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FERMENTATION OF SEWAGE SLUDGE WITH INCREASED CONCENTRATION OF HEAVY METAL IONS

Abstract. In the present work sewage sludge originating from municipal, mechanical-biological wastewater treatment plant was used. In the first stage, the hydrolysis of the mixture of primary and excess sludge was carried out at the temperature of 55°C for 4 days. In the second stage, after the additional insertion of the fermenting sludge mesophilic fermentation was carried out at 37°C for 14 days. The addition of approximately 5 mg/dm^3 of each copper, nickel, and lead ions and of approximately 1 mg/dm^3 of cadmium ions did not disturb the process of sewage sludge mesophilic fermentation. The comparable biogas yields were obtained during the stabilization of both sludges enriched with metals and not enriched. The level of organic matter degradation was at the level of 33% for both sludges (reference and with the metal addition) after the fermentation process.
The low level of the metal ion concentrations in the liquid phase was recorded for fermentation process. The metals were bound to the solid phase of sludge. After the fermentation the highest increase of copper, cadmium, zinc, and chromium content was observed in the organic-sulfide fraction, while of lead in the residual fraction. In case of nickel both fractions: organic-sulfide and exchangeable-carbonate were enriched.

Keywords: heavy metals, fermentation, sewage sludge, sequential extraction.

INTRODUCTION

The modification of sewage sludge fermentation process focuses on several areas, e.g.: searching for more efficient biogas production methods, the improvement of the degree of organic substances decomposition, but also searching for possibilities of shortening the time of the process duration and hygienization of sludges without the use of chemicals. Those aims could be achieved by the use of, inter alia, thermophilic fermentation or the application of two-stage process: thermophilic-mesophilic [3, 8, 11, 12].

Heavy metals in sewage sludge can occur in different forms: as precipitated on sludge flocks, as soluble forms and biopolymers, or as soluble forms accumulated in microorganism cells. Toxicity of the metals depends on their concentration and their chemical forms, which can change during anaerobic fermentation, but also on pH and temperature. The most toxic are metals in their soluble forms, the least toxic are those metals which are in the form of precipitated sludge.

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The limiting value of heavy metal concentration in sludge that can lower the efficiency or prohibit the fermentation process is reported by many authors in a wide range between some to hundreds mg in 1 dm^3. It has been reported that the inhibiting concentration of zinc, copper, nickel, lead, and cadmium in the liquid phase of the sewage sludge equals to 3÷100; 5÷100; 50÷200; 8÷30; 70 mg/dm^3 [7, 10]. It has been proved that when methanogens loose their activity under harmful influence of different compounds, they can still survive even long time in those unfavorable conditions and are able to continue degradation of organic matter afterwards.

During fermentation, the decomposition of the organic substances results in the increase of heavy metals concentrations in the dry matter of stabilized sewage sludge. It is however crucial not only to determine the total heavy metal concentrations in stabilized sludges but most of all to determine their chemical forms of occurrence; since these forms determine their mobility and bioavailability [6, 13].

The aim of this work is to study the influence of heavy metal ions concentration on the mesophilic fermentation of sewage sludge, preceded by the biochemical hydrolysis of the sludge under thermophilic conditions. For the purpose the metal ions were added to the sewage sludge sample and the results were compared with the reference culture – the sludge without metal addition. The fermentation progress was evaluated based on the measurements of biogas quantity and composition but also based on a decrease of organic matter content in the sludge after fermentation. Metal speciation analysis was performed, and revealed some changes in the chemical forms of the metals during the stabilization process of sludge.

MATERIAL AND METHODS

For the experiments the following types of sludges have been used: the mixture of primary and excess sludge (with a ratio of about 4:1) and fermenting sludge. Samples were collected at municipal mechanical-biological wastewater treatment plant in Silesia Voivodeship.

