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THE IMPACT OF EXCESS SLUDGE DISINTEGRATION ON THE CHANGES OF TOTAL ORGANIC CARBON VALUE

WPŁYW DEZINTEGRACJI OSADÓW NADMIERNYCH NA ZMIANY WARTOŚCI CAŁKOWITEGO WĘGLA ORGANICZNEGO

Abstract: The efficiency of conversion of organic substances contained in the excess sludge to the dissolved form is considered as an important factor limiting the process of anaerobic stabilization. Direct effect, occurring in the disintegrated sludge, lysis process is to increase the value of the total organic carbon (TOC), correlates with the increase of the concentration of volatile fatty acids (VFAs). The total organic carbon content is indicative of the supernatant liquid of total organic carbon in dissolved form (DOC) and suspended (SOC). Together with occurring, as a result of biochemical processes, increase the degree of decomposition of organic substances contained in the sludge decreases the value of the ratio of COD to TOC. The aim of the study was to determine the impact of the process of excess sludge disintegration on the changes of the total organic carbon values. The process of chemical disintegration of excess sludge was treated using the selected acidic *ie* HCl, alkaline *ie* KOH and oxidizing reagents *ie* H_2O_2. The modification was carried out at ambient temperature for 6 and 24 h. During sludge disintegration it was noticed the increase of total organic carbon values, disintegration degree as well as the concentration of volatile fatty acids that confirmed the susceptibility of prepared sludge to biodegradation. The highest TOC value of 2150 mg C/dm^3 obtained in case of chemical disintegration of potassium hydroxide at a dose of 12.0 g/dm^3 and preparation time 24 h. For given conditions of preparing a concentration of VFAs was 523 mg CH_3COOH/dm^3.

Keywords: excess sludge; chemical disintegration; total organic carbon (TOC), disintegration degree, volatile fatty acids (VFAs)

Introduction

Sewage sludge generated as a result of the industrial and municipal wastewater treatment, due to the hazardous substances of different nature of origin, have a harmful effect on the environment. In order to increase the effectiveness of conventional methods of disposing of looking for new technological solutions, the implementation of which would provide benefits to both environmental and economic.

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When the chemical modification used in the energy that comes from a chemical reaction, in conjunction with the specific conditions of the reaction. Introduced into the sludge chemical reagents react with the chemical contained therein organic compounds, leading to changes in the physico-chemical parameters of modified sludge. During the chemical modification of the cells lysis and the destruction of cell walls of living microorganisms in sludge is observed and the release of intracellular substances into the liquid supernatant [1, 2].

Disintegration affect the structure of the sludge and the destruction of microbial membranes, leading to lysis of microbial cells. These processes result in increased availability of organic ingredients cells as a substrate for heterotrophic biomass. While water previously associated intracellularly is released. The process of disintegration is also aimed to reduce the forces between water molecules and solid phase of sludge thus facilitating their thickening and dehydration, which may lead the same to the break chemical bonds that hinder the degradation of sludge. The result of these processes is to increase the efficiency of the hydrolysis, the waveform is essentially a spontaneous nature [2, 3].

The heterotrophic microorganisms in a variety of metabolic pathways, preferably using organic carbon as volatile fatty acids. Their content in the substrate may well be characterized by its vulnerability to biochemical decomposition [4].

Chemical methods use the energy from a chemical reaction often in combination with a well defined reaction conditions, *ie* pressure, temperature. To the most popular chemical reagents may include [5, 6]: oxygen (O_2), ozone (O_3), hydrogen peroxide (H_2O_2), hydrochloric acid (HCl), sulfuric acid (H_2SO_4), sodium hydroxide (NaOH), potassium hydroxide (KOH), calcium hydroxide ($Ca(OH)_2$), magnesium hydroxide ($Mg(OH)_2$).

In the structure of excess sludge can be distinguished visible areas of the liquid phase and the solid phase coarse aggregates. The structure of the sludge after chemical disintegration process is characterized by significant fragmentation of the solid particles and increased liquefaction residues. The changes that can be observed in the sludge after the modification process demonstrate improved their susceptibility to biochemical degradation [7–9].

It is important for the processes of biochemical degradation under anaerobic conditions is the relationship between organic carbon and other nutrients, as well as the availability of macro- and microelements in sludge [10].

