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Polybrominated Diphenyl Ethers in Surface Water Sources and Sediments of the Konya Closed Basin and Environmental Risk Assessment

Konya Kapalı Havzası Yüzeysel Su Kaynakları Su ve Sedimentlerinde Polibromlu Difenil Eterler ve Çevresel Risk Değerlendirmesi

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Abstract

Polybrominated diphenyl ethers (PBDEs), which are potentially toxic, are still detected in the environment at ppb levels, despite being banned or restricted in use. In this study, 38 surface water and 28 sediments were taken from 46 sources in the Konya Closed Basin, Türkiye. The samples were investigated for pollution by PBDE congeners (PBDE47, PBDE100, PBDE99, PBDE154, PBDE153). A risk assessment was carried out using the concentrations detected in the water samples for fish, Daphnia magna, and algae. Total PBDE concentrations were determined to be between 0.96-74.5 ng/L in surface water, and between 0.01-0.64 µg/kg dry weight in sediment samples. PBDE47 congener is dominant in water samples, while PBDE100, PBDE153, and PBDE47 dominant in sediment samples. PBDE47 congener poses a high risk for fish, Daphnia magna, and algae in two surface water sources, PBDE154 and PBDE153 pose a high risk for fish in three surface water sources, PBDE154 poses a high risk for Daphnia magna in two, and PBDE153 poses a high risk for Daphnia magna in three surface water sources. The results show that PBDE pollution is present in the surface waters of Konya Closed Basin at levels affecting aquatic life. Water in the basin is used for many purposes, primarily drinking and agricultural irrigation. Commercial products containing PBDE are still in use and are creating waste. An effective waste management plan should be implemented, and micropollutants, including PBDEs, should be regularly monitored in environmental media.

Keyword: Surface water, sediment, polybrominated diphenyl ether, PBDE, pollution, risk.

Öz

Yüksek kalıcılık ve biyoakümülasyon gösteren, potansiyel olarak toksik olan polibromlu difenil eterler (PBDEs), kullanımları yasaklanmış veya kısıtlanmış olmalarına rağmen, çevrede hala ppb seviyelerinde tespit edilmektedir. Bu çalışmada Konya Kapalı Havzasında bulunan 46 yüzeysel su kaynağından 38 yüzeysel su ve 28 sediment örneği alınmıştır. Alınan örneklerde PBDE (PBDE47, PBDE100, PBDE99, PBDE154, PBDE153) kirliliği araştırılmıştır. Su numunelerinde tespit edilen konsantrasyonlar ile balık, Daphnia magna ve alg için risk değerlendirmesi gerçekleştirilmiştir. Su numunelerinde toplam PBDE konsantrasyonları 0.96-74.5 ng/L aralığında, sediment numunelerinde 0.01-0.64 µg/kg kuru ağırlık aralığında tespit edilmiştir. Su numunelerinde PBDE47 kongeneri baskın, sediment numunelerinde PBDE100, PBDE153, PBDE47 kongenerleri baskın tespit edilmiştir. PBDE47 kongeneri balık, Daphnia magna ve alg için iki yüzeysel su kaynağında yüksek dereceli risk, PBDE154 ve PBDE153 balık için üç yüzeysel su kaynağında yüksek dereceli risk, PBDE154 Daphnia magna için iki, PBDE153 Daphnia magna için üç yüzeysel su kaynağında yüksek dereceli risk sergilemektedir. Sonuçlar göstermiştir ki, Konya Kapalı Havzasında bulunan yüzeysel sularda PBDE kirliği akuatik yaşamı etkileyecek derecede mevcuttur. Havzadaki sular içme, tarımsal sulama başta olmak üzere pek çok amaç için kullanılmaktadır. PBDE içeren ticari ürünler halen kullanımdadır ve atık oluşturmaktadır. Özellikle elektronik atıklar için etkili bir atık yönetim planı uygulanmalı, PBDE'ler de dahil olmak üzere mikrokirleticiler çevresel ortamlarda düzenli olarak izlenmelidir.

Anahtar kelimeler: Yüzeysel su kaynağı, su, sediment, polibromlu difenil eter, PBDE, risk.

1 Introduction

The quality of water is deteriorating due to natural and anthropogenic sources. Population growth, rapid development of industry, discharge of waste to the receiving environment without treatment, pesticides and fertilization cause water pollution. The most common pollutants in water resources are nitrate, nitrite, ammonia, heavy metals and toxic compounds [1,2]. Polybrominated diphenyl ethers (PBDEs) are used as fire retardants. They are used in a wide range of products, including building materials, upholstery foam, electrical and electronic equipment, furniture, motor vehicles, aircraft, plastics, polyurethane foams, and textiles [3]. Because of their low cost and excellent flame-retardant effects, PBDEs have been manufactured about 1.5 million tons since the 1970s in global [4, 5]. Routes of entry of PBDEs into the environment are their production, use, and disposal methods such as incineration of waste, storage in landfills, or discharge to urban wastewater treatment plants [3, 6]. There are 209 PBDE congeners which depend on the number of bromine atoms and their position in the aromatic ring. PBDEs are commercially available as three technical mixtures: penta-BDE, octa-BDE, deca-BDE [7, 8]. Penta-BDE, octa-BDE technical mixtures are mostly used in polyurethane foam materials, and deca-BDE technical mixtures are used in electronic materials [9]. Penta-BDE and octa-BDE in 2009, deca-BDE in 2017 were added to the Stockholm Convention. The Stockholm Convention bans or limits the use of persistent organic pollutants [10]. However, the commercial congener deca-BDE is still produced and used in the production

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of cars, trucks and aircraft in some countries [11, 12]. Countries that are parties to the Stockholm Convention are obliged to eliminate PBDE production or reduce their emissions to the environment [13]. Although PBDEs are banned, they are found in trace concentrations in the environment. Studies show that PBDEs are persistent, long-term transportable, bioaccumulating, and toxic compounds in the environment. They have lipophilic characters and so they accumulate in fatty structure. [11]. In many studies, the presence of PBDE compounds has been detected in various environments [14-17]. PBDEs have also been detected in human samples such as breast milk, serum, placenta, and adipose tissue [11, 18, 19]. The bioaccumulation and biomagnification potentials of PBDE congeners are affected by their octanol-water coefficients (logKow) and whether the food web is on land or in water. Logkow values of tetra-BDE and penta-BDE congeners are between 5.9 and 7.2, and they have the greatest biomagnification potential [20, 21]. Among PBDE congeners, highly brominated PBDEs are less toxic than low-brominated PBDE [7]. In addition, low-brominated PBDEs are more mobile in the environment and thus enter the food chain more easily [12]. PBDE congeners may affect thyroid hormones, liver enzymes, and cause DNA damage [7, 14]. PBDEs are designated as group 3 by the International Agency for Research on Cancer (IARC). It is meaning, they are not classifiable as human carcinogens; however, the EPA (Environmental Protection Agency) classified the deca-BDE congener as a compound with suggestive evidence of carcinogenic potential. Despite the potential toxic effects of PBDEs, tolerable ingestion doses of these contaminants are not yet available [22]. Water resources are one of the important environmental media. It can be easily exposed to environmental pollutants. Pollutants found in water can affect both aquatic organisms and other terrestrial organisms, including humans, through the food chain. PBDEs are important environmental pollutants that need to be investigated as explained above. In the Konya Closed Basin Management Plan, PBDE (28, 47, 100, 99, 153,154) in 16 surface water sources in the basin exceeded the environmental quality standard (140 ng/L) given in the Surface Water Quality Regulation. In surface water sources exceeding environmental quality standard, PBDEs were detected in the range of 150-960 ng/L. The target date for reaching the environmental target in the report is stated as 2024 [23].

