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POLYCHLORINATED BIPHENYL (PCB) CONGENER RESIDUES IN FRUTTI DI MARE (SEAFOOD) PRODUCTS

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Abstract. Considering high nutritive value and sensory qualities, the products manufactured of marine invertebrates are a desirable component of human diet. These products enjoy an increasing demand in Poland. However, considerable accumulation of chlorine-organic pollutants in the tissues of marine animals may pose a threat for consumers. The study included determination of the content of PCB congeners (28, 52, 101, 118, 138, 153, and 180 according to IUPAC) in tinned and pickled seafood products purchased in Szczecin's fishshops. In all examined products the analysed congeners were found, only in "Octopus in vegetable oil" PCB 138 was not detected. The highest content, 13.8 $\mu g \cdot kg^{-1}$ m.m. (99.237 $\mu g \cdot kg^{-1}$ lipids), was recorded for PCB congener 153 in "Squid in American sauce". The lowest residue levels were found for PCB 101 (0.002 to 0.07 $\mu g \cdot kg^{-1}$ m.m.). The highest percentage (from among analysed congeners) in majority of examined products was found for PCB 153 (to 95% in "Octopus in vegetable oil") and PCB 180 (to 58% in "Pickled mussels"). The lowest percentage was stated for PCB congeners 101 (to 2.2% in "Shrimps natural") and 52 (to 9.9% in "Greenland shrimps in brine").

Key words: PCB, frutti di mare (seafood), shrimps, molluscs, oysters, crabs, squids, crayfish

INTRODUCTION

Polychlorinated biphenyls (PCB) form a mixture of congeners composed of two phenyl rings with different number and location of chlorine atoms in molecule, which decides about their physicochemical properties, environmental behaviour and toxicity [McFarland and Clarke 1989]. These compounds are characterized by supreme thermal conductivity, high ignition temperature (170-380°C), dielectric properties and high resistance to thermal decomposition. Owing to their properties, they have found application mostly as transformer and capacitor insulators, softening agents for (plastificators), hydraulic fluids, vacuum pomp fluids, as well as many other usages. Their present an-

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nual production is estimated at about 50 thousand tonnes [Bykowski 1997]. Until 1980, global production and wide application of PCBs in different economic branches was valued at 1.2-2 million tonnes, out of which found its way to natural environment. Up to 60% of previous global production of PCBs penetrated together with industrial and municipal effluents into the seas and oceans, out of which 30% was accumulated in littoral bottom sediments [Tanabe 1988].

Considering their lipophilicity, PCBs join quickly in the trophic circulation, accumulating in aquatic organisms. They were found in fish, marine mammals, as well as in other edible sea resources, such as molluscs, oysters and crabs [Falandysz 1987, Zabik et al. 1992, Lee et al. 1996, Castro et al. 1990, Porte and Albaiges 1993, Kuwabara 1989, Ciereszko 2002, Ciereszko and Witczak 2003]. The latter belong to the so-called "frutti di mare" (seafood), having high nutritive value and being a good source of high-quality protein, B-group vitamins, as well as of iodine, selenium, iron, zinc and magnesium. Crustaceans and shellfish contain also a lot of calcium, and oysters plenty of zinc. The fat of these animals is very abundant in fatty acids of n-3 group [Sikorski 2004, Kolanowski 2003 a, b].

Since marine invertebrates enjoy an increasing demand in Poland, the objective of the study was set at determining a degree of pollution in seafood products with PCB residues.

MATERIALS AND METHODS

The study material consisted of tinned and jar-pickled seafood products purchased in Szczecin's retail trading network in 2005 (Table 1).