The first stage of the performed experiments was to hydrolyze the mixture of primary and excess sludge at 55°C in closed 550 cm^3 bioreactors for 4 days. The bioreactors filled with 350 cm^3 of the sludge mixture were placed in a thermostat facilitating the proper temperature. In the second stage, the thermophilically hydrolyzed sludge was mixed with fermenting sludge in the volumetric ratio of 2:1 and was loaded again into the bioreactors. Next, into each bioreactor 1 cm^3 of the solution with the metal ions was introduced. In order to enrich the sludge with heavy metals, the solution of 100 cm^3 doubly deionised water and 0.28 gram of $Pb(NO_3)_2$, 0.665 gram of $Cu(NO_3)_2 \cdot 3H_2O$, 0.709 gram of $NiCl_2 \cdot 6H_2O$, and 0.096 gram of $Cd(NO_3)_2 \cdot 4H_2O$ was used. The concentration of each introduced metal: copper, nickel, lead was approximately 5 mg/dm^3 of sludge, whereas, the concentration of cadmium was at the level of 1 mg/dm^3. After the air was removed from the bioreactors, mesophilic methanogenesis was forced at 37°C for 14 days. The fermenting sludge was inoculated in order to introduce the sufficiently high quantity of mesophilic methanogenic microorganisms. The metal solution was not introduced into the reference culture.

The manometric measurement of the amount of the produced biogas was carried out at 24-hour intervals. The composition of biogas (content of CH_4 and CO_2) was measured every 48 hours with the use of gas chromatography (gas chromatograph with TCD detector, model Agilent GC 6890, Agilent Technologies).

For the sludge before the thermophilic-mesophilic fermentation and during the fermentation at the specified intervals the following parameters were determined: hydration, total solids, volatile and fixed solids (organic and mineral substances) – gravimetric methods, pH – potentiometrically, alkalinity – titration method, volatile fatty acids – distillation method, total organic carbon TOC – infrared spectrometry (carbon analyzer multi N-C, Analytik Jena), copper, nickel, cadmium, lead, zinc and chromium – atomic absorption spectrometry (spectrometer novAA 400, Analytik Jena). Three repetitions were conducted for every analysis.

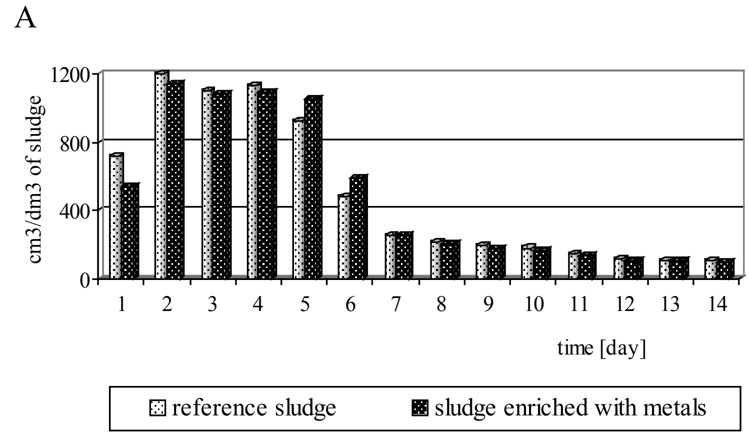
Sequential extraction has been carried out according to BCR procedure (Table 1) in order to quantify forms of occurrence of heavy metals in sludges. The preparation of the necessary reagents and the extraction procedure was carried out according to [9].

