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# The effects of sonication on gravity separation of waste activated sludge

Sonication applied to different phases of wastewater and sewage sludge treatment can be beneficial in many respects. The work reported in the literature is mostly focused on sonication as a disintegration process. However, sonication is also used for conditioning of biological sludge. Disintegrating and conditioning effects of ultrasonic waves depend on the value of specific energy. The aim of the presented work was to perform the analysis of solid/liquid separation of waste activated sludge that was subjected to sonication. The properties and structure of activated sludge changed due to the exposure to ultrasounds. This resulted in some changes in the process dynamics of sedimentation and compression. The results showed that sonication accelerated the process of settling and enhanced compression of the particles. However, thickened sludge obtained by gravity separation showed low dewaterability. Gravitational separation of activated sludge subjected to sonication caused a problem with contamination of the obtained supernatant. The increase in the ultrasounds specific energy resulted in cyclical deterioration in the quality of the supernatant. The concentration of organic substance was as high as for municipal wastewater. As a result the liquid with significant quantities of solid particles can be used as growing medium for microorganisms due to increased biodegradability. Moreover, due to the sonication a layer of suspended sludge is formed. This layer consists of a large number of particles that are not subject to sedimentation. Also, this layer sustains biological activity of living organisms. It is proposed to use activated sludge sonication within separate technological system of gravitational separation. The supernatant and thickened sludge obtained from gravity separation can be considered as valuable products. The observed effects of sonication depended on the following variables: the amplitude of ultrasonic wave and specific energy. It was found that generally there are no significant differences in the observed effects of sonication for different amplitudes required to maintain stable values of specific energy. Sonication effects can be described by mathematical functions (linear regression) that allow the control of ultrasonic treatment.

Keywords: activated sludge, gravity separation, sonication

# Introduction

The application of ultrasounds has a great potential in wastewater and sewage sludge treatment technologies. Ultrasonic wave propagation is primarily a method for disintegration of sewage sludge. Sonication of the excess sludge applied prior to digestion can increase the production of biogas and enhance the reduction of organic matter [1-3]. Sonication can also contribute to minimization of the excess sludge by lysis of microorganisms of activated sludge [4].

The literature reports that parameters of sonication corresponding to specific energy of  $E_s < 1000.0$  kJ/kg TS are classified as a threshold below which only changes in physical properties of sludge are observed. The primary effect of low energy sonication is destruction of sludge floc structure and size reduction of biological agglomerates [5, 6]. After ultrasonic decomposition of flocs, re-flocculation occurs and this results in the formation of sludge agglomerates with different characteristics, including altered settleability [7, 8]. It was also observed that low power sonication is the right tool to stimulate growth and respiratory activity of microorganism or dehydrogenase activity [9, 10].

On the other hand, the energy sonication of about 150 000 kJ/kg TS is considered to be the most advantageous for sewage sludge lysis. Under these conditions, the degree of disintegration is in the range of 50 to 60%. This is generally explained as the transition of substances occurring in the suspension in the soluble [11, 12].

In this study the maximum specific energy applied to the activated sludge did not exceed 7200 kJ/kg TS (which was categorized by the autors as low-energy sonication).

Low-energy sonication can be linked with the problems of solid/liquid separation of activated sludge realized in the secondary clarifiers. The use of low-energy sonication can result in the improvement of settleability and thickening of treated sludge. The aim of this work was to determine the influence of ultrasonic wave propagation on gravity separation of activated sludge. The scope of the presented work also included the experiments conducted to compare the influence of the ultrasonic wave amplitude on the intensity of the sonication effects.

### 1. Materials and methods

#### 1.1. Activated sludge (AS) characteristic

Activated sludge was collected from the municipal wastewater treatment plant (WWTP) in Czestochowa (Poland). The WWTP operates in the technological system UCT. The inflow of wastewater to the WWTP is about 50 000 m<sup>3</sup>/d. Samples of activated sludge were collected from the oxic zone and used immediately for the experiments. The total solids concentration (TS) of activated sludge (AS) varied between 3.55 and 5.16 kg/m<sup>3</sup> and the content of volatile solids (VS) was about 72%.