According to literature data biodegradability usually expressed by comparing the mass of the decomposed during the time period to the mass, which could theoretically be unfolded in stoichiometric proportions. Indicators for indirect assessment of the susceptibility of organic substances contained in the sludge on the biodegradation are COD or BOD_5 and TOC [11].

According to Stelmach et al [12] the maximum rate of biogas production in the case of municipal waste is observed for the carbon content in the compounds dissolved in the liquid approx. 1500 mg/dm^3. It was also found that the maximum rate of biogas production is the C/N ratio within a range of 1.5 to 2.0.

In the high processing temperatures high degree of sludge disintegration and an increase in the concentration of dissolved organic carbon is not synonymous with improvement in susceptibility sludge to biodegradation due to the formation, among others, refracting the compounds [13].

Values of BOD and COD can be influenced of organic nitrogen compounds or inorganic, reducing compounds, which may contribute to an increased demand for oxygen. The total organic carbon (TOC) is currently the only strictly defined, parameter defining the content of organic substances in wastewater and sludge [14].

Therefore, the aim of the research was to determine the effect of the chemical disintegration of excess sludge to changes in the value of total organic carbon.

Material and methods

The substrate for the study was excess sludge, which was taken from the Central Wastewater Treatment Plant "Warta" in Czestochowa. This wastewater treatment plant was the classical mechanical-biological treatment plant, and has been upgrading in terms of nitrogen and phosphorus removal and sludge management with thermal drying. Currently, wastewater is adjusted to increased regulatory requirements.

Table 1

General characteristics of excess sludge used for research

Dry mass [g/dm^3]	VFAs [mg CH_3COOH/ dm^3]	Kjeldahl Nitrogen [mg $N\text{-}NH_4$/ dm^3]	pH [–]	TOC [mg C/dm^3]
10.76 ± 0.15	67.5 ± 1.7	21.6 ± 2.1	7.02 ± 0.03	35.3 ± 1.5

The following physico-chemical designations were made: pH [PN-9/C-04540/05] [15], the dry mass [PN-EN-12879] [16], volatile fatty acids by steam distillation [PN-75/C-04616/04] [17] and Kjeldahl nitrogen [PN-73/C-04576/10] [18]. The evaluate the effectiveness of chemical sludge disintegration was made on the basis of the TOC by spectrophotometric method in the infrared (carbon analyzer multi N/C manufactured by Analytik Jena). Furthermore, the degree of disintegration, assuming as reference the COD of sludge subjected to alkaline hydrolysis, was determined.

The sludge was conditioned by means of 1-mol solution of NaOH for 10 min, at the temperature of 90°C, with unchanged volumetric proportion of the sludge and the solution (1 : 1). For the excess sludge pretreatment in accordance with the above methodology the COD value was equal 8125 mg O_2/dm^3.

The degree of disintegration was estimated according the following formula [19]:

$$DD_{COD} = (SCOD_1 - SCOD_2) / (SCOD_3 - SCOD_2) \cdot 100 \quad (1)$$

where: DD_{COD} – disintegration degree [%];
$SCOD_1$ – SCOD level in the pretreatment sludge, mg O_2/dm^3;
$SCOD_2$ – SCOD level in the unconditioned sludge, mg O_2/dm^3;

$SCOD_3$ – SCOD level in the sludge conditioned chemically 1-mol NaOH with ratio 1 : 1, temp. 90°C for 10 minutes, mg O_2/dm^3.

Results and discussions

During the research have been indications of selected physical and chemical parameters modified sludge, the value of which determines the susceptibility of sludge to biodegradation. Examined the pH of the sludge dry matter and Kjeldahl nitrogen, depending upon the dose of the reagent. Table 2 and 3 show the values of the selected indications physico-chemical sludge modified as appropriate for 6 and 24 h.