Table 1. Scheme of analytic procedures in sequential extraction
Tabela 1. Procedura ekstrakcji sekwencyjnej

Fraction	Sequential extraction procedure per 1g of dry matter sludge	Forms of metals
I	40 cm^3 0.11 M CH_3COOH, temp. 22^0C, shaking for 16 h	Exchangeable and bounded with carbonates
II	40 cm^3 0.5 M $NH_2OH \cdot HCl$ (pH=2), temp. 22^0C, shaking for 16 h	Bounded with Fe and Mn oxides
III	10 cm^3 8.8 M H_2O_2 (pH=2-3), temp. 22^0C, 1 h 10 cm^3 8.8 M H_2O_2, temp. 85^0C, 2 h 50 cm^3 1 M CH_3COONH_4 (pH=2 with HNO_3), temp. 22^0C, shaking for 16 h	Bound with organic matter, sulfides
IV	6 cm^3 65% HNO_3 + 2 cm^3 36% HCl, temp. 100^0C, 2 h	Residual

RESULTS AND DISCUSSION

The amount of the gas generated in the consecutive days of the mesophilic fermentation process of the previously biochemically hydrolyzed sludge, enriched with the metal ions and not enriched (the reference system), in respect to 1 dm^3 of sludge is shown in Figure 1a. The total values of biogas yields are shown in Figure 1b. On the first day of the fermentation process more biogas was generated in the reference bioreactors than in the bioreactors with the addition of the metal ions. During the next three days the quantity of produced biogas in both systems - with the metal ions and without – was comparable. However, on the fifth and sixth day more biogas was generated in the bioreactors with the addition of the metal ions. In total 6890 cm^3 of biogas from 1 dm^3 of sludge was produced in the reference system, whereas, 6770 cm^3 of biogas was produced in the system enriched with the metals.



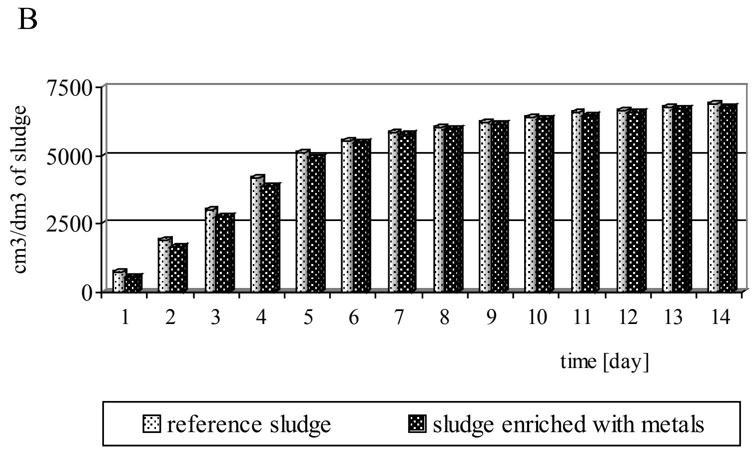


Fig. 1. Yields of biogas produced in the consecutive days (A) of fermentation performed, and cumulative yields of biogas (B)

Rys. 1. Ilość biogazu powstającego w kolejnych dobach (A) prowadzonej fermentacji oraz sumaryczna ilość biogazu (B)

It was calculated that the specific biogas yields for both systems: reference and with additional amount the metal ions, were 1.15 and 1.12 dm^3 from 1 g of the removed dry organic matter of sludge, respectively. The content of methane in the biogas, except of the first day, generated in the reference reactors was at the level of 61÷70%, while for the reactors filled with the sludges enriched with the metal ions it was 62÷69% – Table 2.

Table 2. The content of CH_4 and CO_2 in the biogas
Tabela 2. Zawartość CH_4 i CO_2 w biogazie

Sludge	Content %	Mesophilic fermentation					
		1st day	3rd day	5th day	7th day	10th day	14th day
Reference	CH_4	33.4	62.6	69.9	69.2	64.3	60.8
	CO_2	25.6	30.8	29.4	30.1	30.8	32.4
Enriched with metals	CH_4	28.5	62.8	68.9	68.2	63.8	61.7
	CO_2	23.2	29.7	28.3	31.0	30.5	31.9

The values of the chosen physical-chemical indexes of sludges are shown in Table 3. The values for the sludges before and after hydrolysis are there reported, but also, the values for the sludges before the mesophilic fermentation and on the 14th day of the fermentation for both systems with the metal addition and no addition (reference) are shown.

