Article

Ultrasounds Energy as an Agent of Polyelectrolyte Modification Prior to Sewage Sludge Conditioning

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Abstract: The presented research concerned the phenomenon of polyelectrolyte changes resulting from modification by applying the ultrasonic field. The main aim of this research was to determine the activation degree of this macromolecular chemical compound and its effect on sewage sludge subjected to conditioning and followed by dewatering. The overall goal was to investigate the potential way of reducing the dosage of chemical compounds prior to sewage sludge conditioning. The polyelectrolyte samples were sonicated with the ultrasonic disintegrator UD-20 coupled with a sandwich concentrator. The power output of the generator was 180 W and the ultrasonic field frequency was 22 kHz. To describe the geometrical characteristics of the separated phases, the following parameters were determined: surface area (A_A), perimeter (L_A) and non-dimensional coefficient. With reference to the obtained results, the most significant quantitative changes in shape and size of the separated phases were observed for the ultrasonic field exposure time in the range of 0 to 10 s. This was in agreement with the results observed during dewatering of the investigated sewage sludge. In view of the quantitative analysis of the structure of the polyelectrolyte subjected to the ultrasonic modification, dewatering of sewage sludge was considerably improved by the application of the presented method.

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1. Introduction

In the many scopes of environmental engineering, the new methods of the processes which can eliminate or limit use of chemical reagents are being researched [1,2]. One of the possible solutions in this range is the ultrasonic field energy application [3–5].

A common trend in the selection of technologies to be used in environmental engineering is, in addition to efficiency and economic effects, also ecological considerations. Limiting the amount of chemical reagents is the main factor in reducing the costs of running processes. Replacing them with other physical methods, with the appropriate selection of process parameters, may have an impact on economic efficiency and the environment [6,7]. For example, an overdose of polymers can cause excessive levels of polymers in the water during drainage, affecting the environment. The residual polymers in dewatered sludge cakes may pose a long-term risk to the surrounding environment, especially when the sludge cakes are subject to landfill as the final disposal [8]. European regulations management methods involving storage are now being replaced by methods leading to waste stabilization and safe recycling. Their aim is, amongst other things, to promote pro-ecological management of sewage sludge [9]. According to this trend, a methodology is being searched for within numerous areas, such as water and wastewater treatment and sludge management, which will enable the elimination or reduction of the amount of chemical reagents used.

Chemical compounds used at present as agents for sludge preparation prior to dewatering can penetrate to water and soil, leading to the pollution of the environment. The possibility of reducing the dose of chemical reagents can be achieved, for example,

by combining both methods of sewage sludge preparation (chemical and physical) [10]. Research in this field is focused chiefly on the evaluation of the possibilities of using agents for this purpose, which would be physical in character [11–17].

An ultrasonic field, in a medium subjected to its action, initiates a series of phenomena of a diverse physical, chemical and biological nature. Both their type and intensity are dependent on acoustic field intensity, or the density of ultrasonic energy [18]. The selection of parameters, such as frequency, the amplitude of vibration, and time, which are decisive to field intensity, allows conditions to be obtained which favor the occurrence of specific phenomena [19,20]. Besides these parameters, considerable importance is also ascribed to the properties characterizing the medium exposed to ultrasounds [21,22]. The magnitude of intensity constitutes a criterion for the classification of applications of active ultrasound phenomena associated with irreversible changes in the medium. These are chiefly the secondary processes of ultrasonic cavitation, as well as other processes occurring in the absence of this cavitation, such as separation and coagulation [23,24]. After exceeding the so-called threshold intensity, cavitation occurring in a water medium initiates most of the ultrasonic processes, e.g., sonochemical reactions, depolymerization, or the bactericidal effect [25–27]. One of these methodologies is based on the use of ultrasound. It is known that high-power, low-frequency ultrasounds influence chemical reactions through the phenomenon of cavitation. As a result of the cavitation process, ultrasounds cause changes in the structure and polymer systems [19]. In particular, a remarkable effect of ultrasound on polymers degradation reactions was observed in solution and melt conditions [28–31]. Other authors (Rokhina et al.) [32] proposed the low-frequency ultrasound in biotechnology. Kerminen et al. [33] tested the sonication influence on the atrazine concentration in the sediment slurries.