Table 2

Selected physico-chemical parameters of excess sludge subjected to disintegration using potassium hydroxide, hydrochloric acid and hydrogen peroxide, a contact time with reagent 6 h

Reagent	Dose of reagent	pH [–]	Kjeldahl Nitrogen [mg N-NH_4/dm^3]	Dry mass [g/dm^3]
KOH [g/dm^3]	0.5	8.48 ± 0.08	23.4 ± 0.5	11.23 ± 0.12
	1.0	9.62 ± 0.12	26.2 ± 0.8	12.56 ± 0.14
	3.0	10.92 ± 0.09	32.8 ± 0.6	13.89 ± 0.21
	6.0	12.38 ± 0.01	48.4 ± 1.1	14.68 ± 0.17
	9.0	12.68 ± 0.04	61.6 ± 0.9	15.02 ± 0.19
	12.0	12.84 ± 0.11	83.0 ± 0.7	16.24 ± 0.22
HCl [cm^3/dm^3]	0.5	7.84 ± 0.12	31.1 ± 1.2	11.38 ± 0.23
	1.0	7.14 ± 0.07	38.4 ± 1.7	11.45 ± 0.14
	3.0	6.93 ± 0.05	42.3 ± 0.6	11.98 ± 0.17
	6.0	6.40 ± 0.04	50.7 ± 2.1	12.36 ± 0.23
	9.0	6.05 ± 0.02	53.8 ± 2.0	13.02 ± 0.10
	12.0	5.82 ± 0.07	57.2 ± 1.6	13.89 ± 0.07
H_2O_2 [cm^3/dm^3]	0.5	7.40 ± 0.12	48.3 ± 1.2	11.01 ± 0.11
	1.0	7.45 ± 0.05	46.8 ± 2.4	11.28 ± 0.14
	3.0	7.50 ± 0.07	43.1 ± 2.2	11.89 ± 0.18
	6.0	7.43 ± 0.09	38.6 ± 1.5	11.97 ± 0.09
	9.0	7.48 ± 0.04	38.9 ± 1.6	12.06 ± 0.11
	12.0	7.53 ± 0.10	28.5 ± 1.8	12.35 ± 0.23

With increasing dose, in the case of all used reagents, there was an increase of dry matter and the Kjeldahl nitrogen. It was found that prolonged disintegration significantly influenced the increase in the value of Kjeldahl nitrogen. The highest value of tested indicator of 375 mg N-NH_4/dm^3 obtained for disintegrated sludge by KOH for 24 h, using a dose of 12 g/dm^3. By analyzing changes in the pH of the sludge in case of

Table 3

Selected physico-chemical parameters of excess sludge subjected to disintegration using potassium hydroxide, hydrochloric acid and hydrogen peroxide, a contact time with reagent 24 h

Reagent	Dose of reagent	pH [–]	Kjeldahl Nitrogen [mg N-NH_4/dm^3]	Dry mass [g/dm^3]
KOH [g/dm^3]	0.5	8.74 ± 0.05	38.4 ± 1.5	10.64 ± 0.11
	1.0	8.87 ± 0.07	58.1 ± 2.1	11.74 ± 0.14
	3.0	11.53 ± 0.11	63.7 ± 1.7	13.73 ± 0.21
	6.0	12.46 ± 0.23	187.6 ± 2.3	16.28 ± 0.14
	9.0	12.70 ± 0.25	336.2 ± 3.7	19.44 ± 0.24
	12.0	12.90 ± 0.16	375.6 ± 2.9	24.81 ± 0.08
HCl [cm^3/dm^3]	0.5	7.54 ± 0.32	25.4 ± 1.2	11.45 ± 0.11
	1.0	7.42 ± 0.16	26.5 ± 1.1	11.54 ± 0.21
	3.0	6.84 ± 0.43	35.8 ± 2.6	12.21 ± 0.24
	6.0	7.01 ± 0.24	42.9 ± 3.2	13.32 ± 0.16
	9.0	6.48 ± 0.25	57.2 ± 2.7	14.01 ± 0.31
	12.0	5.98 ± 0.21	58.6 ± 2.4	14.72 ± 0.29
H_2O_2 [cm^3/dm^3]	0.5	7.43 ± 0.11	55.4 ± 1.2	10.32 ± 0.21
	1.0	7.50 ± 0.26	53.3 ± 1.6	10.46 ± 0.18
	3.0	7.51 ± 0.23	52.7 ± 2.7	11.02 ± 0.23
	6.0	7.38 ± 0.18	50.1 ± 2.1	11.56 ± 0.36
	9.0	7.29 ± 0.32	49.5 ± 2.8	11.78 ± 0.54
	12.0	8.04 ± 0.37	49.6 ± 2.2	12.02 ± 0.25

modification of sodium hydroxide and hydrochloric acid were recorded an upward trend and the declining value of the index. While for modified sludge by hydrogen peroxide was observed fluctuations in pH. The prolongation of disintegration time up to 24 h, for all the tested reagents, did not affect the change in pH.