Sample preparation and polychlorinated biphenyl determination was carried out according to PN-EN 1528-1-4 standard. For PCB content analyses, 30-33 g weighed samples were collected, which were lyophilised over 36 hours in LYOLAB 3000 apparatus and then triturated in porcelain mortar. As a reference material, an isooctane solution of 7 PCB congeners was used (28, 52, 101, 118, 153, 138, and 180; NE-N 0813, LGC Promochem GmbH D-46 485 Wesel). In order to determine the recovery size, a known amount of surrogate Pesticides Surrogate Spike Mix (Cat. no. 4-8460, SUPELCO, USA) was added, being an acetone solution of two compounds: decachlorobiphenyl and 2,4,5,6-tetrachloro-m-xylen.

Extraction of PCB congeners with lipids was carried out in a Soxhlet's apparatus within 8 hours, using each time 150 cm^3 n-hexane/acetone mixture (v/v; 3:1). Next, extracts were concentrated in a rotary vacuum evaporator to about 2 cm^3. The thickened extracts were transferred quantitatively with n-hexane into 15 ml weighed tight ground-in stopper glass test-tubes. In order to determine the lipid content, dissolvent was evaporated under a nitrogen atmosphere and residues were desiccated at 60°C. To determine the content of the analysed compounds, the obtained fat was dissolved in 2 cm^3 n--hexane and then purified with 6 cm^3 7% SO_3 in concentrated H_2SO_4. After mixing and separation of layers, the upper n-hexane layer was transferred into clean test-tubes, whereupon samples were rinsed thrice with deionised water and dried on anhydrous Na_2SO_4 bed. The obtained samples were thickened to 1 cm^3 in a nitrogen atmosphere.

Table 1. The characteristics of assortment analysed
Tabela 1. Charakterystyka badanego asortymentu

Product name Nazwa produktu	Manufacturer Producent	Composition Skład
Squid in American sauce Kalmary w sosie amerykańskim	Vigilante – Spain Vigilante – Hiszpania	squids, tomatoes, oil, onion, spices kalmary, pomidory, olej, cebula, przyprawy
Smoked mussels in oil Małże w oleju podwędzane	Graal – Thailand Graal – Tajlandia	smoked mussels, oil, salt małże podwędzane, olej, sól
Pickled mussels Małże marynowane	Vigilante – Spain Vigilante – Hiszpania	mussels, vinegar oil, spices, salt małże, ocet, olej, przyprawy, sól
Smoked oysters in oil Ostrygi podwędzane w oleju	Graal – Thailand Graal – Tajlandia	smoked oysters, salt ostrygi podwędzane, sól
Octopus in vegetable oil Ośmiornice w oleju roślinnym	Garavilla – Spain Garavilla – Hiszpania	octopuses, vegetable oil, salt ośmiornice, olej roślinny, sól
Stuffed squid in oil Kałamarnice nadziewane w oleju	Vigilante – Spain Vigilante – Hiszpania	squids, oil, salt kałamarnice, olej, sól
Crayfish tails in brine Raki ogonki w zalewie	Polanica – Charzyno – PL Polanica – Charzyno – PL	crayfish tails, water, salt, acidity regulator: citric acid, preservatives: E211, E202 ogonki raków, woda, sól, regulator kwasowości: kw. cytrynowy, subst. konserwujące E211, E202
Greenland shrimps in brine Krewetki ziemnowodne w zalewie	AB Halofisk – Sweden AB Halofisk – Szwecja	shrimps, suger, water, salt, citric acid E330 krewetki, cukier, woda, sól, kw. cytrynowy E330
Picnic Shrimps Krewetki Picnic	Laguna – Thailand Laguna – Tajlandia	shrimps, water, salt, acidity regulator: citric acid krewetki, woda, sól, regulator kwasowości: kw. cytrynowy
Crabs natural Kraby w sosie własnym	Laguna – Thailand Laguna – Tajlandia	crab meat, water, salt, acidity regulator: citric acid mięso z krabów, woda, sól, regulator kwasowości: kw. cytrynowy

Analyses were made on GC HP 6890 apparatus equipped with 5973 mass sensitive detector under following chromatograph working conditions:

– carrier gas – helium,
– pressure – 26 psi,
– CPSIL8 CB LOW BLEED (60 m × 250 μm × 0.25 μm; Chrompaq CP 5861) capillary column through-flow – 1.2 ml·min^{-1},
– column oven temperature program: 140°C (0.5 min), increase 10°C/min; 200°C (5 min), increase 5°C/min; 280°C (10 min), increase 30°C/min; 300°C (5 min).

