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EFFECT OF HYBRID METHOD OF EXCESS SLUDGE DISINTEGRATION ON THE INCREASE OF THEIR BIODEGRADABILITY

The main objective of the disintegration of sewage sludge is to obtain a greater susceptibility of sludge on subsequent biotechnological processes. In order to determine the best parameters of the disintegration of excess sludge examined, the effectiveness of hybrid methods, i.e. thermal disintegration aided ultrasonic disintegration. Support thermal method by disintegration by active operation of the ultrasonic field results in high efficiency of the process, estimated by disintegration degree of hybrid modified sludge. Based on the obtained disintegration degree in the excess sludge after the individual thermal method (60 °C, time of heating 3 h) and thermal-ultrasonic method (60 °C, 3h and acoustic power of 214.21 W, sonication time 600s) a significant i.e. 35.98% increase in the disintegration degree of hybrid modified sludge was observed.

1. INTRODUCTION

Disintegration of sewage sludge consists in destruction of its structure using external forces. It should release the components of the cell in order to ensure faster and more intensive stabilization of the sludge. In effect, a change in the structure of sludge and deactivation of microorganisms occurs. This causes a substantial dispersion of the sludge particles which leads to the release of organic components to a liquid phase. Intracellular material after destruction of cell membranes pours out to the outside. Such disintegrated sewage sludge is more susceptible to biodegradation [1]. The criterion of assessment of the obtained effects of disintegration is provided by the increase in COD and VFA levels in sewage sludge. Furthermore, a further technological effect of disintegration is increase in biogas production and reduction in the content of or-

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ganic matter in fermented sludge, resulting in a better dewatering capacity of the stabilized sludge and the opportunities for its use in nature [2, 3]. With respect to the energy introduced to the system, disintegration can be divided into mechanical disintegration, with sludge cells affected mechanically by means of shear forces and changes in the pressure generating stress in the structure of the sludge and non-mechanical disintegration, which includes physical (thermal processing, freezing, osmotic shock, decompression, use of plasma), biological (enzymatic cell lysis, autolysis) and chemical methods (use of acids, alkali and the use of detergents). The most advanced methods and widely used on a technical scale are ultrasound mechanical disintegration and thermal treatment [4]. Choice of the method of disintegration should be based on the technical and economical analysis with respect to such elements as installation capability, desirable effect of disintegration, costs of use of the installation, balance of the costs of use and costs connected with sludge processing [5].

Disintegration of the excess sludge causes a reduction in the volume and mass of waste which is generated during the process in wastewater treatment plants. Consequently, a reduction in the costs of sludge management can be achieved [6]. The most often used method of mechanical disintegration is ultrasound disintegration (sludge sonication) [7]. Sewage sludge is disintegrated with ultrasounds of low frequency and high power, capable of causing ultrasound cavitation in sludge liquid resulting in a series of physicochemical and biochemical processes which occur in the area of active field interaction [8–11]. The effect of cavitation consists in creation of pulsating bubbles in the liquid occurring as a result of local destruction of continuous medium caused by high tensile forces. These forces are created due to sudden local decline in the pressure which might occur during hydrodynamic processes or in an ultrasound field of high intensity [12]. The effect of cavitation occurs only after exceeding a particular intensity of ultrasound wave, so called cavitation threshold, which depends on a number of factors and is regarded to be the cause of active effect of the ultrasound field [13].

A precondition for effective disintegration is application of the field with particular parameters and exposure time which should be adapted to physical and chemical properties, i.e. concentration of solid phase, content of organic matter and sludge particle size. A criterion for effectiveness of disintegration can be, e.g. an increase in the content of organic substances in sludge liquid which is observed after sonication. This parameter is determined based on the level of COD, whereas the consequence of disintegration is intensification of the process of the acid phase of fermentation manifesting itself with an increase in the value and increase rate of volatile fatty acids [14]. The disintegration process of excess sludge causes acceleration of first three phases of methane fermentation. The intensification of these phases results in a previous occurrence and elongation of methane phase and in an increase in biogas amount by ca. 30% [15].