The degree of disintegration of excess sludge pretreatment by the potassium hydroxide was determined. Figure 1 and 2 show the degree of disintegration of excess sludge treated by chemical modification of the 6 and 24 h.

The highest value of 79 and 78% degree of disintegration of excess sludge conditioned with potassium hydroxide was reported for a dose of 6.0 g KOH/dm^3 of excess sludge, the contact time of the reactant 6 and 24 h. For a dose of 9 and 12 g KOH/dm^3 reported decrease in the disintegration degree value which could be due to the heterogeneous nature of the sample. Figure 3 and 4 show the changes in the TOC value and volatile fatty acids concentration of modified excess sludge by potassium hydroxide.

The highest value of TOC was observed for doses of 12.0 g KOH/dm^3 for the time 6 and 24 h respectively 1870 and 2150 mg C/dm^3, while the lowest value of 158 and 218 mg C/dm^3 for dose KOH equal 0.5 g/dm^3. The highest concentration of VFAs,

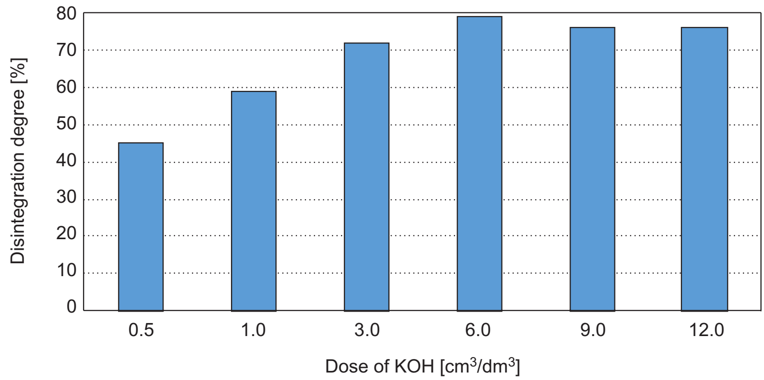

Fig. 1. Changes of the disintegration degree value of excess sludge modified by potassium hydroxide by 6 h

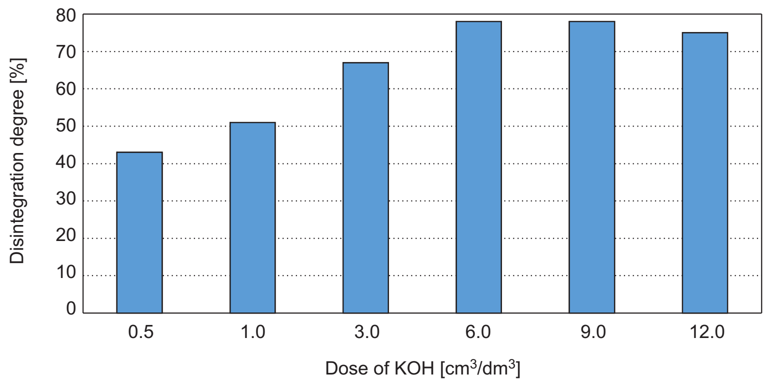

Fig. 2. Changes of the disintegration degree value of excess sludge modified by potassium hydroxide by 24 h