Table 3. The values of chosen indexes of the mixture of primary and excess sludge, before and after thermophilic hydrolysis process, and of fermenting sludge (inoculum), and of sludges before and after mesophilic fermentation
Tabela 3. Wartości wybranych wskaźników mieszaniny osadów wstępnego i nadmiernego, przed i po hydrolizie termofilowej, osadu fermentującego (inokulum) oraz osadów przed i po fermentacji mezofilowej

Indexes	Unit	Hydrolysis sludge		Fermenting sludge	Before fermentation	After fermentation	
		At the beginning	After			reference sludge	sludge + metals
pH	-	6.79	7.50	7.95	7.78	7.96	7.97
Hydration	%	96.03	96.45	97.73	97.31	97.90	97.91
Total solids	g/dm^3	39.64	35.51	22.75	26.89	21.04	20.92
Volatile solids	g/ dm^3	30.32	26.64	14.37	18.39	12.39	12.36
	%	76.5	75.0	63.2	68.4	58.9	59.1
Fixed solids	g/ dm^3	9.32	8.87	8.38	8.50	8.65	8.56
	%	23.5	25.0	36.8	31.4	41.1	40.9
TOC	mgC/ dm^3	876	3590	618	1590	536	570
Alkalinity	mgCa-CO_3/ dm^3	730	2250	3750	3230	4480	4330
Volatile fatty acids	mgCH_3COOH/ dm^3	1110	4200	189	1543	189	270

Thermophilic hydrolysis process resulted in an increase of organic matter quantity in the liquid phase of the sludge. The four-fold increase of the total organic carbon (TOC) concentration was observed (from 876 to 3590 mgC/dm^3), whereas more then the 3-fold increase of volatile acids took place (from 1110 to 4200 mgCH_3COOH/dm^3). The alkalinity of the liquid also increased, from 730 to 2250 mg$CaCO_3$/dm^3.

After mixing of thermally hydrolyzed sludges (primary and excess) with fermenting sludge the concentration of organic carbon in the liquid phase was about 1590 mgC/dm^3. During the mesophilic fermentation, TOC concentration in the liquid phase of sludge decreased from 1590 to 536 mgC/dm^3 in the reference system and for the system with the metal ions addition decreased to 570 mgC/dm^3. This indicates that easily available organic substrate was consumed during the fermentation process. The dry organic matter decreased from 18.39 to 12.39 g/dm^3, which means 32.6% reduction of organic matter in the sludge. During the fermentation of the sludge enriched with the metal ions the comparable organic matter degradation was achieved (32.8%).

During the reference fermentation the concentrations of analyzed metals: nickel, copper, lead, cadmium, zinc and chromium were at the level of 0.1÷0.07; 0.11÷0.05; 0.08÷0.06; 0.016÷0.01; 0.48÷0.27 and 0.08÷0.03 mg/dm^3, respectively (Table 4).

Table 4. The concentrations of heavy metals ions in the sludge liquid on the chosen days of fermentation (reference system)

Tabela 4. Stężenia jonów metali ciężkich w cieczy osadowej w wybranych dobach fermentacji osadów w układzie kontrolnym

Metal	Unit	Before fermentation	During fermentation					
			1st day	3rd day	5th day	7th day	10th day	14th day
Nickel	mg/dm^3	0.154	0.098	0.078	0.074	0.076	0.066	0.070
Copper	mg/dm^3	0.110	0.106	0.086	0.057	0.048	0.052	0.047
Lead	mg/dm^3	0.082	0.078	0.077	0.068	0.063	0.071	0.076
Cadmium	mg/dm^3	0.012	0.014	0.016	0.010	0.009	0.012	0.011
Zinc	mg/dm^3	0.647	0.480	0.415	0.334	0.296	0.304	0.268
Chromium	mg/dm^3	0.106	0.084	0.077	0.055	0.041	0.048	0.032

