Science of the Total Environment 878 (2023) 162966

Contents lists available at ScienceDirect

Science of the Total Environment

journal homepage: www.elsevier.com/locate/scitotenv

The effect of military conflict zone in the Middle East on atmospheric persistent organic pollutant contamination in its north

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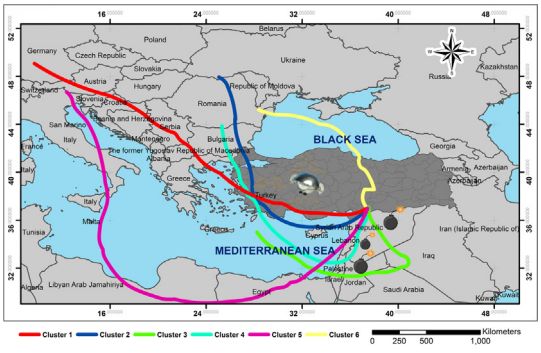
HIGHLIGHTS

- Effect of a military-conflict zone in the Middle East to its north was investigated.
- Passive sampling on a southeast to west transect at ten points in Anatolia
- Data analyses indicate POP transport from the south of the border.
- Back-trajectory analysis provides support.
- Different parts of the study area impacted by LRAT from different regions.

GRAPHICAL ABSTRACT



ARTICLE INFO

Editor: Paromita Chakraborty

Keywords:
Long-range atmospheric transport (LRAT)
Polybrominated diphenyl ethers (PBDEs)
Polychlorinated biphenyls (PCBs)
Organochlorine pesticides (OCPs)
Military conflict
War

ABSTRACT

This study aimed to investigate long-range atmospheric transport of selected POPs released due to the effects of military conflicts in regions to the south of Turkey's borders. Ten locations were selected to deploy passive air samplers at varying distances to the border on a southeast-west transect of the country, proximity-grouped as close, middle, and far. Sampling campaign included winter and transition months when desert dust transport events occur. Hypothesis of the study was that a decreasing trend would be observed with increasing distance to the border. Group comparisons based on statistical testing showed that PBDE-183, Σ_{45}PCB, and dieldrin in winter; PBDE-28, PBDE-99, PBDE-154, *p,p′*-DDE, Σ_{14}PBDE, and Σ_{25}OCP in the transition period; and PBDE-28, PBDE-85, PBDE-99, PBDE-154, PBDE-190, PCB-52, Σ_{45}PCB, *p,p′*-DDE, and Σ_{25}OCP over the whole campaign had a decreasing trend on the transect. An analysis of concentration ratio to the background showed that long-range atmospheric transport impacted the study sites, especially those of close group in comparison to the local sources. Back-trajectory analyses indicated that there was transport from the conflict areas to sites in the close-proximity group, while farther sampling locations mostly received air masses from Europe, Russia, and former Soviet Union countries, followed by North Africa, rather than the military conflict areas. In consequence, decrease in concentrations with distance and its relation to molecular weight through proportions, diagnostic ratios, analysis of concentration ratio to the background, and back-trajectory analyses support the effect of transport from the military-conflict area to its north.

1. Introduction

Persistent organic pollutants (POPs) have become of global interest as these transboundary pollutants are subject to long-range transport including atmospheric and aquatic pathways. These toxic synthetic organic

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http://dx.doi.org/10.1016/j.scitotenv.2023.162966
Received 10 November 2022; Received in revised form 10 March 2023; Accepted 16 March 2023
Available online 21 March 2023

chemicals are ubiquitously found in all environmental compartments including those of remote areas (Daly and Wania, 2005; Qiu et al., 2020; Shen and Wania, 2005) due to their resistance to environmental conditions. Because POPs are a global problem that can lead to serious health effects, the Global Stockholm Convention (SC) for the reduction and elimination of emerging and legacy POPs, that include polybrominated diphenyl ethers (PBDEs), polychlorinated biphenyls (PCBs), and organochlorine pesticides (OCPs), in the environment was signed in 2001, entered into force in 2004, and was updated in 2017 (UNEP, 2017). Global PCB production was 1,325,810 tons between 1930 and 1993 (Breivik et al., 2002; Dai et al., 2016). Although they were never produced, the use of PCBs was banned in Turkey in 1996 (Ministry of Environment and Forestry, 2010). PBDEs were also never produced in Turkey while total production around the world reached 1.3 to 1.5 million tons between 1970 and 2005 (UNEP, 2010). Penta and octa-BDE commercial mixtures were banned in 2009 followed by deca-BDE mixture in 2017 by SC, including Turkey (Kurt-Karakus et al., 2018). OCPs are the most produced legacy POPs, with a global total estimate for only DDT of 4.5 million tons from 1940s to 2005 (Li and Macdonald, 2005). Three million tons of the legacy pesticides were produced by 1998 in Western Europe (25 %), Asia (16 %), Latin America (13 %), Japan (12 %), Africa (2 %), and Eastern Europe (2 %), whereas Turkey was the user of these chemicals and the reported 0,63 kg per hectare usage value was the lowest among others (17.5, 3.5, 4.4, 7.6, 6 kg for Netherlands, USA, Germany and France, Italy, Greece, respectively) (Ministry of Environment and Forestry, 2010).

Today, long-range atmospheric transport (LRAT) is considered to be the primary path of transport for many POPs to remote areas such as Arctic where these chemicals have never been manufactured or used (Corsolini and Sarà, 2017). POPs may be transported in the atmosphere in the gas phase and/or bound to particulate matter. Seasonal temperature changes cause POPs to cycle through the atmosphere, volatilizing and moving through it when it is warm and depositing when it is cold, a phenomenon known as “the grasshopper effect”. If the temperature increases, the rate of POPs passing into the gas phase increases, and becomes available for LRAT (Hageman et al., 2015).

Soil plays an important role as a sink for POPs because its organic matter content renders it open to their accumulation (Sweetman et al., 2005; Dalla Valle et al., 2005). In particular, soils used for agricultural activities are sources or reservoirs of agrochemical POPs that act as primary emission or re-emission (secondary) sources to the atmosphere (Wang et al., 2012). The organic matter content of the soil (SOM) and the proximity to the source point strongly influence the background POPs concentrations (Meijer et al., 2003). For example, Saharan dust storms have been shown to transport pesticides, PAHs, and PCBs (Garrison et al., 2014). Saharan dust outbreaks often observed in western Turkey (Baltaci, 2017) and the Mediterranean Basin (Kabatas et al., 2018) during the transition periods, i.e., spring and fall. Rastgeldi Dogan et al. (2020) reported that City of Sanliurfa, in southeastern Turkey, receives dust from the Sahara Desert, the Syrian desert, and even from the Arabian desert. Turkey, due to its location, is under the influence of air masses transported from different areas. Previous studies showed that pollutants came from northern Europe, parts of Turkey, Ukraine, Russia, and some Balkan regions (Türküm et al., 2008), Russia and Middle Eastern countries (Jordan, Saudi Arabia and Iraq), the north and the south (Uygur et al., 2010), Eastern Mediterranean especially to western Turkey (Kabatas et al., 2014). In addition, there is a clear transport of air masses from Romania to Istanbul, while long-range transport is seen from southern France (Im et al., 2013). Air masses come over Turkey crossing Ukraine, as well as the coastal regions of Bulgaria and Romania during daytime sampling, and the Black Sea during nighttime sampling (Zemmer et al., 2012). Similar findings were reported by back-trajectory and potential source contribution function analyses for POPs measurement at different sites of Turkey (Kuzu and Saral, 2017; Yenisoy-Karakaş et al., 2012). The source areas associated with the measured Σ_{14}PBDE concentrations in İzmir included northern Africa, southern and eastern Europe (Gungormus et al., 2021).

