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SUSCEPTIBILITY OF CONDITIONED EXCESS SEWAGE SLUDGE TO BIODEGRADATION AND DEWATERING

A direct effect of chemical disintegration of excess sludge is an increase in concentration of organic compounds in the sludge liquor, expressed with the levels of soluble chemical oxygen demand (SCOD) and volatile fatty acids (VFAs). The substrate used in the study was activated sludge. The aim of the study was to determine susceptibility of disintegrated excess sludge to biodegradation and dewatering. A SONICS VCX-1500 ultrasonic disintegrator with automated tune-up was used. Thermal disintegration of excess sludge was carried out in water bath with a shaker. Disintegration of excess sludge by the hybrid method was carried out as a combination of the ultrasonic and thermal methods.

1. INTRODUCTION

Disintegration of sludge has been regarded as one of the processes of conditioning. The idea behind this process is to release organic matter and biological water contained in microorganisms' cells and simultaneously lead to the destruction of the bonds between the cells as well as direct destruction of these cells and eventually affect subsequent processing of this sludge (thickening, dewatering, aerobic stabilization) [1–3]. Disintegration accelerates hydrolysis in anaerobic stabilization through release of enzymes contained in microorganisms' cells which were disintegrated through the effect of the conditioning agent [4]. It also influences the effectiveness and course of the processes of thickening, dewatering and flow [5, 6].

There are some difficulties in precise defining borders between the methods of disintegration and classical methods of sludge conditioning. One example is conditioning by means of ultrasound waves, which might be one of the methods of sludge

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conditioning before thickening or dewatering. On the other hands, ultrasounds are considered one of the methods of sludge disintegration, thus contributing to acceleration of hydrolysis [7].

Unlike initial sludge, excess sludge is characterized by a flocculent structure formed by microorganisms' cells using polymers and biopolymers. Sludge can be considered a specific "warehouse" for organic matter which usually accounts for over 70% of dry matter that remains substantially unavailable in the stabilization processes and cannot be biologically decomposed [4].

The degree of disintegration of sewage sludge is determined by means of a variety of laboratory methods which utilize many physical, chemical and biological parameters. Breaking organisms' cells leads to release of organic intracellular organic substances to sludge liquor. The degree of cell destruction might be defined as an increase in protein concentration, DNA, TOC or soluble chemical oxygen demand (SCOD) in sludge liquor [8–11].

The increased effectiveness of anaerobic stabilization depends largely on the use of sludge disintegration processes. The examinations carried out by Xu et al. [12] demonstrated the intensifying effect of excess sludge sonication on the effectiveness of methane fermentation. The effect of this process was an increase in soluble chemical oxygen demand (SCOD) and volatile fatty acids (VFAs) during the process. Volatile fatty acids are an important intermediate product of methane fermentation while the rate of their generation and their quality influence the subsequent phases of anaerobic stabilization, thus causing intensification of biogas generation and increasing the degree of sludge fermentation [13, 14]. The intensifying effect of sludge disintegration was demonstrated in studies carried out by Bougrier et al. [8, 15], where excess sludge disintegrated by means of ultrasounds was used in methane fermentation.

The examinations carried out by Bougrier et al. [8] demonstrated that the biogas production depends on the specific energy input. For specific energy input lower than 3000 kJ/kg of total solids, biogas production linked to the particulate fraction of sludge was constant, even if the concentration of solids decreased. On the other hand, biogas production linked to the soluble part of sludge increased with ultrasound power. Increased overall amount of generated biogas was reported in the studies carried out by Zawieja et al. [16]. In the case of ultrasound disintegrated excess sludge, and the sonification times 5 and 10 min, the biogas yields of 5.15 dm³/g VSS and 9.63 dm³/g VSS were determined with the degree of digestion of 43% and 64%, respectively. During the study ultrasonic disintegrator VCX-1500 with automated tuning (SONICS, USA) was applied. For this device, maximum power output is 1500 W, whereas the frequency of vibration of the ultrasound field is 20 kHz. Moreover, in the case of raw excess sludge biogas yield and degree of digestion of 3.20 dm³/g VSS and 35%, respectively, were recorded.

2. EXPERIMENTAL

The basic substrate in the study was excess activated sludge (90%) and fermented sludge (10%) which represented the inoculum. The sludge was sampled from the Warta Sewage Treatment Plant in Częstochowa. The sludge was characterized by the following parameters: hydration: 99.06–99.09%, dry matter: 9.42–9.66 g/dm³, dry organic matter: 6.67–6.84 g/dm³, dry mineral matter: 2.75–2.82 g/dm³.