The analyses were made triplicate.

RESULTS AND DISCUSSION

The contents of PCB indicator congeners and the sums of PCBs in seafood products are confronted in Tables 2 and 3. The recovery size was at the level of 60-70%, whereas the determinability limit for the analysed compounds was 0.002 $\mu g \cdot kg^{-1}$ on the average.

Table 2. Contents of analysed PCB congeners in processed food frutti di mare adjusted to a wet matter basis, $\mu g \cdot kg^{-1}$ w.m.
Tabela 2. Zawartości analizowanych kongenerów PCB w przetworach z owoców morza w mokrej masie, $\mu g \cdot kg^{-1}$ m.m.

The name of product Nazwa produktu	PCB 28	PCB 52	PCB 101	PCB 118	PCB 153	PCB 138	PCB 180	PCBs
Squid in American sauce Kalmary w sosie amerykańskim	3.596* ±0.290	0.186 ±0.021	0.069 ±0.012	0.251 ±0.031	13.783 ±1.067	0.142 ±0.201	0.048 ±0.001	18.075 ±1.536
Smoked mussels in oil Małże w oleju podwędzane	0.895 ±0.102	0.050 ±0.009	0.009 ±0.005	0.690 ±0.057	2.131 ±0.203	0.180 ±0.034	4.699 ±0.382	8.653 ±0.028
Pickled mussels Małże marynowane	0.233 ±0.014	0.008 ±0.001	0.002 ±0.001	0.475 ±0.002	1.043 ±0.080	0.167 ±0.026	2.681 ±0.314	4.609 ±0.357
Smoked oysters in oil Ostrygi podwędzane w oleju	0.075 ±0.003	0.008 ±0.002	0.011 ±0.002	0.248 ±0.016	1.428 ±0.307	0.298 ±0.099	2.006 ±0.349	4.073 ±0.540
Octopus in vegetable oil Ośmiornice w oleju roślinnym	0.020 ±0.005	0.026 ±0.001	0.004 ±0.002	0.014 ±0.002	3.695 ±0.016	n.w. n.d.	0.094 ±0.005	3.853 ±0.030
Stuffed squid in oil Kałamarnice nadziewane w oleju	0.024 ±0.003	0.050 ±0.013	0.016 ±0.003	0.294 ±0.028	0.240 ±0.046	0.045 ±0.019	0.086 ±0.026	0.754 ±0.069
Crayfish tails in brine Raki ogonki w zalewie	0.038 ±0.008	0.007 ±0.004	0.003 ±0.001	0.023 ±0.002	0.034 ±0.048	0.018 ±0.003	0.041 ±0.016	0.164 ±0.063
Greenland shrimps in brine Krewetki ziemnowodne w zalewie	0.019 ±0.005	0.016 ±0.002	0.002 ±0.001	0.013 ±0.018	0.070 ±0.010	0.009 ±0.012	0.034 ±0.029	0.162 ±0.047
Picnic Shrimps Krewetki Picnic	0.085 ±0.015	0.004 ±0	0.003 ±0	0.005 ±0.007	0.032 ±0.005	0.068 ±0.002	0.044 ±0.011	0.240 ±0.018
Crabs natural Kraby w sosie własnym	0.063 ±0.001	0.008 ±0.004	0.005 ±0.002	0.013 ±0.003	0.060 ±0.030	0.023 ±0.003	0.066 ±0.001	0.238 ±0.025

* 3.596 ± 0.290 – congener mean content ± standard deviation, n.d. – not detected.
* 3,596 ± 0,290 – średnia zawartość kongeneru ± odchylenie standardowe, n.w. – nie wykryto.

