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ORTHO-PCBs IN SEWAGE SLUDGE DURING METHANE FERMENTATION

Abstract. The purpose of this study is to examine the impact of mesophilic and thermophilic digestion on the content of seven congeners (with codes: 28, 52, 101, 118, 138, 153 and 180) in sewage sludge. Mesophilic fermentation occurred for 15 days at the temperature of 37°C (+/-1°C), thermophilic fermentation for 14 days at the temperature of 55°C. This study proved reduction of contents of higher chlorinated PCBs, which, under anaerobic conditions during both mesophilicand termophilic fermentation of the sludge samples, were subject to degradation to lower chlorinated congeners. During mesophilic and termophilic fermentation of the mixture of sewage sludge reduction of total content of PCBs amounted to 92,5% and 47% respectively. It is legitimate to conclude that PCB can transform both under anaerobic or aerobic conditions, which mainly involves dechlorination of higher chlorinated congeners to lower-chlorinated ones.

Keywords: termophilic, mesophilic fermentation; sewage sludge; polychlorinated biphenyls.

INTRODUCTION

For the first time PCBs were synthesized in 1881 by Schmidt and Schulz, and their commercial production started in 1929 in the USA. The mixture of the chemical composition dependent on the presence of chlorine and biphenyl was obtained as a result of the reaction of liquid biphenyl with chlorine in the presence of $FeCl_3$ as the catalyst [1,2]. Because of good dielectric characteristics, non-flammability and chemical resistance, PCBs found application in: insulation materials for filling transformers and condensers, hydraulic liquids for mining devices, additives to paints, lacquers, plasticizers, thermo- hardening materials and copying paper, protection plants agents, preservatives and impregnants [3].

PCBs in sewage sludge derive from following sources: households and industrial plants, wet and dry waste, surface flow, waters being used to wash streets, used transformer oils, dielectric liquids, waste containing PCBs as remains of combustion processes [4-8].

Blanchard and others in the process of examining PCB concentrations in total precipitation (wet and dry) and in sludge cocluded that concentrations in precipitation were from 3 to 4 times higher than in the sludge. The total composition of PCB

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congeners in precipitation and in sludge being examined on the same day was similar – with a higher index of lower chlorinated congeners but different from the total composition of chlorinated biphenyls in sludge. It proves that PCB detected in sludge are mainly of atmospheric origin and the dechlorination takes place in the wastewater tratment plants [5]. Relatively high concentrations of PCB were concluded in sludge during researches conducted during the rain period. High concentration of PCB in sludge was determined in samples taken after the first rain following a long-term drought. It was connected with the surface flow and washing out PCBs from industrial areas. PCB concentrations at the level of about 69% were observed. Then, in the rainy period, the concentration was going down, probably as a result of dilution. The possibilities of PCB penetration into sludge as a result of pouring water onto urban streets were considered. However, that theory is not proved [5]. For the sake of low solubility in water PCBs are removed in the process of sedimentation during wastewater treatment. Formation of the sludge of concentration between 1 – 10 mg/kg d.m. is the effect of that process. In the Poland that PCBs concentrations in sewage sludge might vary within a wide range from 23÷287 μg/kg dm [9,10].