In order to determine the increase in the degree of disintegration of the excess sludge, the process of conditioning with ultrasound field was carried out using a VCS-1500 ultrasonic disintegrator with the frequency of ultrasound field of 20 kHz. The sonotrode was immersed into a vessel containing the excess sludge, at the depth of 5 cm from the vessel's bottom. The volume of the conditioned sample amounted to 0.5 dm³.

Evaluation of the effect of ultrasound field on thickened excess sludge was carried out in five research cycles, with the vibration amplitudes A of 7.85, 15.7, 23.57, 31.2 and 39.25 μm , while sonication time ranged from 60 to 600 s.

The following physicochemical parameters were evaluated:

- dry matter, dry organic matter, dry mineral matter by means of a direct weighing method according to the standard PN-EN-12879,
- volatile fatty acids (VFAs) according to the standard PN-75/C-04616/04,
- chemical oxygen demand by means of a dichromate method based on HACH 2100NIS tests according to ISO 7027.

Measurements of the capillary suction time (CST) were carried out according to the method by Baskerville and Galle [17], based on the measurement of transition of boundary layer of filtrate as a result of suction forces in the used paper (Whatman 17). Gravity densification was measured using 100 cm³ measurement cylinders for 120 min.

Based on the changes in organic matter expressed by means of SCOD (mg O₂/dm³) of the sludge liquor and VFAs (mg CH₃COOH/dm³), the authors determined the most favorable conditions of ultrasonic disintegration.

Thermal disintegration of excess sludge was carried out in water bath with an ELPIN+ shaker. The sludge samples were placed in 0.5 dm³ laboratory flasks. Thermal conditioning was carried out at 60, 70, 80 and 90 °C within the time range of 1–3 h.

The results obtained for ultrasonic and thermal disintegration of the sludge were obtained to select optimum parameters for the combination of the methods using the ultrasonic method first and then the thermal method.

The next stage of the study was a 10-day anaerobic stabilization. The study was carried out for all the mixtures in 0.5 dm³ laboratory flasks used as fermentation chambers. They were securely closed with a glass plug and a fermentation pipe and placed in a laboratory thermostat at 37 °C. Anaerobic stabilization was carried out for the following mixtures:

- mixture A – 90% of non-conditioned excess sludge and 10% of fermented sludge,
- mixture B – 90% of sludge conditioned with ultrasounds (39.25 μm , 600 s) and 10% of fermented sludge,
- mixture C – 90% of sludge conditioned by the thermal method (90 $^{\circ}\text{C}$, 3 h) and 10% of fermented sludge,
- mixture D – 90% of sludge conditioned by the combined ultrasonic and thermal method (39.25 μm , 600 s, 90 $^{\circ}\text{C}$, 3 h) and 10% of fermented sludge.

3. RESULTS

In order to determine the most favorable operational parameters of conditioning of excess sludge with ultrasound field (frequency 20 kHz) using thermal and combined methods, the authors evaluated the effect of selected methods on the SCOD and VFAs levels in the sludge liquor.

Table 1

SCOD in excess sludge liquor
after ultrasonic conditioning [$\text{mg O}_2/\text{dm}^3$]

Sonication time [s]	Amplitude of vibration [μm]				
	7.85	15.7	23.57	31.4	39.25
60	217	367	586	910	1112
120	345	590	866	945	1190
180	554	789	1297	1189	1565
240	676	987	1498	1476	1667
300	776	1245	1567	1787	2012
360	876	1567	1787	1826	2190
420	1067	1754	2119	2108	2589
480	1145	2328	2187	2218	2856
540	1498	2389	2435	2504	3125
600	1610	2676	3287	3651	3819

The SCOD for the unconditioned sample (sample 0) is 174 $\text{mg O}_2/\text{dm}^3$.

In Tables 1, 2 the SCOD values in the sludge liquor after ultrasonic and thermal conditioning, for various amplitudes of vibration (Table 1) and durations (Table 2), respectively, are given. The SCOD for the unconditioned sample was 174 $\text{mg O}_2/\text{dm}^3$. The VFAs contents in the sludge liquor after ultrasonic and thermal treating are given in Tables 3 and 4, respectively.