In this study, the concentrations of PBDE47, PBDE100, PBDE99, PBDE153, and PBDE154 congeners were investigated in 39 surface water and 28 sediment samples taken from the Konya Closed Basin. Additionally, the ecotoxicological risk exhibited by PBDE in surface water resources was evaluated for the test organisms algae, *Daphnia magna*, and fish.

2 Materials and methods

2.1 Water and sediment samples

The Konya Closed Basin in the middle of Türkiye is situated between latitudes 36°51'N and 39°29'N and longitudes 31°36'E and 34°52'E. The basin includes land from 9 provinces. The urban population is 2150514, the rural population is 618695, resulting a total of 2768900 people living within the basin borders. 57% of the basin lands are agricultural lands, 33% are natural areas (steppe, anthropogenic steppe, forest), 7.9% are wetlands and water bodies, and 2% are artificial areas. The rivers in the Konya Closed Basin are mostly seasonal and flood in character. The streams are short due to the closed basin and topography; they disappear in the marshes on the plain. Stream regimes are irregular due to variability in rainfall, and most of them dry up in the summer. The basin, which includes Beyşehir with 34 lakes and 58 rivers, also includes Salt Lake. Salt Lake, which is the second largest lake in Türkiye, meets 40% of the country's salt demand. Beyşehir Lake is the third largest lake in Türkiye. [23, 24]. The State Hydraulic Works have determined surveillance monitoring points for general purposes to monitor water resources that are not at risk. It has also identified operational monitoring points for water resources at risk, where priority substances are discharged, and protection areas within the basin, such as drinking water protection areas and habitat protection areas. Thirty-eight water samples and 28 sediment samples were taken from 46 surface water sources in the Konya Closed Basin. Samples could not be taken from some sample points due to field conditions and water source characteristics. The sampling points include 21 surveillance monitoring points, 18 operational monitoring points, and 2 protected areas. The sampled water resources are used for irrigation purposes in the Konya Closed Basin. Sampling was carried out once a year in April. Although April is expected to be a rainy month in Konya, no rain was observed during the sampling period. The most important pollution sources affecting water bodies in the basin are wastewater discharges, solid waste, agricultural areas, industrial facilities and residential areas. In the Konya Closed Basin Measures Program Summary Report, the potential pressures for PBDEs were stated as discharges from industrial facilities and urban discharges. Table 1 shows the surface water sources from which samples were taken. Figure 1 shows the sample points on the map.

Table 1. Surface water and sediment sampling points

Sample number	Water sources	Monitoring purpose	Water	Sediment
1	Dinamo stream	Surveillance	+	+
2	Sugla stream	Surveillance	+	+
3	Suludere stream	Surveillance	+	+
4	Ulucay stream	Surveillance	+	+
5	Agzıkarahan stream	Surveillance	+	+
6	Melendiz stream	Operational	+	+
7	Karasu stream	Operational	+	+
8	Ilısu river	Surveillance	+	+
9	Ulu Irmak stream	Surveillance	+	+
10	Insuyu stream	Operational	-	+
11	Ozdere stream	Operational	+	+
12	Yanarkac stream	Operational	+	+
13	Uçharman stream	Operational	+	+
14	Uludere Kavaklık stream	Surveillance	+	+
15	Yesildere Kızıllarağırı stream	Surveillance	+	+
16	Yesildere Nalima stream	Surveillance	+	-
17	Yesildere Akköprü stream	Operational	+	+
18	Bozyer stream	Surveillance	+	-
19	Dolav stream	Operational	+	-
20	Kurucay-Hoyuklu-Yazı stream	Surveillance	+	-
21	Donrul-Bag-Kocacay stream	Operational	+	+
22	Ilmen stream	Operational	+	-
23	Salur stream	Operational	+	-
24	Yenisarbademli stream	Surveillance	+	+
25	Derebucak dam	Surveillance	+	-
26	Kavakbasi stream	Operational	+	-
27	Beysehir stream	Operational	+	-
28	Battal stream	Surveillance	+	-
29	Kusla stream	Surveillance	+	-
30	Kırgecit stream	Surveillance	+	-
31	Çiftehan stream	Surveillance	+	-
32	Aydın Kent stream	Operational	+	-
33	Carsamba stream	Surveillance	+	-
34	Gök stream	Operational	+	+
35	Akkaya stream	Surveillance	+	-

36	Göksu river	Surveillance	+	-
37	Ecemis stream	Operational	+	+
38	May dam	Protected	+	+
39	Atlanti irrigation channel	Protected	+	-
40	Avşar dam	Operational	-	+
41	Hizar stream	Operational	-	+
42	Göktepe stream	Unstated	-	+
43	Bıçakcı stream	Unstated	-	+
44	Atınapa dam	Unstated	-	+
45	Tekke stream	Unstated	-	+
46	Kula stream	Unstated	-	+

+: sampled, -: not sampled

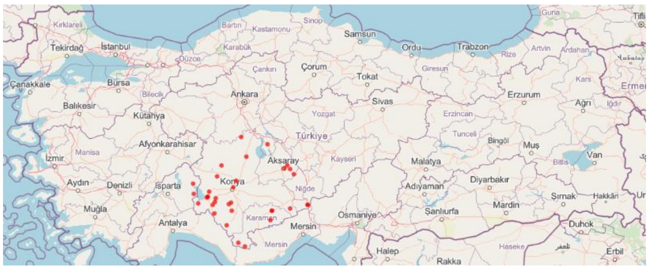

Figure 1. Water and sediment sampling points in Konya closed basin

2.2 PBDE analysis in water and sediment samples

Samples were kept at 4°C until analyzed. No filtering process was applied to the water samples before extraction. PBDE analysis in water samples was performed using the liquid-liquid extraction method in accordance with US EPA Method 3510. A 200 mL water sample was placed in a 250 mL separating funnel. The extraction was carried out with 40 mL of dichloromethane. The separating funnel was shaken for 2 minutes. After allowing the organic layer to separate from the water phase, the next step is to carefully collect the separate layer. The extraction was repeated two times and organic phases were combined. The extracts were dried with anhydrous Na_2SO_4 and reduced to approximately 2 mL with a rotary evaporator. PBDE analysis in sediment samples was performed according to the DFG (German Research Association) S19 multi method. The extraction was carried out with an acetone-water mixture (2:1, v/v) using fifty grams of sediment sample in the dark for 12 hours at 221 rpm. After shaking, approximately 15 g sodium chloride and 100 mL cyclohexane were added and shaken for another 1h. After shaking, shaking approximately 120 mL aliquot of organic phase was taken and dried with 15 g of anhydrous Na_2SO_4. Subsequently, 100 mL of the organic phase was filtered and concentrated to 2 mL using a rotary evaporator. Then, the concentrated organic phase was cleaned up.