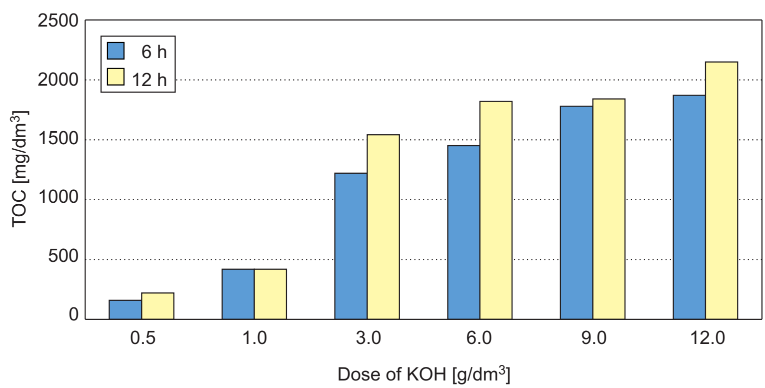

Fig. 3. Changes of the total organic carbon value of excess sludge modified by potassium hydroxide

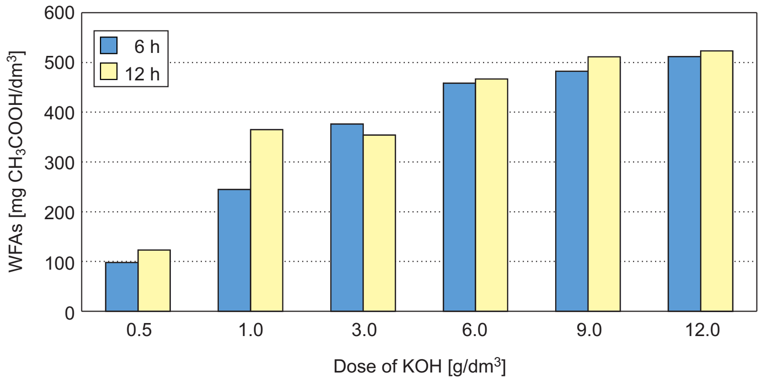

Fig. 4. Changes of the volatile fatty acids concentration of excess sludge modified by potassium hydroxide

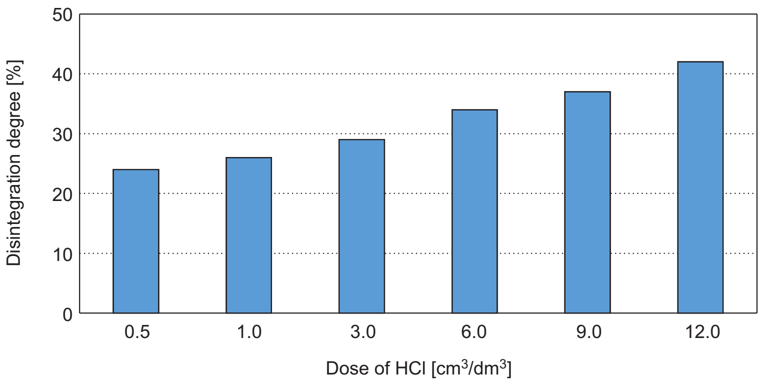

Fig. 5. Changes of the disintegration degree value of excess sludge modified by hydrochloric acid by 6 h

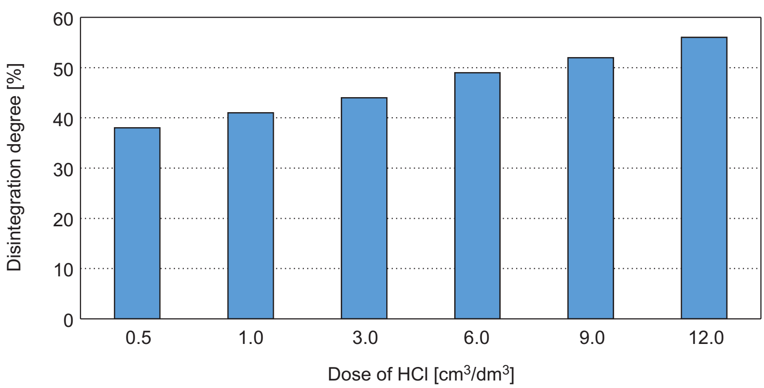

Fig. 6. Changes of the disintegration degree value of excess sludge modified by hydrochloric acid by 24 h