The direct effect of ultrasounds on the flocculants may vary. The separation of electrical charges might occur as a result of strong dispersive action of the passing wave. The activity and capability of adhesion to the surface of sludge particles depends on the wavelength. On the other side, together with the charge separation, also (in certain conditions), the fusion of long-chain radicals originating from the ultrasonic field action may occur. The maintained recombination might be the source of synthesis of new macromolecules of different properties. Based on the analysis of the changes in the structure of the polyelectrolyte subjected to the ultrasonic field, attempts were made to complete the set of data in order to confirm the advantageous effect during dewatering of sewage sludge conditioned with the modified reagent.

Experiments presented in the paper concerning the character of structural changes of ultrasonic-field-modified polyelectrolyte were aimed at determining the level of activation of this macromolecular chemical compound in order to improve its influence on the sewage sludge conditioned before dewatering with the possibility of reducing its consumption [34,35]. The direct ultrasounds' action on the flocculants may be diverse. Because of a powerful dispersion impact of the passing wave, the segmentation of chemical chains may occur. Its activity and ability to grip to the surface of sewage sludge flocs depends on the length of the chains. Simultaneously, together with the separation of the chains, in some defined conditions, joining of long-chain radicals may occur as a result of ultrasonic waves action [36]. Such recombinations may be the source for the synthesis of new macromolecules, perhaps of different properties [37,38].

On the basis of analysis of polyelectrolytes structure changes after sonication, the obtained results were supplemented and broadened to determine the most advantageous course of dewatering tests of sewage sludge conditioned with the modified reagent [39,40].

The main research goal was to determine the possibility of reducing the polyelectrolyte dose after modification. The sonication of polyelectrolytes before sludge conditioning is an issue that is not fully recognized and requires further research. The possibility of reducing the dose of chemical reagents is very important from an environmental point of view and ultrasonic technology may be a clean way to deal with sludge.

2. Materials and Methods

Sewage sludge used in the study was collected from a mechanical-biological sewage treatment plant in Czeszochowa with a capacity of 90,000 m³/d (which corresponds to 314835 PE). This wastewater treatment plant treats both municipal and industrial sewage. The digested sludge was used as the test substrate. The sludge was characterized by the following parameters: capillary suction time (CST)—292 s; dry matter—22.8 g/L; initial hydration—97.64%; dry organic matter (volatile solids)—57.34%; dry mineral matter (ash)—42.66%.

2.1. Experimental Design

The ultrasonic wave energy was applied only to polyelectrolyte, which was then used in the conditioning process of sludge. In experiments, the low-frequency ultrasounds of 22 kHz were used with the variable vibration amplitude of 8 µm (Series I) and 16 µm (Series II). The power output of the generator was 180 W. As the reagents were used C496 HMW, Renfloc 25489 and Praestol 655 BC, cationic polyelectrolytes acting most effectively. Among the three polyelectrolytes, based on the capillary suction time (CST) test, Renfloc 25489 had the lowest values and was selected for further studies. From the test of capillary suction time (CST), the most advantageous polyelectrolyte (Renfloc 25489) dose and the time of acoustic field action were determined. The most favorable dose of this polyelectrolyte was 3.5 mg/g DM and the time of acoustic field action was 0–10 s. The effect of the ultrasonic field on the character of changes in the structure of the polyelectrolyte was estimated from the digital pictures taken with an optical microscope connected with the computer analysis of Sigma Scan Pro 5.0. From the analysis of the separated phases, only 5 to 10 microphotographs in the microscope magnification of 50×, 100× and 200× were taken into consideration.

To describe the geometrical characteristics of the separated phases, the following parameters were determined: surface area (A_A), perimeter (L_A) and non-dimensional coefficient $\left(\eta_A = \frac{(L_A)^2}{4\pi A_A}\right)$. Apart from the parameters of the picture, in the quantitative analysis the statistical parameters, such as:

- percentage of analyzed phases (total area of phases referred to the real area)
- number elements per 1 mm² were taken into consideration. Additionally, the variability indicator characterizing the homogeneity of results dispersion of analyzed stereological features as $\left(\frac{\sigma(x)}{\bar{X}}\right)$ was introduced.

With the reference to the obtained results, the most significant quantitative changes in shape and size of the separated phases were observed for the ultrasonic field exposure time in the range of 0 to 10 s.