Water and sediment extracts were cleaned using the standard column chromatography technique according to US EPA Method 3630C; Silica Gel Cleanup. A glass column with a 1.5 cm diameter and 14.5 cm length was used with glass wool placed at the bottom. The column was cleaned with 50 mL of n-hexane and 50 mL of acetone. Silica gel in n-hexane was added to avoid air bubbles. 50 mL of n-hexane was used to condition the column. Elution was carried out with 70 mL of n-hexane followed by 100 mL of n-hexane/ethyl acetate (1/1, v/v) mixture. Eluents were collected separately. Eluents were concentrated with a rotary evaporator and nitrogen gas.

Qualitative and quantitative analyses of PBDEs were performed on a gas chromatograph (GC, Agilent 6890 N, Agilent Technologies, CA, USA) equipped with a mass selective detector (MSD, Agilent 5973, Agilent Technologies, CA, USA). A DB-5ms capillary column with 250 µm inner diameter, 0.25 µm film thickness, and 30 m length was used for chromatographic separation. Helium was used as the carrier gas.

2.3 Analytical performance

Table 2 presents the results of the limits of detection (LOD), limit of quantifications (LOQ), linear response range, linearity, and repeatability obtained with the GC-MS system for PBDE compounds. The LOD and LOQ values for PBDEs were calculated according to published guidelines at a signal-to-noise ratio (S/N) of 3 and 10, respectively [25]. The repeatability values for the target compounds in the MS detector were determined by calculating the %RSD (Relative Standard Deviation) of the MS responses from five injections of the standard solution at a concentration of 0.1 ng/µL. The repeatability was found to be between 1.2% and 3.1%. Analytical curves were drawn using eight PBDE standards within the concentration range of 0.001-1 ng/µL. The coefficient of determination (R^2) was in the range of 0.9997-0.9999.

Table 2. Analytical parameters of the GC-MS system for PBDEs

PBDEs	LOD (ng/L)	LOQ (ng/L)	Linear response range (ng/µL)	R^2	RSD
PBDE47	0.005	0.016	0.001-1	0.9998	1.2
PBDE100	0.006	0.020	0.001-1	0.9997	2.3
PBDE99	0.007	0.020	0.001-1	0.9999	2.4
PBDE154	0.006	0.020	0.001-1	0.9997	3.1
PBDE153	0.005	0.016	0.001-1	0.9998	2.7

For the recovery and precision assays, samples of surface water were spiked with PBDEs standards at the concentration of 0.1 µg/L and 1 µg/L. The extraction method was applied to both unfortified and fortified surface water samples. The average recoveries of all PBDEs in the surface waters fortified with 0.01 µg/L and 0.1 µg/L were 87% (±5)-99% (±5) and 93% (±5)-101% (±4), respectively (n=4). Also, samples of sediment were spiked with PBDEs standards at the concentration of 0.01 mg/kg and 0.1 mg/kg. The extraction method was applied to both unfortified and fortified sediment samples. The average recoveries of all PBDEs in the sediments spiked with 0.01 mg/kg and 0.1 mg/kg ranged from were 85% (±4)-94% (±6) and 92% (±3)-96% (±5), respectively (n=4). When recoveries of PBDEs were gauged against absolute limits of 70% and 130% [26], it was seen that the extraction and clean-up method gave satisfactory results. The retention times and m/z values of the compounds are given in Table 3.

Table 3. Retention time and m/z value of PBDEs

PBDEs	Retention time, min	Ion (m/z value)
PBDE47	8.09	326, 486, 484
PBDE100	8.82	404, 406, 564
PBDE99	9.06	404, 406, 564
PBDE154	9.62	484, 482, 486
PBDE153	9.93	484, 482, 486

2.4 Risk assessment

The presence of hazardous substances in the aquatic environment may negatively affect the ecosystem. Risk refers to the likelihood of harmful effects occurring in an organism as a result of exposure to hazardous substances [27]. In the study, ecotoxicological risk was assessed using the risk quotient (RQ). Ecotoxicological risk assessment was conducted on fish, *Daphnia magna* and algae. Fish, *Daphnia magna* and algae are good indicators of aquatic toxicity. They represent three different trophic levels in the aquatic environment. The first trophic level, algae, is extremely sensitive to pollutants. *Daphnia magna,* which feeds on algae, is a natural food for fish [28].

The RQ is calculated as the quotient of the measured environmental concentration (MEC) divided by the predicted non-effect environmental concentration (PNEC) of the substance [29]. PNEC is the highest concentration of a substance in an environment at which it does not produce any effects. The PNEC is obtained by dividing the toxicological relevant concentration (EC50 or LC50 values) by a safety factor (f = 1000) [15]. EC50 or LD50 data of PBDE compounds used in this study were obtained from the literature. PNEC and RQ are calculated using equation (1) and equation (2), respectively.

$$PNEC = EC50 \text{ or } LC50/1000 \quad (1)$$

$$RQ = MEC/PNEC \quad (2)$$

Table 4 shows the PNEC values of PBDE for fish, *Daphnia magna*, and algae. When RQ <0.001, this means no risk. If 0.01 ≤ RQ<0.1, then this indicates a low risk. When 0.1 ≤ RQ < 1, it indicates a medium risk; when 1 ≤ RQ, it indicates a high risk [30].

Table 4. PNEC of PBDEs in water [15; 30]

PBDEs	Fish (ng/L)	*Daphnia magna* (ng/L)	Algae (ng/L)
PBDE47	21	18	24
PBDE100	4	4	24
PBDE99	4	4	24
PBDE154	0.69	0.72	7
PBDE153	0.69	0.72	7

3 Results and discussion

3.1 PBDEs detected in water samples

The concentration of PBDEs in water samples has been presented in Figure 2. In water samples, PBDE congeners were detected in the range of 0.13-73.1 ng/L for PBDE47, in the range of 0.08-0.92 ng/L for PBDE100, in the range of 0.09-0.40 ng/L for PBDE99, in the range of 0.16-1.17 ng/L for PBDE154, and in the range of 0.12-1.46 ng/L for PBDE153. According to the environmental quality standards for rivers and lakes, the Surface Water Quality Regulation in force in Türkiye, specifies the maximum allowable total PBDE (PBDE28, 47, 100, 99, 154, 153) concentration as 140 ng/L. PBDE28 was not investigated in this study. The total concentration of the investigated PBDE compounds was determined to range from 0.96 to 74.5 ng/L. It was determined that the total concentration did not exceed the maximum limit value.