Table 3. Contents of PCB congeners in ford processed in marine invertebrates in terms of lipids, $\mu g \cdot kg^{-1}$ lipids
Tabela 3. Zawartości kongenerów PCB w przetworach z bezkręgowców morskich w przeliczeniu na lipidy, $\mu g \cdot kg^{-1}$ lipidów

The product name Nazwa produktu	Per-centage of lipids Procent lipidów	PCB 28	PCB 52	PCB 101	PCB 118	PCB 153	PCB 138	PCB 180	PCBs
Squid in American sauce Kalmary w sosie amerykańskim	13.9	25.888 ±2.626	1.341 ±0.175	0.498 ±0.076	1.800 ±0.182	99.237 ±9.751	1.036 ±1.465	0.344 ±0.007	130.144 ±13.765
Smoked mussels in oil Małże w oleju podwędzane	14.13	6.478 ±1.776	0.357 ±0.125	0.069 0.044	4.982 ±1.211	15.402 ±3.935	1.312 ±0.450	33.487 ±2.725	62.088 ±10.265
Pickled mussels Małże marynowane	12.04	1.940 ±0.218	0.070 ±0.010	0.015 ±0.006	3.949 ±0.184	8.654 ±0.219	1.392 ±0.285	22.225 ±1.461	38.245 ±0.989
Smoked oysters in oil Ostrygi podwędzane w oleju	15.06	0.498 ±0.001	0.053 ±0.015	0.071 ±0.019	1.648 ±0.178	9.443 ±1.630	1.996 ±0.745	13.280 ±1.742	26.987 ±2.416
Octopus in vegetable oil Ośmiornice w oleju roślinnym	6.2	0.314 ±0.053	0.413 ±0.010	0.066 ±0.021	0.225 ±0.014	59.741 ±4.171	n.w. n.d.	1.528 ±0.035	62.281 ±4.128
Stuffed squid in oil Kałamarnice nadziewane w oleju	16.79	0.140 ±0.014	0.300 ±0.073	0.094 ±0.019	1.752 ±0.195	1.434 ±0.294	0.265 ±0.107	0.514 ±0.163	4.498 ±0.476
Crayfish tails in brine Raki ogonki w zalewie	1.38	2.783 ±0.875	0.505 ±0.194	0.241 ±0.114	1.652 ±0.069	2.265 ±3.203	1.262 ±0.029	2.939 ±0.776	11.647 ±3.144
Greenland shrimps in brine Krewetki ziemnowodne w zalewie	1.58	1.161 ±0.078	1.053 ±0.359	0.153 ±0.069	0.955 ±1.350	4.463 ±0.369	0.653 ±0.924	2.375 ±2.327	10.812 ±5.320
Picnic Shrimps Krewetki Picnic	1.16	7.334 ±1.597	0.316 ±0.011	0.236 ±0.029	0.431 ±0.610	2.768 ±0.507	5.895 ±0.432	3.771 ±0.759	20.751 ±2.369
Crabs natural Kraby w sosie własnym	0.79	7.994 ±0.815	1.104 ±0.571	0.663 ±0.152	1.589 ±0.227	7.904 ±4.646	2.858 ±0.114	8.442 ±0.855	30.553 ±6.396

The presence of all analysed congeners was stated in all examined products, apart from ”Octopus in vegetable oil”, for which PCB 138 was not detected.

The lowest fat content was found in products in natural sauce, while the highest in those in oil marinade, whereas the lipid content in the examined products ranged 0.79% in “Crabs natural” to 16.78% in “Stuffed squid in oil” (Table 3).