Thermal methods can be divided into low-temperature methods at temperatures below 100 °C and high-temperature methods, occurring at higher temperatures. One

example of high-temperature processes of sludge disintegration is thermolysis. The process of thermolysis consists in heating the excess sludge to a temperature at which the cells of the microorganisms contained in the sludge and biopolymers which create the sludge are decomposed. Disintegration and decomposition of the cells cause a release of the biological material (which is collected in microorganism cells) to the solution, significantly increasing the content of total organic carbon in water phase. According to Podedworna and Umiejewska [7], at ca. 150 °C, around 25% of organic carbon present initially in the activated sludge is gradually dissolved to sludge liquid, causing an adequate increase in total organic carbon (TOC). The increase in the temperature of the process of disintegration causes an increase in the reaction rate and the degree of solid phase conversion; at 250 °C, the degree of conversion amounts to 50%. Dissolving the activated sludge is a result of the process of hydrolysis of complex organic compounds such as proteins, polysaccharides and lipids, which, at a higher temperature and in the presence of water, create mono- and oligomers. During heating of excess sludge at higher temperatures, a partial decomposition of organic compounds contained in the solution and creation of simple compounds of CO₂, CO, CH₄ and CH₃OH can be observed.

The process of thermolysis causes a reduction in the content of dry matter, improvement in sedimentation properties and filterability. Thermolysis is usually carried out at 160–180 °C and under the pressure of 1.0 MPa by means of water steam, with the heat from the process used for heating purposes, e.g. heating tap water, whereas the sludge, after completion of the process, is transferred to fermentation chambers. This dewatered sludge is more stable, devoid of an unpleasant odour and easy to be stored [7].

Chemical methods of disintegration utilize energy of chemical reactions combined with the conditions at which a particular reaction is supposed to occur, e.g. temperature, pressure etc., which contribute to various methods of their classification.

According to Fukas-Płonak et al. [16], hybrid disintegration by means of homogenizing stirring and sonication is more effective compared to any of the individual methods of disintegration. Immediate effects of disintegration of excess sludge were expressed by means of the indexes of dispergation of sludge flocs, lysis of microorganism cells and the index of changes in filtration properties. These effects were reflected by enhanced technological effects of the processes of methane fermentation. They included a reduction in the amount of organic matter and enhanced dewatering effect. The results of the investigations confirmed that hybrid disintegration is much more effective than individual disintegration.

2. EXPERIMENTAL

In order to define the most advantageous conditions of thermal conditioning of excess sludge, examinations were carried out at 50 °C, 70 °C and 90 °C, of the sludge

heated for 1.5 h, 3 h, 4.5 h and 6 h. The sludge was placed in laboratory flasks and secured from air access with glass plugs with a liquid-column gauge and placed in water bath. Sample volume was 500 cm³.

The choice of the most advantageous parameters of disintegration with an ultrasound field was made using a VCX 1500 disintegrator manufactured by SONICS (USA) with a frequency of sonication of 20 kHz and power of 1500 W. In order to identify the most advantageous conditions of sonication, the disintegration effect of ultrasounds was checked through increasing exposure time with varied vibration amplitude. Five research cycles were carried out with the following parameters: (39.25 μm = 100%): vibration amplitude $A = 7.85, 15.7, 23.57, 31.4, 39.25$ μm, exposure time t from 60 s to 600 s. The acoustic power of ultrasonic field was in the range 94.62–223.35 W.

In order to determine the most advantageous parameters of disintegration of the excess sludge by means of hybrid methods, the effectiveness of thermal disintegration supported with ultrasound disintegration was investigated. In the first stage, sludge samples (450 cm³) were processed thermally for 3 h at 60 °C, 70 °C, 80 °C, 90 °C, followed by ultrasonic field with the acoustic power: 187.18 W, 189.75 W, 213.32 W, 214.21 W estimated for the selected best oscillation amplitude and sonication time i.e. $A = 31.4, 39.25$ μm and $t = 300, 600$ s.