Table 2

SCOD in excess sludge liquor
after thermal conditioning [$\text{mg O}_2/\text{dm}^3$]

Duration of exposition [h]	Temperature of exposition [$^{\circ}\text{C}$]			
	60	70	80	90
1	1276	1932	1987	2143
2	1605	2405	2188	2658
3	1978	2712	2715	3273

The SCOD for the unconditioned sample (sample 0) is $174 \text{ mg O}_2/\text{dm}^3$.

Table 3

VFAs in excess sludge liquor after ultrasonic
conditioning [$\text{mg CH}_3\text{COOH}/\text{dm}^3$]

Sonication time [s]	Amplitude of vibration [μm]				
	7.85	15.7	23.57	31.4	39.25
60	59	78	137	198	216
120	76	93	157	232	243
180	115	122	187	237	251
240	132	136	218	242	263
300	145	149	221	245	275
360	156	162	227	253	279
420	175	177	234	264	286
480	185	197	243	275	312
540	197	212	254	287	334
600	208	215	263	312	354

VFAs for unconditioned sample (sample 0) is $37 \text{ mg CH}_3\text{COOH}/\text{dm}^3$.

Table 4

VFAs in excess sludge liquor after
thermal conditioning [$\text{mg CH}_3\text{COOH}/\text{dm}^3$]

Duration of exposition [h]	Temperature of exposition [$^{\circ}\text{C}$]			
	60	70	80	90
1	168	201	217	224
2	174	215	227	257
3	211	225	254	282

VFAs for unconditioned sample (sample 0) is $37 \text{ mg CH}_3\text{COOH}/\text{dm}^3$.

Thermal conditioning or by means of ultrasound field led to changes in the structure of sludge through breaking the bonds between flocs, causing an increase in the value of SCOD and VFAs in the sludge liquor.

The results concerning the most satisfactory values of SCOD in the excess sludge liquor conditioned with ultrasound field and by the thermal method were used to select the parameters of conditioning of the excess sludge in the combined method. It was found that the increase in vibration amplitude, temperature of thermal processing and exposure time causes increase in SCOD and VFAs in the excess sludge liquor.

The highest value of SCOD after sonication was recorded for the exposure time of 600 s for the vibration amplitude $A = 39.25 \mu\text{m}$ (the sonication density of 0.42 W/cm^3). Apula and Sanin [18] applied the sonication density of 0.51 W/cm^3 for 15 min noticing a significant increase in SCOD concentration from 50 (unsonicated sludge) to $2500 \text{ mg O}_2/\text{dm}^3$ (sonicated sludge). The sonication frequency of the device was 24 kHz and the maximum power input was 400 W. BMP tests showed 15 min of sonication led to the most enhanced anaerobic digestion with the highest methane production.

With regard to thermal processing in the temperatures considered, the highest level of SCOD and VFAs was found for the exposure time of 3 h (Table 2 and 4). According to Neyens and Baeyens [19], thermal pretreatment in the temperature range of 60–180 °C can destroy walls of cells and makes the proteins accessible for biological degradation.

In the next stage of the study, concerning anaerobic stabilization of the modified sludge, the following parameters were used for ultrasonic disintegration: sonication time – 600s, vibration amplitude – $39.25 \mu\text{m}$. In the case of thermal disintegration, the sludge before stabilization was subjected to thermal conditioning at 90 °C for 3 h. All the above parameters were used in the hybrid ultrasonic and thermal method.

3.1. ANAEROBIC STABILIZATION

During the first stage of the study, mixture A was subject to 10-day methane fermentation. The next stage involved methane fermentation of the prepared sludge, i.e. mixtures B, C and D (Fig. 1).

Figure 2 presents the dry matter content recorded on consecutive days of anaerobic stabilization of the non-conditioned excess sludge and its mixtures after physical modification with the methods used in the study. The reduction in dry mass for mixtures A, B, C and D was 15.6%, 28.06%, 28.99% and 38.9%, respectively.