During the Balkan conflicts between 1991 and 1999, both military and industrial facilities were destroyed. With the destruction of the industrial plant in Kragujevac, Serbia, a large amount of pollutants (PCBs) were released into the environment (Turk et al., 2007). United Nations Environment Program (UNEP) identified that 2500 kg of PCBs leaked out into Morava river in Kragujevac (UNEP, 1999). Later, Radonic et al. (2009) reported that the concentrations of PAHs and PCBs varied by more than three orders of magnitude at different sampling points, including the background areas in Bosnia and Herzegovina, Croatia, and Serbia.

Large quantities of POPs may be released into the environment and be subject to transport during war, creating particular source areas. Therefore, areas of conflict/war to the south of Turkey specifically Iraq, Syria, Lebanon, and Palestine/Israel may be sources of POPs for which the literature is very limited (Gevao et al., 2022a, 2022b). Based on this hypothesis, a 7-month passive sampling campaign was conducted by proximity grouping ten sampling points in Anatolia on a transect from the southern border region of the country, as close and middle distance, to the west (Izmir) and northwest (Bursa) forming the far distance group. Climatology of the sampling sites was studied by monthly cluster analysis of the air mass back-trajectories for each sampling site.

2. Material and methods

2.1. Study area and sample collection

Polyurethane foam disk passive air samplers (PUF-PAS), that are useful and cost-effective tools for monitoring of POPs (Harner et al., 2004; Melymuk et al., 2021), were concurrently deployed at ten sites in this study. Sampling sites were selected on a southeast to west transect in Turkey: Adana, Gaziantep, Osmaniye, Karaman, Malatya, Denizli, Isparta, Mugla, Izmir, and Bursa (Fig. 1). The sampling points were selected to be outside the city centers to minimize the urban effect. Samplers were placed at least 2 m above the ground level. Adana, Gaziantep, Osmaniye, Karaman, and Malatya were grouped as the close group in proximity to the border while Isparta, Denizli, and Mugla constituted the middle-proximity group. In the far distance group, samples were collected in Izmir and Bursa, representing different directions for air mass flow. Passive samplers were deployed for a month for 7 months between December 2018 and July 2019. Duplicate sampling was performed every two months at one sampling location in each group (Adana, Isparta, Bursa). Additional information about sampling and sampling locations are given in the Supplementary Material (SM) Table S1.

2.2. Chemicals and reagents

Analytical grade solvents and anhydrous-Na_2SO_4 (granulated, trace organic analysis grade) were procured from Merck (Merck EMD Millipore, USA). Recovery compounds PCB-14, PCB-65, PCB-166 and PBDE-77, PBDE-181, and depuration compounds PCB-30, PCB-107, and PCB-198 were purchased from AccuStandard (New Haven, CT, USA). $^{13}C_{12}$-labelled PCB-9, PCB-15, PCB-32 (Cambridge Isotope Laboratories (MA, USA)) were also used as depuration compounds and $^{13}C_{12}$-PCB-105 (Cambridge Isotope Laboratories (MA, USA)) was used as internal standard. Target chemicals of this study are listed in the Table S2.

2.3. Sampler preparation and deployment

Flame-retardant-free certified polyurethane foam (PUF) disks (Tisch Environmental, Ohio, USA) were cleaned using a series of organic solvents (at least 18 h each, acetone, 1:1 Acetone: Hexane; Hexane) on a Soxhlet apparatus and dried under vacuum. PUF disks were spiked with the depuration compounds (120 ng each except d_6-ɣ-HCH: 200 ng) prior to sampling to determine sampled air volume (Shoeib and Harner, 2002) based on the rate of chemical uptake being equal to the rate of depuration compound loss. In order to reduce the effects of particle deposition, UV (sunlight) and wind speed on the sampling rate, PUF discs were placed in

