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Effect of chemically conditioned FOG fraction on methane co-fermentation with excess sewage sludge with regard to heavy metals concentration

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ABSTRACT

The paper presents the results of research on chemical pretreatment of fats, oils, and grease fraction (FOG fraction) with Fenton's reagent (100 mg Fe^{2+} L^{-1}; 5,000 mg H_2O_2 L^{-1}). Chemical pretreatment was performed at ambient temperature for 30 min, at initial pH 3.0. It caused partial hydrolysis of FOGs. Pretreatment with Fenton's reagent was followed by methane co-fermentation of FOGs/excess sewage sludge mixtures. Advanced oxidation processes processing allowed for an increase in biogas production only at about 7% compared to the control sample (not treated FOG and the excess sludge mixture). Pretreatment with Fenton's reagent under conditions described above did not significantly affect the heavy metals. Co-fermentation had more influential impact on their content in the solid phase and supernatants. The results indicate that pretreatment with low doses of strong oxidants may be useful in improving FOGs processing in WWTPs, because it does not increase significantly leachability of heavy metals.

Keywords: Heavy metals; Grease; Oils; Fats; Methane digestion; Fenton's reagent

1. Introduction

Advanced oxidation processes (AOPs) are used both in wastewater treatment and in pretreatment of sewage sludge. One of the AOP methods used for chemical conditioning and pretreatment of sewage sludge is Fenton's reagent. It is a mixture of H_2O_2 and Fe^{2+}. Classical Fenton's reaction is performed in acidic environment (the optimal pH value is considered to be between 3 and 5). The simplified scheme of the process can be described as follows:

$$H_2O_2 + Fe^{2+} \rightarrow Fe^{3+} + OH^- + OH^{\bullet} \quad (1)$$

Hydroxyl radicals ($OH^{\bullet}$) which are generated during Fenton's reaction are strong chemical oxidants. They do not react selectively with most organic compounds present in the environment of the reaction. In fact, the mechanism of hydroxyl radicals formation is more complex [1].

In sewage sludge pretreatment, Fenton's reagent is used mainly to improve dewaterability of sewage sludge. Exemplary doses of H_2O_2 and Fe^{2+}, and other technological parameters of Fenton's reagent used in

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sewage sludge pretreatment are listed in Table 1. Based on the data from Table 1, it can be stated that the doses of Fenton's reagent used in sludge treatment technologies are in most cases at the level of one or more than 1000 mg L^{-1} or per 1 kg of sewage sludge. Time of process is relatively short (up to 90 min) and initial pH is usually about 3.0. Under optimal conditions, even over 90% reduction of capillary suction time and specific resistance can be achieved. Fenton's reagent and some other AOPs are also used for improving degradation of toxic organic micropollutants present in the sludge [7–11], in order to improve aerobic biodegradation of excess sludge [12] and speed-up hydrolysis of sewage sludge before further methane digestion [13]. AOPs are used mainly in pretreatment of excess sludge, since this kind of waste material contains cells of bacteria and other components which are less suitable for biodegradation than primary sludge [12].

Doses of Fenton's reagent applied to the sludge in order to increase the susceptibility of excess sludge for biodegradation were about 10 times lower than those used to improve dewaterability [12]. A decrease in pH during the process was observed. Aerobic degradation of sludge was improved (sludge was stable within 10 d), however, after this time, decomposition of sludge occurred which worsened sedimentation properties of the activated sludge.

Apart from excess sewage sludge, also fats, oils, and grease fraction (FOG) caught in WWTPs in grease traps is a substratum difficult to biodegrade. It is connected with the fact that greasy waste material contains not only lipids but also other organic and inorganic contaminants which may be resistant to

Table 1
Doses of H_2O_2 and Fe^{2+}, and other technological parameters of Fenton's reagent used in treatment and conditioning of sewage sludge