The major part of the sediment is being used as organic fertilizers or sewage sludge-amended soils [11]. Rural usage of the sewage sludge is one of the main ways of their utilization. The use of the sewage sludge to fertilize fields and meadows in European Union and North America is on average of 30%. In Great Britain that amount is 40% (that means about 300 000 tonnes d.m. of sewage sludge/1 year). In Poland about 20 % of sewage sludge is used in agriculture [12]. Only if heavy metals contained in sewage sludge occur in permissible amounts, the sewage sludge can be used as a fertilizer. Only USA and some European countries (Holland, Germany, Austria, France) introduced in the norms tolerable and border concentrations of PCBs in sewage sludge being used as fertilizers. Their acceptable accumulation in the soil within 10 years was also given and amounted to 1,2 mg/m^2. At the same time it was stated that if one of the above mentioned compounds exceeds acceptable amounts, the sewage sludge cannot be used in agriculture. The European Union, seeing the problem of global threat to the environment from polychlorinated biphenyls, found it necessary to prepare corresponding regulations which would determine acceptable concentrations of these compounds in sewage sludge used in agriculture. For this reason, EU member states proposed, on 27th April 2000, a draft directive, referred to as the Sludge Directive [12]. It is important for the nearest future to point to the method of wastewater treatment which would allow for high level of removal of polychlorinated biphenyls [2,5,6]. The purpose of this study is to examine the impact of thermophilic and mesophilic digestion on the content of seven congeners (with codes: 28, 52, 101, 118, 138, 153 and 180) in sewage sludge.

EXPERIMENTAL PROCEDURES

Sewage sludge from municipal wastewater treatment plant, with 25% of total amount of the sewage being industrial wastewater typically produced in metallurgy, textile and food industry.

The research material included sediments mixture of primary and excess sludge (S+N) and fermenting sludge (F). The samples were taken once, the fermenting sludge was taken from the bottom of closed digestion chamber.

Process of mesophilic and thermophilic digestion occurred in two stages. During first stage, hydrolysed substrates i.e. the mixture (S+N) were inoculated with fermenting sludge with 1:2 ratio and the mixture of $(S+N+F)_m$ was obtained. The fermenting sludge was added in order to inoculate with bacterial microflora adapted to provide mesophilic digestion. The sludge prepared using this method were then subjected to mesophilic methane fermentation at the temperature of $37^oC \pm 1^oC$. The process of mesophilic methane fermentation took 15 days. In the second stage the inoculation for the process of thermophilic fermentation was from microorganisms which were present in the fermenting sludge. In order to adapt microorganisms to anaerobic thermophilic conditions, the sludge after transport from waste treatment plant was placed in glass bioreactors in thermostat at the temperature of 55 ± 1^oC and periodically reinforced with enriched glucose bed. The nutrient for feeding was prepared as recommended for this type of cultures [13]. The process of fermentation of the adapted sludge was monitored through measurement of the generated biogas and its composition through determination of the content of CH_4 and CO_2. Then, the sludge adapted to the temperature of 55^oC was mixed in volumetric ratio of 1:10 with fermenting sludge which was sampled from separate digestion chamber in wastewater treatment plant. This fermenting sludge was uses as an inoculation material which was mixed with a volumetric ration of 1:2 with the mixture of primary sludge and excess sludge and the mixtures of sludges $(S+N+F)_t$ was obtained. Incubation was carried out for 14 days at the temperature of 55 ± 1^oC.

Both processes were carried out at tightly closed bioreactors with option to measure the amount of the produced biogas and to sample it for analysis. The selected properties of the sludge, both before, during and after of the digestion, were determined, i.e. moisture, dry residue, residue after ignition (organic substance), post-ignition loss (mineral substance) while in supernatant liquor: pH, alkalinity, total organic carbon (TOC), volatile fatty acids (VFA). Determination was carried out with three repetitions.

In order to perform PCB extraction, the sludge was centrifuged and dried. Next the sludge was put into the conical flask and covered with 30 cm^3 of hexane. The flask was then placed for 30 minutes in ultrasonic bath and was sonicated. The obtained extract was purified through passing through previously conditioned columns of BAKERBOND spe Silica Gel (SiOH). The eluate was then concentrated in the stream of nitrogen. In order to extract PCBs, the SPE method was employed for BAKERBOND spe octadecyl C_{18} columns [5,10].

Qualitative and quantitative analysis of the selected congeners of PCBs was carried out by means of CGC-MS method. The procedure of extraction and qualitative and quantitative measurements of PCBs in sewage sludge samples were performed respectively during three repetitions, maintaining the same determination conditions. Accuracy of determination was assessed giving the values of initial standard deviations.

