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RESEARCH ARTICLE

Presence of persistent organic pollutants in a breeding common tern (*Sterna hirundo*) population in Ireland

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Abstract Persistent organic pollutants (POPs) are chemical compounds of environmental concern due to their toxic, persistent nature and their ability to bio-accumulate in biological tissue. Seabirds, for often being at the top of the food web, have been used as monitors of environmental pollutants. Adverse effects caused by POPs have been reported in common terns (*Sterna hirundo*) since the 1970s. Egg shell thinning, embryo and hatchling deformities have been reported for this species. Environmental legislation, such as the Oslo-Paris Convention (OSPAR), has agreed on the monitoring of concentration of POPs in common terns. This study set out to investigate contemporary concentrations of polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), organochlorine pesticides (OCPs) and brominated flame retardants (BFRs) in common terns breeding in Ireland, along with congener profiles. Investigation was conducted in live (n = 15) and dead birds (n = 20) to test for the efficiency of different methodologies using preen oil and feathers versus liver and preen gland. Mean concentrations of POPs followed the order: PCB (36.48 ng/g ww feather) > PAH (30.01 ng/g ww feather) > OCP (13.36 ng/g ww feather) > BFR (1.98 ng/g ww feather) in live birds; and PAH (46.65 ng/g ww preen gland) > PCB (44.11 ng/g ww preen gland) > OCP (15.15 ng/g ww liver) > BFR (5.07 ng/g ww liver) in dead birds. Comparison of contaminant results with toxicity pre-established levels concluded that this population of common terns in Ireland is not at risk of anomalies caused by POPs. However, some levels are higher in comparison to the ones established by OSPAR's EcoQO and must be monitored periodically.

Keywords Common tern · *Sterna hirundo* · Persistent organic pollutants · PCB · PAH · OCP · BFR

Highlights • First detected levels of persistent organic pollutants (POPs) in common terns (*Sterna hirundo*) in Ireland.
• PCBs, PAHs, OCPs and BFRs were detected in feathers and preen oil of live birds (n = 15).
• PCBs, PAHs, OCPs and BFRs were detected in liver and preen gland of dead birds (n = 20).
• Comparisons were made with levels of toxicological importance and EcoQO monitoring values.

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Introduction

Persistent organic pollutants (POPs) are chemical compounds of environmental concern due to their environmentally resilient and toxic nature. Such compounds are generally man-made or the result of anthropogenic activities and have become ubiquitous in the environment (Jones and de Voogt 1999; Pariatamby and Kee 2016). POPs have been used for many purposes in industrial, commercial and agricultural activities (Stockholm Convention 2001; Van Den Brink 1997), but in past decades have been found to cause ill-effects on humans and, mainly wildlife (Jones and de Voogt 1999; Stockholm Convention 2001).

Persistent organic pollutants such as polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), organochlorine pesticides (OCPs) and brominated flame retardants (BFRs) have been found to cause endocrine disruption and to have carcinogenic effects (Barron et al. 1995; UNEP 2011). These compounds can be biomagnified along the food web reaching levels of toxicological importance in top predators (Jaspers et al. 2006). In birds, for instance, PCBs were

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found responsible for egg shell thinning in many raptor species in the 1970s causing concerning population decline (Tanabe et al. 1984). POPs have been correlated to low reproductive success in fish-eating birds (Giesy et al. 1994), embryonic abnormalities (Gilbertson and Fox 1977), reduced growth (Gilbertson and Fox 1977) and physiological and biochemical alterations (Elliott et al. 1989). When such severe ill-effects were brought to light by research, legislation throughout the world imposed ban or restriction to most well-known POPs (Stockholm Convention 2001). The Stockholm Convention came into force in 2004 and with it, the need to monitor concentrations and levels in all environmental matrices, including biota (Stockholm Convention 2001).

Measuring the concentration of pollutants in birds is often done through destructive sampling, where a certain number of birds were sacrificed, although sometimes found dead, and serve as proxy for a given population. Such sampling would involve the collection of internal organs such as the liver, muscle or brain (Falkowska et al. 2016; Roscales et al. 2011). Eggs are an alternative to destructive sampling (Elliott et al. 2005; Moore and Tatton 1965; Mora et al. 2016; Peck et al. 2016), but when certain species of birds lay a single egg per season, care should be taken to make sure such species would relay. Non-destructive sampling techniques became necessary and feathers started being used as a proxy for contamination levels in internal organs (Jaspers et al. 2007; Jaspers et al. 2011; Van den Steen et al. 2007). Additionally, preen oil has also been regarded as a non-destructive technique (Wang et al. 2015; Yamashita et al. 2007).