The effectiveness of examined disintegration techniques, based on the percentage increase of the value of COD, i.e. the degree of disintegration (A_{COD}) in accordance with formula [17]:

$$A_{\text{COD}} = \frac{\text{COD}_D - \text{COD}_0}{\text{COD}_A - \text{COD}_0} \quad (1)$$

where: A_{COD} – disintegration rate, %, COD_D – COD of disintegrated sludge in the supernatant, mg O₂/dm³, COD_0 – COD of raw sludge in the supernatant, mg O₂/dm³, COD_A – COD of reference sludge sample, subjected to chemical hydrolysis, using 1 M NaOH in a 1:1 ratio, at 90 °C for 10 min, mg O₂/dm³.

The chemical oxygen demand of the reference sludge sample, subjected to chemical hydrolysis was 4086 mg O₂/dm³.

In order to identify the changes in the structure of the sludge after conditioning by selected methods, microscope preparations were prepared. Observations were carried out with an Olympus BX 41 microscope with photographic equipment using 10× magnification.

During the investigations, excess sludge from the Warta Sewage Treatment Plant in Częstochowa, Poland was used, which is a mechanical and biological plant. Excess activated sludge samples were taken immediately before mechanical densification. The dry matter content and dry organic and mineral matter contents of the sludge subjected to analysis were 9.66 g/dm³, 6.84 g/dm³ and 2.82 g/dm³, respectively, while

its hydration was ca. 99.03%. The chemical oxygen demand of the supernatant liquid of raw sludge was 185 mg O₂/dm³.

3. RESULTS AND DISCUSSION

The first step was to identify the changes in the degree of disintegration which was determined for the excess sludge disintegrated with an ultrasound field having a particular acoustic power as well as vibration amplitude and exposure time and in the sludge disintegrated thermally at a selected temperature for the assumed heating time. In the case of modification with the ultrasound field, the highest degree of disintegration 85.66% was found for the acoustic power 214.21 W, i.e. the amplitude of 39.25 μm and sonication time of 600 sec (Table 1).

Table 1

Determination of the disintegration degree A_{COD} of excess sludge undergoing disintegration by ultrasonic field

A	Time of sonication [s]	Energy delivered to the sonotrode [J]	Acoustic power [W]	COD [mg O ₂ /dm ³]	COD _{dis.} /ChZT ₀	A_{COD} [%]
sample 0	0	–	–	185	–	–
7.85 μm	60	5911	98.52	210	1.14	0.64
	120	11354	94.62	350	1.89	4.22
	180	18489	102.72	577	3.12	10.02
	240	23876	99.48	630	3.41	11.38
	300	29736	99.12	783	4.23	15.29
	360	34569	96.03	968	5.23	20.02
	420	42696	101.66	1154	6.24	24.78
	480	48753	101.57	1245	6.73	27.10
	540	54338	100.63	1397	7.55	30.99
	600	62271	103.79	1562	8.44	35.21
15.7 μm	60	8092	134.87	375	2.03	4.86
	120	16394	136.62	632	3.42	11.43
	180	24695	137.19	889	4.81	18.00
	240	32670	136.13	1030	5.57	21.61
	300	40644	135.48	1285	6.95	28.13
	360	46753	129.87	1545	8.35	34.77
	420	58643	139.63	1828	9.88	42.01
	480	65943	137.38	2225	12.03	52.16
	540	75205	139.27	2534	13.70	60.06
	600	82251	137.09	2540	13.73	60.21

Table 1

Determination of the disintegration degree A_{COD} of excess sludge undergoing disintegration by ultrasonic field

23.57 μm	60	10523	175.38	570	3.08	9.84
	120	21035	175.29	855	4.62	17.13
	180	31649	175.83	1366	7.38	30.20
	240	42014	175.06	1502	8.12	33.67
	300	52497	174.99	1638	8.85	37.15
	360	61589	171.08	1840	9.95	42.32
	420	73707	175.49	2072	11.20	48.25
	480	84013	175.03	2240	12.11	52.54
	540	96205	178.16	2345	12.68	55.58
600	107278	178.8	2754	14.89	65.69	
31.4 μm	60	11129	185.48	840	4.54	16.75
	120	20485	170.71	975	5.27	20.20
	180	33796	187.76	1202	6.50	26.00
	240	41289	172.04	1490	8.05	33.37
	300	56155	187.18	1795	9.70	41.17
	360	62158	172.66	1845	9.97	42.44
	420	79451	189.17	2028	10.96	47.12
	480	87863	183.05	2185	11.81	51.14
	540	102020	188.93	2604	12.99	56.74
600	113849	189.75	2967	16.08	71.62	
39.25 μm	60	12704	211.73	1098	5.94	23.34
	120	20458	170.48	1210	6.54	26.21
	180	38499	213.88	1580	8.54	35.67
	240	53603	223.35	1762	9.52	40.32
	300	64177	213.92	2034	10.99	47.28
	360	78456	217.93	2285	12.35	53.69
	420	90865	216.35	2612	14.12	62.06
	480	103256	215.12	2885	15.59	69.04
	540	115922	214.67	3275	17.70	79.01
600	128528	214.21	3512	18.98	85.66	