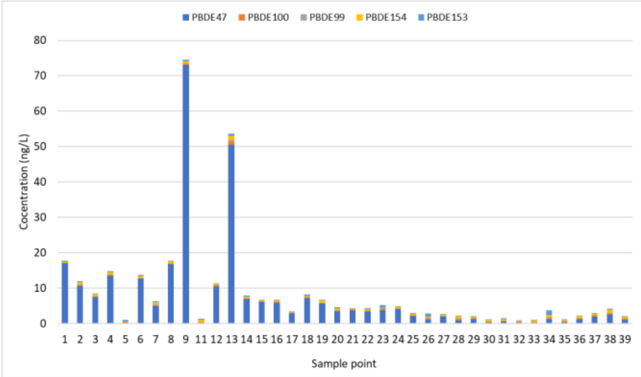

Figure 2. PBDE concentrations detected in surface waters

Figure 3 shows the distributions of PBDE congeners in each sample. In water samples, the PBDE47 congener ranged from 9.60% to 98.05%, PBDE100 from 0.28% to 26.55%, PBDE99 from 0.31% to 19.85%, PBDE154 from 0.73% to 53.66%, and PBDE153 from 0.62% to 38.11%. According to Figure 3, PBDE47, PBDE154, and PBDE153 congeners were detected dominantly in 81.6%, in 13.16%, and 5.26%, of the water samples, respectively. The PBDE47 congener was detected in the highest concentrations in water samples. It was followed by PBDE153, PBDE154, PBDE100, and PBDE99 congeners. Penta-BDE contains PBDE47 (36.11%), PBDE99 (41.49%), PBDE100 (9.22%), PBDE153 (4.49%), and PBDE154 (3.18%). Octa-BDE contains PBDE153 (rate of 4.07%) and PBDE154 (rate of 0.51%) [3]. The high concentrations of PBDE47 in water samples were attributed to it being one of the main components of penta-BDE. PBDE99 was detected at low concentrations, however, it was also found in high amounts when part of the penta-BDE mixture. In literature studies, PBDE47 was detected more frequently than PBDE99 [31, 32]. This is thought to occur through atmospheric deposition, transport of contaminants, or debromination of the PBDE99 congener [32, 33]. PBDE47 was detected as the dominant congener in the water. Ge et al. [33] detected the PBDE47 congener predominantly in water samples. PBDE47 and PBDE99 congeners are the degradation products of PBDE209, and PBDE209 is still used in some countries. High brominated congeners may decompose over time and form low brominated congeners. Increasing temperatures in summer may cause the decomposition of congeners [34].

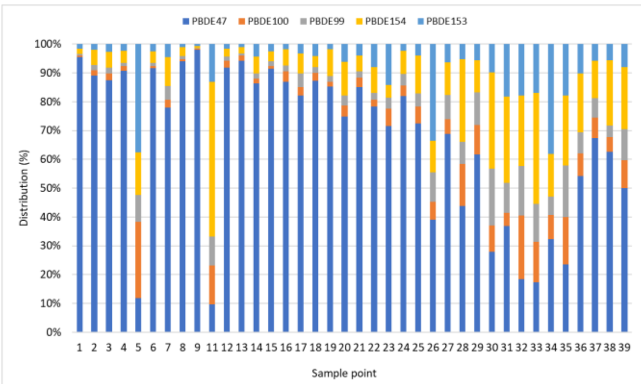

Figure 3. Distributions of PBDEs in water samples

Total PBDE concentrations were higher at sample points 9 and 13, compared to other sample points. Pollutant sources around the sample areas included traffic, agriculture, residential areas, fishing, urban and domestic discharges, and industrial discharges.

In the sample points where concentrations were detected to be high, urban discharges flow into water resources, near agricultural residential areas. Olaniyan et al. [35] stated that the primary PBDE sources were industrial discharges, inadequate waste disposal, atmospheric pollution from local and imported e-waste, and surface runoff from residential and agricultural areas.

There are 9 organized industrial zones and 58 industrial areas in Konya. The sectors of machinery and equipment, metal products, food products, rubber and plastic, furniture, wood and cork products, leather products, automotive, and non-metallic mineral products are located in Konya province [36]. The Konya Closed Basin partially covers the provinces of Isparta, Nigde, Ankara, Aksaray, Nevsehir, and Karaman [23]. There are also important industrial sectors in these regions. PBDEs are applied as flame retardants in a wide variety of consumer products. Since PBDEs are additives that are not chemically bound to products, they can leak into the air. They can be adsorbed onto atmospheric particles and accumulate in water [37]. Some studies have reported that PBDEs are not treated in wastewater treatment plants but accumulate, especially in treatment sludge, and reach the receiving environments [38]. Industrial plant flue gases and wastewater are important sources of PBDEs. It has been reported that atmospheric PBDE concentrations in industrial areas are higher than in urban and residential areas [39]. Atmospheric transport and accumulation are the most important factors in the spread of PBDEs. In the Konya Closed Basin Measures Program summary report, it was stated that PBDE pressures in the basin originate from point-source industries such as machinery, weapons, automotive, metal, chrome plating, and the hunting industry sectors [23].

Table 5 shows the PBDE concentrations detected in surface waters in previous literature and in this study. The PBDE47 congener was detected in China at lower concentrations in 2017, 2018, 2019 and 2021 than in this study, while other congeners were detected at similar concentrations. PBDE100 and PBDE153 concentrations were found to be similar between this study and South Africa, but PBDE47 was detected at levels below the detection limit (dl) levels in South Africa. Concentrations of PBDE99 and PBDE153 were found to be higher in Korea than the concentrations reported in this study. The concentration of the PBDE47 congener was also lower in Korea than in this study. The PBDE47 concentration was similar in Bulgaria and to that in this study, while other congeners were detected at higher concentrations in Bulgaria than in this study. The concentration of PBDE154 detected in Nigeria is similar to that of this study, while the concentration of PBDE47 is lower.

In Pakistan, PBDE congeners, except for the PBDE47 congener, were detected at higher concentrations than those reported in this study. When studies conducted in the Czech Republic and France were examined, PBDE47 congener was detected at low levels in France, while other congeners were found in similar concentrations. Studies on PBDE pollution in Türkiye have focused on the atmosphere. Cetin [40] and the Summary report of Konya Closed Basin measures program [23] investigated PBDEs in surface waters. The concentrations detected in the study conducted in 2006 in Izmir, Türkiye, were found to be lower than those found this study. Summary report of Konya closed basin measures program ΣPBDE (28, 47, 100, 99, 154, 153) results are given.

In this study, the concentration of PBDE47 was generally higher than that reported in other studies. These comparisons have shown that surface waters in our country are contaminated with the pollutant congener PBDE47.