Persistent organic pollutant concentrations in common terns (*Sterna hirundo*) have been measured in many parts of the world since the 1960s (Bosveld et al. 1995; Gilbertson et al. 1976; Scharenberg 1991; Van Den Brink and Bosveld 2001; Custer et al. 2001). POPs were found to cause death, feminization of male embryos and other embryonic developmental abnormalities in this species (Becker et al. 1993; Fox 1976; Hays and Risebrough 1972; Hoffman et al. 1998; Hoffman et al. 1993; Scharenberg 1991). Since then, toxicity levels over which embryonic development would be affected have been established (Hays and Risebrough 1972; Hoffman et al. 1998; Scharenberg 1991).

Monitoring of POPs in eggs of common terns is one of the Oslo-Paris Convention's (OSPAR) Ecological Quality Objectives (EcoQO) (OSPAR 2010). EcoQOs establish threshold contaminant levels for certain species and parties must monitor levels to meet the treaty's requirements (Dittmann et al. 2012).

Common terns are highly migratory seabirds, globally distributed, with tropical wintering areas in the south and northern breeding areas (Austin 1953). Their diet consists mainly of fish (Massias and Becker 1990). In Ireland, there are over two and a half thousand pairs of breeding common terns (Mitchell et al. 2004). Main threats to common tern populations are habitat loss and pollution (Mitchell et al. 2004). To our knowledge, there are no persistent organic pollutant data for common terns breeding in Ireland. Most recent published data for closely related species such as roseate (*Sterna dougallii*) and sandwich (*Sterna sandvicensis*) terns date from 1965 (Koeman et al. 1967). Given the absence of data in Ireland for a species of conservation importance, the research presented here intended to (1) gather contemporary data on concentrations of PCBs, PAHs, OCPs and BFRs in common terns breeding in Ireland; (2) investigate congener profiles, along with destructive and non-destructive sampling methods, using preen oil and feathers in live birds, and liver and preen gland in corpses found in breeding colonies; and (3) investigate contaminant levels of toxicological importance.

Material and methods

Sampling location

Rockabill is a 0.9 ha island located 7 km off the north coast of county Dublin, Ireland (Grid Ref. O320627). Rockabill is home to approximately 2000 pairs of common terns, along with 1550 pairs (47% of the entire European population) of roseate terns (*S. dougallii*) and smaller numbers of breeding Arctic terns (*Sterna paradisaea*), black-legged kittiwakes (*Rissa tridactyla*) and black guillemots (*Cepphus grylle*) (Burke et al. 2016). The major disturbance to tern nests on the island is predation by great black-backed gulls (*Larus marinus*) (Burke et al. 2016). Common tern diet composition consists mostly of Clupeids, Sandeels and Gadoids (Burke et al. 2016).

Dead birds sampling

Necropsies

In total, 38 common tern corpses were collected at Rockabill colony, during the breeding seasons of 2015–2016. Birds were necropsied following Van Franeker (2004) methodology. When possible, sex, age class and cause of death were inferred. Preen gland and liver were collected from 20 birds for persistent organic pollutants (POP) analysis. All 38 stomachs were additionally analysed for plastic litter according to Van Franeker (2004) by sieving contents through a 1 mm mesh sieve. All retained solids were collected in petri-dishes and air-dried overnight. Only a single piece of plastic (fragment) was found in all stomachs analysed. Mass of the item was 0.1538 g and it was perforating the stomach lining, causing an ulcer.

Liver and preen gland extractions

In total, 20 livers and preen glands were analysed from necropsied birds. All utensils were previously washed using *n*-hexane (VWR Analar Normapur). Tissue samples (liver and preen gland) were cut into small pieces. Preen gland samples also had remaining feathers removed. Samples were weighed in beakers to the nearest 0.0001 g. A solvent mixture of three parts of hexane and one part of acetone (Merck SupraSolv) was added to samples (approximately 30 ml). Samples were spiked with internal standards (PAH 24D, 13C PCB and BFR, OCP Pesticide Mix 20). Samples were homogenised using an UltraTurrax (IKA T10 Basic) for 1 min, then 20 ml of pure water was added to the sample, and the mixture was homogenised again for another minute. Samples were transferred to centrifuge tubes and placed on the centrifuge (Hehich Zentrifugen Mikro 220R) for 5 min at 4000 rpm. Using disposable pipette tips, the top layer (solvent) was transferred to glass vials. The cleaning process was achieved by placing 2 g of pre-treated (300 °C for 3 h, with 5% weight by water) silica gel (Molekula) in a glass column for each sample. The solvent layer in the glass vials was then poured into the glass column followed by a solvent mixture of 60 ml of hexane and 10 ml of acetone. Clean samples were collected in a conical flask by opening the tap of the glass column. Samples were then evaporated in the TurboVap LP (Biotage) to approximately 1 ml and transferred into GC vials.