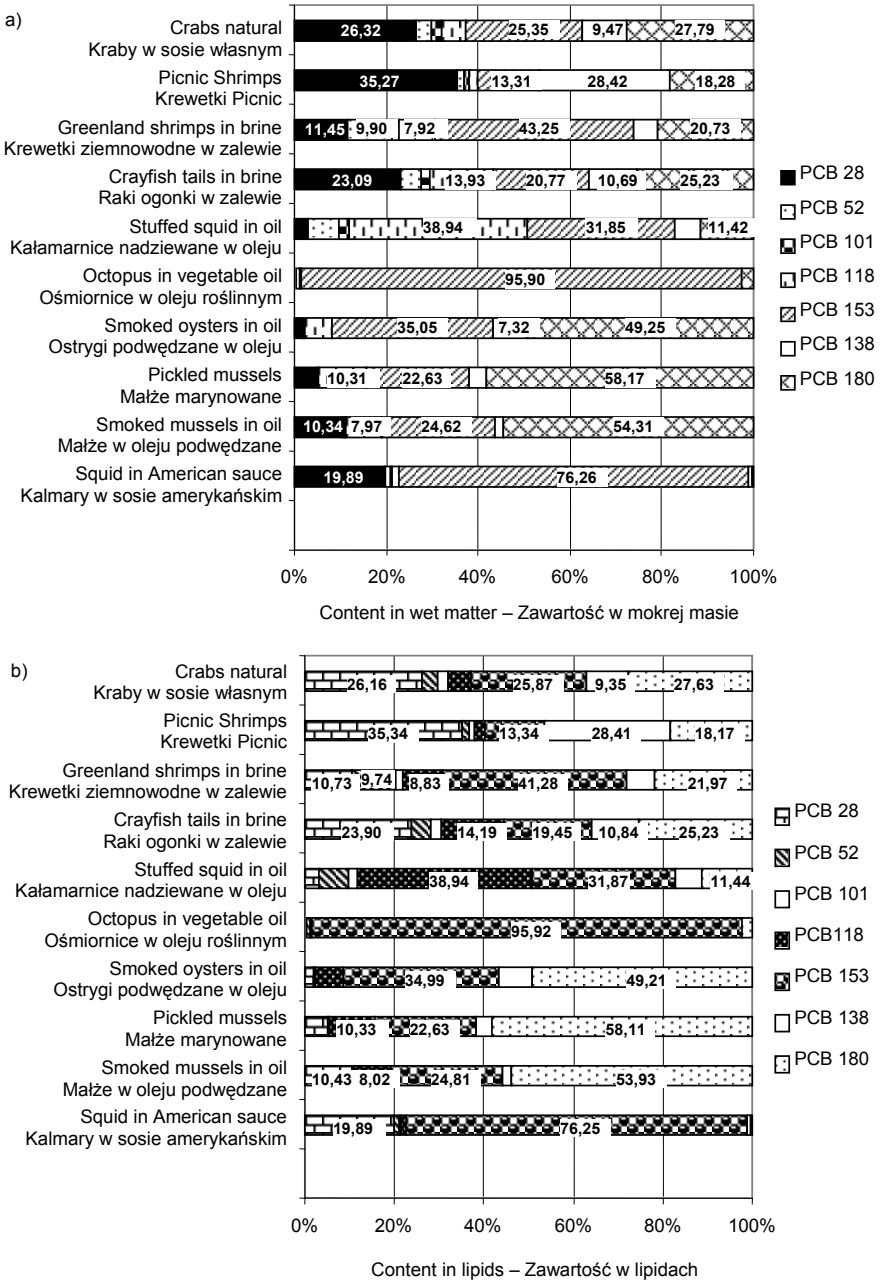

Fig. 1. The percentage participation of PCB congeners in the analysed products: a) in wet matter, b) in lipids

Rys. 1. Procentowy udział kongenerów PCB w analizowanych produktach: a) w mokrej masie, b) w lipidach

The PCB sum in wet matter ranged 0.162 μg·kg^{-1} in "Greenland shrimps" to 18.075 μg·kg^{-1} w.m. in squids. On the other hand, total PCB content converted to lipids ranged 4.498 μg·kg^{-1} in "Stuffed squid in oil" to 130.144 μg·kg^{-1} in "Squid in American sauce".