Organic waste	Purpose of the chemical conditioning	Dose of H_2O_2	Dose of Fe^{2+}	Fe^{2+}: H_2O_2	Other parameters of the process	Effects of chemical stabilization	Refs.
Waste activated sludge	Improving of dewatering properties	178 mg g_{VSS}^{-1}	211 mg g_{VSS}^{-1}	1.2	Initial pH 3.8	Reduction of capillary suction time of 98.25% because of microbial cells lysis as a result of sludge flocs disintegration	[2]
Waste activated sludge	Improving of dewatering properties and filterability	No data	No data	8:1	pH 2.5–3.0; t = 2–3 h, T = 60–70°C	Up to 81% improvement of filterability, 12% improvement of dewaterability (measured as CST [capillary suction time])	[3]
Excess activated sludge	Improving of dewatering properties	3,000 mg L^{-1}	6,000 mg L^{-1}	2:1	At 30°C, t = 30 min; initial pH 3.0	Decrease of specific resistance in 90%	[4]
Excess activated sludge	Improving of dewatering properties of the sludge	2,000–6,000 mg L^{-1}	1,000–6,000 mg L^{-1}	1:1–6:1	Initial pH < 3.0; t = 5–45 min	93% decrease of specific resistance of the sludge; 43% decrease of capillary suction time at dose of Fe^{2+} equal to 5,000 mg L^{-1} and H_2O_2 equal to 6,000 mg L^{-1}	[5]
Concentrated sewage sludge	Improving of dewatering properties of sewage sludge, improving degradation of organic solids	5,000–50,000 mg kg dm^{-1}	1,670 mg kg dm^{-1}	1:0.3–1:0.03	Initial pH 3; t = 60–90 min	Optimal dose 1,670 mg Fe^{2+} kg dm^{-1} and 1,670 mg H_2O_2·kg dm^{-1}. Increase of total nitrogen in supernatant, decrease of COD and BOD of supernatant	[6]

iodegradation [9]. When grease from the traps undergoes methane digestion, the contact time of at least 8–10 d is required before it begins to break down [13]. It means that if COD reduction of supernatant or biogas production must be maximized, degradation of the grease scum can be a rate-limiting step [14]. Also Sincero and Sincero [15] emphasized that grease is one of the most stable organic compounds which is not easily decomposed by micro-organisms. Despite the problems with the rate of biodegradability, fats present in wastewater are one of the main sources of biogas. The biogas production rate (BPR) for fats and grease is very high (961 m^3 of biogas per ton) compared to other substrates (e.g. BPR value for food scraps is equal to 265 m^3 of biogas per ton; for chicken manure only 80 m^3 of biogas per ton) [16]. That is the reason why grease is a desired substratum in the methane digestion process. Technologies that improve the biodegradability of this waste material are thus of great interest. One of the promising technologies seems to be chemical pretreatment. Chemical pretreatment methods based on pH value of the reaction environment can be divided into alkaline and acidic. Both pretreatment technologies liberate glycerol and fatty acids from fats. In alkaline environment, however, soaps are generated, which are equally resistant to degradation [15]. Under acidic conditions, soap generation does not occur [17], and as a result it seems they are a better solution for pretreatment of FOGs separated during mechanical treatment of municipal wastewater. The experiments on the use of Fenton's reagent on FOGs degradation improvement have not been carried out up to now. As the process is conducted in acidic environment, it can potentially cause leaching of heavy metals from the solid phase to the supernatant.

The aim of the present investigation was to evaluate the effect of chemical pretreatment of FOGs with Fenton's reagent on heavy metals during methane co-digestion of FOGs and excess sewage sludge under mesophilic conditions.

2. Materials and methods

2.1. Sampling procedure

FOG fraction caught in a grease trap of wastewater treatment plant in southern Poland was used in the study. The random samples of the FOGs were taken three times directly from an aerated separator. They were homogenized with MPW-302 homogenizer (MPW, Poland) before further treatment. Homogenized samples of the FOGs underwent pretreatment with Fenton's reagent then.