Live birds sampling

In total, 15 common terns were hand caught at Rockabill colony, county Dublin during the breeding season, under licence no. C124/2015 and C125/2015 from National Parks and Wildlife Service (NPWS), in July 2015. Birds were weighed, had their wingspan measured and were ringed if they had not been previously ringed. Preen oil cotton swabs were collected by exposing the preen gland and gently pressing it to express the oil. Swabs were placed in sterile glass jars with foil covered lids. Furthermore, six breast feathers were collected from each individual and kept in paper envelopes. Preen oil samples were kept frozen at −80 °C, whilst feather samples were kept at room temperature until analysis.

Preen oil extraction

All utensils were previously washed using methanol (Merck SupraSolv). Cotton swabs were transferred into glass beakers by using metal forceps. Sample jars were then rinsed with SupraSolv methanol to remove any remaining preen oil in the glass jar. This methanol was also poured into the beaker containing the corresponding cotton swab. In total, 150 ml of methanol was poured into each beaker (in three aliquots). Contents were stirred for 1 min each time. Samples were spiked with internal standards (PAH 24D, 13C PCB and BFR, OCP Pesticide Mix 20). Only the liquid sample was then transferred to another beaker and covered with aluminium foil. Samples were placed in a TurboVap LP (Biotage) to evaporate the volume to approximately 1 ml. Using disposable glass pipettes, the remaining sample was transferred into previously labelled GC vials. Samples were kept frozen at −80 °C until subsequent analysis using gas-chromatography/mass-spectrometry (GC/MS).

Feather extraction

All utensils were previously washed with methanol. Samples of four feathers per bird were placed in individual beakers. Feathers were washed with distilled water, using forceps to separate the barbs, and stirred. They were left soaking for 20 min and then left to dry in folded tissue paper for 2 h or until fully dried. After drying, each sample was weighed to the nearest 0.0001 g and placed inside a beaker with 15 ml of 37% HCl (Merck EMSURE) and 20 ml of a solvent mixture of two parts of hexane and one part of acetone. Samples were spiked with internal standards (PAH 24D, 13C PCB and BFR, OCP Pesticide Mix 20). Beakers were covered with aluminium foil and put in the oven at 37 °C overnight (in total for approximately 15 h). Consequently, 40 ml of a solvent mixture of three parts of hexane and one part of acetone was added to each sample. Samples were then placed within a separation funnel and shaken vigorously. The subsequent aqueous layer was removed by opening the tap on the separation funnel and pouring the liquid into a beaker. The remaining lipid layer was decanted into previously labelled glass vials. This separation procedure was repeated by placing the aqueous layer back into the separation funnel and adding 20 ml of fresh hexane/acetone solvent mixture. Samples were transferred into a TurboVap LP (Biotage) and evaporated under a nitrogen stream until approximately 1 ml remained. Samples were subsequently transferred to pre-labelled GC vials using disposable glass pipettes. Samples were kept frozen at −80 °C until subsequent analysis using GC/MS.

Gas-chromatography mass-spectrometry

Liver, preen gland, preen oil and feather solvent extractions were then analysed for polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), organochlorine pesticides (OCPs) and brominated flame retardants (BFRs) using gas-chromatography/mass spectrometry (Agilent GC-MS (5977E)) equipped with an auto-sampler. GC/MS was run in EI mode, with a J&W 30 m BD1 MS column, with helium being the carrier gas. Quality control was guaranteed by the use of blanks per batch of samples and certified reference materials (CRMs). For preen gland, preen oil and liver analysis, cod liver oil (Commission of the European

Communities, Community Bureau of Reference – BCR. Reference Material n° 349. Chlorobiphenyls in cod liver oil n° 0831) was used as a CRM and for feather analysis, fish tissue (NIST 1947 Lake Michigan Fish Tissue. U.S. Department of Commerce, National Institute of Standards and Technology, Gaithersburg, MD 20899).

Statistical analysis

Statistical analysis was carried out using R (R Core Team 2015), version 3.2.3 and 'prcomp' package. To investigate the potential relationships between matrices, such as preen oil and feathers and liver and preen gland, Pearson's correlation was computed for each group of contaminants. This was done in two ways: through a correlation matrix at the individual level and through aggregated data. A correlation matrix was combined with hierarchical clustering using complete hierarchical clustering method. The input to a hierarchical clustering algorithm consists of the measurement of the similarity (or dissimilarity) between each pair of objects. The goal of the clustering algorithm is then to partition the objects into homogeneous groups, such that the within-group similarities are large compared to the between-group similarities. Aggregated data on the other hand uses means and standard deviations of each congener to compute correlation by homogenising individual samples.