2.2. Sonication Experiment

Sonication of the tested polyelectrolyte was carried out with the ultrasonic disintegrator UD-20 type with a “Sandwich” type immersion concentrator. Ultrasonic field energy (E_s) generated by a “sandwich” ceramic transducer in the disintegrator was introduced to the tested medium through a probe. The following sonication parameters were used: vibration amplitude $A = 8 \mu\text{m}$ (Series I) and $A = 16 \mu\text{m}$ (Series II) and sonication time: 0, 2, 4, 6, 8, 10 s. The volume of the sonicated polyelectrolyte samples was 0.1 L (100 cm³) in a vessel with a diameter of 1.8 cm. According to Śliwiński [13], energy values can be defined as the acoustic field that describes the transport of energy through waves. The intensity of the sound is defined as the amount of energy which is transported by the acoustic waves over time of 1 s per area of the surface which is perpendicular to the direction of wave propagation. The intensity of the acoustic wave was calculated from the formula:

$$I = N/S \text{ [W/cm}^2\text{]} \quad (1)$$

N—acoustic power (W)

S—area of the surface that the wave passes through (cm²)

2.3. Analytical Methods and Statistical Procedures

The following analytical designations were used in the course of the conducted tests:

- Dry matter and initial hydration were determined based on the PN-EN-12880 standard.
- The capillary suction time was measured by the Baskerville and Galle method by measuring the filtrate transit time between the rings with diameter of 32 and 45 mm. Whatman 17 paper was used for the measurement.
- The sigma Scan Pro 5.0 program was used for computer quantitative image analysis in conjunction with optical microscopy.
- The measurement of the viscosity of the polyelectrolyte was carried out on a RC 20 rheometer ("Rheotec"). A CC-45 tip with the following parameters was used for the measurement:
 - speed gradient $0 \div 1032 \text{ s}^{-1}$
 - shear stress $0 \div 195 \text{ Pa}$;
 - plastic viscosity $0.02 \div 15 \text{ Pa}\cdot\text{s}$;
 - sample volume 100 cm^3 .

The measurement time of one polyelectrolyte sample was 120 s for the assumed rotational speed of the measuring vessel $v = 200 \text{ s}^{-1}$. The device worked in conjunction with a computer equipped with the Rheo 2000 program. The measurement error was $\pm 1\%$ of the maximum value of the measurement.

Each treatment was performed in triplicate and average values and standard deviations were obtained.

2.4. Statistical Analysis

The study of the relationship between mass phenomena and processes enables the recognition of the causal relationships between them. Correlation calculus deals with the detection and recognition of relationships between variables representing mass phenomena. Correlation means the interconnection, interdependence of some phenomena or objects. The linear correlation coefficient R is the ratio of the sum of the product of the deviations of individual values of the X and Y variables from their arithmetic means to the product of the standard deviations of both variables and the total sample size. The factor is always a number between -1 and 1 .

3. Results and Discussion

In the diagram (Figure 1), the changes of average values of A_A , L_A , η_k for the samples of Renfloc 25489 polyelectrolyte in the function of the time of ultrasonic field action were presented. On the basis of completed observations, it was stated that the most significant quantitative changes of shape and size of crystallized phases were found in samples exposed to ultrasonic field in the time range of 0–10 s. Therefore, the subsequent results interpretation was limited to the time range mentioned above. In addition, the analysis of microphotographs of the investigated polyelectrolyte structure concerned the sonification time up to 10 s, related to the most pronounced quantitative changes of crystallized phases.

Figure 1. Sonification time-dependent changes of average values of (a) surface area (b) perimeter, and (c) shape coefficient for the Renfloc 25489 slides.

The observation of the polyelectrolyte structure before and after sonification led to the statement that obtained pictures of separated phases revealed meaningful differences in shape and sort of formed crystals in comparison to the structure of non-sonificated polyelectrolyte (initial sample). This sample, in microscopic observation, was an arrangement of compact, long ramifications of regular pattern. 2-s ultrasonic field exposure caused some

change of this regular structure, which can be described as shorter ramifications (dendrites), similar shape and sort to the initial sample, though the obtained dendrites were thinner and more elongated. The increase of sonication time led to further changes in the observed structure of investigated polymer. The microphotograph of Renfloc 25489 sonicated for 6 s displayed a distinctly different picture of formed structure (Figure 2), which differed from the initial sample. The obtained crystals were less regular, shorter and partially deformed. On the other hand, the tested polyelectrolyte after 8-s exposure to ultrasounds presented a regular contour of crystals with many lateral, uniform dendrites. The gained structure was tinier and very symmetric, and its shape was non-linear. However, after 10-s sonication, the picture of the tested reagent structure showed dendrites clearly differing morphologically from the initial sample. This time they were thinner, tinier, with dispersed, quite regular crystals.