Table 5. PBDEs detected in surface waters worldwide (ng/L)

Country	PBDE47	PBDE100	PBDE99	PBDE154	PBDE153	Reference
China	Rainy season 0.021-0.394 Dry season <dl-1.82	Rainy season 0.041-0.961 Dry season 0.017-2.56	Rainy season 0.077-1.22 Dry season <dl -3.32	Rainy season 0.002-0.205 Dry season <dl -0.048	Rainy season <dl -0.213 Dry season <dl -0.094	[3]
China	Rainy season 0.13-0.73 Dry season 0.05-4.5	Rainy season 0.21-0.54 Dry season 0.10-0.20	Rainy season 0.15-0.29 Dry season 0.03-0.12	Rainy season 0.15-0.88 Dry season <dl-0.09	Rainy season 0.17-0.28 Dry season <dl-0.09	[16]
China	0.38	0.01	0.08	0.05	<dl	[31]
China	0.96	0.0043	1.4	0.0069	2.8	[41]
South Africa	<dl	Rainy season <dl-0.082 Dry season <dl-0.0329	-	-	Rainy season <dl -0.131 Dry season <dl	[35]
Korea	<dl-6.15	<dl	<dl-2.46	<dl-0.21	<dl-10.4	[34]
Bulgaria	50-120	4	4-18	4-10	12-18	[42]
Nigeria	Rainy season 6.63,8.69 Dry season 4.64, 6.64	Rainy season 1.32, 4.14 Dry season 0.78, 1.44	Rainy season 4.52, 5.43 Dry season 3.57, 4.30	Rainy season 2.43, 3.81 Dry season 1.57, 1.27	Rainy season 4.03, 3.28 Dry season 2.68, 3.83	[17]
Pakistan	0.08-13.2	0.06-14.0	0.05-14.4	0.04-7.13	0.04-11.9	[43]
Czech Republic	27.44	-	0.963	0.835	0.316	[44]
France	0.046-0.205	0.013-0.029	0.054-0.181	0.009-0.028	0.014-0.038	[45]
Türkiye, Izmir	Dissolved phase	Dissolved phase	Dissolved phase	Dissolved phase	Dissolved phase	[40]

	Summer 0.04 Winter 0.0095 Particle phase Summer 0.034 Winter 0.011	Summer 0.011 Winter 0.0034 Particle phase Summer 0.011 Winter 0.0038	Summer 0.057 Winter 0.014 Particle phase Summer 0.053 Winter 0.016	Summer 0.0054 Winter 0.0016 Particle phase Summer 0.0057 Winter 0.0018	Summer 0.0082 Winter 0.0025 Particle phase Summer 0.0083 Winter 0.0027	
Türkiye, Konya	0.13-73.1	0.07-0.92	0.09-0.40	0.16-1.17	0.13-1.46	In this study

<dl: below detection limit -: not analyzed

3.2 PBDEs in sediment samples

Figure 4 shows the concentrations of PBDEs detected in sediment samples. PBDE concentrations in sediment samples were determined as 0.01 to 0.098 µg/kg dry weight for PBDE47, 0.01 to 0.464 µg/kg dry weight for PBDE100, 0.01 to 0.032 µg/kg dry weight for PBDE99, 0.0003 to 0.161 µg/kg dry weight for PBDE154, and 0.004 to 0.041 µg/kg dry weight for PBDE153. Total PBDE concentrations in sediment samples were determined to be 0.01-0.64 µg/kg dry weight.

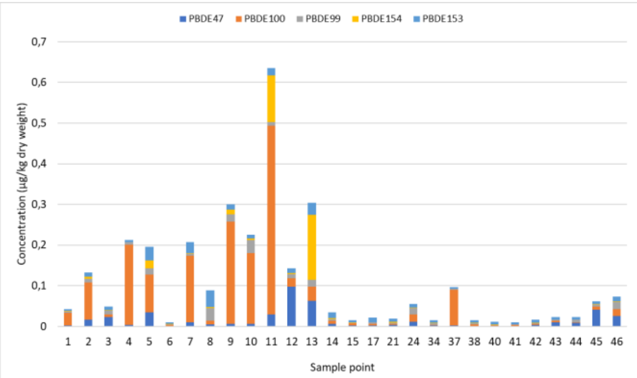

Figure 4. PBDE concentrations detected in sediments

Figure 5 shows the distributions of PBDE congeners detected in each sediment sample. In sediment samples, the PBDE47 congener ranged from 1.73% to 68.60%, PBDE100 from 6.34% to 92.77%, PBDE99 from 1.30% to 34.73%, PBDE154 from 0.29% to 52.96%, and PBDE153 from 2.31% to 55.97%. When Figure 5 is examined, PBDE47, PBDE100, PBDE153, and PBDE154 congeners are detected dominantly in 21.4%, 39.3%, 35.7%, and 3.6% of sediment samples, respectively. The PBDE100 congener was detected in the highest concentrations in sediment samples. It was followed by PBDE154, PBDE47, PBDE153, and PBDE99 congeners. The highest total PBDE concentration was detected at sample point 11. Urban discharge is released into the surface water source. The congener distribution profile in sediment samples differed from that in water samples. While PBDE47 congener was detected as the dominant congener in 81.6% of water samples, PBDE100 and PBDE153 were detected in 39.3% and 35.7% of sediment samples, respectively. PBDE47 was detected as a dominant congener in 21.4% of sediment samples. High molecular weight congeners are more likely to accumulate in sediment, while low molecular weight congeners tend to accumulate in water [46].