A principal component analysis (PCA) was used to investigate which congeners contributed most to the variance in each group of contaminants. The principal components were extracted to represent the patterns encoding the highest variance in the data set. However, in many high-dimensional data sets, the most dominant patterns, i.e. those captured by the first principal components, are those separating different subgroups of the samples from each other. The first principal component (PC1) captures the maximum variance and will determine the direction of highest variability in the data. The following components (e.g. PC2, PC3, etc.) capture the remaining variance. The same analysis was then used to investigate if live sampling (e.g. preen oil and feathers) can potentially serve as a proxy for organs (e.g. liver and preen gland). Congeners with over 50% of values below the level of detection (LOD) were excluded from statistical analysis (Jaspers et al. 2008).

Results

Live birds—preen oil and feathers

In total, 16 PCBs were detected in preen oil and feathers. The mean concentration of ∑PCBs was 4.23 ng/g ww preen oil (range 1.78–9.11 ng/g ww) and 36.48 ng/g ww feathers (range 14.96–113.48 ng/g ww). The mean concentration of ∑7 PCBs was 3.45 ng/g ww preen oil and 27.25 ng/g ww feathers (Table 1). Results from the PCA showed that in preen oil, the three first components (PC1, PC2 and PC3) explained 72% of the variance, whilst in feathers, they explained 91%. In preen oil, the congeners that contributed most to PCB burden were PCBs 118, −153 and −138, whilst in feathers, highest contributions came from PCBs 101, −149 and −138.

Twelve PAH congeners were detected in preen oil and feathers. The mean concentration of PAHs was 10.52 ng/g ww preen oil (range 6.42–18.74 ng/g ww) and 30.01 ng/g ww feathers (range 18.53–53.46 ng/g ww) (Table 2). PCA results showed that in preen oil, the three first components explained 69% of the variance; and in feathers, 63%. Congeners that mostly contributed to PAH burden in preen oil were chrysene, benzo(b)fluoranthene and benzo(a)anthracene, whilst for feathers were pyrene, fluoranthene and benzo(b)fluoranthene.

Fifteen OCPs were detected in feather and preen oil. The mean concentration of OCPS was 3.69 ng/g ww preen oil (range 2.86–5.02 ng/g ww) and 13.36 ng/g ww feathers (range 6.23–25.01 ng/g ww) (Table 3). PCA results showed that the first three components retained 59% of the variance for preen oil and 94% for feathers. Congeners that had the highest contribution to PAH burden were heptachlor, dieldrin and pp-DDE in preen oil, and Endrin, a-HCH and heptachlor in feathers.

In total, six BFRs were detected in feathers and preen oil. The mean concentration of BFRs was 1.86 ng/g ww preen oil (range 1.54–2.20 ng/g ww) and 1.98 ng/g ww feathers (range 1.87–2.90 ng/g ww) (Table 4). The first three components in the principal component analysis explained 84% of the variance in preen oil, and 75% in feathers. Congeners that contributed most to BFR burden in preen oil were BFRs 47, −99 and −100, and BFRs 100, −154 and −183 in feathers.

Congener profiles differed between feathers and preen oil. That was confirmed by the correlation matrices combined with hierarchical clustering. Correlations were either negative or very low between congeners. Aggregated data on the other hand showed a strong correlation between feathers and preen oil for BFR (0.97), PCB (0.73) and PAH (0.72), and a moderate correlation for OCP (0.51).

Dead birds—liver and preen gland

In total, 16 PCBs were detected in liver and preen gland. The mean concentration of PCBs was 41.43 ng/g ww liver (range 11.01–103.93 ng/g ww) and 44.11 ng/g ww preen gland (range 4.74–115.6 ng/g ww). The mean concentration for ∑7 PCBs was 35.34 ng/g ww liver and 34.85 ng/g ww preen gland (Table 1). The three first principal components explained 82% of the variance in liver and 85% in preen gland. In liver, the congeners that contributed most to PCB burden

Table 1 PCB mean concentrations (ng/g ww) ± standard deviation (SD) separated per congener, detected in preen oil, feathers, liver and preen gland. Seven PCBs(Σ7) are −28, −52, −101, −118, −153, −138 and −180