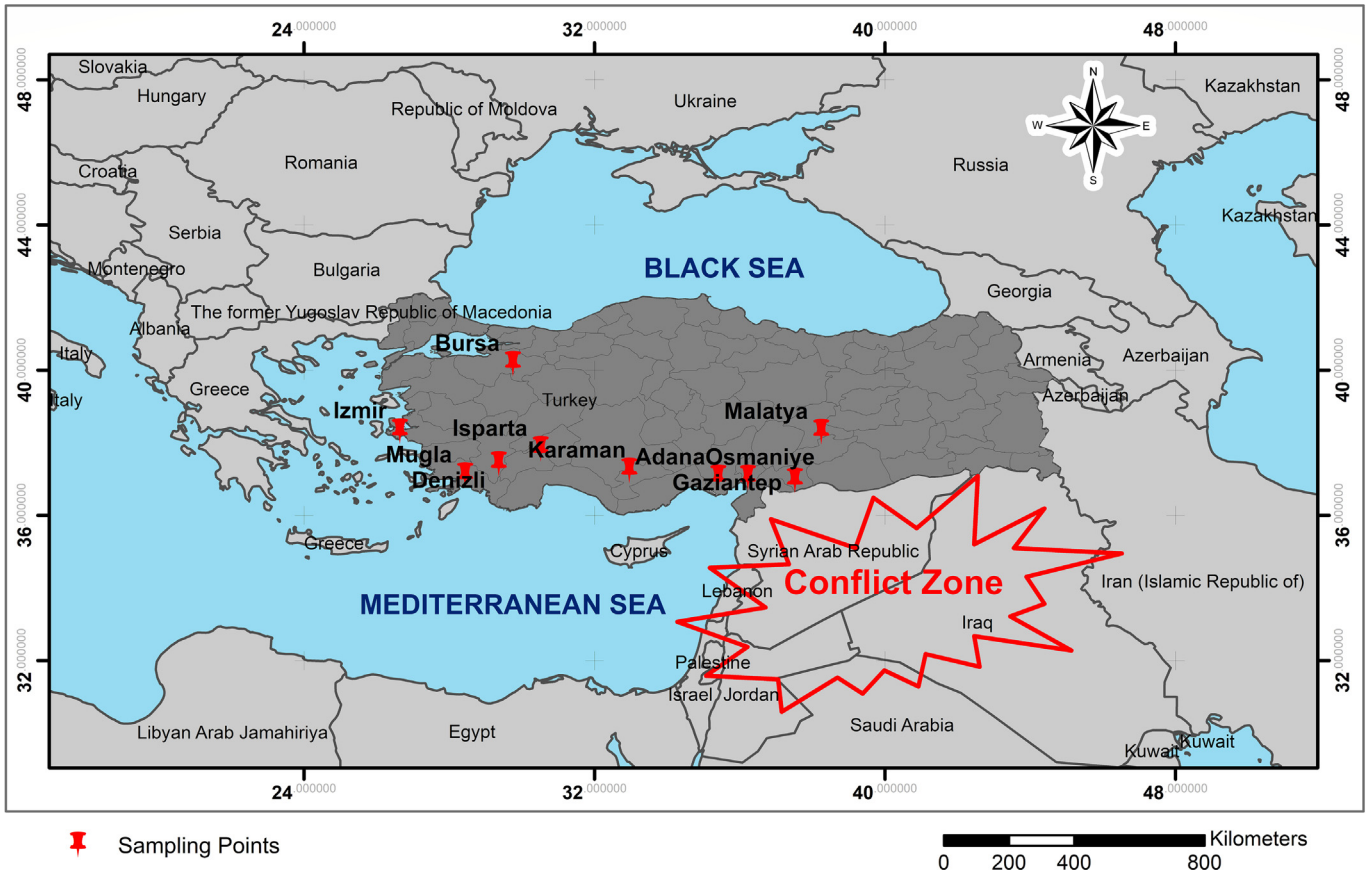

Fig. 1. Sampling sites.

stainless steel chambers at the sampling sites (Tuduri et al., 2006). More detailed information on sampler preparation is given in Table S3.

2.4. Extraction

Just prior to extraction, PUF disks were spiked with recovery surrogate chemicals (50 ng PCB-14, PCB-65, PCB-166, and 25 ng PBDE-77, −181). Then they were extracted with acetone:hexane mixture (1:1 and 300 mL) on a Soxhlet extractor for 18 h. Further details on sample preparation and clean-up of extracts are given elsewhere (Kurt-Karakus et al., 2018). Later final volume was reduced to 1 mL isooctane, and all extracts were spiked with internal standard (12 ng of $^{13}C_{12}$PCB-105) before instrumental analysis. Samples were kept at −20 °C until instrumental analysis.

2.5. Instrumental analysis

PDBEs (14 congeners), PCBs (45 congeners), and OCPs (25 compounds) were the targeted POPs in this study. While OCPs and PBDEs were analyzed in negative chemical ionization mode, PCBs were analyzed in electron impact ionization using a GC–MS (Agilent, 7890B GC and 5977 MSD). PCB-41/64 and PCB-90/101 were the co-eluting PCB congeners. Details of the analysis parameters are shown in Table S4.

2.6. QA/QC

All glassware were pre-cleaned at 450 °C and rinsed with acetone and hexane mixture (1:1) before sampling and sample preparation processes. Field and laboratory blanks were processed, and recoveries of surrogate compounds were determined to ensure the satisfactoriness of sample processing and analysis procedures. The Instrument Detection Limit (IDL) was the lowest signal producing level that is distinguishable from a reagent blank at a signal to noise ratio of 3:1 (Ripp, 1996) while the Method Detection Limit (MDL) was calculated with mean of blanks +3 × SD (3 ≈ Student t-value of single tailed 99th percentile t statistics, USEPA, 2016). IDL was used to calculate MDL using sampling volumes for target chemicals that were not detected in the blank samples. MDLs and IDLs are shown in Tables S5, S6, S7). The mean recovery efficiency of complete sample preparation procedure (extraction and concentration) were 84 ± 14 % and 84 ± 13 % for PBDE-77, PBDE-181, and 91 ± 13 %, 86 ± 15 %, 84 ± 16 % for PCB-14, PCB-65, PCB-166, respectively.

2.7. Deriving sampled effective air volumes

Atmospheric concentrations of targeted compounds were calculated by dividing accumulated amount of compound in PUF disk (ng/sampler) by corresponding effective air volume (V_{AIR}, m^3). Site-specific effective air volumes (V_{AIR}) were determined using the equation by Shoeib and Harner (2002), which assumes that the uptake was linear ($R_{sampling}$; m^3/day). The sampling volumes of PUF-PAS calculated depending on the desorption of the depuration compounds spiked before sampling, and the GAPS (Global Atmospheric Passive Sampling) template (version:2021_v10, received from Tom Harner with personal communication) was used for calculations (Harner, 2016; Parnis et al., 2016). The average uptake rate was determined to be 6.11 ± 1.27 m^3/day, for which the variation occurred (Table S1) due to variation in meteorological variables (Klánová et al., 2008; Tuduri et al., 2006).

2.8. Statistical analysis

Normal distribution assumption for congeners was tested using Shapiro Wilk test ($\alpha = 0.05$). Pearson correlation analysis for the normally-distributed congeners and Spearman correlation analysis for the remaining ones were performed to evaluate the distance-concentration relationship. Kruskal-Wallis and ANOVA tests were performed to test differences in

median and mean concentrations between groups. Principle Components Analysis (PCA) was conducted for the three proximity groups on all detected compounds through the whole sampling period, considering an Eigenvalue of > 1 for significance and loadings of > 0.5. All statistical analyses were conducted with SPSS v23.

2.9. Back-trajectory analysis

The Hybrid Single-Particle Lagrangian Integrated Trajectory (HYSPLIT) model is useful for the study of long-range transport of air masses (Zeng and Hopke, 1989; Draxler et al., 1999). In this study, monthly cluster analysis was performed with HYSPLITv4 model in order to determine the main routs of air masses received at each sampling location. In this study, monthly global reanalysis (.gbl) meteorological datasets were used for back trajectory analysis. Six hour trajectories were constructed through the whole sampling period. The orbital frequency initiates an orbit from a single point and altitude each 6 h. It then sums the frequency at which the trajectory passes over a grid cell, and finally normalizes it to the total number of trajectories or endpoints. An orbital can intersect a grid cell one or more times (Draxler et al., 1999). Trajectories were clustered per 24 h. The simulated heights were set to 500 m and 1000 m above ground level (AGL). The cluster analysis of Gaziantep is given as an example in Fig. S1 in SM for the first month of sampling. The number of clusters required for cluster analysis was calculated by calculating the percent change in total spatial variance (TSV) (Fig. S1(c)) to the sum of SPVAR (Spatial Variance). Large changes in TSV mean that different trajectories are combined in the same cluster, and the required number of clusters is found before the large percent change in TSV. Another possibility is to use a percentage change criterion (20 % or 30 %) (Draxler et al., 1999). In this study, 30 % change was used to obtain required cluster number as can be seen in Fig. S1(b) and same time in Fig. S1(c). Cluster analysis of the samples are given in SM from Fig. S2 to Fig. S21 (Number at the bottom left corner refers to sampling month).

