intensification of metabolic processes of activated sludge microorganisms. It was observed that low power sonication is the right tool to stimulate growth and respiratory activity or dehydrogenase activity [16,17].

The aim of the study was to analyze the effect of industrial sewage sludge preparation with the ultrasonic field in which the disintegration potential of ultrasonic waves was determined based on the dependence of dissolved organic carbon (DsOC) and total organic carbon on the acoustic energy supplied.

2. Experimental part

2.1. Substrate

Excess sludge obtained from the sewage treatment plant in a company from the dairy industry was used in the study. Samples were taken directly from the pressure pipe draining excess sludge from the secondary settling tank. The company's sewage treatment plant is a facility operating under high substrate load, with the resulting excess sludge being dewatered and used for agricultural and natural purposes. No technological system is used in the plant to process sludge (anaerobic stabilization, oxygen stabilization). Only post-production wastewater and municipal wastewater from the plant site flow into the treatment plant. Characterization of industrial sludge is shown in Table 1.

2.2. Methodology

2.2.1. Ultrasonic treatment

A Sonics VC750 ultrasound processor was used for sludge sonication, generating the ultrasound field with the frequency of vibration of f_{UD} = 20 kHz. Power in the device was adjusted digitally as a percentage of maximal sonotrode amplitude. The maximum amplitude of the ultrasound wave was determined by the geometry of the sonotrode used. A 19 mm diameter sonotrode with a maximum amplitude of 61 μm was used in the study. The use of the Sonics disintegrator allowed for monitoring of the final value of the acoustic energy supplied to the sample *(E)* expressed in Joules at given values of amplitude and propagation time of ultrasound wave. Four vibration amplitudes were used in the study for sludge sonication: A_1 = 24.4 μm (40% of maximum sonotrode power), A_2 = 36.6 μm (60%), A_3 = 48.8 μm (80%), and A_4 = 61.0 μm (100%). The times of ultrasound wave propagation (seven preset times of ultrasound wave propagation at 3 min intervals) for each of the preset amplitudes were t_S = 180; 360; 540; 720; 900; 1,080; and 1,260 s. The volume of samples of the sonicated substrates was always constant and amounted to V_0 = 1,000 cm^3 in a vessel with a diameter of S = 78.5 cm^2 at room temperature. The maximum specific energy supplied to the sludge did not exceed 7,200 kJ/kg TS. Specific energy (E_S) was calculated from Eq. (1):

$$E_S = \frac{E}{V \cdot \mathrm{TS}}, \mathrm{kJ/kgTS} \tag{1}$$

$$E = P \cdot t_S, J \tag{2}$$

where *E*-amount of acoustic energy in Joules (watts seconds) supplied to the probe (energy monitor of ultrasonic processor) (J); *P*, acoustic power (W); t_S, sonication time (s); TS, total solids in WAS (waste activated sludge) samples (kg/m^3); *V*, sample volume (m^3).

The size of the applied amplitude of ultrasound wave vibrations and the time of its propagation constituted the value of acoustic energy *E* supplied and were the basis for calculating the remaining operational parameters of the sonication process. Changes in the value of acoustic energy are presented in the chart below as a dependence on the applied sonication times t_S = 180; 360; 540; 720; 900; 1,080; and 1,260 s and vibration amplitudes of A_1 = 24.4 μm, A_2 = 36.6 μm, A_3 = 48.8 μm, and A_4 = 61.0 μm. Such a range of sonication process variables caused that the acoustic energy supplied to the sludge sample was different and characteristic for the appropriate combination of the amplitude *(A)* and sonication time (t_S) (Fig. 1.).