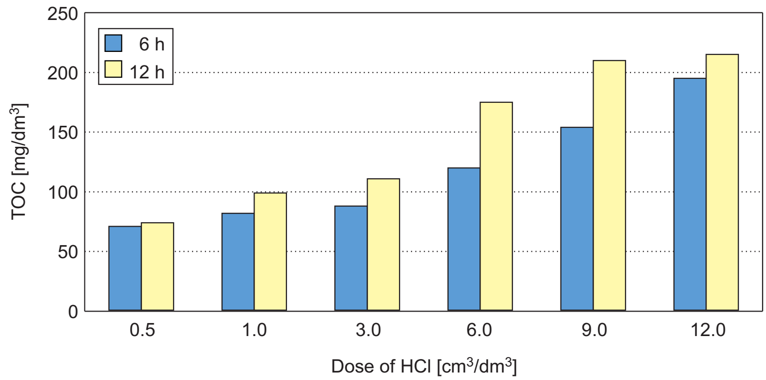

Fig. 7. Changes of the total organic carbon value of excess sludge modified by hydrochloric acid

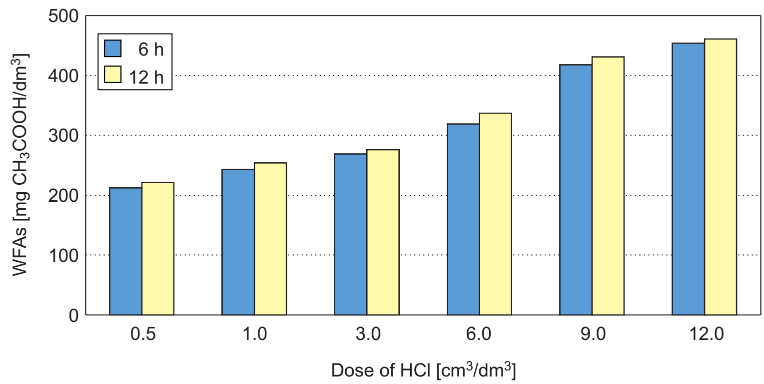

Fig. 8. Changes of the volatile fatty acids concentration of excess sludge modified by hydrochloric acid

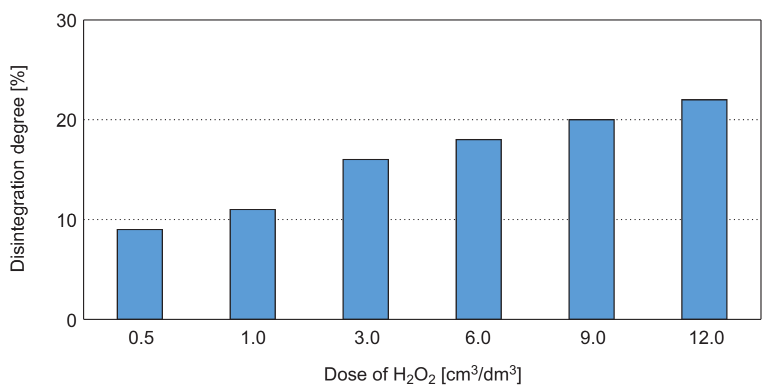

Fig. 9. Changes of the disintegration degree value of excess sludge modified by hydrogen peroxide by 6 h

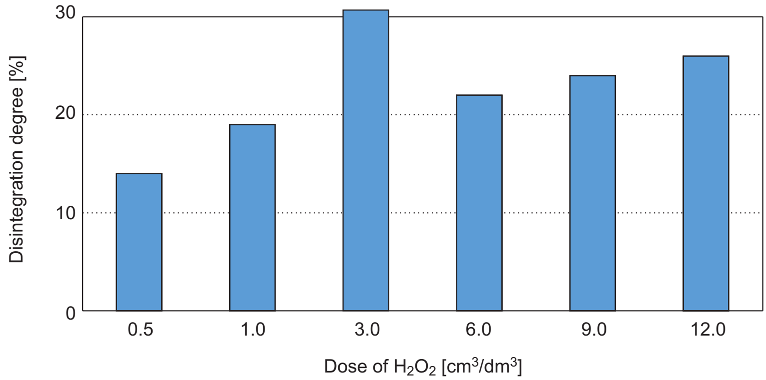

Fig. 10. Changes of the disintegration degree value of excess sludge modified by hydrogen peroxide by 24 h