From the technological standpoint, obtaining the degree of disintegration of 71.62% for acoustic power 189.75 W and lower amplitude (31.4 μm) and time of sonication 600 sec is a satisfactory result and further elongation of the time of sonication and increasing the value of amplitude seems to be unnecessary due to increased energy expenditure. The study carried out by Tomczak-Wandzel et al. [18] demonstrated that subjecting the mixture of initial and excess sludge mixed at the volume ratio of 3:7 to disintegration with an ultrasound field with field intensity of 84 W/cm² and frequency of 24 kHz resulted in obtaining the degree of disintegration of ca. 27%. Based on the literature data [19, 20] and the obtained results of the investigations, it was found that low ultrasound frequency is conducive to obtaining a high degree of disintegration.

According to Bougrier et al. [21], the degree of disintegration of sludge during the ultrasound disintegration depends mainly on the energy supplied.

Table 2

Determination of the disintegration degree
of excess sludge undergoing thermal disintegration

Temperature [°C]	Time of heating [h]	COD [mg O ₂ /dm ³]	COD _{dis} /COD ₀	A _{COD} [%]
sample 0	–	185	–	–
60	1	1305	7.05	28.64
	2	1815	9.81	41.68
	3	2192	11.85	51.32
70	1	1950	10.54	45.13
	2	2321	12.55	54.62
	3	2666	14.41	63.44
80	1	2014	10.89	46.77
	2	2360	12.76	55.61
	3	2860	15.46	68.40
90	1	2202	11.90	51.57
	2	2758	14.91	65.79
	3	3125	16.89	75.17

During thermal disintegration, the highest degree of disintegration which amounted to 75.17% was reported for the sludge heated at 90 °C for 3 h (Table 2). The lower degrees of disintegration amounting to 51.32, 63.44 and 68.40% were obtained for the temperature of 60, 70, 80 °C and time of 3 h. The study carried out by Janosz-Rajczyk et al. [19] confirmed that conditioning the sludge at 70 °C after 6 h of disintegration contributed to a substantial increase in the concentration of volatile fatty acids in sludge liquid, from 2100 mg CH₃COOH/dm³ to 3300 mg CH₃COOH/dm³ and a reduction in the concentration of organic substances in the disintegrated sludge which occurs as a result of thermal hydrolysis and the process of removal of volatile compounds. For example, in the case of alkaline disintegration, during the process carried out at ambient temperatures, Penaund et al. [22] obtained the 65% degree of disintegration with the dose of 5 g NaOH/dm³. In the case of acidic disintegration initiated with an introduction of a suitable dose of peracetic acid, the highest degree of disintegration (229%) was obtained for the dose of 5 cm³ CH₃COOOH/dm³ of the sludge and the time of 1 h.

Support for thermal disintegration with another factor of physical nature, i.e. an active ultrasound field, caused an increase in the degree of disintegration of excess sludge subjected to modification with the combined method (Table 3). As a result of

disintegration by means of the combined method, i.e. combination of thermal disintegration with a temperature of 60, 70, 80, 90 °C and ultrasonic field of 214.21 W acoustic power (field vibration amplitude: 39.25 μm , sonication time: 600 s) and a 90.62, 95.60, 97.60 and 98.48% degree of sludge disintegration, respectively, was obtained (Table 3). The combined method gave the 39.3, 32.16, 27.2 and 23.31% increase in the degree of disintegration of the prepared sludge at 60, 70, 80 and 90 °C, respectively. Such a significant increase with respect to the thermal method results from the fact that the ultrasonic field of 214.21 W acoustic power caused a considerable increase in the concentration of organic substances in a dissolved form as early as at the stage of the independent method of ultrasound disintegration.