3. Results and discussion

3.1. Ambient air concentrations

The median, mean, and geometric mean values were calculated for the proximity groups (Table S8). Turkey is under the influence of southern winds especially in transition seasons (Spring and Autumn) (Rastgeldi Dogan et al., 2018). For this reason, the concentrations were divided into two seasonal groups, winter (December–February) and spring (March–August), and it was investigated whether a clearer proximity relationship could be observed in the transition season. However, the frequency of southerly winds was the highest in winter in the close sampling group, while the far group was mostly under the influence of northerly air masses originating from Europe and Russia.

3.1.1. PBDEs

The levels of studied PBDE congeners in each group are shown for each congener in Fig. 2 (a, b, c). The average of Σ_{14}PBDEs concentrations were 517 pg/m^3, 247 pg/m^3, and 395 pg/m^3 in close, middle, and far groups, respectively. The dominant congeners and their contributions to Σ_{14}PBDEs in this study were from PBDE-47 (33.88 pg/m^3–7%), PBDE-100 (31.16 pg/m^3–6%), PBDE-183 (15.55 pg/m^3–3%), PBDE-190 (95.95 pg/m^3–19%), and PBDE-209 (325.23 pg/m^3–63%) based on the seven-month mean concentration. Countrywide studies are limited for Turkey. Kurt-Karakus et al. (2018) reported annual Σ_{14}PBDE concentrations of 183 ± 260 pg/m^3 and 200 ± 387 pg/m^3 for urban and rural sites across the country, respectively. Countrywide annual average was found to be Σ_{14}PBDE 191 ± 329 pg/m^3 with contributions from PBDE-190 (42 %), PBDE-99 (24.4 %), and PBDE-47 (20.6 %), while the average concentrations measured along the southeast-west transect in this study were higher than those both of the urban and rural countrywide levels.

Congener profiles differed in the middle and far groups in terms of PBDE-209 contribution being lower (~20 %) compared to the close group. Far group differed from the others as it included PBDE-17 and -28 (<10 %). PBDE-209 is considered to have low bioavailability and a tendency to bind tightly with soil and sediment (Ji et al., 2017; Tian et al., 2011). It is hypothesized that the instant increases in temperature during explosion and combustion results in release of POPs such as PCDD and PAHs (Lammel et al., 2013). Therefore, higher concentration/contribution in the close group may be related to the war zone because of its tendency of binding soil. The lower contribution of PBDE-209 (85.4 pg/m^3–22%) and considerable contributions of PBDE-17 (17.2 pg/m^3–4%) and PBDE-28 (28.4 pg/m^3–7%) in far group may be indications of atmospheric transport from short and long distances.

Overall, average Σ_{14}PBDE concentrations in winter and transition seasons were similar (237 and 234 pg/m^3, respectively). Congener concentration profiles were similar except PBDE-190 (90.1 and 120 pg/m^3) and PBDE-209 (137 and 388 pg/m^3). Average concentrations of the proximity groups were similar in the whole, winter, and transition periods for majority of the congeners except for PBDE-190 and PBDE-209 that had close > far > middle ranking. The differences were greater for the latter. The average concentrations of close, middle, and far groups for PBDE-209 were 325, 51.4, and 85.4 pg/m^3 during the whole sampling duration, 137, 35.0, and 52.5 pg/m^3 in winter, and 388, 62.4, 107 pg/m^3 in the transition periods. The above ranking, i.e., gradient towards the middle and southeastern locations having higher concentrations than those of western Turkey, may indicate that in addition to the local sources southeastern locations impacted by atmospheric transport from the south of the border whereas western locations by that from Europe and Eurasia. In fact, there is a relation between congener molecular weight (MW) and proportion of close and far groups in the total of the three groups (Fig. S22). The proportion decreases by MW for close group while it increases for far group, which may be related to the above argument (please also see Section 3.2 PCBs). There are supporting findings by the back-trajectory analysis presented in Section 3.2. The lighter congeners have higher LRAT potential (Wania and Dugani, 2003), and higher concentration and proportion of lighter congeners at Izmir and Bursa stations may implicate the effect of both short and LRAT as there are known sources such as those in Aliaga and Dilovasi industrial areas (Besis et al., 2017; Cetin et al., 2018; Gungormus et al., 2021; Kuzu and Saral, 2017).

3.1.2. PCBs

The mean Σ_{45}PCB concentration was 2150 pg/m^3, 1482 pg/m^3, and 2163 pg/m^3 in close, middle, and far groups, respectively. Concentrations in each group are illustrated in Fig. 2 (d, e, f). The dominant congeners with ≥5 % contributions (from high to low) to Σ_{45}PCB concentration in the close group were PCB-123, -44, -118, -203, -18 while they were PCB-118, -203, -41/64, -189, -170 in the middle group, PCB-123, -118, -203, -170 in the far group. The concentration and percent contribution values for the above listed congeners are given in SM (Table S9). Coplanar PCB-123 had the highest contribution in close and middle groups which is mostly found in surface soil (Salihoglu et al., 2013).

Seven-month average concentrations in close, middle, and far groups for PCB-44 (310; 45.2; 39.1 pg/m^3), and PCB-74 (35.0; 25.4; 22.5 pg/m^3) were in decreasing order from close to far group. For many congeners, however, close and far group averages were both higher than those of middle group (PCB-123 and -49) or middle group was higher than at least one of close or far groups (PCB-41/64, -170, -189, -203). Dominant congeners differ both seasonally and between groups.

The highest concentration in the atmosphere of Turkey was measured in the industrial zone of Aliaga, a district of Izmir to the north of the city. Σ_{35}PCB concentrations in the air varied between 349 and 94,363 pg/m^3 with dominance of low molecular weight congeners PCB-18, PCB-28, PCB-31, PCB-33, PCB-52, and PCB-49 (Aydin et al., 2014). Σ_{41}PCB concentration at rural, suburban, urban and industrial/urban sites varied from 177 pg/m^3 to 41,781 pg/m^3 with a mean value of 4152 pg/m^3. The highest Σ_{41}PCB concentration was measured at an industrial/urban site which is