#### 1.2. Ultrasonic treatment

The experiments were carried out with the ultrasonic disintegrator VC750 (Sonics). The ultrasonic apparatus was operated at frequency of 20 kHz using probe with a tip diameter of 19 mm and maximum applied power of 750 W. Sludge samples ( $V_0 = 1.0 \text{ dm}^3$ ) were sonicated in a glass vessel at ambient temperature. Specific energy ( $E_s$ ) was calculated from Eq. (1):

$$E_{\rm S} = \frac{E}{V \times TS'}, \ kJ \ kgTS^{-1}$$
(1)

$$\mathbf{E} = \mathbf{P} \cdot \mathbf{t}_{\mathrm{s}}, \ \mathbf{J} \tag{2}$$

where:

- E amount of acoustic energy in Joules (watt · second) that is delivered to the probe (energy monitor of ultrasonic processor), J,
- P acoustic power, W,
- t<sub>s</sub> sonication time, s,
- TS total solids concentration in treated AS samples, kg/m<sup>3</sup>,
- V sample volume, m<sup>3</sup>.

The applied ultrasonic probe (i.e. a horn) generates ultrasonic waves with the maximum amplitude of 61.0  $\mu$ m (percentage of maximum amplitude 100%). For this study two amplitudes were used: A<sub>1</sub> = 30.5  $\mu$ m (50%) and A<sub>2</sub> = 61.0  $\mu$ m (100%). The obtained power of the ultrasonic wave at amplitude A<sub>1</sub> = 30.5  $\mu$ m was P<sub>1</sub> = 47.4±4.24 W, at A<sub>2</sub> = 61.0  $\mu$ m was P<sub>2</sub> = 121.6±4.87W. Sludge sonication times for the amplitude A<sub>1</sub> = 30.5  $\mu$ m (50%) were following: t<sub>s</sub> = 82, 157, 232, 306, 380, 545, 527 and 598 s, and for the amplitude A<sub>2</sub> = 61.0  $\mu$ m were t<sub>s</sub> = 30, 60, 90, 120, 150, 180, 210 and 240 s. The combination of applied amplitudes (power P<sub>1</sub>, P<sub>2</sub>) and corresponding propagation times (t<sub>s</sub>) of ultrasonic wave allowed to obtain similar values of acoustic energy (Eq. (2)) and also specific energy.

# 1.3. Analytical methods

Chemical  $(COD_S)$  and biochemical oxygen demand  $(BOD_S)$  were measured in the supernatant obtained after solid/liquid separation. 5 ml samples were homogenized (60 seconds, homogenizer IKA T10, 11500 rpm/min) and  $COD_S$  was measured with a spectrophotometer (HACH DR 5000). BOD was measured without prior supernatant homogenization. Determination of the biochemical oxygen demand was carried out with the manometric method using a set of WTW OXI TOP with continuous measurement of the momentary value of BOD. Maintaining the right conditions of BOD measurements makes the reaction as the first-order reaction kinetics, hence the resulting variable was also the value of the five-day biochemical oxygen demand (BOD<sub>5-S</sub>) and rate constant (k) of biochemical oxidation (Eq. (3)) [13]. Other parameters such as total solids (TS), volatile total solids (VTS), sludge volume index (SVI), total solids in supernatant (X<sub>E</sub>) and capillary suction time (CST) were determined according to the Standard Methods for the Examination of Water and Wastewater [14]:

$$\mathbf{y} = \mathbf{L}(1 - e^{-\mathbf{k}\mathbf{t}}) \tag{3}$$

where:

- k reaction rate constant,  $d^{-1}$ ,
- L full oxygen demand of the first stage of biochemical oxidation (carbon compounds), mg  $O_2/dm^3$ ,
- y oxygen demand at time t, mg  $O_2/dm^3$ .

#### 1.4. Statistical analysis

The results are presented in the graphs as the arithmetic means. The statistical dispersion of data around the arithmetic mean was characterized by the standard deviation. The objective of this study was to determine the significance of the amplitude as the fundamental variable affecting the sonication energy with the Mann-Whitney U-test. The Mann-Whitney U-test is a nonparametric test that allowed the comparison between each two groups of independent variables (amplitudes  $A_1 = 30.5 \ \mu m$ ,  $A_2 = 61.0 \ \mu m$ ). This assumption was used to verify the hypothesis. If the calculated value of the probability test (p-value) is less than the accepted significance level alpha ( $\alpha = 0.05$ ), then the null hypothesis is rejected. Otherwise there was no reason to reject the null hypothesis.

It also examined that the relationship between the parameters of ultrasonic treatment (values of specific energy  $E_s$  and amplitude A) and studied parameters of gravitational separation is a linear regression. Therefore, the coefficient of determination ( $R^2$ ) for the equation of linear regression was calculated.