Table 1
Selected physicochemical and technological properties of the collected sludge

Index	Mean ± SD
Dry matter (d.m.)	23.97 ± 6.71 g/L
Dry organic matter	16.78 ± 4.81 g/L
Concentration of total carbon in a sample of sludge	8.05 ± 1.74 g C/L
Concentration of total nitrogen in the sample of sludge	1.84 ± 0.59 g N/L
Concentration of total dissolved carbonTDC	32.53 ± 11.3 mg C/L
Concentration of dissolved organic carbonDsOC	21.73 ± 7.63 mg C/L
Concentration of total dissolved nitrogenTDN	12.63 ± 4.23 mg N/L

SD, standard deviation.

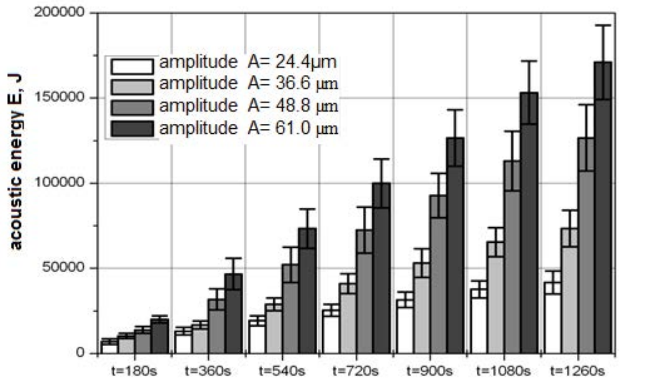

Fig. 1. Changes in acoustic energy as a function of sonication time and ultrasound wave amplitude.

The quantity that links the characteristics of the sludge with the energy characteristics of the ultrasound wave is the sonication energy (E_S). The obtained values E_S results from the amount of acoustic energy and mass concentration of the sludge studied (Fig. 2).

2.2.2. Physicochemical analyses

Analytical determinations used in the research were determined according to the Standard Methods [18] carried out based on the following methods and devices:

- Determination of the loss on ignition for a dry matter of the sludge according to EN 12,879,
- Determination of concentrations of total dissolved nitrogen (TDN), total carbon, organic and inorganic carbon using the TOC Analytik Jena multi N/C analyzer (Analytik Jena Multi N/C 2100-Germany). The dissolved fraction was obtained by separation of suspensions and colloids contained in the supernatant liquid using the centrifugal force field (centrifuging time: 15 min, coefficient of separation: 10,000 rcf), followed by filtration through a 0.45 µm pore diameter filter (membrane–cellulose acetate).

All the physicochemical properties of samples were conducted in triplicate and the mean values with standard deviation (SD) are presented.

3. Results and discussion

Preliminary experiments concerning substrate sonication and preparation effects were conducted based on the dependence of total carbon and total organic carbon concentrations vs. acoustic energy supplied into the sonicated sample (Figs. 3 and 4). It is remarkable that increasing the sonication power led to a cyclical increase in the concentrations of the indices studied. With extreme times and amplitudes of the propagated ultrasonic wave, the concentrations of total dissolved carbon (TDC) ranged from TDC = 52.4 mg C/L (A_1 = 24.4 µm, t_S = 180 s, $E \approx 7$ kJ) to TDC = 1,577.0 mg C/L (A_4 = 61.0 µm, t_S = 1,260 s, $E \approx 171$ kJ). Similarly, high values were recorded for concentrations of

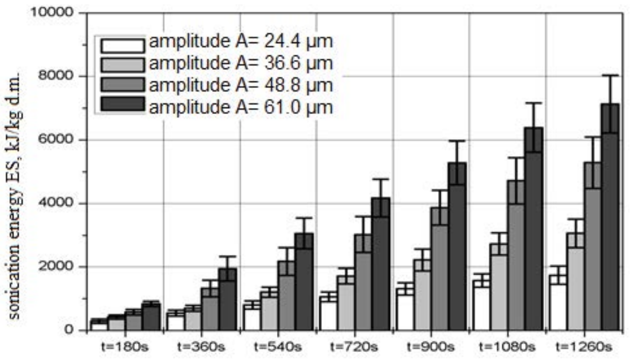

Fig. 2. Dependence of sonication energy on the dry mass of the sonicated sludge.