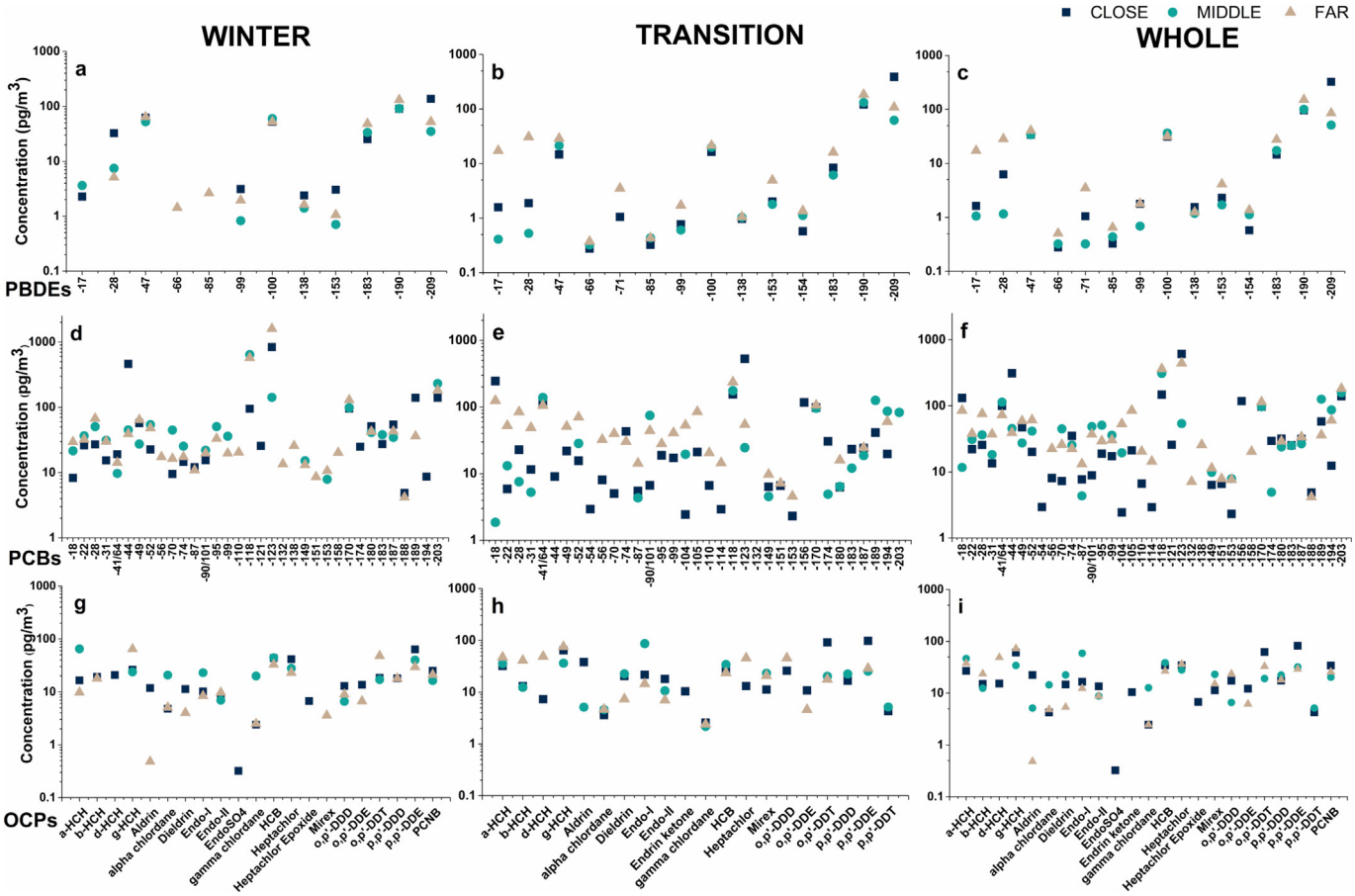

Fig. 2. The mean PBDE (a, b, c), PCB (d, e, f), OCP (g, h, i) concentrations (pg/m^3) in winter, transition, and whole sampling periods.

nearly 4 times higher than the urban site. The reported concentration at the industrial/urban site was between 180 pg/m^3 and 41,781 pg/m^3 for which contribution from PCB-18 was the highest both in winter and summer at the industrial sites while PCB-33 along with PCB-18 had the highest contribution at the urban site (Cetin et al., 2017). Due to the ban on the use of PCBs and containing materials, unintentional lower chlorinated congener emissions from industrial regions to Turkey's atmosphere were reported (Aydin et al., 2014; Odabasi et al., 2009).

Sampling sites in far group are located nearby industrial areas. Aliaga in İzmir, is the main industrial area including petroleum refinery, petrochemical complex, shipbreaking/scrap iron dockyards, scrap-processing iron steel plants, steel rolling mills, a natural gas-fired power plant, a harbor, heavy road and rail traffic (Aydin et al., 2014). Industrial areas of Bursa include factories of motor vehicles and automotive parts, textile and food industries (BCCI, 2021). Birgül et al. (2017) reported that 4-Cl and 3-Cl chlorinated compounds were dominant groups which were resulted from industrial emission and urban effect. In this study, 3-Cl and 4-Cl groups were dominant in far group, former of which has a higher LRAT potential than highly chlorinated congeners (Greenwood et al., 2007). Western Turkey is known to receive POPs via LRAT from Europe and Eurasia (Gungormus et al., 2021; Mulder et al., 2015). Thus, Izmir and Bursa may have high ambient air POP concentrations due to having large metropoles, and they are located nearby industrial areas, in addition to contribution from LRAT. Evaluation of average homolog-group concentrations revealed that the 3-Cl in far (236 pg/m^3) group is 1.2 times the close (192 pg/m^3) group, and 2.4 times the middle (97.1 pg/m^3) group. The concentration of close-proximity group is 1.8 times that of the far group for 4-Cl congeners. The whole campaign, winter, and transition season average concentrations of 7-Cl homolog group were higher in close group than that of far group by about 1.5 times. The relation between congener MW and proportion of close and far groups in the total of the three groups are shown in SM (Fig. S22) for the two seasonal and whole sampling period. Although not as apparent as for PBDEs, there seems to be a subtle relation between congener MW and proportion of close and far groups in the total of the three groups (Fig. S22). The proportion decreases by MW for close group while it increases for far group. If higher concentrations and proportions of higher MW congeners in close group may be attributed to war activity, i.e., sudden disruption and increases of temperature in soil with explosions, the decreasing concentration gradient towards middle group from southeastern Turkey may be attributed to the war zone. In the meantime, higher concentrations and proportions of lower MW congeners in far group may be attributed to the effects of the local industrial emissions and LRAT from the North which could explain the decreasing concentration gradient towards middle group from western Turkey. The movement of air masses during our sampling period, i.e., clustered back-trajectories (presented in Section 3.2 and SM, Figs. S2 – S21), support the above argument.

3.1.3. OCPs

Turkey has large agricultural lands where OCPs were used from 1945 to 1985. Although the use of OCPs has been prohibited in many countries including Turkey, residues are still found in environmental compartments with wide range of concentrations such as air (Can-Güven et al., 2022; Kurt-Karakus et al., 2018) and soil (Akça et al., 2016; Can-Güven et al., 2022). In general, seven-month average OCP concentrations were higher in close group and the differences were increased in transition period (Fig. 2g, h, i). Averages of dominant (with >5 % contribution to the total POP-pesticide concentration) compounds were, in decreasing order, ΣDDT, ΣHCH, PCNB, heptachlor, and HCB in close group; ΣHCH, ΣDDT, α-endosulfan, and HCB in middle group; and ΣDDT, ΣHCH and heptachlor, in far group. The concentration and percent contribution values for the above listed congeners are given in SM (Table S9).