### 1.5. Sedimentation and thickening

A sedimentation column with a height of 1.0 m and a diameter of 0.1 m was used for the experiments. The active sedimentation column height was 0.9 m (H<sub>0</sub>). The total volume of sludge in the column was  $V_c = 7.0 \text{ dm}^3$ . 1 dm<sup>3</sup> sludge sample ( $V_0 = 1.0 \text{ dm}^3$ ) was subjected to sonication and transferred to the settling column to fill it with 7.0 dm<sup>3</sup> of sonicated sludge. A settling test was performed for the suspension of solids (AS) with the uniform concentration (for each TS of AS samples) in the settling column for 120 minutes. The interface height was recorded at regular time intervals every 5 minutes and used to plot the settling curve. On the basis of the sedimentation curves determined with the Talmange and Fitch geometric method it was determined zone settling velocity ( $V_s$ ) and retention time for obtaining the desired thickened sludge ( $t_u$  - thickening time). Geometric constructions that were used for determination of parameters of gravity separation process are presented in Figure 1.



Fig. 1. Graphical representation of the Talmadge and Fitch method [15, 16]

# 2. Results and discussion

Gravity separation of sonicated sludge allowed to increase the settling velocity but this effect significantly depended on the sonication energy (Fig. 2). When a low-energy sonication ( $E_S < 2000 \text{ kJ/kg TS}$ ) was applied, no significant acceleration of sedimentation phase was observed. The application of sonication in the energy range of  $E_S = 2000 \div 3000 \text{ kJ/kg TS}$  doubled the zone settling velocity ( $V_S = 0.31 \text{ mm/s}$ ) in comparison to non-sonicated activated sludge ( $V_S =$ 0.16 mm/s). Above this energy threshold the significant increase in sedimentation rate of activated sludge particles occurs.



Fig. 2. Changes in the zone settling velocity (V<sub>s</sub>) as a function of specific energy (E<sub>s</sub>) and vibrations amplitude (A)

The sludge volume index (SVI) is a very important parameter in the processes of designing and operating the secondary settling tanks. In the experiments the untreated sludge showed a very disadvantageous value of SVI = 180 cm<sup>3</sup>/g. Ultrasonic treatment definitely allowed to improve the sludge settleability (Fig. 3). Ultrasonic treatment at doses  $E_S > 3000 \text{ kJ/kg TS}$  resulted in the reduction of the sludge volume index to the level of recommended values (120 cm<sup>3</sup>/g). The obtained maximum value of SVI amounted to 86 cm<sup>3</sup>/g ( $E_S > 6000 \text{ kJ/kg TS}$ ). In the activated sludge process, the significant reduction in the values of sludge volume index can indicate negative effect on the floc structure and indirectly on biological activity of microorganisms.



Fig. 3. Changes in the sludge volume index (SVI) as a function of specific energy  $(E_s)$  and vibrations amplitude (A)

Sonication of AS resulted also in rapid reduction of time required for sludge thickening (t<sub>U</sub>) that were determined by using the Talmadge and Fitch construction. Reduction of the t<sub>U</sub> value will result in the reduction of volume of facilities for separation phase (Fig. 4). Sonication at  $E_S > 6000 \text{ kJ/kg TS}$  lead to a significant decrease in thickening time to  $\overline{t_U} > 30 \min(\overline{t_U} = 65 \min$  for non-sonicated AS).



Fig. 4. Changes in thickening time (t<sub>U</sub>) as a function of specific energy (E<sub>s</sub>) and vibrations amplitude (A)

Thickened activated sludge obtained from gravitational separation was used for dewaterability studies. Sonication caused a significant deterioration in the ability to their dewatering as indicated capillary suction time test (CST) (Fig. 5). The obtained values of CST were CST = 83.2 s for non-sonicted sludge and CSK = 495 s for the sludge sonicated at a dose  $E_s > 6000 \text{ kJ/kg TS}$ , respectively. The increase in the CST probably resulted from a significant proportion of bound water in the structure of thickened sludge. This phenomenon can be associated with ultrasonic cell lysis and release of biologically and chemically bound water to the aqueous phase (originally before lysis in the intracellular form) and hydrophilic biopolymers.



Fig. 5. Changes in capillary suction time (CST) as a function of specific energy (Es) and vibration amplitude (A)

The required effect of gravity separation is also to obtain clear liquid that means that the concentration of particulates in the supernatant should be at the lowest. This condition cannot be fulfilled in case of separation of AS subjected to sonication. Ultrasonic activity caused that after the separation process the supernatant showed the properties of a liquid that contained significant quantities of solid particles. The mass of the particles remaining in the suspended phase was very high. Applying the sonication dose of  $E_S > 6000 \text{ kJ/kg TS}$ , about 30% of the total solids of AS remained in the supernatant instead of precipitating to the layer of thickened sludge (Fig. 6).