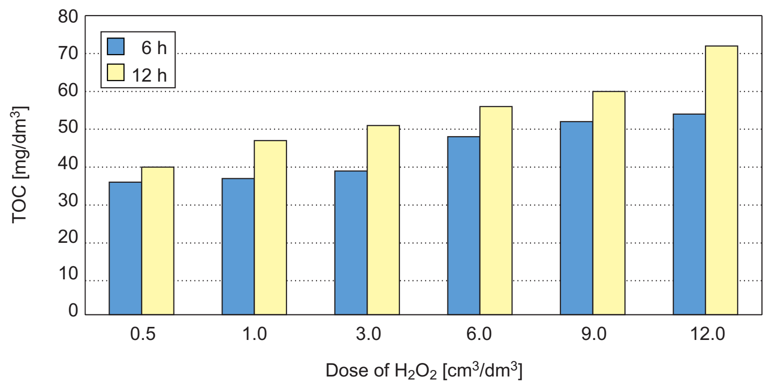

Fig. 11. Changes of the total organic carbon value of excess sludge modified by hydrogen peroxide

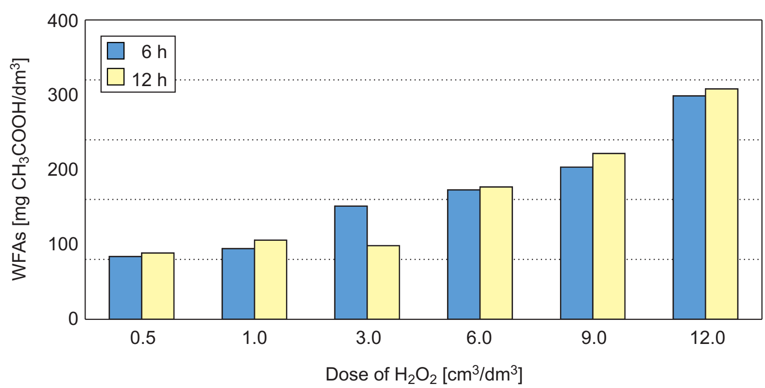

Fig. 12. Changes of the volatile fatty acids concentration of excess sludge modified by hydrogen peroxide

respectively 512 and 523 mg CH_3COOH/dm^3 was observed for the highest dose of the reagent 12.0 g KOH/dm^3 of sludge, and two periods of preparation. The lowest concentration of KOH was reported for a dose of 0.5 g KOH/dm^3 and was, respectively, for 6 h – 314 mg CH_3COOH/dm^3 and 24 h – 342 mg CH_3COOH/dm^3. Figure 5 and 6 show the changes in disintegration degree of excess sludge modified with hydrochloric acid.

The highest values of the disintegration degree of excess sludge treated with hydrochloric acid 42 and 56% was observed at a dose of 12.0 cm^3 of HCl/dm^3 of sludge and 6 and 24 hours. Figure 7 and 8 show the changes in the TOC value and volatile fatty acids concentration of excess sludge modified by hydrochloric acid.

From the Fig. 3 it can be seen that the value of TOC increased with the increase of reagent dose. The lowest value was observed for a dose of 0.5 cm^3 of HCl/dm^3 *ie* for the time 6 h – 71 mg C/dm^3 and for the 24 h – 74 mg C/dm^3. The value obtained for the highest dose of 12.0 cm^3 of HCl/dm^3 was for a time of 6 h – 195 mg C/dm^3 and for 24 h – 215 mg C/dm^3. The highest concentration of VFAs, respectively 454 and 461 mg CH_3COOH/dm^3 was observed for the highest dose of the reagent 12.0 cm^3 HCl/dm^3 of sludge, and both period of preparation. The lowest concentration of VFAs was reported for a dose of 0.5 g KOH/dm^3 and was, respectively, for 6 h – 212 mg CH_3COOH/dm^3 and for 24 h – 221 mg CH_3COOH/dm^3.

Figure 9 and 10 show the changes in the degree of disintegration of excess sludge modified with hydrogen peroxide.

The highest degree of disintegration of excess sludge chemically modified by hydrogen peroxide 22% was reported for a dose of 12.0 cm^3 H_2O_2/dm^3 of sludge and pretreatment time equal 6 h. For 24 h the highest value of disintegration degree equal 32% was reported for a dose of 3.0 cm^3 H_2O_2/dm^3 of sludge. Figure 11 and 12 show the changes in the TOC value and volatile fatty acids concentration of excess sludge modified by hydrogen peroxide.