Commercial DDT mixture was consisted of 77 % *p-p'* isomer and 15 % *o-p'* isomeric impurity. Dichlorodiphenyldichloroethylene (DDE) and dichlorodiphenyldichloroethane (DDD) are the main metabolites and environmental degradation products (World Health Organization, 1979). In the environment, *p,p'*-DDT is converted to *p,p'*-DDE by UV (Pacyna et al., 2003) and > 1 of *p,p'*-DDT/*p,p'*-DDE indicate new usage of this chemical whereas < 1 past usage (Jaward et al., 2004) or points to LRAT (Iwata et al., 1995). In the current study, ratio of average *p,p'*-DDT/*p,p'*-DDE concentration (ranged between 0.04 and 0.20) indicated past usage or LRAT with mean values of 0.04 in transition and 0.05 in the whole sampling periods in close group. *p,p'*-DDT not detected in winter period in any of the groups. In the middle group in transition period the ratio was < 1 similar to close group whereas *p,p'*-DDT was not detected during whole sampling campaign. The non-detection or very low concentrations of *p,p'*-DDT along with 20 pg/m^3 to 30 pg/m^3 average *p,p'*-DDE concentrations indicated to the LRAT or past usage (Iwata et al., 1995; Jaward et al., 2004).

The ratio of α-HCH/γ-HCH isomers < 3 indicates lindane as the main source of environmental HCHs, whereas a ratio ranging between 3 and 7 indicates technical HCH as the source of HCHs (Ullah et al., 2019; Adeleye et al., 2016; Venier and Hites, 2014). Values of the ratio were < 3 in close, middle and far groups in this study indicated that lindane is the source of ambient air HCH in the current study, which is in agreement with Can-Güven et al. (2022). The *α/β*-endosulfanand *trans/cis*-chlordane ratios differentiate between the ongoing and past use of these OCPs. In this study TC/CC ratios in close and far groups varied from 0.5 to 0.66 indicating the LRAT of these pollutants during winter and transition seasons and also over the whole sampling campaign. On the other hand, in the middle group these values were > 1 implying that volatilization from fresh sources (Hung et al., 2002, 2005). In Turkey, use of endosulfan has been banned in 2008 (Çok, 2011).

The technical mixing ratio of endosulfan is approximately 70 % *α*-endosulfan and 30 % *β*-endosulfan (Chakraborty et al., 2010). The *α/β*-isomer ratio in this mixture is approximately 2.3. Since *β*-endosulfan isomer is more reactive than *α*-endosulfan isomer, it readily converts to *α*-endosulfan (Nasir et al., 2014). Therefore, a higher *α/β* ratio than the technical mix indicates the effect of aged endosulfan applications or LRAT, while a ratio close to that of the technical mix indicates new application (Pozo et al., 2011; Shunthirasingham et al., 2010). While the *α/β*endosulfan ratio was <2.33 in the close and far proximity groups (over all time periods; winter, transition, whole), indicating volatilization from old use or LRAT, this ratio was 8.3 in the middle group, which is in agreement with Can-Güven et al. (2022). It is reported that pesticide consumption in different agricultural zones in Turkey varies regionally due to agricultural and climatic characteristics (Çok, 2011). The areas for heavy agricultural activities such as the Mediterranean and Aegean accounted for 24 % and 19 %, while it was 18 %, 11 %, and 10 % for Central Anatolia - Thrace, the Black Sea, and Eastern Anatolia, of the total pesticide usage in Turkey (Çok, 2011). There is only one obsolete stock of POP-pesticides mainly (94 %) α-HCH stored in a warehouse in Kocaeli, northwest Turkey (Vijgen et al., 2019; Tauw, 2017).

Evaluation of whole sampling period show that aldrin (22.3 pg/m^3, 5.1 pg/m^3, 0.48 pg/m^3), *β*-endosulfan (13.5 pg/m^3, 8.8 pg/m^3, 8.8 pg/m^3), PCNB (34.1 pg/m^3, 20.3 pg/m^3, 25.7 pg/m^3), *o,p'*-DDT (62.3 pg/m^3, 19.2 pg/m^3, 32.9 pg/m^3), *p,p'*-DDE (82.4 pg/m^3, 32.1 pg/m^3, 29.2 pg/m^3), HCB (34.3 pg/m^3, 38.6 pg/m^3, 26.7 pg/m^3) show decreasing concentration from close to far while β-HCH (15.1 pg/m^3, 12.4 pg/m^3, 23.7 pg/m^3), heptachlor (34.6 pg/m^3, 27.9 pg/m^3, 36.5 pg/m^3), *p,p'*-DDD (17.4 pg/m^3, 22.4 pg/m^3,17.9 pg/m^3) were nearly the same in close and far groups. Southeastern Turkey includes large prominent agricultural areas from 1 to 5 million decare (TUIK, 2020). Although not as large, agriculture is one of the main economic activities in Western Turkey. Thus, the fact that having measured comparable OCP concentrations in close and far groups may be associated with local agricultural lands (Başar, 2001; Hepcan et al., 2013; Kaymakci et al., 2000) suppressing the effect of transport from the war zone. However, similar to PBDEs, there is an apparent relation between congener MW and proportion of close and far groups in the total of the three groups (Fig. S24) indicating that their transport from the south of the border is plausible. Although most of OCPs were showing a decreasing trend both in winter and transition periods, only HCH and DDT species differed.

Various studies investigated atmospheric OCPs in Turkey, such as in urban (Kurt-Karakus et al., 2018), suburban (Ugranli et al., 2016), rural (Lammel et al., 2015), and industrial (Lammel et al., 2015; Odabasi and Cetin, 2012) areas of Turkey. A countrywide study by Kurt-Karakus et al. (2018) showed that the ΣDDT was the pollutant group with the highest average concentration. Concentrations of ΣHCH, HCB, ΣDDT, and Σendosulfan were reported to be higher at urban areas than rural sites. The highest concentration for urban and rural areas were reported in samples collected between August 2014 and October 2014. Σ_{22}OCP concentrations were reported as 1050 pg/m^3 and 551 pg/m^3 for urban and rural areas, respectively. Concentrations in this study were lower than those of urban but close to rural levels reported by Kurt-Karakus et al. (2018). Lower concentrations (Σ_{10}OCP 122 ± 89 pg/m^3) was reported for the urban area in Gemlik, Bursa (Cindoruk et al., 2020). Lammel et al. (2015) collected passive samples in Bursa and Urla, Izmir. In Urla, the average Σ_9OCP concentration was measured as 43 pg/m^3 with higher contributions from HCB (12.3 pg/m^3) and *α*-HCH (11.2 pg/m^3). The mean Σ_9OCP concentration in Bursa was 25.7 pg/m^3 and *p,p'*-DDE had the highest contribution (6.9 pg/m^3).