Fig. 6. Effect of specific energy  $(E_s)$  and vibrations amplitude (A) on total solids concentrations in the supernatant  $(X_{\rm E})$ 



Fig. 7. Effect of specific energy  $(E_{s})$  and vibrations amplitude (A) on  $COD_{s}$  value in the supernatant

The increased mass concentration of solid particles in the supernatant resulted in the increased concentration of organic matter. Energy intervals used in the study caused a gradual increase in the chemical oxygen demand of the supernatant up to  $1400 \text{ g O}_2/\text{m}^3$  (Fig. 7).

The growth response to increasing sonication energy was also observed through the five-day biochemical oxygen demand. The range of observed mean BZT<sub>5-S</sub> values was from 187 to 443.7 g  $O_2/m^3$  (Fig. 8). The obtained supernatant demonstrated physical and chemical parameters typical for municipal wastewater streams supplied to a wastewater treatment plant.



Fig. 8. Effect of specific energy  $(E_S)$  and vibrations amplitude (A) on BOD<sub>5</sub>-s value in the supernatant

In addition to the BOD<sub>5-S</sub>, it is also important to determine the value of rate constant (k). From a technological point of view, decomposition rate constant can define the quality of the substrate (i.e. supernatant) as a medium substance for microorganisms. The increase in the (k) values from  $k = 0.26 d^{-1} (E_S < 1000 \text{ kJ/kg TS})$  to  $k = 0.37 d^{-1}$  at  $E_S > 6000 \text{ kJ/kg TS}$  indicated that biodegradation can be intensified by ultrasonic wave propagation (Fig. 9). This is an important aspect of ultrasonic wave interaction that gives opportunities for further improvement of the characteristics of the sludge liquids that can be uses as growing medium for microorganisms.

Also, statistical analysis for this study was performed. The regression analysis indicated that the relationship between the specific energy and the investigated variables can be defined as a linear correlation. However, there was a value fluctuation as indicated by standard deviation. The acceptable value of the correlation coefficient, especially for SVI,  $t_U$ , CST,  $X_E$  and k, had a value greater than  $R^2 = 0.5$ . The coefficients of determination exceeded  $R^2 > 0.7$  for linear regression of specific energy and parameters including:  $V_S$ , COD<sub>S</sub> and BOD<sub>5-S</sub>.



Fig. 9. Changes in  $BOD_{5-S}$  rate constant of biochemical oxidation (k) as a function of specific energy ( $E_S$ ) and vibrations amplitude (A)

The average values of the studied parameters indicated that the application of the amplitude  $A_2 = 61.0 \ \mu m$  resulted in the intensification of sonication effect. Based on the probability test (Mann-Whitney U-test) in most cases (V<sub>S</sub>, SVI, t<sub>U</sub>, X<sub>E</sub>, COD<sub>S</sub>, BOD<sub>5-S</sub> and k) there was no influence of the amplitude on the sonication effects. P-values exceeded the significance level of  $\alpha = 0.05$ . Therefore there was no reason to reject the null hypothesis that assumed no differences between the compared data sets (amplitudes: A<sub>1</sub> = 30.5  $\mu m$  and A<sub>2</sub> = 61.0  $\mu m$ ). In consequence, two opposing conclusions regarding the variation in the population of results between the amplitudes A<sub>1</sub> and A<sub>2</sub> were formulated. It should be pointed out that the U-test results and the final assessment depend on the interpretation of the researchers.

# Conclusions

Based on the obtained results it can be concluded that sonication of AS performed in order to improve the clarifying and thickening function of a secondary settling tank has many limitations.

It is proposed to use activated sludge sonication within separate technological system of gravitational separation. The supernatant and thickened sludge obtained from gravity separation can be considered as valuable products. Sludge liquids can be recycled to the biological system as a readily biodegradable substrate. Thickened sludge can be targeted to digester as excess sludge which also has the characteristic of readily biodegradable substrate. It can be pointed out that the application of low-energy ultrasounds can be a technique associated with the work of flow or sequencing biological reactors.