From among the whole examined assortment, the highest content – 13.8 μg·kg^{-1} w.m. (99.237 μg·kg^{-1} lipids) – was stated for PCB 153 congener in "Squid in American sauce". The content of PCB 101 congener residues was the lowest and amounted to 0.002 to 0.07 μg·kg^{-1} w.m. (Table 2 and 3).

When analysing the contents of respective PCBs in the examined products, it was found that, both in wet matter and lipids, the highest percentage was for PCB 153 (to 95% in "Octopus in vegetable oil") and PCB 180 (to 58% in "Pickled mussels"; Fig. 1 a, b). On the other hand, the lowest percentage was stated for PCB 101 congener (max. 2.2% in "Shrimps natural) and PCB 52 (max. 9.9% in "Greenland shrimps in brine").

Food raw materials of marine origin are mainly eaten in the form of products submitted to technological and culinary processing, which leads in general to the loss of part of PCBs [Zabik 1992]. This is connected most frequently with the leakage of part of fat together with PCB compounds dissolved in it and their co-distillation with water vapour.

Despite high health and flavoury qualities of the meat and organs of marine animals, attention should be paid to a considerable accumulation of different chemical pollutants, including PCB compounds, in them. Degree of environmental contamination has a crucial effect on the level of PCB residues in fish, crustaceans and edible shellfish tissues, while salinity degree and water temperature as well as individual or seasonal factors influence it indirectly. Crustaceans and molluscs caught in the littoral zone are characterized by large ability of pollutant bioaccumulation due to sedentary life, being thus good bio-indicators for local contamination of aquatic environment.

According to Falandysz [1987], the water regions polluted with PCB compounds were: North America Great Lakes, North Sea littoral zone, Mediterranean Sea and Baltic Sea, while average PCB residue levels in mussel from the Baltic Sea ranged 0.06--0.14 mg·kg^{-1}, whereas 0.004 mg·kg^{-1}-0.47 mg·kg^{-1} in that from the North Sea. Kuwabara et al. [1989] stated that PCB content in shrimp and squid coming from the North Sea was 0.1 mg·kg^{-1} w.m. and 0.03 mg·kg^{-1} w.m., respectively. On the other hand, Porte and Albaiges [1993] determined the average level of PCB indicator congener sum in molluscs and crabs caught by the Catalonian coast to 10 ng·g^{-1} to 91 ng·g^{-1}. In the latter study, the highest concentration in all samples was found for PCB 153. Examination of PCB residues in molluscs from the south-eastern Baltic [Lee et al. 1996] showed that penta- and hexachlorinated biphenyls had accumulated in the highest concentrations, with PCB 153 prevailing among congeners.

The results obtained in the present study confirm the above-mentioned findings. More chlorinated congeners, e.g. PCB 153 and PCB 180, are also more lipophilous and have longer half-life, which affects their domination in fat tissues of living organisms [Falandysz 1999]. The PCB 153 congener was found in the highest concentration in all samples, and its content in "Squid in American sauce" reached almost 13.8 μg·kg^{-1} w.m. The highest content of PCB 180 (2.68 μg/kg w.m.) was found in the pickled mussels.

Significantly lower contents ($p < 0.05$) were stated for PCB 101 and PCB 52 in all examined products (Fig. 1 a, b).

According to EPA (Environmental Protection Agency), the limit of PCBs contents for water, in which fish exist, equalls 0.05 ppb [ATSDR 2000]. In European countries

different values of MPL (maximum permissible level) for indicating PCB congeners are established. In Germany MPL for sea fish and crustaceans falls within the range from 0.08 $mg \cdot kg^{-1}$ for the congeners PCB 28, PCB 52, PCB 101, PCB 180 to 0.1 $mg \cdot kg^{-1}$ for the congeners PCB 138 and PCB 153. For freshwater fish MPL for these compounds equalls from 0.2 to 0.3 $mg \cdot kg^{-1}$. However, in the Netherlands different values concern herring and mackerel (from 0.12 $mg \cdot kg^{-1}$ for PCB 52 to 0.36 $mg \cdot kg^{-1}$ for PCB 180) and different are for other fish (except eel). The values are within the range from 0.04 $mg \cdot kg^{-1}$ for PCB 52 to 0.12 $mg \cdot kg^{-1}$ for PCB 180 [Żmudzki 2004].