Also during the fermentation of the sludge enriched with the metals, the concentrations of the metal ions in liquid phase of the sludge were at a low level – Table 5. The concentration of nickel, copper, lead, cadmium, zinc and chromium already after the first day of the fermentation process was 0.20, 0.24, 0.23, 0.04; 0.52; 0.1 mg/dm^3, respectively and the values were decreasing until the seventh day of the process. It is in agreement with the observation that heavy metal ions may precipitate in the form of sulfides as reported in literature [7, 10]. It resulted in a non-disturbed fermentation process.

Table 5. The concentrations of heavy metals ions in the sludge liquid on the chosen days of fermentation of sludge enriched with metals ions
Tabela 5. Stężenia jonów metali ciężkich w cieczy osadowej w wybranych dobach fermentacji osadów wzbogaconych jonami metali

Metal	Unit	Before fermentation	During fermentation					
			1st day	3rd day	5th day	7th day	10th day	14th day
Nickel	mg/dm^3	4.93	0.202	0.134	0.116	0.107	0.104	0.103
Copper	mg/dm^3	4.81	0.236	0.207	0.165	0.143	0.130	0.122
Lead	mg/dm^3	5.17	0.225	0.196	0.131	0.118	0.128	0.126
Cadmium	mg/dm^3	0.942	0.037	0.024	0.020	0.020	0.022	0.017
Zinc	mg/dm^3	0.647	0.518	0.427	0.380	0.315	0.318	0.261
Chromium	mg/dm^3	0.106	0.091	0.082	0.046	0.033	0.033	0.027

The averaged concentrations of heavy metals in the sludge fractions, before and after mesophilic fermentation process, together with standard deviations are shown in Table 6. The percentage distribution of the metals over the stabilized sludge fractions is shown in Figure 2.

The highest concentration of nickel in the sludges before the stabilization was present in the exchangeable-carbonate and organic-sulfide fractions, on the other hand the smallest concentration was found in the residual fraction. The fermentation process resulted in nickel enrichment mainly in the exchangeable-carbonate fraction, and in smaller enrichment in the organic-sulfide fraction of the sludges. The distribution of nickel over the fractions: exchangeable-carbonate, iron and manganese oxides, organic-sulfide and residual was 48, 18, 31, and 3%, respectively. The enrichment in nickel of all fractions during fermentation process was already reported in previous research [4, 14]. Copper was bound to organic matter and sulfides (92%) before fermentation and this fraction was enriched after the fermentation process. Copper shows high affinity for organic compounds [2].

Before the stabilization of the sludge, lead was present in the organic-sulfide fraction and residual fraction. The increase of lead concentration after fermentation was observed in the residual fraction. The high content of lead in the residual fraction of fermented sludges was already reported by Fuentes et al. [5, 6]. In case of cadmium the increase was observed in the organic-sulfide fraction in the stabilized sludge. The distribution of cadmium in this fraction was 63%, while, 24% of total cadmium content was found together with iron and manganese oxides.

Table 6. Heavy metals speciation in sludge before and after fermentation
Tabela 6. Specjacja metali ciężkich w osadach przed i po fermentacji