Gevao et al. (2022a) deployed samplers along a northwest-southeast transect across the Middle East at several sites (agricultural, industrial, remote, urban, and background) which were close to the war zone. The average Σ_{24}OCP concentrations of the subject countries were Lebanon at 1684 pg/m^3 > Turkey at 78.7 pg/m^3 > Oman at 55 pg/m^3 > Kuwait at 42 pg/m^3 > Saudi Arabia at 19.1 pg/m^3. The highest mean Σ_{24}OCP concentrations were measured in Lebanon and Turkey which are the surrounding countries of the war zone. Estellano et al. (2017) measured ΣDDT and ΣHCH air concentrations in urban and rural areas of Italy, the mean values of which ranged from 2.9 pg/m^3 to 50.8 pg/m^3 and 4.9 pg/m^3 to 10.7 pg/m^3, respectively. It can be concluded that the levels measured in this study are within the levels reported in the literature.

There are studies with various types of environmental samples over the extended Middle East concentrating on PBDEs in Kuwait such as in indoor and ambient air (Gevao et al., 2006, 2013). The remaining are on OCPs in air in Kuwait (Gevao et al., 2018), in foodstuff (Ahmad et al., 2010) and dates in Saudi Arabia (El-Saeid and Al-Dosari, 2010). However, there is only a few studies in the military conflict area limited to OCPs in Lebanese soils (Bashour et al., 2004; Helou et al., 2019). In addition, Gevao et al. recently reported atmospheric OCP (2022a) and PCDD/F (2022b) concentrations across Middle East (in order from South to North: Oman, Kuwait, Lebanon, and Turkey). PCDD/Fs were at higher average concentrations in the South (Oman and Kuwait) associated with industries and waste incineration. The average OCPs, on the other hand, were at similar levels in Saudi Arabia, Kuwait, Oman, and Turkey, but two orders of magnitude lower than that in Lebanon. While Lebanon and Turkey can be associated with agriculture, the remaining three countries may not. The difference between Lebanon and Turkey, therefore, could indicate its south has source regions for Turkey. However, there is no report of atmospheric and soil POP levels in between Lebanon and Turkey, i.e. Syria and Iraq, while a study by Douabul and Al-Timari (2014), which measured OCP levels in river water and sediments, fish, and other aquatic organisms in Iraq and Arabian Gulf, show that OCPs have been used and present in the environment since the late 1970s. In addition, there is a huge amount of e-waste in the middle east countries (Forti et al., 2020), which may be sources of PBDEs (Rahman et al., 2001) and PCBs (Chen et al., 2014).

Although higher concentrations were measured mostly in the close and middle regions, there was a certain decrease towards far group, for which higher concentrations were measured for only a few compounds. The findings of this study indicate contribution of both the south of the border and agriculture in southeast Turkey in the higher close-group concentrations with comparison to middle and far groups.

3.1.4. Proximity group hypothesis testing and PCA

Differences in congener concentrations in the three proximity groups were tested using ANOVA and Kruskal Wallis tests, for those that have normal and non-normal distributions, respectively. Results of the hypothesis testing are presented in Table 1 for congeners with significant difference (up to $\alpha = 0.10$) for winter, transition, and the whole sampling periods for Izmir, Bursa, and the two together forming the far group. The congeners with completely decreasing and increasing concentrations from southeastern to western Turkey are also identified. The following congeners had decreasing (close > middle > far) concentrations in at least one of the nine cases in Table 1: PBDE-28, -47, -99, -99, -100, -138, -153, -183, -209, Σ_{14}PBDE, PCB-44, -74, -118, -123, aldrin, dieldrin, HCB, γ-HCH, *p,p'*-DDE, *o,p'*-DDD, o,*p'*-DDT, γ-HCH, heptachlor, *α*- and *β*-endosulfan, PCNB, Σ_{25}OCP, however, among those only concentrations of PBDE-183, -209, PCNB, *p,p'*-DDE, Σ_{25}OCP differed significantly between the three proximity groups ($p < 0.05$). Therefore, the aforementioned four compounds and Σ_{25}OCP are the best candidates for indicators of transport from southeastern to western Turkey.

Principal Component Analysis was applied to identify congeners with similar variation, which may be an indication of originating from the same type of source. PCA analysis was performed for the close, middle and far groups by including all the data belonging to each group. Loading plots are shown in SM, Fig. S25 a, b, c, for close, middle, and far groups, respectively. PBDE congeners in close group were compiled into three locales roughly by molecular weight. In middle group, PBDE-99 and PBDE-138 were individually separated while PBDE-47, -183, and -190 formed a third compilation. In far group, however, nine congeners were all separated showing no compiling. PCB congeners were compiled on to three locales as Σ_{45}PCB, PCB-31, and the remaining congeners for close group (Fig. S25d), while for far group (Fig. S25e) only PCB-28 and -31 piled together, and PCB-52, PCB 41/64, and Σ_{45}PCB appeared separately. OCPs, on the other hand, were piled into five locales in close group whereas *p,p'*-DDE showed separately with HCB in middle group and alone in far group.

Table 1
Results of hypothesis testing for concentration differences in proximity groups.

IZMIR-BURSA TOGETHER		ONLY IZMIR		ONLY BURSA	
Congener	p	Congener	p	Congener	p
WINTER					
PBDE-183[a]	0.028	PCB-18[a]	0.049	PCB-18	0.057
Σ_{45}PCB	0.006			Σ_{45}PCB	0.037
Dieldrin	0.030			Dieldrin	0.040
				α-Endosulfan	0.034
				Σ_{25}OCP	0.046
TRANSITION					
PBDE-28	0.006	PBDE-183	0.057 ↓	PBDE-99[a]	0.049
PBDE-99	0.046	PBDE-154	0.007	PBDE-85[a]	0.062
PBDE-154	0.010	PCNB	0.046	PBDE-154[a]	0.016
p, p'-DDE	0.024	*p, p'*-DDE	0.024	*p, p'*-DDE	0.024
Σ_{14}PBDE	0.050			Σ_{14}PBDE	0.034
Σ_{25}OCP	0.018			Σ_{25}OCP	0.034 ↓
WHOLE					
PBDE-28	0.008	PBDE-154	0.007	PCNB[a]	0.048
PBDE-99	0.016	PBDE-209	0.013 ↓	PBDE-28	0.002
PBDE-85	0.046	PCB-90/101	0.041	PBDE-85	0.057
PBDE-154	0.010	Σ_{45}PCB	0.021	PBDE-99	0.006
PBDE-190	0.021	PCNB	0.016 ↓	PBDE-154	0.011
PCB-52	0.013	*p,p'*-DDE	0.003 ↓	PBDE-190	0.062
Σ_{45}PCB	0.008	Σ_{25}OCP	0.009 ↓	PBDE-209	0.021
p,p'-DDE	0.003 ↓			PCB-52	0.032
Σ_{25}OCP	0.005 ↓			*p,p'*-DDE	0.005
				HCB	0.025
				Σ_{25}OCP	0.009

↓: Close > Middle > Far Concentration.
[a] ANOVA, Remaining: Kruskal Wallis.