2.2. Chemical pretreatment of FOGs with Fenton's reagent

Chemical pretreatment of FOGs was carried out on a laboratory scale at ambient temperature from 19 to 20°C. Fe^{2+} was introduced to the samples as $FeSO_4{\cdot}7H_2O$ solution (10%); 30% H_2O_2 was used simultaneously. The dose of Fe^{2+} was equal to 100 mg L^{-1}, whereas H_2O_2 to 5,000 mg L^{-1}. The doses of reagents were chosen based on preliminary experiments. The initial pH was equal to 3.0 and it was achieved by adding concentrated H_2SO_4. Chemical pretreatment lasted for 30 min. The samples were continuously stirred on a shaker with horizontal motion. Chemically stabilized FOGs samples were mixed with excess sludge (1:3 v/v) and further co-digested under mesophilic conditions (see Section 2.3).

2.3. Methane co-digestion of chemically stabilized grease–raw sewage sludge mixtures

Methane co-digestion of chemically stabilized grease and excess sewage sludge was carried out in closed bioreactors under mesophilic conditions (37°C) for 20 d. The bioreactors were filled with one part of digested sewage sludge (inoculum of methanogens) and four parts of chemically pretreated FOG–excess sewage sludge mixture (substratum). The control sample (a mixture of not chemically treated FOGs and excess sludge) was run simultaneously. During the process, each 24 h measurements of generated biogas were performed using a manometric method. The composition of biogas (CH_4 and CO_2) was analyzed every 24 h with a chromatograph equipped with TCD detector, model Agilent GC 6890, and Agilent technologies.

2.4. Analytical methods

In the samples of FOGs and sewage sludge or their mixtures of dry matter or supernatants, the following physicochemical parameters were determined: pH—potentiometrically; chemical oxygen demand (COD)—by a standard dichromate method; dry matter—by a mass balance method (drying at 105°C); volatile suspended solids—by loss of ignition (ignition at 550°C); and lipids—by Soxhlet extraction with petroleum ether. All analyses were done according to Standard Methods [18].

The total content of selected heavy metal ions (Zn, Cu, Cd, Ni, and Pb) in dry matter and supernatants was analyzed after concentrated HNO_3 and HCl (1+3—aqua regia) digestion. The analyses were done before and after chemical pretreatment of FOGs. Chemically pretreated FOGs were mixed with excess

sewage sludge and after that, they underwent methane co-digestion. Heavy metals were also analyzed in samples before and after methane co-digestion. The content of metals was detected by an atomic absorption spectrometry method, using a spectrometer novAA 400, Analytic Jena, Germany.

Sequential extraction of heavy metals in the samples was performed with modified Tessier procedure [19]. The details of extraction procedure are given in Table 2. To analyze the fractions of heavy metals, 1 g of an air-dried sample was used.

3. Results and discussion

The physicochemical characteristics of FOG fraction used in the study and changes of its properties after chemical treatment are given in Table 3. Before chemical pretreatment, FOG´ pH was slightly acidic. COD of supernatant was equal to 2,500–3,000 mg O_2 L^{-1}, which is typical for this kind of waste material [20]. The greasy waste collected from grease trap contained only 38–41% of lipids. According to the literature, [9] it is also typical for this kind of waste since FOG fraction collected in grease trips represents only 10% of the lipids brought to the wastewater treatment plant. FOGs obtained in aerated separators are usually difficult to degrade by micro-organisms. They are also polluted by other organic and inorganic contaminants, which is visible when we analyze the volatile suspended solids content in FOGs. In our study, it is in the range of 55–61% and it means that grease waste used in the study contained 39–45% of inorganic solids. The total concentration of five analyzed heavy metals in FOGs was between 1,967.2 and 2,063.7 mg kg^{-1}. The most abundant heavy metal compound was zinc, followed by copper and nickel. The least abundant compound was cadmium. Lead concentration was only slightly higher than cadmium one. Zn was also the most abundant pollutant in FOG supernatant. The least abundant ones were Cu and Ni. Compared to the concentrations of heavy metals in other waste materials separated during wastewater treatment, it can be stated that they were at comparable levels to the ones present in sewage sludge [19,21], however, concentrations of individual heavy metal ions in various sewage sludge are very variable [21].

Since FOG fraction separated during mechanical treatment of wastewater may be resistant for microbial decomposition in our research work, it underwent chemical stabilization with Fenton's reagent. The doses of the reagent were lower than those used for improving dewatering properties by other authors [3–6], because we intended to achieve only partial decomposition of organic matter in order to make the greasy wastes more susceptible for anaerobic biodegradation.