PCB	Live common terns		Dead common terns	
	Preen oil (ng/g ww) ± SD	Feathers (ng/g ww) ± SD	Liver (ng/g ww) ± SD	Preen gland (ng/g ww) ± SD
PCB 18	0.03 ± 0.03	0.71 ± 0.74	0.07 ± 0.04	1.25 ± 4.59
PCB 28	0.07 ± 0.06	2.42 ± 1.83	0.76 ± 0.63	0.97 ± 1.14
PCB 31	0.06 ± 0.05	2.27 ± 1.94	0.65 ± 0.62	0.83 ± 0.84
PCB 52	0.20 ± 0.18	6.75 ± 3.86	1.94 ± 1.69	2.89 ± 4.48
PCB 44	0.15 ± 0.18	2.51 ± 1.40	0.98 ± 0.80	1.83 ± 4.45
PCB 101	1.13 ± 0.54	9.16 ± 7.41	5.64 ± 3.37	5.36 ± 5.51
PCB 118	0.69 ± 0.43	3.88 ± 3.67	5.51 ± 3.48	5.80 ± 5.74
PCB 105	0.08 ± 0.06	0.74 ± 0.93	0.68 ± 0.53	2.00 ± 3.17
PCB 149	0.30 ± 0.13	2.65 ± 2.59	3.10 ± 3.64	1.71 ± 2.08
PCB 153	0.68 ± 0.35	2.45 ± 2.41	11.71 ± 6.69	9.69 ± 9.43
PCB 138	0.44 ± 0.23	2.26 ± 2.13	8.25 ± 5.28	6.92 ± 6.41
PCB 156	0.09 ± 0.05	0.19 ± 0.22	0.28 ± 0.26	0.78 ± 0.71
PCB 180	0.24 ± 0.14	0.33 ± 0.24	1.53 ± 1.35	3.22 ± 3.18
PCB 170	0.04 ± 0.04	0.08 ± 0.08	0.24 ± 0.28	0.42 ± 0.53
PCB 194	0.02 ± 0.01	0.07 ± 0.06	0.07 ± 0.07	0.29 ± 0.74
PCB 209	0.01 ± 0.01	0.01 ± 0.01	0.02 ± 0.02	0.15 ± 0.41
∑ all PCBs	4.23 ± 0.30	36.48 ± 2.47	41.43 ± 3.33	44.11 ± 2.68
∑ 7 PCBs	3.45 ± 0.34	27.25 ± 2.81	35.34 ± 3.69	34.85 ± 2.68

were PCBs 153, −138 and −180, whilst in preen gland, the highest contributions came from PCBs 138, −153 and −118.

Fifteen PAH congeners were detected in preen gland and only 13 in liver. The mean concentration of PAHs

Table 2 PAH mean concentrations (ng/g ww) ± standard deviation (SD) separated per congener, detected in preen oil, feathers, liver and preen gland

PAH	Live common terns		Dead common terns	
	Preen oil (ng/g ww) ± SD	Feathers (ng/g ww) ± SD	Liver (ng/g ww) ± SD	Preen gland (ng/g ww) ± SD
Acenaphthylene	1.00 ± 0.01	0.23 ± 0.12	0.19 ± 0.10	0.72 ± 0.28
Acenaphthene	ND	ND	ND	2.78 ± 1.62
Fluorene	ND	ND	ND	5.07 ± 2.20
Phenanthrene	3.89 ± 2.45	9.99 ± 3.34	7.03 ± 8.92	7.59 ± 3.36
Anthracene	0.50 ± 0.56	0.67 ± 0.26	0.88 ± 1.29	0.43 ± 0.43
Fluoranthene	0.63 ± 0.38	3.21 ± 1.53	2.17 ± 2.34	0.83 ± 0.45
Pyrene	ND	ND	3.72 ± 5.27	2.69 ± 1.23
Benzo(a)anthracene	0.43 ± 0.54	0.54 ± 0.66	0.99 ± 1.54	3.29 ± 4.33
Chrysene	0.76 ± 0.64	0.49 ± 0.60	0.39 ± 0.62	1.51 ± 1.12
Benzo(b)fluoranthene	0.45 ± 0.55	0.48 ± 0.15	2.91 ± 7.12	5.99 ± 8.27
Benzo(k)fluoranthene	0.43 ± 0.36	0.23 ± 0.08	0.85 ± 1.97	5.46 ± 15.48
Benzo(a)pyrene	0.62 ± 0.71	0.70 ± 1.27	4.06 ± 6.16	4.43 ± 4.03
Indeno(1,2,3-CD)pyrene	1.21 ± 1.09	12.67 ± 8.60	3.25 ± 4.56	0.89 ± 1.31
Dibenzo(a,h)anthracene	0.38 ± 0.41	0.62 ± 0.05	0.84 ± 0.72	1.42 ± 2.24
Benzo(g,h,i)perylene	0.22 ± 0.18	0.18 ± 0.20	0.36 ± 0.36	3.55 ± 4.11
∑PAH	10.52 ± 0.94	30.01 ± 4.06	27.64 ± 1.92	46.65 ± 2.13

ND not detected

Table 3 OCP mean concentrations (ng/g ww) ± standard deviation (SD) separated per congener, detected in preen oil, feathers, liver and preen gland