RESULTS AND DISCUSSION

Physico-chemical properties of sewage sludge

Physico-chemical properties of the fermenting (F) sludge and the mixtures of (S+N) and $(S+N+F)_m$ during mesophilic digestion are presented in Table 1.

Table 1. Physico-chemical properties of fermenting (F), mixture of (S+N) and $(S+N+F)_m$ during mesophilic fermentation

Tabela. 1. Fizyczno-chemiczne właściwości osadów ściekowych: fermentującego (F) mieszaniny (S+N) oraz $(S+N+F)_m$ podczas fermentacji mezofilowej

Properties	Unit	Sewage sludge		Mesophilic fermentation, days			
		F	S+N	Before	7	10	15
pH	-	8.06	7.28	7.95	7.91	7.88	7.84
Alkalinity	$mgCaCO_3/dm^3$	3450	1660	2840	4090	4160	4400
VFA	$mgCH_3COOH/dm^3$	407	2350	1270	360	380	220
TOC	mgC/dm^3	602	4120	1630	612	570	445
Moisture	%	97.5	96.01	97.0	97.5	97.5	97.7
Dry residue	g/dm^3	25.3	39.9	29.7	25.3	24.2	23.4
Organic substances	g/dm^3	9.9	13.6	10.2	10.4	10.3	10.2
	%	39.1	34.1	34.4	41.1	42.6	43.6
Mineral substances	g/dm^3	15.4	26.3	19.5	14.9	13.9	13.2
	%	60.9	65.9	65.6	58.9	57.4	56.4

Sludge (S+N) showed the content of dry residue of 39.9 g/dm^3, organic substances of 34.1% and mineral substances 65.9%. Overall alkalinity during mesophilic fermentation of the mixture of sludge $(S+N+F)_m$ rose whereas the VFA concentration decreased from 1270 to 220 mg CH_3COOH/dm^3. During mesophilic fermentation with termophilic hydrolysis, the TOC concentration in the liquid decreased from 1630 to 445 mgC/dm^3. Value of dry residue, organic and mineral substances loss decreased. Moisture of the studied sludge reached over 97%.

Physicochemical properties of the fermenting (F) sludge and the mixtures of (S+N) and $(S+N+F)_t$ during thermophilic digestion are presented in Table. 2.

Table 2. Physico-chemical properties of fermenting (F), mixture of (S+N) and $(S+N+F)_t$ during thermophilic fermentation

Tabela 2. Fizyczno-chemiczne właściwości osadów ściekowych: fermentującego (F) mieszaniny (S+N) oraz $(S+N+F)_t$ podczas fermentacji termofilowej

Properties	Jednostka	Sewage sludge		Termophilic fermentation, days czas			
		F	S+N	Before	3	7	14
pH	-	7.75	7.13	7.78	7.88	7.81	7.99
Alkalinity	$mgCaCO_3/dm^3$	2880	720	2000	3050	3600	4050
VFA	$mgCH_3COOH/dm^3$	231	1200	643	1414	643	317
TOC	mgC/dm^3	409	775	653	952	907	866
Moisture	%	97.9	96.6	97.4	97.7	97.9	98.1
Dry residue	g/dm^3	21.4	34.2	26.2	22.8	20.5	18.8
Organic substances	g/dm^3	13.1	26.3	18.1	14.5	12.7	10.8
	%	61.2	76.9	69.1	63.6	62.0	57.5
Mineral substances	g/dm^3	8.3	7.9	8.1	8.3	7.8	8.0
	%	38.8	23.1	30.9	36.4	38.0	42.6

Assessment of the quality of primary + excess (S+N) and fermenting (F) sludge revealed that the values of selected indexes differed considerably. Fermenting sludge was characterized by lower dry matter; content of organic substances amounted to 61.2% with mineral substances at the level of 38.8%. Sludge (S+N) showed the content of dry residue of 34.2 g/dm^3, organic substances of 76.9% and mineral substances 23.1%. Sludge fluid (S+N) was characterized by higher concentration of VFA (1200 $mgCH_3COOH/dm^3$), TOC (775 mgC/dm^3) whereas higher alkalinity was demonstrated by sludge fluid (F). The sludge was characterized by the degree of moisture from 97.4% to 98.1%.