Chemical pretreatment of grease with Fenton's reagent caused a decrease in pH accompanied by COD increase. COD increase was connected with a decrease in dry matter (about 2–4% decrease). Due to those changes, it was expected that after chemical pretreatment, FOGs would be more suitable for biodegradation than untreated greasy wastes. The total concentration of heavy metals in FOG fraction dry matter after chemical pretreatment increased slightly, which compared to the 2–4% decrease in the d.m. content indicates that AOPs treatment only slightly affected individual heavy metals concentration. An increase in heavy metal concentration in sewage sludge was connected with the fact that the amount of metals in the sample was stable (they do not evaporate and do not undergo biological transformations), whereas the part of dry matter was degraded by micro-organisms. The constant value of heavy metals compared to the lower amount of dry matter caused a

Table 2
Scheme of extraction procedure of heavy metals in grease–sewage sludge mixtures

Fraction of heavy metals	Characteristics of the fraction	Analytical procedure
I	Exchangeable metals	10 mL 1 M $MgCl_2$; pH 7; shaking for 1 h at ambient temperature
II	Bounded with carbonates	20 mL 1 M CH_3COONa, acidification to pH 5; shaking for 5 h at ambient temperature
III	Bounded with Fe and Mn oxides	20 mL 0.04 M $NH_2OH{\cdot}HCl$ in 25% (v/v) CH_3COOH for 6 h at 95°C
IV	Bounded to organic matter and sulfates	5 mL 0.02 M HNO_3 + 5 mL 30% H_2O_2; pH 2; shaking for 2 h at 85°C followed by adding 5 mL 30% H_2O_2 (pH 2; shaking for 3 h at 85°C) and 10 mL 3.2 M CH_3COONH_4 in 20% (v/v) HNO_3 (at ambient temperature for 0.5 h)
V	Residual	3 mL 10 M HNO_3 + 3 times 2 mL 30% H_2O_2; shaking for 1 h, at temperature of boiling

Table 3
Selected physicochemical properties of FOGs fraction before and after chemical pretreatment with Fenton's reagent

Parameter	Unit	Value			
		FOG before chemical pretreatment		FOG after chemical pretreatment with 100 mg Fe^{2+} L^{-1} and 5,000 mg of H_2O_2 L^{-1}	
pH	–	6.5–6.7		5.9–6.0	
COD	$mgO_2\,L^{-1}$	2,500–3,000		2,700–3,300	
Dry matter	$g\,L^{-1}$	72.5–81.5		69.5–79.0	
Volatile suspended solids	% of dry matter	55–61		51–57	
Lipids	% of dry matter	38–41		35–38	
Heavy metal		Bounded to the solids, $mg\,kg^{-1}$	In supernatant, $mg\,L^{-1}$	Bounded to the solids, $mg\,kg^{-1}$	In supernatant, $mg\,L^{-1}$
Pb		4.1–6.5	0.02–0.08	3.9–6.1	0.02–0.09
Zn		1,750–1,800	0.21–0.35	1,820–1,950	0.25–0.65
Cu		180–210	0.05–0.08	180–185	0.06–0.10
Cd		3.1–5.2	0.011–0.032	3.0–4.8	0.014–0.035
Ni		30–42	0.10–0.15	25–38	0.15–0.18

false increase in the concentration of these inorganic micropollutants. Cadmium and nickel concentration in FOGs decreased. That suggests that they were the most leachable compounds. Cu was at the similar level as in not-treated sludge. Zn concentration increased to 1,820–1,950 mg kg d.m.$^{-1}$.

Simultaneously, it was expected that heavy metals in the samples pretreated with Fenton's reagent would be more leachable than untreated ones. In order to confirm that thesis, chemically pretreated grease was mixed with excess sludge from WWTP. The samples prepared in this way were mixed with digested sewage sludge to inoculate methanogens. Methane co-digestion of the sample which contained a mixture of not chemically pretreated FOGs and excess sludge (also inoculated with digested sewage sludge) was running simultaneously (the control sample). The results obtained during methane co-digestion experiment are summarized in Table 4. Biogas potentials of chemically pretreated and control samples are presented in Fig. 1.