OCP	Live common terns		Dead common terns	
	Preen oil (ng/g ww) ± SD	Feathers (ng/g ww) ± SD	Liver (ng/g ww) ± SD	Preen gland (ng/g ww) ± SD
a-HCH	0.17 ± 0.15	2.78 ± 1.55	0.88 ± 1.04	0.33 ± 0.22
HCB	0.08 ± 0.05	0.09 ± 0.06	1.46 ± 2.04	0.07 ± 0.06
g-HCH	ND	ND	1.23 ± 3.67	2.43 ± 2.96
b-HCH	ND	ND	0.43 ± 1.94	0.35 ± 1.56
Heptachlor	0.17 ± 0.13	1.77 ± 2.67	0.25 ± 0.39	0.39 ± 0.20
Aldrin	0.01 ± 0.02	0.13 ± 0.19	0.12 ± 0.15	0.35 ± 0.25
Isobenzan	0.02 ± 0.02	0.11 ± 0.24	0.09 ± 0.07	0.37 ± 0.60
Isodrin	0.02 ± 0.02	0.26 ± 0.40	0.45 ± 1.06	0.20 ± 0.12
Heptachlor epoxide	0.01 ± 0.02	0.15 ± 0.29	0.34 ± 0.38	0.32 ± 0.17
op-DDE	0.20 ± 0.24	0.17 ± 0.21	0.63 ± 0.63	0.42 ± 1.36
pp-DDE	0.49 ± 0.49	0.21 ± 0.24	1.77 ± 4.34	0.14 ± 0.23
Dieldrin	0.03 ± 0.02	0.29 ± 0.33	0.91 ± 1.25	0.43 ± 1.00
pp-DDT	0.01 ± 0.02	0.39 ± 0.41	0.41 ± 0.43	0.15 ± 0.20
Endrin	0.18 ± 0.23	2.83 ± 4.97	1.28 ± 1.28	3.70 ± 6.03
Endosulphan B	0.40 ± 0.42	0.50 ± 0.81	1.16 ± 1.12	0.42 ± 0.41
pp-DDD	1.87 ± 0.01	2.84 ± 1.51	2.93 ± 1.09	2.90 ± 2.19
op-DDT	0.03 ± 0.03	0.84 ± 0.94	0.81 ± 0.92	0.51 ± 0.52
∑OCP	3.69 ± 0.45	13.36 ± 1.04	15.15 ± 0.69	13.48 ± 1.05

ND not detected

was 27.64 ng/g ww liver (range 4.49–78.76 ng/g ww) and 46.65 ng/g ww preen gland (range 12.34–124.37 ng/g ww) (Table 2). The first three components of the PCA explained 61% of the variance in preen oil and preen gland equally. Congeners that mostly contributed to PAH burden in liver were phenanthrene, fluoranthene and pyrene, whilst for preen gland were phenanthrene, acenaphthene and fluoranthene.

Seventeen OCPs were detected in liver and preen gland. The mean concentration of OCPS was 15.15 ng/g ww liver (range 4.84–38.08 ng/g ww) and 13.48 ng/g ww preen gland (range 4.80–28.85 ng/g ww) (Table 3). The three first components explained 52% of the variance in preen oil and the same in feathers. Congeners that had the highest contribution to OCP burden were dieldrin, HCB and pp-DDE in liver, and op-DDT, dieldrin and op-DDE in preen gland.

In total, seven BFRs were detected in liver and preen gland. The mean concentration of BFRs was 5.07 ng/g ww liver (range 2.04–18.81 ng/g ww) and 4.37 ng/g ww preen gland (2.06–8.53 ng/g ww) (Table 4). Principal components 1, 2 and 3 explained 83% of the variance in liver and 78% in preen gland. Congeners that contributed most to BFR burden in liver were BFRs 99, −153 and −100, and BFRs 47, −28 and −99 in preen gland.

Table 4 BFR mean concentrations (ng/g ww) ± standard deviation (SD) separated per congener, detected in preen oil, feathers, liver and preen gland

BFR	Live common terns		Dead common terns	
	Preen oil (ng/g ww) ± SD	Feathers (ng/g ww) ± SD	Liver (ng/g ww) ± SD	Preen gland (ng/g ww) ± SD
BFR 28	0.31 ± 0.02	0.30 ± 0.02	0.37 ± 0.12	0.45 ± 0.14
BFR 47	0.48 ± 0.11	0.58 ± 0.24	0.93 ± 0.66	0.84 ± 0.68
BFR 100	0.34 ± 0.04	0.33 ± 0.04	0.77 ± 0.61	0.69 ± 0.45
BFR 99	0.32 ± 0.05	0.37 ± 0.07	0.78 ± 0.97	0.61 ± 0.43
BFR 154	0.38 ± 0.10	0.37 ± 0.05	0.45 ± 0.23	0.41 ± 0.14
BFR 153	ND	ND	0.70 ± 1.65	0.52 ± 0.34
BFR 183	0.03 ± 0.02	0.03 ± 0.04	1.07 ± 0.72	0.85 ± 0.69
∑BFR	1.86 ± 0.55	1.98 ± 0.16	5.07 ± 0.22	4.37 ± 0.16

ND not detected

Results from the correlation matrices between congeners of liver and preen gland showed weak or negative correlations. Aggregated data correlation, however, showed a strong correlation between liver and preen gland for PCB (0.96) and BFR (0.94), but a weak correlation for PAH (0.6) and OCP (0.55).