In Poland the regulation of the European Union Commission, concerning maximum permissible levels of chloroorganic contaminants in foodstuffs with respect to dioxin and dioxin-like PCB, is currently in force. The latest Regulation No 199/2006 (UE Official Journal, Regulation from 3rd of February 2006) gives the value of MPL for total dioxins, furans and dioxin-like PCBs in fish meat and products obtained from fish as 8 $pg_{WHO\text{-}PCDD/F\text{-}PCB\text{-}TEQ}$ /g live-wight.

The detected residues of the analysed congeners and total PCB in seafood products were at a low level and did not pose a threat for consumer's health considering their average daily uptake of 8.5-11 μg/person with food in Poland (compared to only 0.53 μg/person daily in Great Britain and 4-50 μg/person in Japan) [Falandysz 1999].

CONCLUSIONS

1. In all examined products the analysed compounds were found, except PCB 138, which was not detected in "Octopus in vegetable oil".

2. The highest level, 13.783 $\mu g \cdot kg^{-1}$ w.m. (99.237 $\mu g \cdot kg^{-1}$ lipids), was stated for PCB 153 in "Squid in American sauce".

3. The highest percentage was found for PCB 153 (to 95%), while the lowest for PCB 101 and PCB 52 (max. 9.9%).

4. The contents of respective congeners and total PCB were at a low level, much more below the allowable concentrations (according to FDA).

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POZOSTAŁOŚCI KONGENERÓW PCB W PRZETWORACH Z OWOCÓW MORZA

Streszczenie. Przetwory z bezkręgowców morskich, ze względu na wysoką wartość żywieniową i walory sensoryczne, są pożądanym składnikiem diety człowieka. Produkty te cieszą się rosnącym popytem w Polsce. Jednak znaczne kumulowanie się zanieczyszczeń chloroorganicznych w tkankach zwierząt morskich może stanowić niebezpieczeństwo dla konsumentów. Badania obejmowały określenie zawartości kongenerów wskaźnikowych PCB (o numerach według IUPAC 28, 52, 101, 118, 138, 153, 180) w konserwach i marynatach z owoców morza, zakupionych w szczecińskim handlu detalicznym. Analizę chromatograficzną wykonano w aparacie GC HP 6890 D 5973 z kolumną CPSIL8 CB LOW BLEED (60 m × 250 μm × 0,25 μm). We wszystkich badanych produktach stwierdzono obecność analizowanych kongenerów, nie wykryto jedynie PCB 138 w „Ośmiorni-

cach w oleju roślinnym". Spośród badanego asortymentu, największą zawartość – 13,8 $\mu g \cdot kg^{-1}$ m.m. (99,237 $\mu g \cdot kg^{-1}$ lipidów) notowano dla kongeneru PCB 153 w „Kalmarach w sosie amerykańskim". Najniższe poziomy pozostałości stwierdzono dla PCB 101 (od 0,002 do 0,07 $\mu g \cdot kg^{-1}$ m.m.). Największym udziałem procentowym (spośród 7 analizowanych kongenerów wskaźnikowych) w większości produktów charakteryzował się PCB 153 (do 95% w „Ośmiornicach w oleju roślinnym") oraz PCB 180 (do 58% w „Małżach marynowanych"). Natomiast najmniejszy udział procentowy miały kongenery PCB 101 (do 2,2% w „Krewetkach w sosie własnym") i PCB 52 (do 9,9% w „Krewetkach ziemnowodnych w zalewie").

Słowa kluczowe: PCB, owoce morza, krewetki, małże, ostrygi, kraby, kalmary, raki

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