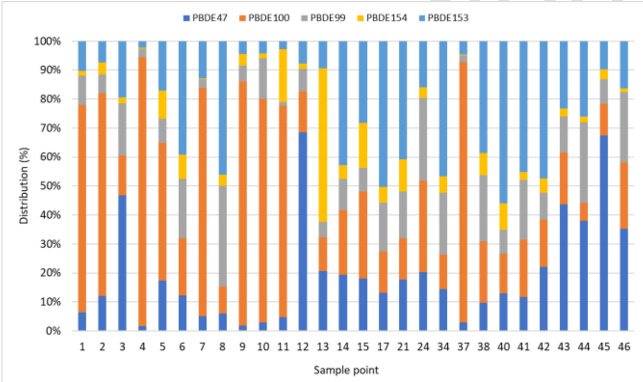

Figure 5. Distributions of PBDEs in sediment samples

Oloruntoba et al. [17] reported that the amount of PBDEs detected in the sediment of artificial wetland ponds located in e-waste storage areas was approximately three times much as than that in water. They stated that PBDEs adsorb suspended solids and accumulate in the bottom sediment due to their hydrophobic character. Due to the slow flow of stream waters, PBDE congeners in water can be adsorbed onto suspended solids and then accumulate in the bottom sediment [32]. Lee et al. [34] reported that 95% of the PBDE in the sediment was PBDE209 in 2003, but, in contrast, 59%, 25%, and 6.6% of the PBDE in the sediment were PBDE209, PBDE47, and PBDE99 in 2013. They thought that lower-brominated congeners increased because of debromination of PBDE209 in the environment over time. Wei et al. [31] stated that PBDE47 was the main congener detected in water and sediment samples. Table 6 shows the concentrations of PBDEs detected in sediments worldwide. The concentration of PBDE47 is lower than that found in Pakistan, Nigeria, Korea, South Africa and Hong Kong. The concentration of PBDE100 is similar to the concentrations detected in China, Korea, Hong Kong, France and lower than the concentrations detected in Nigeria and Pakistan. The concentration of PBDE99 was lower than the concentrations detected in Pakistan, Nigeria, Korea, Hong Kong, ans was similar to the concentrations detected in China and France. The concentrations of PBDE154 and PBDE153 were also lower than the concentrations detected in Pakistan, Nigeria, Hong Kong, and were similar to the concentrations detected in China, South Africa, France. It is observed that the PBDEs in Pakistan Nigeria and Hong Kong are at higher concentrations than in other countries. It may be due to the lack of an effective waste management system. In China, the PBDE209 congener, which was not investigated in this study, is actively used frequently detected in various environmental settings [3]. With this comparison, it was observed that the concentrations detected in sediment samples were lower or similar to the world average. Both water and sediment samples were taken from 20 of the sampling points. Total PBDE concentrations in water and sediment samples taken from the same point are given in Figure 6. No correlation (R^2=0.09) was found between the total PBDE concentrations detected in water and sediment. Water and sediment samples were collected from streams. At some sampling points, the water flow rate was very high, while at others, it was quite slow. PBDEs need sufficient time to adsorb suspended particles before accumulating in sediment [17]. The low correlation between PBDE concentrations in water and sediment is thought to be primarily due to variations in flow rate.

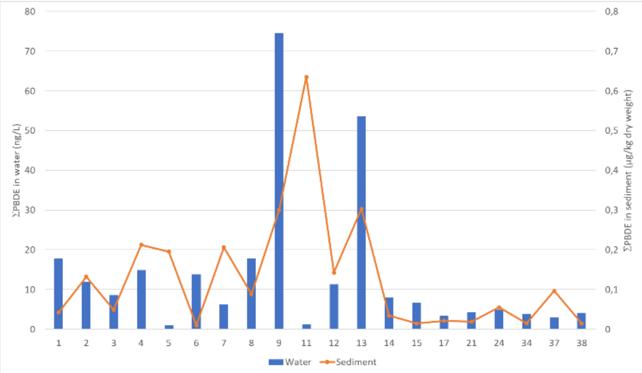

Figure 6. ΣPBDE in water and sediment samples

The distribution rates of congeners in total PBDE concentrations detected in water and sediment samples are given in Figure 7. Figure 7 shows that 87% of the PBDE concentration detected in water samples is attributed to the PBDE47 congener, and the distribution of other congeners is between 2% and 5%. PBDE47 was detected predominantly in water samples. In sediment samples, the detected PBDE concentration is composed of the following: 56% PBDE100, 14% PBDE47, 11% PBDE153, 11% PBDE154, and 8% PBDE99. The PBDE100 congener was prevalently determined in sediment samples

Table 6. PBDEs detected in sediment worldwide (ng/g dry weight)

Country	PBDE47	PBDE100	PBDE99	PBDE154	PBDE153	Reference
China	Rainy season 0.0139-0.190 Dry season <dl-0.0305	Rainy season 0.0135-0.168 Dry season 0.000666-0.082	Rainy season 0.0185-0.296 Dry season 0.000470-0.207	Rainy season <dl -0.00749 Dry season 0.000203-0.0128	Rainy season 0.00148-0.0245 Dry season 0.000032-0.0226	[3]
China	Rainy season 0.09-7.6 Dry season <dl-3.6	Rainy season 0.10-3.8 Dry season <dl-0.19	Rainy season 0.03-0.92 Dry season <dl-1.3	Rainy season 0.05-2.4 Dry season <dl-0.52	Rainy season 0.13-3.5 Dry season <dl-0.64	[16]
China	3.6	0.14	0.36	0.11	0.02	[31]
China	2.3	0.27	3.0	0.51	3.6	[41]
South Africa	Rainy season 0.00344-0.105 Dry season 0.000314-0.404	Rainy season <dl-0.712 Dry season 0.00023-0.218	-	-	Rainy season 0.000805-0.411 Dry season 0.000077-0.104	[35]
Korea	<dl-21.0	<dl-0.26	<dl-8.01	<dl	<dl	[34]
Nigeria	Rainy season 7.91-14.9 Dry season 13.6-8.48	Rainy season 14.71-12.5 Dry season 6.42-14.1	Rainy season 14.1-18.6 Dry season 11.2-15.3	Rainy season 2.23-6.42 Dry season 5.35-3.04	Rainy season 14.4-23.2 Dry season 9.28-16.4	[17]
Pakistan	0.06-15.9	0.02-16.8	0.08-24.2	0.03-8.55	0.02-14.3	[43]
France	0.19-0.78	0.09-0.540	0.34-0.299	0.05-0.1	0.07-0.13	[45]
Hong Kong	0.36-2.55	0.01-0.23	0.3-8.46	0.06-2.45	0.23-5.36	[47]
Türkiye	0.01-0.098	0.01-0.464	0.01-0.32	0.0003-0.161	0.004-0.41	In this study

<dl: below detection limit -: not analyzed

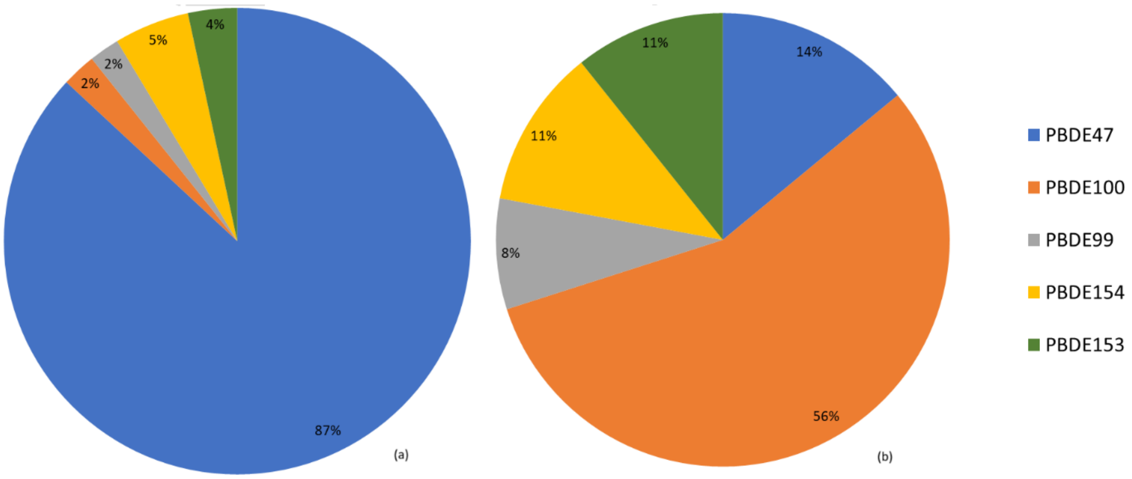

Figure 7. Distributions of total PBDE compounds detected in Konya Closed Basin (a) water, (b) sediment