Table 5 presents the most beneficial values of SCOD and VFAs obtained during 10-day acid fermentation of the excess sludge. During fermentation of mixture A, the maximum level of volatile fatty acids (VFAs) was found on the 3rd day and amounted to $531 \text{ mg CH}_3\text{COOH}/\text{dm}^3$ whereas the maximum value of SCOD ($1145 \text{ mg O}_2/\text{dm}^3$) was found on the 4th day of acid fermentation. During each of the processes for the

mixtures B, C and D, the maximum values of volatile fatty acids (VFAs) were reached on the 2nd day of the process. The levels of VFAs for the mixtures were: mixture B – 1385 mg CH₃COOH/dm³, mixture C – 1020 mg CH₃COOH/dm³, mixture D – 1491 mg CH₃COOH/dm³. The maximum levels of SCOD were recorded for each of the mixtures at the beginning of the fermentation process. They were: mixture B – 3752 mg O₂/dm³, mixture C – 3105 mg O₂/dm³, mixture D – 3614 mg O₂/dm³.

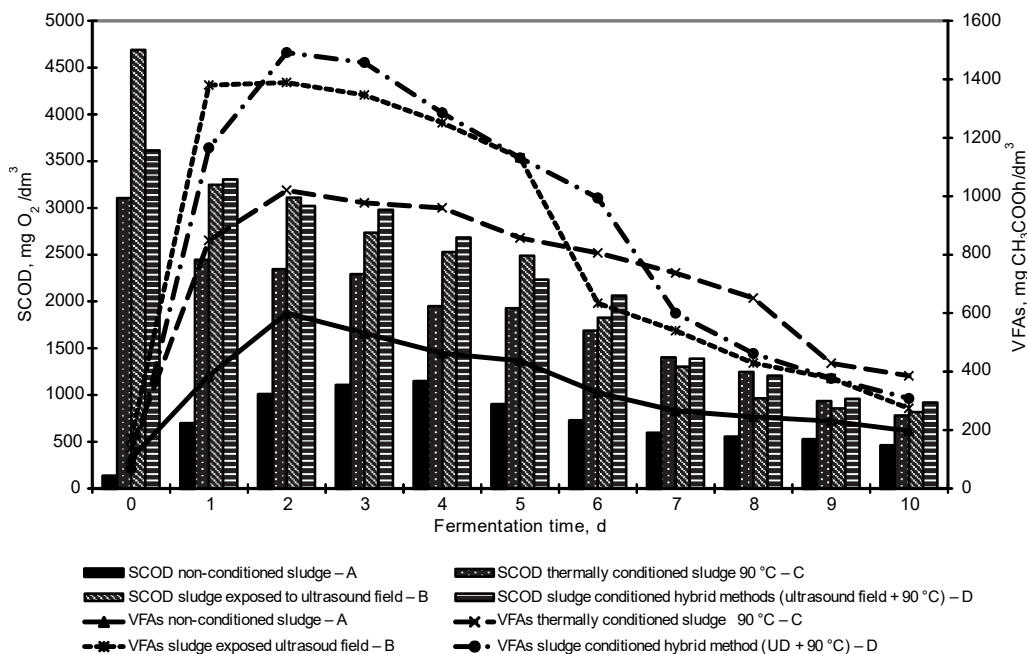


Fig. 1. SCOD and VFAs values recorded during 10-day anaerobic stabilization of the excess sludge without conditioning and after physical modifications by independent methods and a hybrid method (mixtures A–D); UD – ultrasound field

Table 5

Most beneficial values of SCOD and VFAs obtained during 10-day anaerobic stabilization of non-conditioned (mixture A) and conditioned excess sludge (mixtures B, C, D)

Indicator	Mixture			
	A	B	C	D
VFAs ₀ , mg CH ₃ COOH/dm ³ (value before methane fermentation for non-prepared sludge)	68	171	137	68
VFAs _{max} , mg CH ₃ COOH/dm ³ (maximum value for 10 days of the methane fermentation)	531 (3rd day)	1385 (2nd day)	1020 (2nd day)	1491 (2nd day)
SCOD _{max} , mg O ₂ /dm ³ (maximum value for 10 days of the methane fermentation)	1145 (4th day)	3752 (0th day)	3105 (0th day)	3614 (0th day)