Both control and FOG/excess samples after co-digestion can be classified as well as digested ones. Chemical stabilization of FOG fraction did not affect significantly the removal of organic compounds of the solid phase. As can be seen from Fig. 1, it affected, however, production of biogas. In the samples which contained chemically pretreated FOGs about 7% higher of the total biogas production was achieved.

An increase in biogas generation was especially visible over the first seven days of co-digestion. It confirmed that pretreatment with Fenton's reagent caused hydrolysis of the part of FOG fraction separated from wastewater. The total biogas potential was equal to 402 and 428 mL g_{VSS}^{-1}, for pretreated and control samples, respectively. It was lower than the one achieved for fats [16], which was connected with fact that FOGs contain many non-biodegradable contaminants. Both

Table 4
Changes in selected physicochemical properties of FOGs/excess sewage sludge mixtures during methane co-digestion of chemically pretreated and control samples

Parameter	Unit	Value			
		Before co-digestion		After co-digestion	
		Control sample	FOG/excess sludge	Control sample	FOG/excess sludge
pH	–	7.1–7.2	6.7–6.9	7.2–7.5	7.3–7.5
COD	$mg\,O_2\,L^{-1}$	550–680	1,205–1,840	320–350	300–340
Dry matter	$g\,L^{-1}$	20.3–22.8	42.3–45.8	14.8–15.2	34.6–35.5
Volatile suspended solids	% of dry matter	66–68	63–65	41–45	39–42

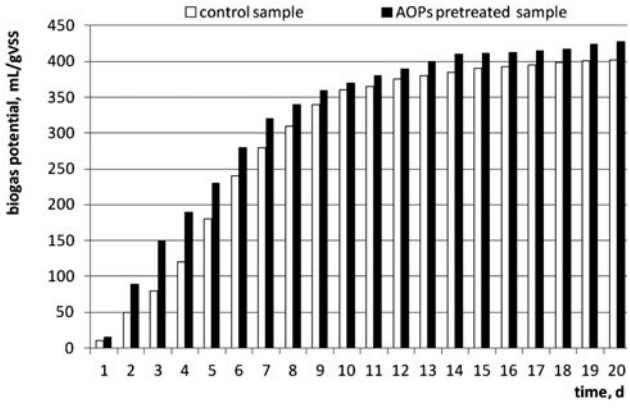

Fig. 1. Biogas potentials of chemically pretreated and control samples during co-digestion of FOGs and excess sewage sludge.

in the samples which contained chemically pretreated FOGs and in the control samples, CH_4 content was at the level of 65–70%. It means that chemical pretreatment of grease did not change significantly biogas composition.

Fractions of heavy metals in FOGs/excess sludge mixtures before and after chemical stabilization are presented in Fig. 2. In the control sample, before the co-digestion process, in a residual fraction, the dominant percentage share was lead. Chemical oxidation with Fenton's reagent slightly decreased the percentage share of Pb in a residual fraction. Also copper in FOG/excess sludge mixtures (both control and chemically pretreated) was strongly bounded to organic matter and a residual fraction.

The most leachable heavy metals of five analyzed during the study were Ni and Cd. Chemical pretreatment, however, slightly decreased the content of these metals in a solid fraction, which indicates that they were probably leached into supernatants. Percentage differences in shares between the control and Fenton's pretreated samples were within several percent. This indicates that chemical pretreatment did not significantly affect heavy metals concentration in the mixtures. It was probably connected with the fact that the dose of Fenton's reagent was rather low and did not cause any drastic changes in FOGs structure.

After co-digestion, both in the control and pretreated sample, copper was a heavy metal connected mainly with residual and organic fractions (Fig. 3). Based on the literature data [22,23], it can be concluded that copper is the metal which shows significant affinity to organic matter. Also lead was rather nonleachable, since over 80% of it was bounded with residual and organic fractions. The percentage share of residual bounded lead was higher in Fenton's pretreated sample than in the control one. Nickel turned out the most leachable compound of five analyzed metals, which was also stated by other authors, e.g. Lasheen and Ammar [22]. Methane co-digestion caused a simultaneous increase in the cadmium content in the fractions IV and V, however, no significant differences were observed between control and chemically stabilized sludge. It indicates that methane co-digestion had a greater effect on heavy metals fractionation in the solid phase than chemical pretreatment of FOGs.