3.3 Risk assessment

The calculated RQs are given in Table 7. In Table 7, PBDE47 congener showed a high risk for all trophic levels in 5.26% of the samples, and a moderate risk for fish in 55.3%, for *Daphnia magna* in 60.5%, and for algae in 52.7% of the samples. PBDE100 showed moderate risk for fish and *Daphnia magna* in 2.63% of the samples, while the remaining samples exhibited either no risk or low risk. PBDE99 showed low risk for fish and *Daphnia magna* in all samples. For algae, the analysis showed a low risk in 21% of the samples, while no risk was observed in the other samples. PBDE154 showed high risk in 7.9% of the samples for fish and 5.3% of the samples for *Daphnia magna*, and moderate risk in the remaining samples. PBDE153 caused high risk in 7.9% of the samples for fish and *Daphnia magna*, and moderate risk in the other samples. PBDE153 and PBDE154 showed moderate risk in 7.9% of the samples for algae, while no risk was detected in the other samples. PBDE47, PBDE154, and PBDE153 posed a high risk to aquatic organisms. Only the PBDE99 congener posed no significant risk. Moderate risk was observed in at least one sample point and at least one trophic level for all other congeners. In the study, the aquatic organisms least adversely affected by PBDEs were algae. This result was also observed in the studies conducted by Xiong et al. [15, 48]. Liu et al. [30] determined low risk for algae and moderate risk for fish and *Daphnia magna* in their risk assessment of PBDEs using the RQ method and emphasized that more attention should be paid to the aquatic environment. Xiong et al. [15] stated that RQ values for fish, *Daphnia magna*, and algae were less than 1 at all sampling points, and no ecotoxicological risk was expected. Li et al. [49] determined that the RQ value was <0.1 in surface waters in China and evaluated it as a low ecological risk. In this study, the ecotoxicological risk of PBDEs has been assessed. Considering that other pollutants are also present in aquatic environments along with PBDEs, the actual risk is likely higher than what has been identified.

Table 7. RQ of water samples (No risk; Low risk; Medium risk; High risk)

Sample point	PBDE47			PBDE100			PBDE99			PBDE154			PBDE153		
	Fish	*Dahphnia magna*	Algae	Fish	*Daphnia magna*	Algae	Fish	*Daphnia magna*	Algae	Fish	*Daphnia magna*	Algae	Fish	*Daphnia magna*	Algae
1	0.8115	0.9468	0.7101	0.0265	0.0265	0.0044	0.0249	0.0249	0.0042	0.4531	0.4342	0.0447	0.4149	0.3976	0.0409
2	0.5058	0.5901	0.4426	0.0531	0.0531	0.0088	0.0581	0.0581	0.0097	0.9061	0.8684	0.0893	0.3319	0.3181	0.0327
3	0.3560	0.4153	0.3115	0.0531	0.0531	0.0088	0.0415	0.0415	0.0069	0.6796	0.6513	0.0670	0.3319	0.3181	0.0327
4	0.6425	0.7496	0.5622	0.0619	0.0619	0.0103	0.0415	0.0415	0.0069	0.9061	0.8684	0.0893	0.4979	0.4771	0.0491
5	0.0060	0.0070	0.0052	0.0707	0.0707	0.0118	0.0249	0.0249	0.0042	0.2265	0.2171	0.0223	0.5808	0.5566	0.0573
6	0.6029	0.7034	0.5276	0.0265	0.0265	0.0044	0.0415	0.0415	0.0069	0.7929	0.7598	0.0782	0.4979	0.4771	0.0491
7	0.2325	0.2713	0.2035	0.0442	0.0442	0.0074	0.0747	0.0747	0.0125	0.9061	0.8684	0.0893	0.4149	0.3976	0.0409
8	0.7971	0.9300	0.6975	0.0354	0.0354	0.0059	0.0498	0.0498	0.0083	0.7929	0.7598	0.0782	0.2489	0.2386	0.0245
9	3.4810	4.0611	3.0458	0.0531	0.0531	0.0088	0.0581	0.0581	0.0097	0.7929	0.7598	0.0782	0.6638	0.6361	0.0654
11	0.0060	0.0070	0.0052	0.0442	0.0442	0.0074	0.0332	0.0332	0.0055	1.0194	0.9769	0.1005	0.2489	0.2386	0.0245
12	0.4975	0.5804	0.4353	0.0707	0.0707	0.0118	0.0415	0.0415	0.0069	0.4531	0.4342	0.0447	0.2489	0.2386	0.0245
13	2.4069	2.8081	2.1061	0.2299	0.2299	0.0383	0.0996	0.0996	0.0166	1.6990	1.6282	0.1675	0.8298	0.7952	0.0818
14	0.3260	0.3804	0.2853	0.0354	0.0354	0.0059	0.0332	0.0332	0.0055	0.6796	0.6513	0.0670	0.4979	0.4771	0.0491
15	0.2925	0.3412	0.2559	0.0177	0.0177	0.0029	0.0249	0.0249	0.0042	0.3398	0.3256	0.0335	0.2489	0.2386	0.0245
16	0.2793	0.3258	0.2444	0.0619	0.0619	0.0103	0.0332	0.0332	0.0055	0.5663	0.5427	0.0558	0.1660	0.1590	0.0164
17	0.1355	0.1580	0.1185	0.0265	0.0265	0.0044	0.0415	0.0415	0.0069	0.3398	0.3256	0.0335	0.1660	0.1590	0.0164
18	0.3404	0.3972	0.2979	0.0531	0.0531	0.0088	0.0415	0.0415	0.0069	0.4531	0.4342	0.0447	0.4979	0.4771	0.0491
19	0.2697	0.3147	0.2360	0.0265	0.0265	0.0044	0.0332	0.0332	0.0055	0.9061	0.8684	0.0893	0.1660	0.1590	0.0164
20	0.1666	0.1944	0.1458	0.0442	0.0442	0.0074	0.0415	0.0415	0.0069	0.7929	0.7598	0.0782	0.4149	0.3976	0.0409
21	0.1762	0.2056	0.1542	0.0354	0.0354	0.0059	0.0249	0.0249	0.0042	0.3398	0.3256	0.0335	0.2489	0.2386	0.0245
22	0.1618	0.1888	0.1416	0.0265	0.0265	0.0044	0.0249	0.0249	0.0042	0.5663	0.5427	0.0558	0.4979	0.4771	0.0491
23	0.1798	0.2098	0.1573	0.0796	0.0796	0.0133	0.0498	0.0498	0.0083	0.3398	0.3256	0.0335	1.0787	1.0337	0.1063
24	0.1918	0.2238	0.1678	0.0442	0.0442	0.0074	0.0498	0.0498	0.0083	0.5663	0.5427	0.0558	0.1660	0.1590	0.0164
25	0.1019	0.1189	0.0892	0.0442	0.0442	0.0074	0.0332	0.0332	0.0055	0.5663	0.5427	0.0558	0.1660	0.1590	0.0164
26	0.0539	0.0629	0.0472	0.0442	0.0442	0.0074	0.0747	0.0747	0.0125	0.4531	0.4342	0.0447	1.4106	1.3518	0.1390
27	0.0899	0.1049	0.0787	0.0354	0.0354	0.0059	0.0581	0.0581	0.0097	0.4531	0.4342	0.0447	0.2489	0.2386	0.0245
28	0.0455	0.0531	0.0399	0.0796	0.0796	0.0133	0.0415	0.0415	0.0069	0.9061	0.8684	0.0893	0.1660	0.1590	0.0164
29	0.0611	0.0713	0.0535	0.0531	0.0531	0.0088	0.0581	0.0581	0.0097	0.3398	0.3256	0.0335	0.1660	0.1590	0.0164
30	0.0156	0.0182	0.0136	0.0265	0.0265	0.0044	0.0581	0.0581	0.0097	0.5663	0.5427	0.0558	0.1660	0.1590	0.0164
31	0.0276	0.0322	0.0241	0.0177	0.0177	0.0029	0.0415	0.0415	0.0069	0.6796	0.6513	0.0670	0.4149	0.3976	0.0409
32	0.0084	0.0098	0.0073	0.0531	0.0531	0.0088	0.0415	0.0415	0.0069	0.3398	0.3256	0.0335	0.2489	0.2386	0.0245
33	0.0084	0.0098	0.0073	0.0354	0.0354	0.0059	0.0332	0.0332	0.0055	0.5663	0.5427	0.0558	0.2489	0.2386	0.0245
34	0.0588	0.0687	0.0515	0.0804	0.0804	0.0134	0.0604	0.0604	0.0101	0.8237	0.7894	0.0812	2.1121	2.0241	0.2082
35	0.0144	0.0168	0.0126	0.0531	0.0531	0.0088	0.0581	0.0581	0.0097	0.4531	0.4342	0.0447	0.3319	0.3181	0.0327
36	0.0587	0.0685	0.0514	0.0442	0.0442	0.0074	0.0415	0.0415	0.0069	0.6796	0.6513	0.0670	0.3319	0.3181	0.0327
37	0.0959	0.1119	0.0839	0.0531	0.0531	0.0088	0.0498	0.0498	0.0083	0.5663	0.5427	0.0558	0.2489	0.2386	0.0245
38	0.1235	0.1440	0.1080	0.0531	0.0531	0.0088	0.0415	0.0415	0.0069	1.3592	1.3026	0.1340	0.3319	0.3181	0.0327
39	0.0515	0.0601	0.0451	0.0531	0.0531	0.0088	0.0581	0.0581	0.0097	0.6796	0.6513	0.0670	0.2489	0.2386	0.0245