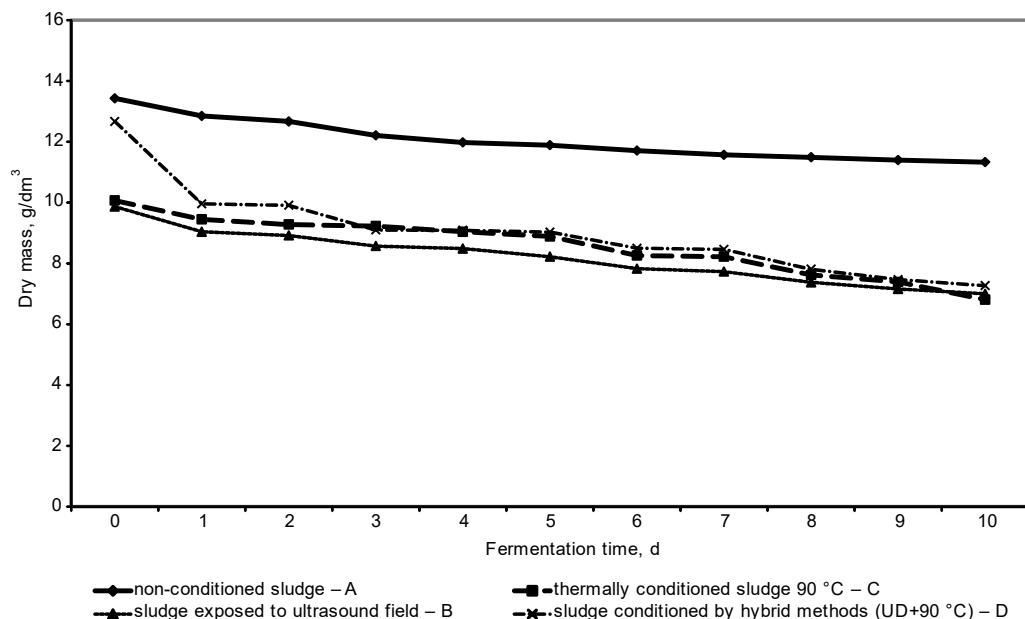


Fig. 2. Dry mass content recorded during 10-day anaerobic stabilization of the excess sludge without conditioning and after physical modifications by independent methods and a hybrid method (mixtures A–D); UD – ultrasound field

The results suggest the intensifying effect of the disintegration methods studied on hydrolysis and acidogenesis (which are the initial stages of anaerobic stabilization) in all the mixtures studied where the basic component was the prepared excess sludge. Regarding the changes in the values of SCOD and VFAs observed during stabilization of mixtures B (excess sludge after disintegration with ultrasound field with $A = 39.25 \mu\text{m}$ and exposure time of 600s) and D (hybrid disintegration method), no significant differences were found in the indices studied.

The effects of disintegration recorded during the ongoing process of anaerobic stabilization of the sludge prepared by hybrid (combination of disintegration performed under the field with $A = 29.25 \mu\text{m}$ and during sonication 600 s and thermal disintegration carried out at 90 °C) and independent ultrasonic method indicate that the use of ultrasound field with power of 1500 W, frequency of 22 kHz, established sonication time and the selected vibration amplitude showed strong disintegration effect, comparable to the efficiency recorded during the disintegration conducted by the hybrid method. Therefore, in further studies, especially because of the economy of the process, it is advisable to use the ultrasound field of shorter sonication time and the lower value of the amplitude.

According to Thiem et al. [20], the methane yield of the sonicated sludge is directly related to the VFAs concentration. Quarmby et al. [21] observed that the biogas production in a solubilised sludge increased by 15% due to sonication at 356 W·min compared to control sample (unsonicated sludge). Moreover, the effect of sonication on VFAs production was evaluated. Their concentration for the control sample in the digester increased from the initial value of 1100 mg/dm³ to 1400 mg/dm³ on the second day of digestion process. After complete digestion, the VFAs concentration decreased to 86 mg/dm³. For the sludge sample sonicated at 356 W·min, the VFAs initial concentration in the digester increased to 1300 mg/dm³, and further increase in VFAs concentration (to 1800 mg/dm³ on the second day of digestion process) was observed. The increment in VFAs production due to sonication amounted to 22%. The observed increase in the amount of VFAs was the result of increased susceptibility to biodegradation for sludge subjected to disintegration.

3.2. CAPILLARY SUCTION TIME AND THICKENING OF EXCESS SLUDGE AFTER 10-DAY ANAEROBIC STABILIZATION

Higher values of capillary suction time (CST, Fig. 3) were reported after initial conditioning of the sewage sludge. The highest value of this index was found in the sludge conditioned with ultrasounds (1710 s), followed by the hybrid method (1082 s), while the lowest value was observed for thermal conditioning (206 s).