Live versus dead

Results from the PCA comparing feathers as a proxy for liver (Fig. 1) and preen oil as a proxy for preen gland (Fig. 2) showed a clear separation between the two types of sample (live and dead), with much clustering in live bird samples, whilst dead bird samples show larger variance between individuals.

Discussion

To our knowledge, this is the first study to provide data on persistent organic pollutants in common terns in Ireland. Whilst there are data on POPs from sandwich and roseate terns in Ireland, this originates from the 1960s, and it is comprised of only two OCP congeners (Koeman et al. 1967).

Total PCB concentrations were ninefold higher in feathers (36.48 ng/g) than in preen oil (4.23 ng/g) in live

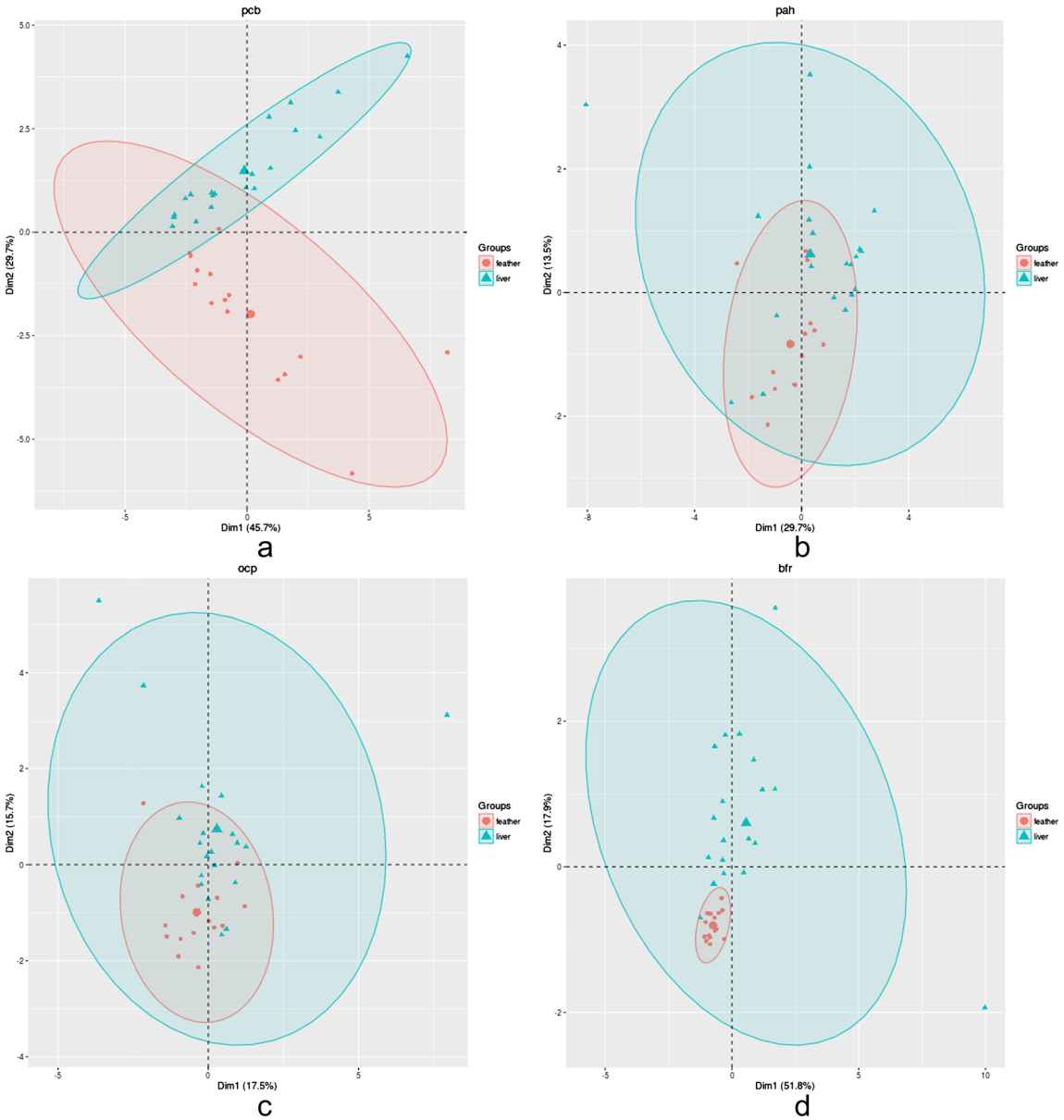

Fig. 1 Principal component analysis (*PCA*) comparing feathers (*red dots*) as a proxy for liver (*blue triangles*) for PCB (**a**), PAH (**b**), OCP (**c**) and BFR (**d**). There is a clear separation between the two groups, with feathers being much more clustered together, whilst liver samples appear to be more spread. *Ellipses* drawn around individual samples show a 95% concentration of points