It was connected with the fact that FOGs were pretreated with a relatively low dose of Fenton's reagent and its content in the mixture with excess sludge was only 33%. Nickel and zinc turned out the most leachable heavy metals of five analyzed also after the co-digestion process.

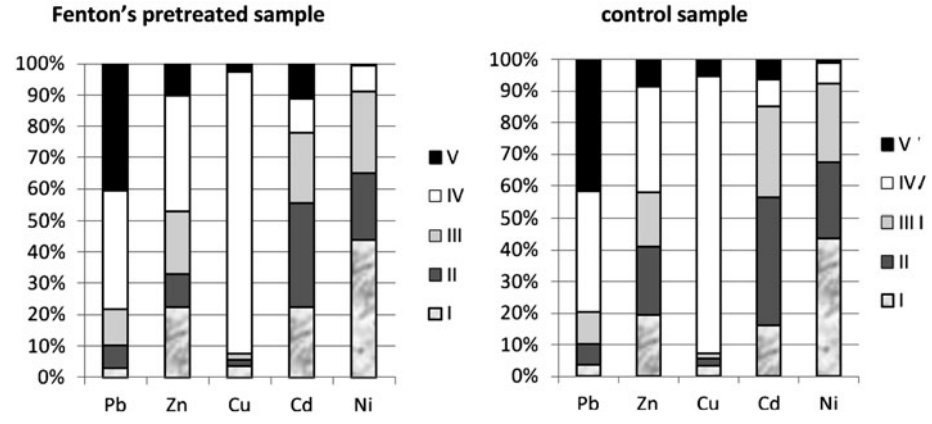

Fig. 2. Fractions of heavy metals in sewage sludge/FOG mixture before methane co-digestion (chemically stabilized vs. control sample): I—exchangeable, II—bounded with hydrocarbons, III—bounded with Fe and Mn, IV—bounded to organic matter, and V—residual.

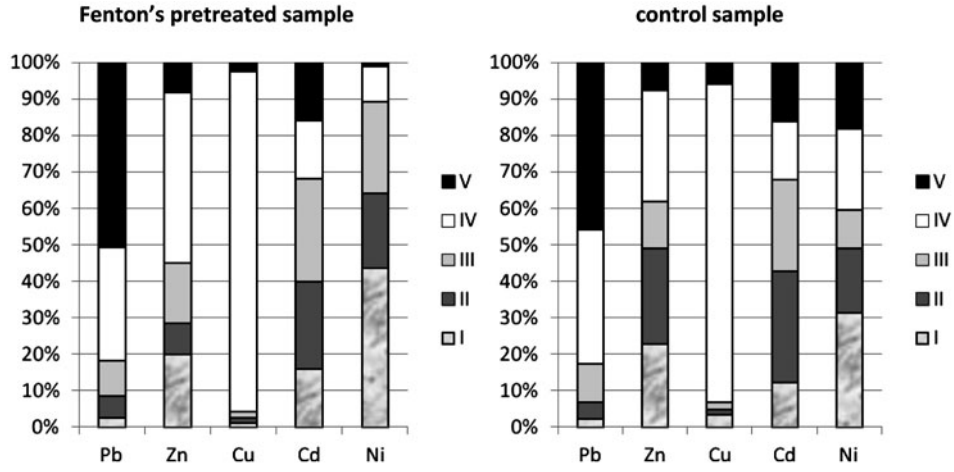

Fig. 3. Fractions of heavy metals in sewage sludge/chemically pretreated FOG mixture after methane co-digestion (chemically stabilized vs. control sample)): I—exchangeable, II—bounded with hydrocarbons, III—bounded with Fe and Mn, IV—bounded to organic matter, and V—residual.

The analysis of the total selected heavy metals concentration in the solid phase (Fig. 4) showed that methane co-digestion caused an increase in the total heavy metals concentration.