4 Conclusion

In this study, PBDE47, PBDE100, PBDE99, PBDE154, and PBDE153 congeners were investigated in water and sediment samples. Samples were collected from surface water in the Konya Closed Basin. PBDEs were detected in the range of 0.13-73.1 ng/L for PBDE47, 0.08-0.92 ng/L for PBDE100, 0.09-0.40 ng/L for PBDE99, 0.16-1.17 ng/L for PBDE154, and 0.12-1.46 ng/L for PBDE153 in water samples. Total PBDE concentration ranged from 0.96 to 74.5 ng/L in water samples. PBDEs in sediment samples were found to be 0.01-0.098 µg/kg dry weight for PBDE47, 0.01-0.464 µg/kg dry weight for PBDE100, 0.01-0.032 µg/kg dry weight g for PBDE99, 0.0003-0.161 µg/kg dry weight for PBDE154, and 0.004-0.041 µg/kg dry weight for PBDE153. Total PBDE concentrations were determined to be 0.01-0.64 µg/kg dry weight in sediment samples. While the PBDE47 congener was dominant in water samples, however, the PBDE100, PBDE153, and PBDE47 congeners were dominant in sediment samples. The dominance of PBDE47 congener suggests the presence of the commercial penta-PBDE mixture. When the obtained results are compared with other studies conducted worldwide, it has been determined that the surface water resources in the Konya Closed Basin are contaminated with PBDEs. The identification of high pollution levels in water resources receiving urban and industrial discharges has emphasized the need to focus on industrial areas. The risk assessment results showed thatPBDE47, PBDE154, and PBDE153 congeners were detected to present a high risk to fish, *Daphnia magna* and algae at some sample points. This situation has demonstrated that there is PBDE contamination in surface waters at levels that can affect aquatic life. The risk assessment was conducted individually for each congener. Congeners and other environmental pollutants are present in water, and when their toxic effects on living beings are combined, it may result in more serious risks.

There is no information on whether PBDE is used in our country. However, detectable levels of PBDE have been detected in different environmental environments media [50] Products containing PBDE were imported before becoming a party to the Stockholm Convention. The regulation on persistent organic pollutants is in force in our country. The list of substances subject to restriction is in Annex 2 of the regulation. PBDEs are included in this list. The production, use and marketing of products containing PBDEs are permitted in some exceptional cases. The use of items containing PBDEs, which were in use before the regulation came into force, is permitted. Electronic devices, vehicles, etc., especially those containing PBDE, should be disposed of appropriately after their useful life ends. During the processing of these wastes, parts containing PBDE must be separated and stored appropriately. The import of second-hand electronic products into our country is prohibited. However, restricting the second-hand electronics market within the country would prevent the use of those that have reached the end of their life. Raising public awareness and encouraging the collection of electronic waste are crucial. PBDEs are micropollutants with high logKow values (PBDE47: 6.55, PBDE99: 7.13, PBDE100: 6.86, PBDE153: 7.62, PBDE154: 7.39; [14]). Therefore, they can show high accumulations in seafood, agricultural products, terrestrial organisms, and humans over time. The surface water resources examined in the study are used for irrigation purposes and may cause PBDEs to reach the food chain.

In the study we conducted in the Konya Closed Basin, it was determined that surface water resources were contaminated with PBDEs and posed an ecological risk. The sampling points in the study are adequate and representative. However, only a single sampling was conducted may limit the generalizability of the results. It is recommended that future studies include more sampling efforts. To fully determine the extent and source of pollution, detailed research is also required in the water, atmosphere, soil and products grown in the region.

5 Author contribution statements

1: methodology, sampling, analysis, writing, reviewing, and editing

2: methodology, sampling, analysis, writing, reviewing, and editing

3: methodology, sampling, analysis, writing, reviewing, and editing

6 Ethics committee approval and conflict of interest statement

There is no need to obtain permission from the ethics committee for the article prepared.

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