Metal	Unit	Fraction	Before fermentation	After 14th day of fermentation	
				reference sludge	sludge enriched with metals
Nickel	mg/kg d.m.	I	141.0 ± 4.5	197.0 ± 6.0	315.0 ± 11.0
		II	59.7 ± 1.4	72.6 ± 3.6	127.1 ± 6.4
		III	86.1 ± 3.6	123.5 ± 10.5	202.0 ± 8.8
		IV	8.4 ± 1.2	10.2 ± 0.5	15.6 ± 1.2
		∑	295.2	403.3	659.7
Copper	mg/kg d.m.	I	2.0 ± 0.5	2.1 ± 0.3	2.8 ± 0.2
		II	1.7 ± 0.2	1.3 ± 0.1	3.3 ± 0.2
		III	231.0 ± 8.0	286.0 ± 9.0	542.0 ± 14.0
		IV	15.6 ± 2.2	28.0 ± 1.3	39.2 ± 2.4
		∑	250.3	317.4	587.3
Lead	mg/kg d.m.	I	4.3 ± 0.4	5.1 ±0.1	4.6 ± 0.3
		II	2.7 ± 0.5	3.5 ± 0.2	3.4 ± 0.1
		III	29.4 ± 4.0	16.0 ± 1.8	27.1 ± 2.7
		IV	33.4 ± 6.5	62.0 ± 2.3	288.0 ± 5.6
		∑	69.8	86.6	323.1
Cadmium	mg/kg d.m.	I	0.32 ± 0.08	0.43 ± 0.04	2.11 ± 0.32
		II	0.78 ± 0.06	1.10 ± 0.18	18.6 ± 0.6
		III	2.07 ± 0.16	2.92 ± 0.24	28.9 ± 1.8
		IV	0.19 ± 0.04	0.20 ± 0.07	1.22 ± 0.20
		∑	3.36	4.65	50.8
Zinc	mg/kg d.m.	I	244.0 ± 12	171.0 ± 3	194.0 ± 2
		II	555.0 ± 16	678.0 ± 6	702.0 ± 5
		III	828.0 ± 27	1325.0 ± 14	1280.0 ± 11
		IV	112.0 ± 2	98.0 ± 8	121.0 ± 4
		∑	1739.0	2272.0	2297.0
Chromium	mg/kg d.m.	I	0.8 ± 0.1	0.5 ± 0.2	0.6 ± 0.2
		II	0.6 ± 0.1	0.4 ± 0.1	0.3 ± 0.1
		III	213.0 ± 7	259.0 ± 5	261.0 ± 3
		IV	3.5 ± 0.8	24.3 ± 2.2	17.0 ± 1
		∑	217.9	284.2	278.9

Fraction: I – exchangeable, and carbonates-bound, II – Fe/Mn oxides-bound, III – organic matter/sulfides-bound, IV – residual

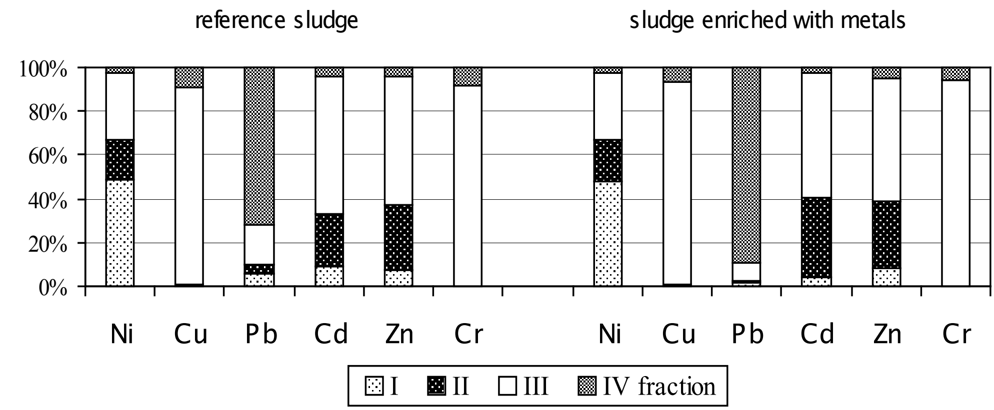


Fig. 2. Percentage of metals in fractions of sludge (I – exchangeable, and carbonate, II – Fe and Mn oxides, III – organic matter/sulfide, IV – residual) after fermentation

Rys. 2. Procentowa zawartość metali we frakcjach osadów (I – wymienna i węglanowa, II – tlenków Fe i Mn, III – organiczno-siarczkowa, IV –l' pozostałościowa) po fermentacji