Biodegradation of organic compounds during thermophilic digestion of the mixture of sewage sludge $(S+N+F)_t$ was confirmed by the decline in dry residue and residue after ignition. Dry residue decreased from 26,2 to 18.8 g/dm^3, residue after ignition from 18.1 to 10.8 g/dm^3. Moisture of the studied sludge was over 96.5%. Overall alkalinity of sludge fluid rose from 2000 to 4050 $mgCaCO_3/dm^3$, VFA concentration decreased on 14th day of the process to 317 mg CH_3COOH/dm^3 and did not exceed the limit of 2000 mg CH_3COOH/dm^3. During thermophilic digestion, the content of TOC in the fluid decreased to 866 mgC/dm^3.

PCBs in sewage sludge

Table 3 and Figure 1 compares the results of PCBs concentration in sewage sludge before and during mesophilic fermentation with termophilic hydrolysis. Their total concentration did not exceed permissible level of 0.8 mg/kg d.m. according to the Sewage Sludge Directive [12].

Table 3. PCBs concentration [µg/kg d.m.] in sewage sludge during mesophilic fermentation
Tabela 3. Stężenie PCB w osadach ściekowych podczas fermentacji mezofilowej

Congeners	Before fermentation			Mesophilic fermentation, days		
	F	S+N	S+N+F	7	10	15
PCB 28	nd[1]	0.29	0.35	0.62	0.27	nd
PCB 52	1.75	0.25	1.86	2.38	1.17	0.24
PCB 101	3.48	0.37	0.69	1.64	1.43	1.38
PCB 118	nd[1]	0.67	6.69	1.16	0.60	nd
PCB 138	0.75	0.27	1.11	nd	nd	nd
PCB 153	0.82	0.24	10.41	nd	nd	nd
PCB 180	0.33	0.27	0.53	0.07	0.03	nd
∑PCB	7.13	2.36	21.64	5.87	3.50	1.62