3.2. Back-trajectory analysis

HYSPLIT is one of the most widely used atmospheric transport and dispersion models which has been empolyed to identify possible source regions of measured pollutants or to identify air masses that may have affected a location under investigation (Lin et al., 2003; Stohl et al., 1998). Uncertainties associated with calculated trajectories originate from vertical transport, sparse meteorological data, numerical inaccuracies during calculation, sub-grid scale phenomenon, assumptions about turbulence, convection, evaporation, and condensation contribute to various errors in backward trajectory calculations (Ashbaugh et al., 1985; Kahl, 1993). Cluster analysis, that groups the individual trajectories of an ensemble into a smaller number of clusters, tend to average out errors and uncertainties associated with single backward trajectories (Stohl, 1998). Back-trajectory clusters for the seven sampling months at each location were drawn for two heights (500 m, 1000 m) above ground level (agl). All clusters are presented in SM, Fig. S2 to S11 for 1000 m, Figs. S12 and S21 for 500 m. The back-trajectories showed that there is considerable variation in contributing trajectories over both sampled time and locations.

However, southeastern locations were rarely connected to far group locations to investigate transport of POPs from the war zone to western Turkey. In fact, the southeastern sampling locations received air masses from the south of the border, specifically Adana, Gaziantep, Osmaniye, and Malatya with higher percentages between December and April, indicating that the war zone may have an effect on their POP levels. The highest cluster percentage (73 %) was found for Gaziantep in winter season when higher concentrations were measured instead of transition period when lower concentrations measured especially after April. Back-trajectory clusters for the far group locations are presented in SM, Figs. S3 and S13 for Bursa, and S7 and S17 for Izmir, showing both locations mainly received air masses originating and passing through Europe and Eurasia. Two prominent examples are shown in Fig. S7 for Izmir and Fig. S15 for Gaziantep in 2019 when the POP concentrations were relatively high compared to the other months, especially in the transition period. Thus, taking LRAT as a source of POPs, western Turkey was impacted by areas in Europe and Eurasia while southeastern Turkey was impacted by areas in south of the border, the war zone. There are reports in the literature supporting these findings (Gungormus et al., 2021; Rastgeldi Dogan et al., 2018).

3.2.1. Comparison of Local Emissions and LRAT

Halse et al. (2012) evaluated contribution of local and LRAT of pollutants using ratio of concentration at a sampling location to the concentration at a location considered as background (R). $R > 1$ would indicate the influence of local sources (LS) while $R > 2$ would imply predominance of LS. Meanwhile, $R \leq 1$ would indicate the predominance of LRAT (Halse et al., 2012).

The ratio R was adopted as $C_{Sampling}/C_{Gaziantep}$ in this study. Gaziantep is the entrance sampling point for air masses received from the south of the border, and it was the point that received the most incoming air masses over the war zone (see Fig. S5 and Fig. S15), hence Gaziantep was used as the background location. Calculation of R values indicated that atmospheric transport might be the source of many congeners at locations in all three proximity groups ($R < 1$). The calculated R values are listed in Table S13 along with the percentage apportioned to LRAT. Probably due to historical reasons, i.e., PCBs being the first group to be banned/phased out in comparison to the other two groups investigated in this study, and due to urban areas being PBDE and agricultural areas being OCP sources, there were higher number of PCB congeners with larger percentages apportioned to LRAT (≥ 50 %) especially in Close group (8 congeners) followed by Middle (5 congeners) and Far (2 congeners) groups. PCB-44 and -49 were the common congeners of the three proximity groups while PCB-183 was the common congener between Close and Middle groups. PBDE-66 and -85 were the congeners that may be brought forward with ≥ 80 % in Close and ≥ 35 % in Middle and Far groups while *α*- and *γ*-HCH were the OCPs with ≥ 50 % for LRAT.

In the literature, high availability of PBDE-66 in biota has been associated with the degradation of higher brominated PBDEs (Meng et al., 2008; Wang et al., 2008) while both PBDE-66 and PBDE-85 may originate from photo-degradation of PBDE-209 (Bezares-Cruz et al., 2004; Zeng et al., 2008). However, they were used only in minor percentages in the PentaBDE mixture (La Guardia et al., 2006). Observation of the highest PBDE-66 and PBDE-85 concentrations in the group closest to the war zone support that they may be indicators of LRAT from the conflict zone to its north. Gevao et al. (2022a) reported evidence for current and past usage – re-volatilization of aged DDTs based on *p,p'*-DDE/*p,p'*-DDT ratio, and current usage of lindane or technical HCH considering α/γ HCH ratio across the middle east, which renders the conflict zone as a potential source area in support to the findings of this study and their LRAT potential (Wu et al., 2010).

4. Conclusion

This study aimed to examine the long-range transport of POPs that are released into the environment due to the effects of the military conflicts in countries next to our south border. Our hypothesis was that there could be a significant contribution from the military conflict zone to atmospheric POP levels in Turkey. It was tested by investigating concentration-distance relationship and back trajectory analysis. In addition, contribution of local sources versus LRAT was examined. Various central tendency measures showed increasing concentration trends with proximity to the war zone. Local emissions vs. LRAT analysis indicated that many congeners in all three regions may be dominated by LRAT. On the other hand, statistical testing provided a limited support.

The concentration of POPs measured by monthly passive air sampling were higher in the close and far groups than in the middle group during the winter months. While monthly back-trajectory analyses pointed to the military conflict zone for the close group, Europe, Russia, and the former Soviet Union countries were indicated as source areas for the far-proximity group. The increasing contribution with increasing MW in the close sampling group and the increasing contribution with decreasing MW in the far group indicated that southeast Turkey was impacted by POP transport from the war zone while west – northwest of the country impacted by POP transport from the areas northwest – north – northeast to the country.

CRediT authorship contribution statement

Ilknur Ayri: Writing – original draft, Investigation, Formal analysis, Visualization. **Mesut Genisoglu:** Investigation, Formal analysis. **Aysun Sofuoglu:** Conceptualization, Funding acquisition, Supervision, Writing – review & editing. **Perihan B. Kurt-Karakus:** Methodology, Investigation, Writing – review & editing. **Askin Birgul:** Investigation. **Sait C. Sofuoglu:** Methodology, Funding acquisition, Project administration, Writing – review & editing.

Data availability

Data will be made available on request.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

This work was supported by The Scientific and Technological Research Council of Türkiye (TUBITAK) with Grant #118Y254. We thank the volunteers (Ali Emrah Çetin, Ali İhsan Öztürk, Doğan Sözbilen, Hasan Demir, Nevzat Özgü Yiğit, Yaşar Ede, Şamil Özden, Y. Alparslan Argun) who helped us to deploy the samplers, collected the samples, and more importantly for taking good care of the samplers during the course of the study.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.scitotenv.2023.162966.

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