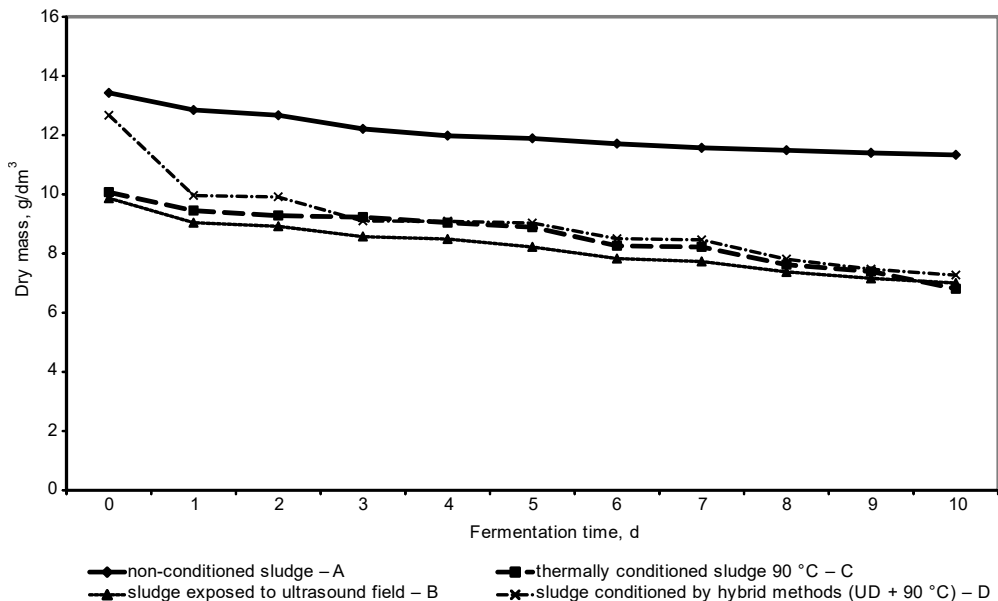


Fig. 3. Capillary suction time of non-conditioned sewage sludge and initially conditioned one by various methods during 10-day anaerobic stabilization

The values from 40 to 60 s were noted for the non-conditioned sludge. Before the beginning of the fermentation process, both sludge conditioned with ultrasounds and by the hybrid method showed high values of CST which were reduced during stabilization. The highest decrease was recorded for the sludge conditioned with ultrasound field on the first day, with the CST reduced by ca. 1000 s. The trend to reduce the CST continued until the 3rd day of fermentation and then the CST did not change on the subsequent days.

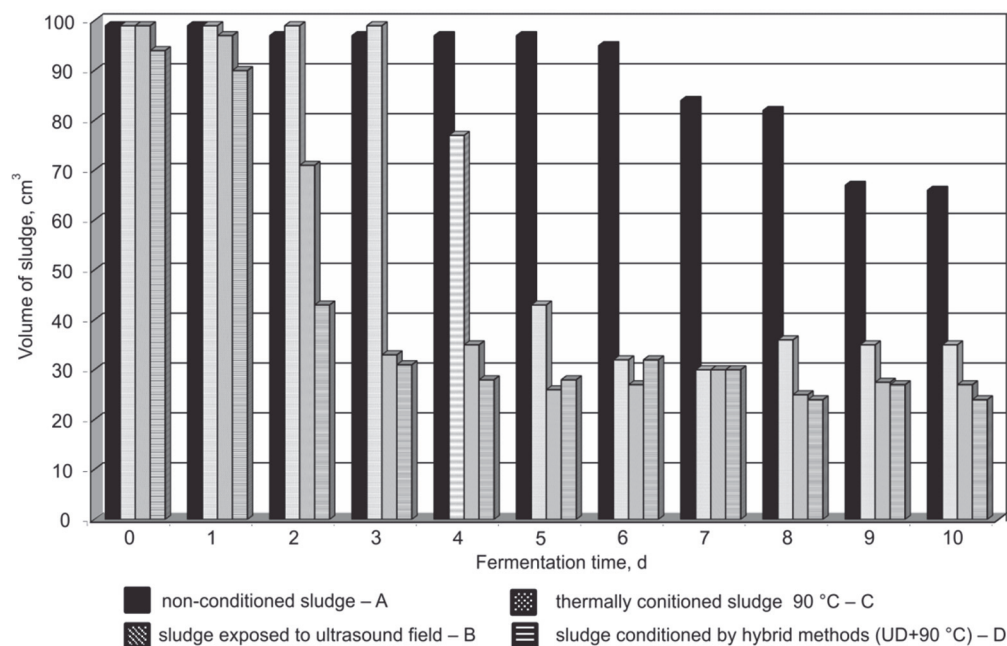


Fig. 4. Thickening of non-conditioned sewage sludge and initially conditioned one by various methods during 10-day anaerobic stabilization