Figure 2. Dependence of viscosity and microstructure of tested polyelectrolyte Renfloc 25489 on its sonication time. Photographs of the sonicated polyelectrolyte (Series I— $A = 8 \mu\text{m}$ and Series II— $A = 16 \mu\text{m}$): time 0, 2, 6, 10 s, respectively.

Together with microstructure observation, the viscosity measurement of this reagent was led. The measurement was performed for the same ultrasonic field exposure time (0, 2, 4, 6, 8, 10 s). For the Series I ($A = 8 \mu\text{m}$), the obtained result indicated a slight influence of short sonication time on viscosity changes of tested polyelectrolyte (Figure 2). For the initial sample, the measured viscosity value was 0.048 Pa·s and after 8 and 10 s of sonication, it remained almost unchanged; though 2-s ultrasonic field exposure resulted in an insignificant increase of this parameter value to 0.05 Pa·s.

The presented course of Renfloc 25489 polyelectrolyte viscosity changes for Series II ($A = 16 \mu\text{m}$) indicated a more significant influence of the acoustic field on this parameter. In this case, the viscosity decrease together with elongation of sonication time was obtained, which, after 10 s of ultrasonic field action, was 0.015 Pa·s. In the graphical assembly of test results (Figure 2), a choice of polyelectrolyte microphotographs was presented (sonication time 0, 2, 6, 10 s). The photographs confirm the obtained result, which was the increase in the efficiency of tested sludge dewatering after conditioning with modified polyelectrolyte (Figure 3). This may state the influence of ultrasonic field on the flocculating properties of the polyelectrolyte.

The confirmation of structural changes observed on the microphotographs were the quantitative tests carried out for the Series I and II of specimens, which allowed for determination and comparison of surface area size, its perimeter and shape coefficient of phases arisen under ultrasonic field action. It was affirmed that increase of ultrasonic field amplitude ($A = 16 \mu\text{m}$) Series II caused more intensive pulverization of analyzed structures.

Figure 3. Capillary suction time of sewage sludge conditioned with sonicated polyelectrolyte.

For the Series I ($A = 8 \mu\text{m}$), the surface area changed its average value from 0.0025 mm^2 (initial sample) to 0.0018 mm^2 for the specimen after 10-s sonification. This parameter gained its peak value of 0.014 mm^2 for 2-s sound application and it was five times more than for the initial sample. The perimeter changed its value from 0.23 mm for the initial sample to 0.26 for the sample after 10-s sonification. However, the non-dimensional shape coefficient was 2.58 for the initial sample and then increased to the top value of 5.95 for the sample exposed to 6-s sonification (Figure 1).

Simultaneously, the value of the perimeter of the observed phases increased and was the highest (over three times higher in comparison to the initial sample) for 2-s ultrasonic field action, which correlated with the observed tendency in the case of surface area. In the case of Series II of tests, after the quantitative analysis carried out, it was stated that amplitude growth caused at the same time an increase of tinier structures and phases number, which was confirmed by the results of the average surface area, perimeter and shape coefficient changes.

In the sample mentioned above, the highest value of surface area was obtained for the specimen after 8 s of sonification and it was 0.032 mm^2 . On the other hand, the lowest value was obtained for the 10-s exposure of the ultrasonic field and it was 0.00056 mm^2 , which compared to the initial sample was an over four times lower result. Further increase of sonification time did not influence significantly the changes of the characterized parameter.

Similarly, for the average perimeter of analyzed phases, the decrease of this parameter was observed for 2 and 4 s of sonification to 0.13mm and 0.18mm, respectively, from the initial value of 0.23mm. The same tendencies were noticed for the third of the characterized parameters—non-dimensional shape coefficient. This coefficient changed its value from the initial 2.58 to 1.97 for the sample sonificated for 2 s. The highest value of 4.13 was reached for 6-s sonification, and 3.16 for 10-s exposure of the ultrasonic field.

Comparing the results of quantitative analysis carried out for the Renfloc 25489 polyelectrolyte (Series I and II), it can be noticed that application of higher ultrasonic field amplitude caused worsening of values of analyzed parameters. It was confirmed by pictures of samples structure included in this series of tests. The obtained picture of analyzed polyelectrolyte structure exposed to 2-s sonification showed clearly formed phases, but tinier and torn in comparison to the initial sample. Separated dendrites were less numerous and their shape and sort hardly corresponded to the structures observed before (Figure 2).