It can be explained by the fact that during co-digestion, the removal of volatile solids occurs, while heavy metals can only change the fraction with which they are bounded or can be leached to the supernatant. The same quantity of heavy metals compared to the lower content of solids in the sample causes a visible increase in the heavy metals content in the solid phase. An increase in heavy metals in the solid phase of chemically pretreated FOG/excess sludge mixture was slightly higher than in the control sample. Zinc was the most abundant heavy metal in the solid phase. The least abundant ones were lead and nickel with concentrations over one thousand lower than zinc.

The total concentration of the analyzed metals in supernatants was rather low (the total five metals

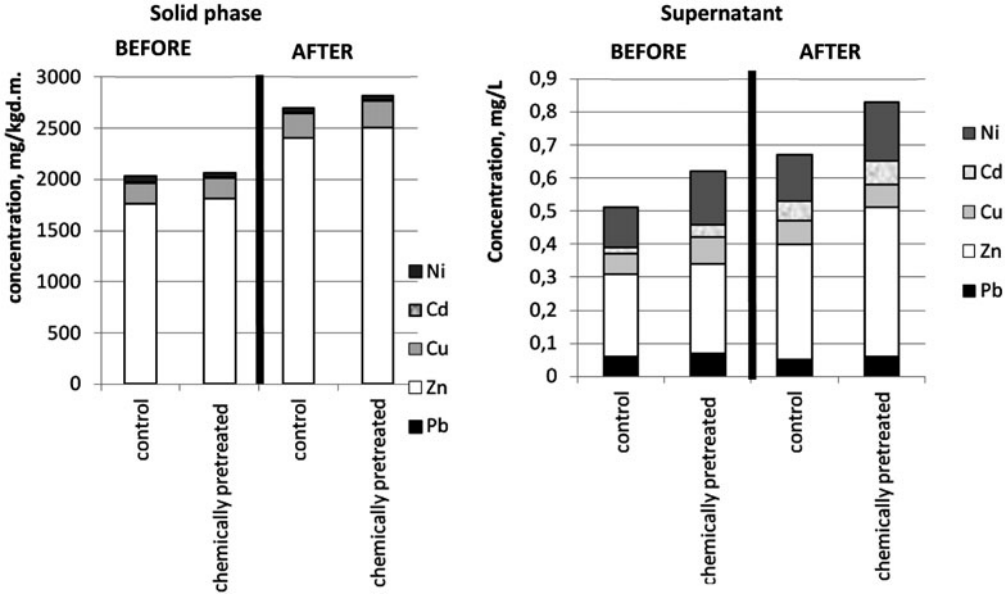

Fig. 4. Changes in average total heavy metals concentrations in sewage sludge and supernatant as a result of methane co-digestion.

concentration was at the level not higher than 1 mg $L^{-1}$of supernatant). The content of most leachable metals (Zn and Ni) increased more significantly as a result of co-digestion. About 17% increase in the total content of heavy metals was observed in the control sample of supernatants; about 25% in the samples which contained some amendment of chemically pretreated FOGs. Concentrations of heavy metals in all samples were rather low and during one-time introduction should not affect methane digestion. It, however, can accumulate in the solid phase of sewage sludge.

4. Conclusions

Based on the results of the research work, it can be stated that:

(1) Chemical pretreatment of FOGs with Fenton's reagent at a dose of 100 mg Fe^{2+} L^{-1} and H_2O_2 to 5,000 mg L^{-1} caused partial decomposition of this waste material. It allowed for an increase of biogas production only at about 7%.
(2) Chemical pretreatment did not significantly affect the fractions of heavy metals in FOGs. The co-digestion process had more influential impact on the inorganic micropollutants in the solid phase of FOG/excess sewage sludge mixtures and caused some changes in chemical forms of heavy metals.
(3) The effects observed throughout the study are promising, but it is necessary to check the results of higher doses of Fenon's reagent on the biogas generation effectiveness.
(4) Concentrations of heavy metals in all samples were rather low, and in the case one-time introduction of supernatant to the inflow to WWTP should not affect methane digestion. However, heavy metals can accumulate in the solid phase of sewage sludge.

Acknowledgments

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