Table 3

Determination of the disintegration degree of excess sludge undergoing disintegration by the hybrid method, i.e. thermal disintegration aided with ultrasonic field

Temperature /acoustic power	The time of exposition (heating/sonication)	COD [mg O ₂ /dm ³]	COD _{dis.} /COD ₀	A _{COD} [%]
60°C/187.18 W	3 h/300 s	2787	15.06	66.99
60°C/189.75 W	3 h/600 s	3065	16.09	74.15
60°C/213.82 W	3 h/300 s	3280	17.70	79.14
60°C/214.21 W	3 h/600 s	3729	20.2	90.62
70°C/187.18 W	3 h/300 s	2921	15.79	70.44
70°C/189.75 W	3 h/600 s	3176	17.17	77.00
70°C/213.82 W	3 h/300 s	3298	17.83	80.15
70°C/214.21 W	3 h/600 s	3898	21.07	95.60
80°C/187.18 W	3 h/300 s	3322	18.00	80.21
80°C/189.75 W	3 h/600 s	3728	20.20	91.59
80°C/213.82 W	3 h/300 s	3374	18.20	81.54
80°C/214.21 W	3 h/600 s	3976	21.70	97.60
90°C/187.18 W	3 h/300 s	3576	19.33	87.30
90°C/189.75 W	3 h/600 s	3965	21.43	97.32
90°C/213.82 W	3 h/300 s	3670	19.83	89.72
90°C/214.21 W	3 h/600 s	4010	22.76	98.48

The studies conducted by Gonzo et al. [23] and Chu et al. [24] confirmed that the efficiency of the conditioning process depends on both the ultrasonic vibrations and the temperature.

Compared to the independent method of disintegration at elevated temperatures in the range of 60–90 °C and heating time 3 h, by the combined method, for the highest value of acoustic power 214.21 W, 35.98%, 33.88%, 21.32% and 23.31% increase in

the degree of disintegration was obtained (Fig. 1). The highest increase of the disintegration degree in the excess sludge

by the thermal-ultrasonic method (60 °C, 3 h and acoustic power 214.21 W, sonication time 600s) was the result of supporting activity of ultrasonic field, as a factor which increases the concentration of organic substances in dissolved form.

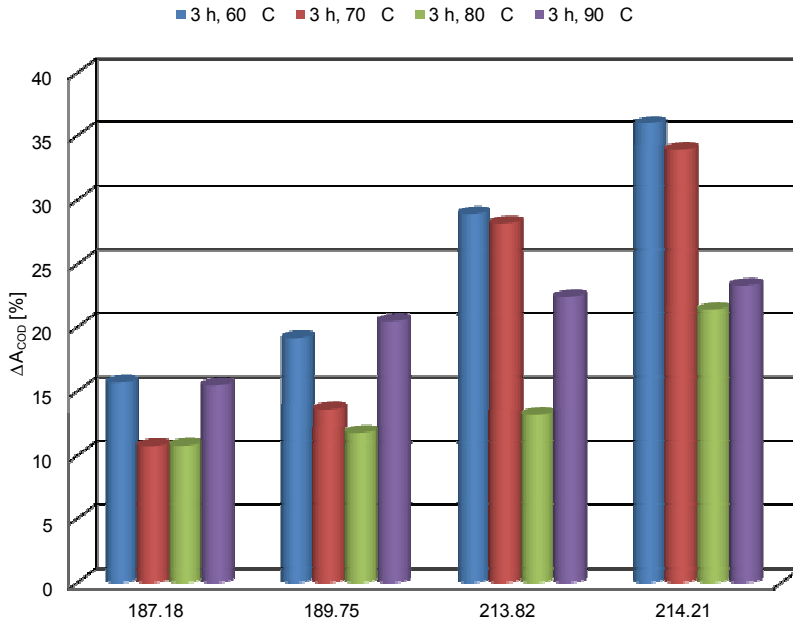


Fig. 1. Increase in the disintegration degree of excess sludge (ΔA_{COD}) treated by the hybrid method in relation to the disintegration degree of the sludge subjected to the method of self-disintegration (thermal disintegration)