The process of gravity thickening was the most intensive during the first 30 min. Figure 4 presents the volume of the sludge studied on each day of fermentation after 30 min thickening. It was found that the sludge which was the most susceptible to thickening was the sample after conditioning with the hybrid method. The biggest decrease occurred after the 2nd day, when sludge volume was 43 cm³. On the consecutive days, the volume was still decreasing but the changes were smaller. The highest degree of thickening for the sludge conditioned with ultrasounds was observed in the 3rd day (33 cm³) and on the 5th day (43 cm³) for the thermally conditioned sludge. The volume of the sludge conditioned by this method reached similar value (29 cm³) on the 7th day, which did not change until the end of the fermentation process. Among the sludge samples studied, the poorest thickening was found for the non-conditioned sludge (mixture A),

which sedimented gradually, with the first noticeable decrease in its volume observed on the 7th day (84 cm³).

According to Akin et al. [22, 23] increased level of disintegration positively affects intensification of the process of hydrolysis and conditions the effectiveness of anaerobic stabilization.

4. CONCLUSIONS

The aim of the study was to analyze excess sewage sludge after physical conditioning by exposing to ultrasound field, by the thermal and the hybrid method being a combination of both. Based on the results obtained for SCOD levels in sludge liquor, the most favorable conditions of preparation have been determined. The processes of 10-day anaerobic stabilization of the mixtures studied were carried out and the changes in SCOD, VFAs, dry matter content, CST and thickening were analyzed. It was found that application of the thermal method and the combination of these methods increases the degree of sludge disintegration due to release of organic matter contained in microorganisms' cells, thus contributing to acceleration of the first phases of anaerobic stabilization such as acidogenesis.

An essential effect of anaerobic stabilization of excess sludge after modification by methods studied with respect to anaerobic stabilization of non-conditioned excess sludge was reduction in dry matter content. With regard to anaerobic stabilization of excess sludge after disintegration with the combined methods, no essential effect of combination of the methods studied (compared to the independent ultrasonic method) on process effectiveness i.e. degree of reduction in dry mass, changes in SCOD and generation of VFAs from hybrid modification of sludge was found.

Capillary suction time of the initially conditioned excess sludge decreased upon increasing anaerobic stabilization time. The sludge after initial conditioning showed considerably higher capillary suction time compared to that observed for the non-prepared sludge (ca. 50 s). The highest CST was obtained for the sludge conditioned with the ultrasound field (1710 s). On the last day of anaerobic stabilization, all the mixtures studied had similar CST of ca. 70 s.

Analysis of the results obtained after thickening process revealed the effect of the conditioning methods on the thickening degree. Sludge after previous conditioning demonstrated improved sedimentation properties. The process of sludge thickening occurred much faster than in the non-conditioned sludge and its volume after 30 min was much lower.

The most favorable effect of the exposure of the sludge to ultrasound field can be found for the amplitude of $A = 39.25 \mu\text{m}$ and sonication time of 600 s, with ca. 22-fold increase in SCOD compared to its level observed in the control sample.

The most promising effect of thermal conditioning on the sludge can be observed for the temperature of 90 °C and exposure time of 3 h, with ca. 18-fold increase in SCOD compared to its level observed in the case of the control sample.

The biggest reduction in dry matter (38.9%) is observed for the sludge conditioned using a combined method, whereas the lowest degree of reduction (15.6%) was found for the sludge which was not initially modified.

Regarding anaerobic stabilization of the excess sludge after disintegration by the methods studied, the most favorable and similar SCOD values were obtained in both mixture B (ultrasound field of 39.25 mm and exposure time of 600 s), as well as mixture D (hybrid disintegration) at the beginning of the process: 3752 mg O₂/dm³ and 3614 mg O₂/dm³, respectively. The maximum levels of VFAs occurred on the 2nd day of the anaerobic stabilization process. Stabilization process is very effective in reduction in CST.

Initial conditioning of excess sludge after stabilization leads to decrease in the volume of sludge after gravity thickening. The most intensive thickening is observed for sewage sludge initially conditioned by the hybrid method.

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