nd[1] – not deteced

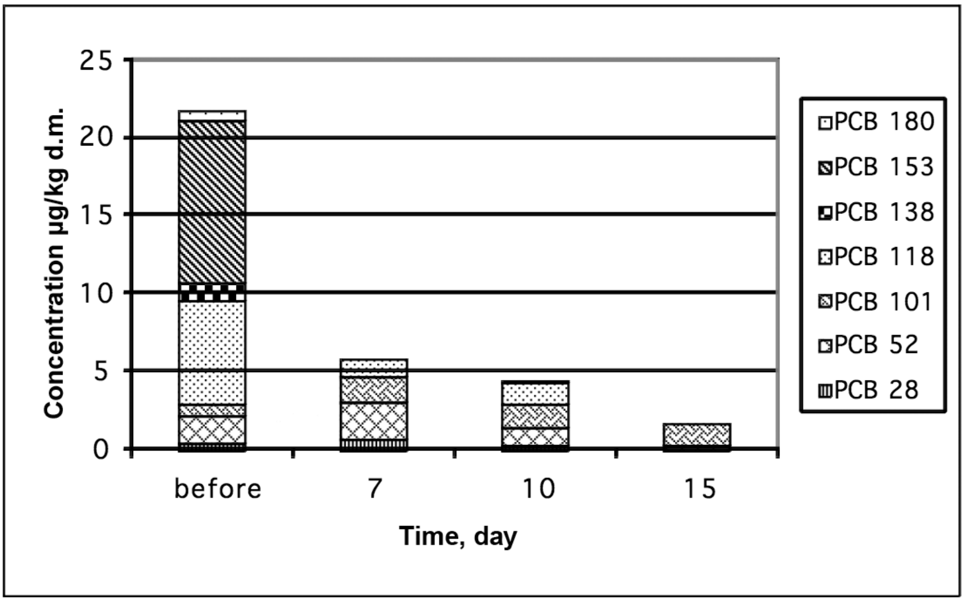

Fig. 1. Concentration of PCBs in sewage sludge $(S+N+F)_m$ before, during and after mesophilic fermentation
Rys. 1. Stężenie PCB w osadach ściekowych $(S+N+F)_m$ przed, podczas i po fermentacji mezofilowej

Before of mesophilic fermentation in the mixture of $(S+N+F)_m$ all seven congeners of PCB were determined; their total concentration amounted to 21.64 µg/kg d.m., whereas the highest contents were determined for hexachlorobiphenyl with code 153 (10.41 µg/kg d.m.) and pentachlorobiphenyl with code 118 (6.69 µg/kg d.m). Comparison of total concentration of PCBs throughout all the monitored days of the process of fermentation revealed that this value was highest on the zero day (21.64 µg/kg d.m.)

and resulted mainly from higher percentage of PCB 118 and PCB 153 isomers, which amounted to 31% and 48%, respectively. On the seventh day of the process of fermentation no higher chlorinated congeners of PCB (with codes 138 and 153, representative for hexachlorobiphenyls) were observed, whereas the content of lower chlorinated PCBs (28, 52, 101) with the total content of 4.64 μg/kg d.m. and nearly 2-time higher than their initial contents determined before of the fermentation process. Two lower chlorinated congeners of PCB, with codes 52 and 101, were determined on the 15th day; their contents were 0.24 μg/kg d.m. and 1.38 μg/kg d.m., respectively. During mesophilic fermentation of the mixture $(S+N+F)_m$, reduction of total content of PCBs amounted to 92.5%. This study proved reduction of contents of higher chlorinated PCBs, which, under anaerobic conditions during fermentation of the sludge samples, were subject to degradation to lower chlorinated congeners. It is legitimate to conclude that PCBs can transform both under anaerobic or aerobic conditions, which mainly involves dechlorination of higher chlorinated congeners to lower-chlorinated ones [14-17]. The bacteria which developed under anaerobic conditions dominated the process of dechlorination of PCBs with higher number of chlorine atoms in the molecule, whereas PCBs are transformed into the lower chlorinated biphenyls [17,18]. Performance of this process depends on saturation of the molecules with chlorine, their substitution in ortho, meta or para positions, since the study proved that chlorine atoms substituted in meta and para position are easier and faster biodegraded while those substituted in ortho position inhibit biological decomposition of PCBs [17-19].

Table 4 and Figure 2 present the results of qualitative and quantitative changes in PCBs in sewage sludge $(S+N+F)_t$ before and during thermophilic digestion.

Table 4. PCBs concentration [μg/kg d.m.] in sewage sludge during termophilic fermentation
Tabela 4. Stężenie PCB w osadach ściekowych podczas fermentacji termofilowej

Congeners	Before fermentation			Termophilic fermentation, days			
	F	S+N	S+N+F	3	7	10	14
PCB 28	0.14	5.48	7.39	7.39	5.16	9.57	4.09
PCB 52	0.09	4.26	4.62	4.87	2.52	14.28	5.67
PCB 101	1.68	3.34	3.60	3.30	2.79	1.01	1.46
PCB 118	0.15	3.25	4.19	4.54	2.90	2.09	2.01
PCB 138	0.37	2.67	4.05	4.01	2.40	1.47	0.87
PCB 153	2.32	2.60	3.39	0.29	0.21	1.69	1.13
PCB 180	0.40	2.37	3.71	3.91	2.42	1.17	1.05
∑PCB	5.15	23.95	30.94	28.30	18.39	31.28	16.27