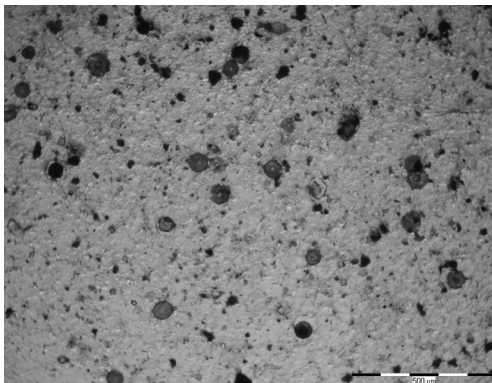


Fig. 2. Structure of raw excess sludge

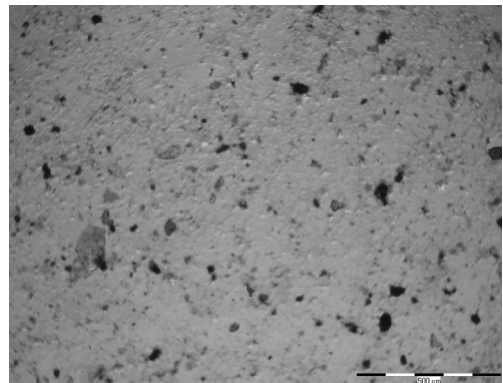


Fig. 3. Structure of excess sludge disintegrated by ultrasonic field of acoustic power 189.75 W

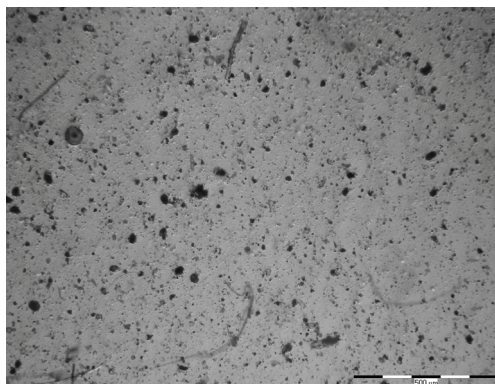


Fig. 4. Structure of excess sludge disintegrated by ultrasonic field of acoustic power 214.21 W ($A = 39.25 \mu\text{m}$, sonication time – 600 s)

($A = 31.4 \mu\text{m}$, sonication time – 600 s)

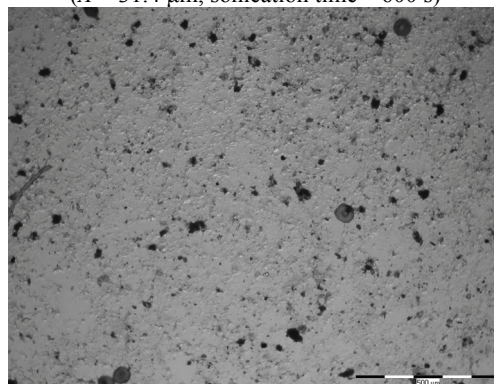


Fig. 5. Structure of excess sludge disintegrated by thermal method at 60 °C for 3 h

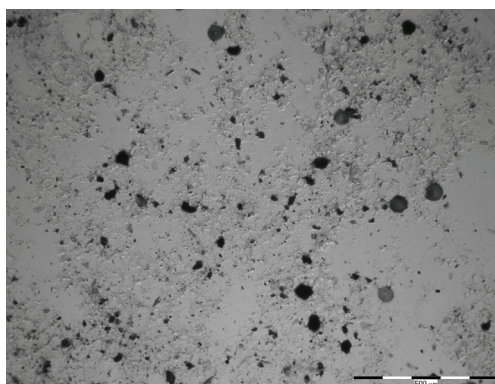


Fig. 6. Structure of excess sludge disintegrated by thermal method at 90 °C for 3 h