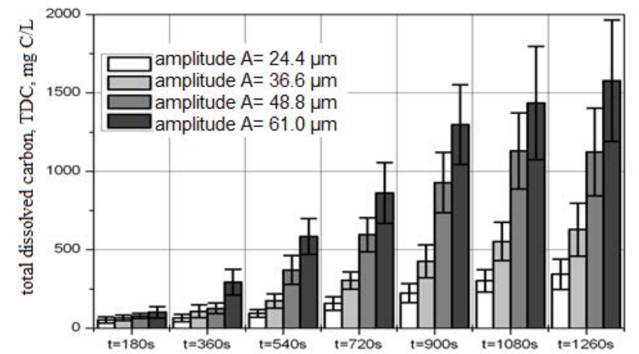

Fig. 3. Changes in the concentration of total dissolved carbonTDC in the sludge liquor of the sonicated substrates vs. vibration amplitude and sonication times.

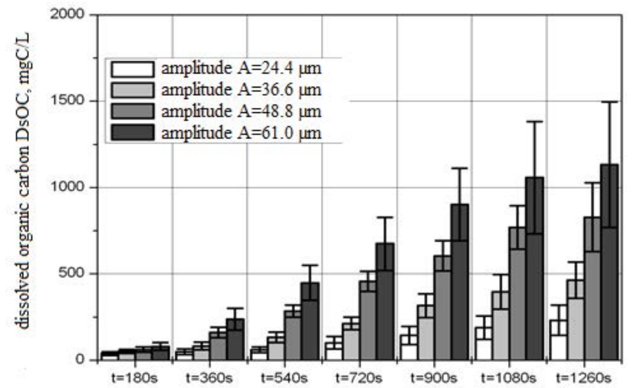

Fig. 4. Changes in the concentration of dissolved organic carbon DsOCin the sludge liquor of the sonicated substrates vs. vibration amplitude and sonication times.

DsOC in the range of from DsOC = 38.4 to 1,131.0 mg C/L. Therefore, it is clear that the process of sludge sonication is a disintegration process causing the breakdown and decomposition of the structure of sludge aggregates, tearing of cell walls and the release of intracellular material into the sludge liquor. It should be noted that the concentration of total carbon and especially the concentration of organic carbon in sludge liquor should be considered as a source of an easily available substrate in biological processes, thus providing significant support for the efficiency of the methane fermentation process, especially the hydrolytic phase of this process. Usually, changes in the chemical properties of sewage sludge after treatment with ultrasonic field are expressed by chemical oxygen demand (COD) in the sludge supernatant. The higher the soluble chemical oxygen demand (SCOD) value is, the higher the release degree of organic matter is, the more thoroughly the sludge floc and cell structure are destroyed [19]. As reported by Deng et al. [20] the dissolved COD, NH_3–N, NO_3–N, and other substances in the leachate have changed after ultrasonic treatment. This indicates that due to the sonochemical properties of ultrasound. Solid COD in sludge is decomposed into dissolved COD, resulting in the discharge of internal water from bacterial micelles.

Since nitrogen has the second largest (following carbon) content in the composition of microorganisms, the concentrations of TDN were also determined (Fig. 5). The increase in amplitude and sonication time, and thus in sonication energy, also resulted in the increase of this ratio from the value of TDN = 36.2 to 336.2 mg N/L. Concentrations of total nitrogen presented on the graph confirm the conclusion that sonication causes disintegration of microorganisms that are part of sludge aggregates. It should also be stressed that the concentrations of dissolved forms of carbon and nitrogen are even several dozen times higher than the values characteristic of non-prepared sludge.

After ultrasonic pretreatment destroys the strength and structure of microorganisms, a large amount of organic matter enclosing in cells is released into water, which is easy to make use of other microorganisms [21]. Chiu et al. [22] showed that, can be increased from 36% to 89% and the ratio of soluble N from 34% to 42% by low-frequency ultrasound treatment (0.12 W/mL).