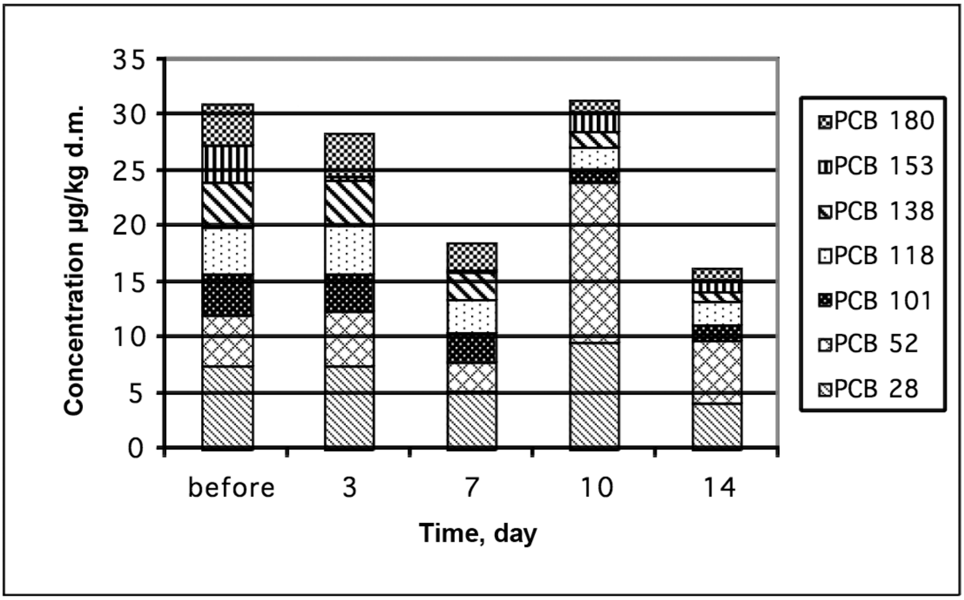

Fig. 2. Concentration of PCBs in sewage sludge $(S+N+F)_t$ before, during and after termophilic fermentation

Rys. 2. Stężenie PCB w osadach ściekowych $(S+N+F)_t$ przed, podczas i po fermentacji termofilowej

The analysis revealed presence of all the analysed congeners in the sludge. Their total concentration did not exceed permissible level of 0.8 mg/kg d.m. according to the Sewage Sludge Directive [12].

Before the process of thermophilic digestion in the mixture of $(S+N+F)_t$, all seven congeners of PCB were determined: their total concentration was 30.94 μg/kg d.m., with the highest concentrations obtained for trichlorobiphenyl with code 28 (7,39 μg/kg d.m.) and tetrachlorbiphenyl with code 52 (4,62 μg/kg d.m). Comparison of total concentration of PCB in all the monitored days of the process of fermentation revealed that the concentration was the highest before the process. On the third day of the process of fermentation, concentrations of individual congeners were comparable, whereas concentration of PCB 153 was reduces and was almost 12-time lower compared to concentration before the process of fermentation. On the tenth day it was found that the total concentration of lower chlorinated PCBs doubled the previous level, whereas in higher chlorinated PCBs this value decreased twice. During next days, the concentration of individual congeners in the mixture of sludge was on the decrease. After the process of thermophilic digestion, all the determined congeners of PCBs were still present. However, their total concentration was reduced by 47% on the fourteen day of the process. On the 7th day of the process, concentration of tri- and tetrachlorobiphenyls amounted to 41.8%. It should be emphasized that on the tenth day of fermentation, total concentration of lower chlorinated PCBs rose, whereas this level decreased for higher chlorinated PCBs. The percent concentration of PCBs with lower and higher concentration of chlorines amounted to 76.2 and 23.8%, respectively. The sludge after fermentation showed low content of five higher chlorinated PCBs. Insignificant percentage of higher chlorinated PCBs in the studied mixture after the process of thermophilic digestion can be explained by their capability of bioaccumulation (the more chlorine atoms in biphenyl molecule the better biosorption) and/or biological degradation as a result of which higher chlorinated PCBs were transformed into lower chlorinated PCBs. According to Borja et al., (2005) kinetics of this process of dechlorination depends on the composition of a particular population of microorganisms. The content of a particular culture in sewage sludge is affected by the environmental conditions such as: availability of the sources of carbon, hydrogen and other electron donors, presence or lack of electron acceptors other than PCBs, competition with other microorganisms, presence of toxic pollutants, temperature and pH [14].