As a result of 8-s sonification, the pulverization of observed phases was even more pronounced and their shape visibly differed from those observed previously. Apart from appearing crystals, at the same time, straight, stretched fragments of structure, almost without dendrites, were observed.

The picture of very tiny, sparsely situated crystals was presented in the microphotographs for the 6 and 10-s ultrasonic field exposure. The developed structure may

confirm the depolymerization of the macromolecular compound after the sonification process performed.

The presented results documented certain dependences between ultrasonic field parameters, observed structures of separated phases and parameters of quantitative analysis. The average values of surface area for the specimen of Renfloc 25489 polyelectrolyte (Series I) were in the range of the strongest ultrasonic field action (0–10 s) over seven times higher than those obtained for the Series II. Similar changes were noticed for the average perimeter value and for the shape coefficient.

The following regularities were observed when the sewage sludge was conditioned with the most favorable dose of ultrasonic polyelectrolyte (3.5 mg/g DM). Capillary suction time values were the lowest when the polyelectrolyte was sonicated for 2–4 s (Figure 3). These values were lower by about 50% in relation to the conditioning of the sludge with non-sonicated polyelectrolyte. In addition, the values of final hydration of sewage sludge conditioned with polyelectrolyte sonicated for 2 s decreased by 2.5% in relation to the use of non-sonicated polyelectrolyte. This proves that the use of polyelectrolyte dose after ultrasonic modification to sewage sludge conditioning may next improve the parameters of sewage sludge processing, for example, dewatering. The changes of values of polyelectrolyte viscosity after ultrasonic field action were not significant (Figures 4 and 5). In addition, the increase of the values of the resistivity of conditioned sludge were observed (Figure 6). Changes and relationships between the structure of conditioned sewage sludge and modified in the ultrasonic field polyelectrolyte are shown in Figure 7.

Figure 4. Viscosity of polyelectrolyte after ultrasonic field action.

Figure 5. Final hydration of sewage sludge conditioned with sonicated polyelectrolyte.

Figure 6. Specific resistance of sewage sludge conditioned with sonicated polyelectrolyte.

Figure 7. Structure of modified polyelectrolyte and sewage sludge conditioned with sonicated polyelectrolyte.

4. Conclusions

Ultrasound-assisted modification of polyelectrolyte was investigated for the first time in this study. According to the research goal, the modification of macromolecular polymers by using the ultrasonic field can bring about measurable effects in the form of improved parameters of sludge dewatering. The ultrasonics multidirectional influence on the analyzed substances was taken into consideration. The modifying action of ultrasonic field on the high-molecular polymers used in the process of sewage sludge conditioning before dewatering was confirmed. It was noticed that the properly selected time of sonication, determined individually for each polyelectrolyte, is important. The changes in the indices characterizing sludge prepared with sonicated polyelectrolytes, observed in the tests, may be associated with the intermolecular regrouping of the polyelectrolyte under the influence of ultrasonics.

Application of the ultrasonic field results in changes of inner structure of polyelectrolytes and these changes intensify the polyelectrolyte activity on sewage sludge processing parameters.

The most significant quantitative changes concerning shape and size of crystallized phases of investigated polyelectrolyte were observed in the short range of sonification time (0–10 s) and vibration amplitude of 8 μm . The surface area changed its average value from 0.0025 mm^2 (initial sample) to 0.0018 mm^2 for the specimen after 10-s sonification. For the same value of sonication time, the perimeter changed its value from 0.23 mm for the initial sample to 0.26 mm for the ultrasonicated sample.

The depolymerization effect, which may occur for the laid-out conditions of the acoustic field, did not cause significant changes of the sonicated polyelectrolyte viscosity.

For Series I, the viscosity values of the sonicated polyelectrolyte did not change. For Series II, a decrease in viscosity was obtained along with the extension of the sonication time, which after 10 s of the ultrasonic field operation was 0.015 Pa·s.

Low values of the sonication time of the polyelectrolyte (2–4 s) improved the testing dewatering properties of the sewage sludge (CST, final hydration and specific resistance). The capillary suction time of the sludge conditioned with the ultrasonicated polyelectrolyte after 4 min of sonication was 28 s, and the final hydration value was 80.8%.

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