Since the main objective of the study was to determine the most effective conditions of ultrasonic preparation, it was found that the decisive factor would be to consider the indices of total dissolved and organic carbon and acoustic energy as an operational parameter of the sonication process. The quotient of the above values allows for linking the economics of the process with the disintegrating effect (Figs. 6 and 7). The graphs show a tendency to increase the TDC/E (the quotient of concentrations of total dissolved carbon and acoustic energy) and DsOC/E (the quotient of the concentrations of dissolved organic carbon and acoustic energy). More detailed observations can be made based on Figs. 8 and 9. The positions of points of quotients TDC/E and DsOC/E allowed for the determination of the optimal sonication time of 15 min. A further increase in the sonication time leads to an increase in the preparation energy, whereas no increase occurs in the generated concentrations of organic and total carbon. It should be noted that the values of the quotients studied were the highest for the highest amplitude of A_4 = 61.0 µm. The other amplitudes do not have such a strong effect. For the amplitude of A_3 = 48.8 µm, the experimentally determined best sonication time is 18 min. For the other two amplitudes, this is the sonication time of 21 min (A_1 = 24.4 µm, A_2 = 36.6 µm).

Zhang et al. [23] to assess the effectiveness of ultrasonic disintegration, including sediment characteristics and lysis efficiency, they used the index, kWh/kg SCOD-increase. The results showed that the energy efficiency of sludge sonication was in the range of 102–347 kWh/kg SCOD-increase. Also studies have shown, that the initial first minutes of sonication were most effective; hence intensive-short treatment was preferred. Show et al. [24] also demonstrated the effectiveness of

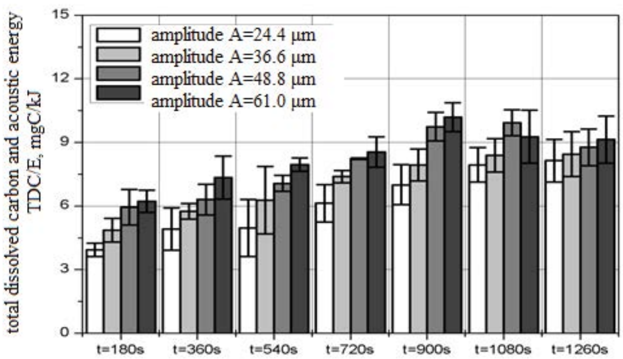

Fig. 6. Quotient of concentrations of total dissolved carbon and acoustic energy (TDC/E) vs. amplitudes and sonication times.

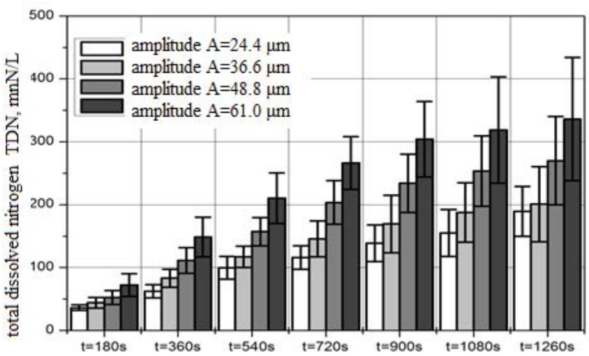

Fig. 5. Changes in the concentration of total dissolved nitrogen TDN in the sludge liquor of the sonicated substrates vs. vibration amplitude and sonication times.