Observation of the results obtained for changes in the concentration of PCBs during thermophilic digestion in the studied sludge revealed reduction in the concentration of the analysed PCBs. After fermentation in the studied mixture of sewage sludge, low concentration of higher chlorinated PCBs was demonstrated. Therefore, one can expect that the process of thermophilic digestion does not cause accumulation of polychlorinated biphenyls in sewage sludge.

Distribution of PCBs in sludge and their tendencies towards reduction in total concentration during the process of thermophilic digestion is supported in the studies by other authors. Benabdallah El-Hadaj demonstrated reduction in total PCB concentration (28, 52, 101, 138, 153 and 180) amounted to 83.5% on the last day of thermophilic digestion [19].

CONCLUSION

1. The process of mesophilic with termophilic hydrolysis and termophilic fermentation in the mixtures of sludge total PCBs concentration decreased.
2. Absence of higher chlorinated PCBs in the sludge after the process of fermentation can be explained by biological degradation of polychlorinated biphenyls under anaerobic conditions, which in consequence led to transformation of higher chlorinated into lower chlorinated PCBs.
3. Methane fermentation has positive effect on biodegradation of higher chlorinated PCBs. During mesophilic and termophilic fermentation of the sewage sludge reduction of total content of PCBs amounted to 92,5% and 47% respectively.

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ORTO-PCB W OSADACH ŚCIEKOWYCH PODCZAS FERMENTACJI METANOWEJ

Streszczenie. W pracy przedstawiono wyniki badań zmian zawartości wybranych kongenerów PCB o kodach: 28, 52, 101, 118, 138, 153, 180. Przedmiotem badań były osady ściekowe pochodzące z miejskiej oczyszczalni ścieków. Prowadzono proces fermentacji mezofilowej i termofilowej. Zhydrolizowany substrat tj. mieszaninę osadów wstępnego i nadmiernego (S+N) zaszczepiono osadem fermentującym i otrzymano mieszaninę osadów $(S+N+F)_m$. Tak przygotowany osad poddawano fermentacji metanowej mezofilowej w temperaturze 37°C (+/-1°C). Proces fermentacji termofilowej prowadzono przez 14 dni w temperaturze 55 ± 1°C. Odpowiednio przygotowany osad fermentujący użyto jako materiał zaszczepiający, który zmieszano w stosunku objętościowym 1:2 z mieszaniną wstępnego z nadmiernym i otrzymano mieszaninę osadów $(S+N+F)_t$. Badania wykazały, że zarówno fermentacja mezofilowa jak i termofilowa wpływa korzystnie na biodegradację PCB. Proces stabilizacji beztlenowej spowodował zmniejszenie stężenia siedmiu oznaczanych kongenerów PCB w mieszanie osadów $(S+N+F)_m$ oraz $(S+N+F)_t$. Podczas fermentacji mezofilowej zmniejszenie sumarycznego stężenia PCB w mieszaninie osadów wynosiła 92,5 %, a po fermentacji termofilowej 47% Uważa się, że takie zmiany stężenia PCB były powodowane biodegradacją tych mikrozanieczyszczeń.

Słowa kluczowe: fermentacja: mezofilowa, termofilowa; osady ściekowe. polichlorowana bifenyle.