The results obtained in the presented work allow formulation of the following conclusions:

- 1. The effect of ultrasonic preparation was to accelerate the zone settling velocity and improve the settleability of activated sludge. The energy dose of  $E_S = 5000 \div 7000 \text{ kJ/kg}$  TS increased the sedimentation velocity to  $V_S > 0.6 \text{ mm/s}$ , reducing the sludge volume index below the value of  $100 \text{ cm}^3/\text{g}$ .
- 2. The increase in sonication energy allowed to reduce the time required for thickening. Gravity separation of sonicated activated sludge can be carried out within the time of 30 minutes.
- 3. The increase in the ultrasound specific energy resulted in cyclical deterioration in the quality of the supernatant. The concentration of organic substance in supernatant was as high as for raw municipal wastewater ( $E_s > 5000 \text{ kJ/kg TS}$ ,  $COD_s \approx 1000 \text{ mg } O_2/\text{dm}^3$ ).
- 4. Liquid with significant quantities of solid particles can be considered as suspended activated sludge layer. After 2 hours of thickening in the supernatant nearly one-third of the dry mass of the activated sludge remains in the suspended form (ES > 6000 kJ/kg TS, XE  $\approx 1.5$  g/dm<sup>3</sup>). This layer sustains biological activity of living organisms as demonstrated by BOD test and values of rate constant of biochemical oxidation.
- 5. It is possible to describe mathematically the effects of sonication by linear correlation. The values of coefficients indicated that the linear regression could be correct for the estimation of obtained results approximately in about 50÷80%. Therefore, sonication is the subject to traditional engineering optimization which enables the prediction of the effects of activated sludge preparation.
- 6. With reference to the Mann-Whitney U-test, it was found that increasing the amplitude of the ultrasonic wave did not affect the efficiency of the sonication (in comparison with the constant specific energy). However, it is suggested to use possibly high amplitude for sonication. This leads to reduction of the sonication time by increasing the power of ultrasonic wave.

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#### Efekty grawitacyjnej separacji sonifikowanego osadu czynnego

Dotychczasowe doświadczenia laboratoryjne wskazują, że proces sonifikacji można wykorzystywać niemalże na każdym etapie oczyszczania ścieków i przeróbki osadów. W większości przypadków sonifikację traktuje się jako metodę dezintegracji, w tym dezintegracji osadów nadmiernych kierowanych do komór fermentacyjnych. Sonifikacja może być również wykorzystana jako czynnik kondycjonujący osady ściekowe. Dezintegracyjne bądź kondycjonujące oddziaływanie fali ultradźwiękowej jest silnie uzależnione od energii wprowadzonej do sonifikowanego medium. Celem przedstawionej pracy była analiza efektywności separacji grawitacyjnej sonifikowanej zawiesiny osadu czynnego. Widocznym efektem nadźwiękawiania osadu czynnego było rozbicie struktury kłaczków. Rezultatem sonifikacji były również silne zmiany w dynamice procesu sedymentacji oraz kompresji cząstek. Odnotowano znaczące zwiększenie prędkości sedymentacji cząstek oraz skrócenie czasu zagęszczania. Uzyskane osady zagęszczone charakteryzowały się jednak ograniczoną podatnością na odwadnianie. Kolejną wadą rozdziału grawitacyjnego sonifikowanych zawiesin było pogorszenie jakości cieczy nadosadowych. Stężenia zanieczyszczeń w cieczy nadosadowej odpowiadały charakterystyce jakościowej dopływających do oczyszczalni ścieków komunalnych. Zdaniem autorów uzyskane produkty rozdziału grawitacyjnego można rozpatrywać jako łatwobiodegradowlane substraty dla biocenozy osadu czynnego. Ciecze nadosadowe można scharakteryzować jako łatwo biodegradowalny substrat organiczny. Ponadto cząstki stałe pozostające w cieczy nadosadowej tworzą stan zawieszonego

osadu czynnego. Powstaje w ten sposób specyficzna warstwa filtracyjna, zachowująca aktywność biologiczną. Miejsce zastosowania niskoenergetycznej sonifikacji, takiej jak w zrealizowanych badaniach, widzi się jako proces prowadzony w wydzielonym ciągu technologicznym opartym na zagęszczaczu osadów sonifikowanych. Uzyskane produkty rozdziału grawitacyjnego o wysokiej podatności na biodegradację, tj. ciecze nadosadowe oraz osady zagęszczone, można dozować do reaktorów osadu czynnego. Obserwowane efekty oddziaływania fali ultradźwiękowej zależały od badanych parametrów operacyjnych, tj. amplitudy drgań oraz właściwej energii sonifikacji. Stwierdzono, że na ogół nie ma znaczących różnic w zaobserwowanych efektach w zależności od wielkości zastosowanej amplitudy przy stałej wielkości energii sonifikacji wprowadzonej do osadów. Drugi wniosek wypływający z analizy matematycznej dotyczy możliwości opisu zmian wartości badanych parametrów w funkcji zwłaszcza właściwej energii sonifikacji za pomocą regresji liniowej.

Słowa kluczowe: osad czynny, separacja grawitacyjna, sonifikacja