Increase the value of TOC was observed with increasing doses of a reagent. For the disintegration time of 6 h and dose of 0.5 cm^3 H_2O_2/ dm^3 recorded lowest value of TOC 36 mg C/dm^3, while for a 24 h a value of 40 mg C/dm^3. The highest value of TOC 54 and 72 mg C/dm^3, respectively for the time 6 and 24 h obtained for 12.0 cm^3 H_2O_2/dm^3 of sludge. The highest concentration of VFA, respectively 373 and 385 mg CH_3COOH/dm^3 was observed for the highest dose of the reagent 12.0 cm^3 H_2O_2/dm^3 of sludge, and both period of preparation. The lowest concentration was observed at a dose of 0.5 cm^3 H_2O_2/dm^3, and was, respectively, for 6 h – 105 mg CH_3COOH/dm^3 and for 24 h – 111 mg CH_3COOH/dm^3.

Conclusions

Based on the results obtained the following conclusions were drawn:

1. By subjecting the excess sludge chemical disintegration was observed with increasing doses of reagent increase the value of the degree of disintegration, increase the value of TOC and concentration of VFAs. Increasing the value of these indicators may indicate an increase in the biodegradability of sludge, which will be confirmed in further studies regarding the process of anaerobic stabilization of modified sludge.

2. It has been found that the extension of the modification time for 24 h in the case of the concentration of VFAs did not significantly influence the value of the test indicator.

3. The highest TOC value of chemically modified excess sludge 2150 mg C/dm^3 was reported for a dose of 12.0 g KOH/dm^3 and preparation time 24 h.

4. The highest degree of disintegration of excess sludge modified chemically, *ie* 79% were obtained with a dose of 6.0 g KOH/dm^3 and preparation time 6 h.

5. The highest concentration of VFAs 523 mg CH_3COOH/dm^3 of sludge obtained for excess sludge pretreatment at 12.0 cm^3/dm^3 for 24 h. For the time 6 h a concentration of VFAs was 512 mg CH_3COOH/dm^3.

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Abstrakt: Efektywność przemiany substancji organicznych zawartych w osadach nadmiernych do formy rozpuszczonej jest traktowana jako ważny czynnik limitujący przebieg procesu stabilizacji beztlenowej. Bezpośrednim efektem procesu lizy zachodzącego w dezintegrowanych osadach jest wzrost wartości ogólnego węgla organicznego (OWO) oraz korelujący z nim wzrost wartości stężenia lotnych kwasów tłuszczwych (LKT). Ogólny węgiel organiczny jest wskaźnikiem zawartości w cieczy nadosadowej całkowitego węgla organicznego w formie rozpuszczonej (RWO) i zawieszonej (ZWO). Wraz z zachodzącym w wyniku procesów biochemicznych wzrostem stopniem rozkładu substancji organicznych zawartych w osadach maleje wartość ilorazu ChZT do OWO. Celem badań było określenie wpływu procesu dezintegracji osadów nadmiernych na zmiany wartości całkowitej węgla organicznego. Proces chemicznej dezintegracji osadów nadmiernych prowadzono, stosując wybrane reagenty kwaśne (HCl), zasadowe (KOH) i utleniające (H_2O_2). Modyfikację przeprowadzono w temperaturze pokojowej w ciągu 6 i 24 godzin. Podczas procesu dezintegracji osadów nadmiernych odnotowano wzrost wartości ogólnego węgla organicznego, a także stężenia lotnych kwasów tłuszczowych, co potwierdziło podatność preparowanych osadów na biodegradację. Najwyższą wartość OWO 2150 mg C/dm^3 uzyskano w przypadku chemicznej dezintegracji wodorotlenkiem potasu w dawce 12 g/dm^3 i w czasie preparowania 24 h. Dla podanych powyżej warunków preparowania uzyskano stężenie LKT, wynoszące 523 mg CH_3COOH/dm^3.

Słowa kluczowe: osady nadmierne, ogólny węgiel organiczny (OWO), stopień dezintegracji, lotne kwasy tłuszczowe (LKT)