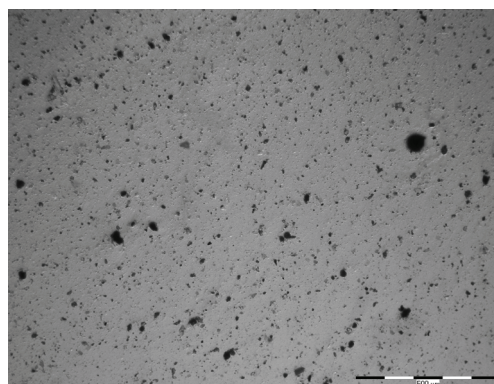


Fig. 7. Structure of excess sludge disintegrated by hybrid method, i.e. thermal-ultrasound disintegration (60 °C, 3 h, ultrasonic field 214.21 W)

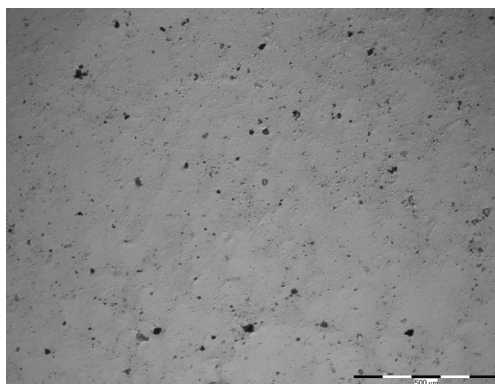


Fig. 8. Structure of excess sludge disintegrated by hybrid method, i.e. thermal-ultrasound disintegration (90 °C, 3 h, ultrasonic field 214.21 W)

Whereas obtaining of the 23.31% increase in the degree of sludge disintegration in the case of the combined method at 90 °C compared to the independent method results from the fact that thermal hydrolysis during preparation caused a considerable increase in concentrations of dissolved organic substances as early as at the stage of the independent method of thermal disintegration (Fig. 1). Tables 1–3 present changes in the degree of disintegration of excess sludge subjected to conditioning with the discussed methods. Changes in the structure correlate to the disintegration degree obtained for sludge samples subjected to individual methods of preparation.

The structure of the non-conditioned excess sludge is characterized by clusters of particles in solid phase (Fig. 2), whereas the structure of the sludge exposed to an ultrasound field was characterized by a substantial fragmentation of solid phase and observed spaces of liquid phase (Figs. 3, 4). A similar tendency in dispersion of sludge particles was observed after thermal treatment of the sludge. The structure of the sludge subjected to the thermal method was characterized by substantial liquidizing, which resulted from thermolysis of microorganism cells (Figs. 5 and 6). In the case of disintegration by the hybrid method being a combination of thermal modification at 60 °C (heating time 3 h) and ultrasonic field of acoustic power 214.21 W a significant increase of the liquefaction degree of sludge particles was observed compared to the independent method of preparation.

During the thermal disintegration at 90 °C supported with ultrasonic field of acoustic power 214.21 W, the highest destruction of the solid phase was obtained (Fig. 8).

4. CONCLUSIONS

A direct effect of excess sludge disintegration by means of physical and hybrid methods was an increase in the concentration of organic substances in sludge liquid, expressed with the level of chemical oxygen demand (COD), which occurred as a result of the initiation of the processes of lysis in microorganism cells. This increase was estimated based on the degree of sludge disintegration.

The most advantageous effect of active ultrasound field on excess sludge was found for the 214.21 W acoustic power ($A= 39.25 \mu\text{m}$ and sonication time of 600 s), which gave 85.66% degree of disintegration.

In the case of thermal disintegration of excess sludge, the most advantageous parameters were: the temperature of 90 °C and heating time of 3 h, which resulted in 75.17% degree of excess sludge disintegration.

During disintegration carried out by means of the hybrid methods, i.e. combination of thermal disintegration of the sludge at the 90 °C and ultrasound disintegration of 214.21 W acoustic power, a 98.48% disintegration degree was obtained, i.e. compared to the independent method of thermal disintegration, a 23.31% increase of the degree was noticed.

With respect to the independent method of disintegration at 60 °C and heating time 3 h, the combined method for gave the highest value of 214.21W acoustic power 35.98% increase in the degree of disintegration of the prepared sludge (Fig. 1). The highest increase of disintegration degree in the excess sludge by the thermal-ultrasound method was the result of supporting action of ultrasonic field, which increased the concentration of organic substances in dissolved form.

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