Zinc was mainly found in the organic-sulfide and iron-manganese oxides fractions in sludge analyzed before fermentation. After the fermentation the decrease of content was observed for exchangeable and carbonates fraction, although organic-sulfide fraction and iron-manganese oxides fraction became enriched in the metal. After the fermentation 58% of total zinc content was present in the organic-sulfide fraction. The highest concentration of chromium was found in the organic-sulfide fraction (98%). After the fermentation the concentration of the metal increased in this very fraction. Much smaller increase was observed in the residual fraction of the sludges. It has been also reported in literature [1, 5, 6, 13] that copper and chromium were mainly bound to organic-sulfide fraction in the sewage sludge stabilized during anaerobic treatment.

During the fermentation of the sludge enriched with the addition of the following metals: copper, nickel, cadmium, and lead, it was confirmed that the dominant role in copper, and chromium bonding plays organic-sulfide fraction, and for lead the dominant is the residual fraction (the compounds insoluble in practice). All fractions became enriched with nickel, mostly the exchangeable-carbonate and the organic-sulfide fraction. In the case of cadmium, its concentration mainly increased in the organic-sulfide fraction, less in the hydrated iron and manganese oxides fraction.

CONCLUSIONS

The aim of this work is to study the influence of Cu, Ni, Cd, Pb ions concentration on the mesophilic fermentation of sewage sludge, preceded by the biochemical hydrolysis of the sludge under thermophilic conditions.

Based on the performed experiments the following conclusions can be drawn:

- The addition of approximately 5 mg/dm^3 of each Cu, Ni, and Pb ions and of approximately 1 mg/dm^3 of Cd ions did not disturb the process of sewage sludge fermentation. The comparable biogas yields were obtained during the stabilization of both sludges enriched with metals and not enriched. The level of organic matter degradation was at the level of 33% for both sludges after the fermentation process.
- The low level of the metal ion concentrations in the liquid phase was recorded for both fermentation processes: zinc below 0.6 mg/dm^3, the other metals below 0.3 mg/dm^3. The metals were bound to the solid phase of sludge.
- The fermentation process has not caused the accumulation of heavy metals (except Ni) in the mobile fractions of sludge.

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FERMENTACJA OSADÓW ŚCIEKOWYCH PRZY ZWIĘKSZONYM STĘŻENIU JONÓW METALI CIĘŻKICH

Streszczenie. W badaniach wykorzystano osady ściekowe, pochodzące z miejskiej, mechaniczno-biologicznej oczyszczalni ścieków. W pierwszym etapie przeprowadzono hydrolizę mieszaniny osadów wstępnego z nadmiernym w temp. 55°C w czasie 4 dób, w drugim etapie po wprowadzeniu dodatkowo osadu fermentującego fermentację mezofilową w temp. 37°C przez okres 14 dób. Wprowadzenie dodatkowych ilości jonów miedzi, niklu, ołowiu (ok. 5 mg/dm^3 każdego metalu) oraz kadmu ok. 1 mg/dm^3 nie zakłóciło przebiegu procesu fermentacji mezofilowej osadów ściekowych. Uzyskano podczas stabilizacji osadów wzbogaconych metalami i niewzbogaconych porównywalną ilość biogazu oraz stopień rozkładu substancji organicznych ok. 33%.
Stwierdzono niskie stężenia jonów metali w cieczach podczas prowadzenia fermentacji. Metale związane były w osadach. Po fermentacji najwyższy wzrost zawartości cynku, miedzi, kadmu i chromu stwierdzono we frakcji organiczno-siarczkowej, natomiast ołowiu we frakcji pozostałościowej osadów. W przypadku niklu wzbogaceniu uległa zarówno frakcja organiczno-siarczkowa, jak i wymienno-węglanowa.

Słowa kluczowe: metale ciężkie, fermentacja, osady ściekowe, ekstrakcja sekwencyjna.