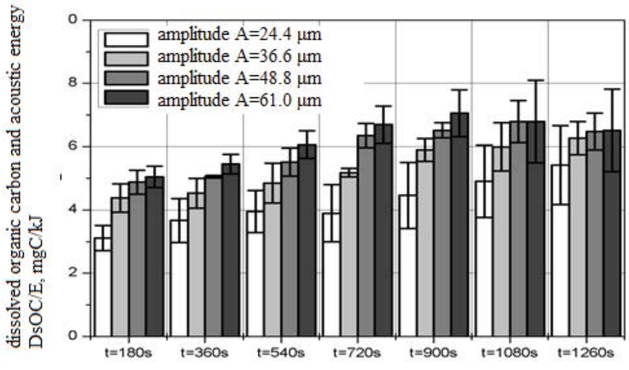

Fig. 7. Quotient of concentrations of dissolved organic carbon and acoustic energy (DsOC/E) vs. amplitudes and sonication times.

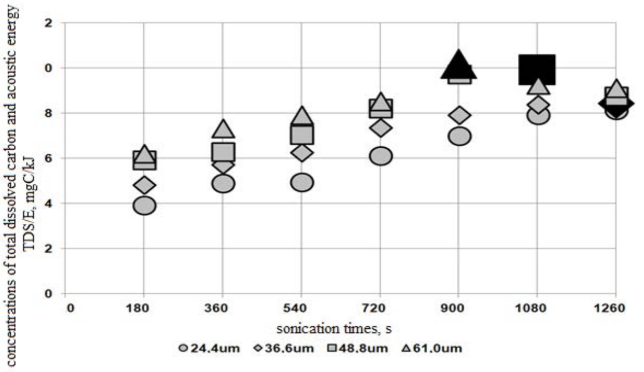

Fig. 8. Mean values of the quotient of the concentrations of total dissolved carbon and acoustic energy TDC/Ewith the choice of the most effective sonication conditions.

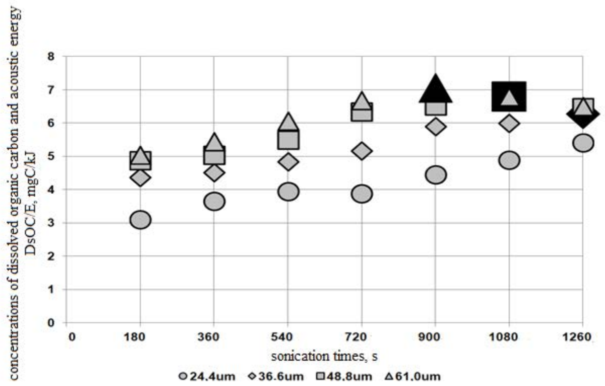

Fig. 9. Mean values of the quotient of the concentrations of dissolved organic carbon and acoustic energy DsOC/Ewith the choice of the most effective sonication conditions.

short sonification times, the most vigorous particle disruption was achieved in the initial period of sonication, which subsided subsequently.

4. Conclusions

A number of 28 research combinations based on the application of variable operational conditions of sonication and repetitions of the determinations guaranteed the choice of the most favorable conditions for the preparation of sludge by means of ultrasonic wave and formulation of the following conclusions:

- Increasing the ultrasound energy (by increasing the amplitude of vibrations and sonication times) causes a cyclical increase in concentrations of dissolved forms of carbon and nitrogen, which corresponds to an increase in the degree of disintegration of the substrates.
- From the technological point of view, the most favorable sonication conditions were (respectively, for the amplitudes studied):
  - A_4 = 61.0 μm, t_S = 900 s ($E \approx 127$ kJ, E_S = 5,278 kJ/kg d.m.)
  - A_3 = 48.8 μm, t_S = 1,080 s ($E \approx 113$ kJ, E_S = 4,717 kJ/kg d.m.)
  - A_2 = 36.6 μm, t_S = 1,260 s ($E \approx 74$ kJ, E_S = 3,063 kJ/kg d.m.)
  - A_1 = 24.4 μm, t_S = 1,260 s ($E \approx 42$ kJ, E_S = 1,740 kJ/kg d.m.)
- Use of the lowest tested amplitude A_1 = 24.4 μm shows numerous technical and technological limitations (low sonication energy, small progress of the disintegrating effect).

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