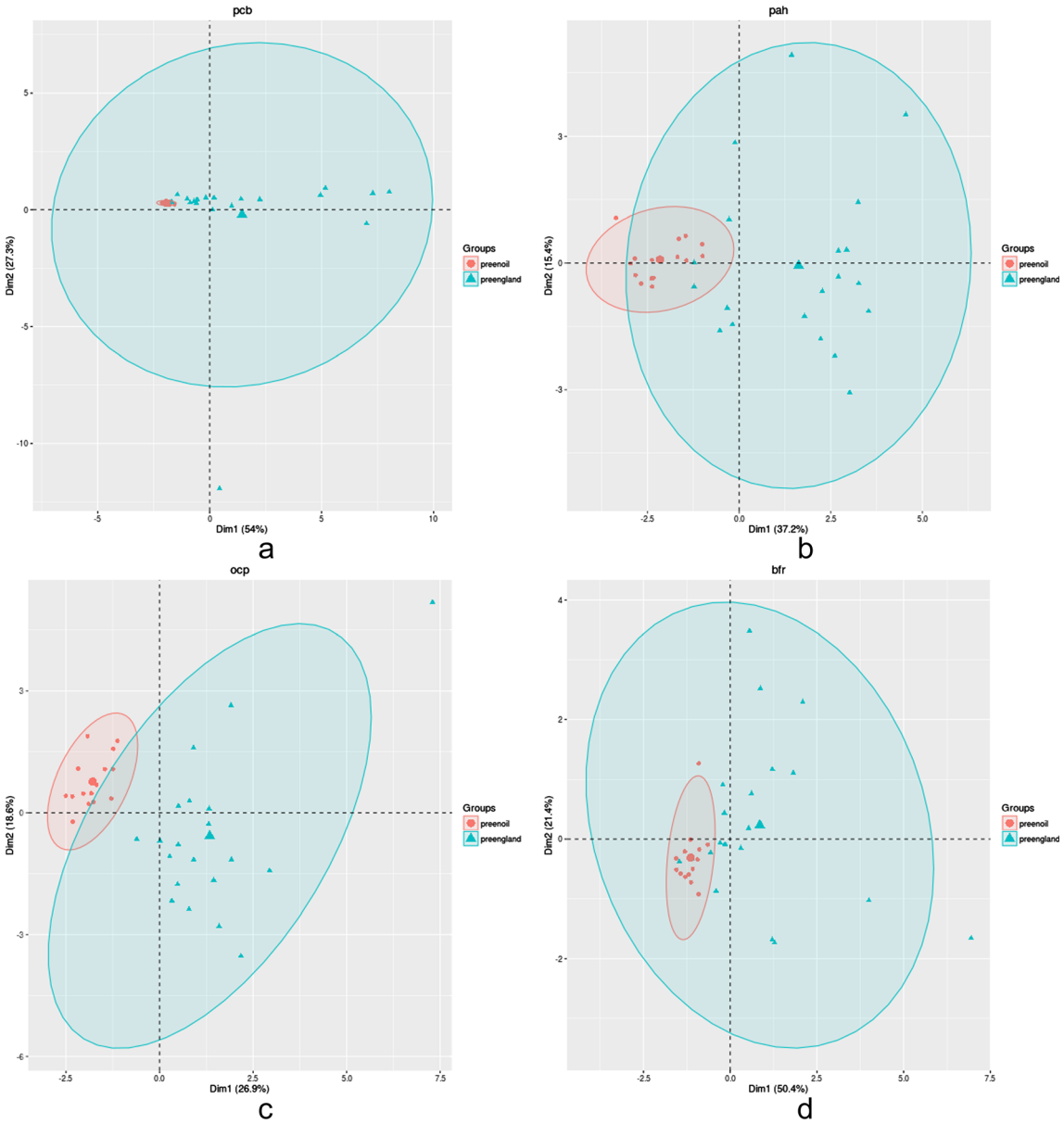

Fig. 2 Principal component analysis (*PCA*) comparing preen oil (*red dots*) as a proxy for preen gland (*blue triangles*) for PCB (**a**), PAH (**b**), OCP (**c**) and BFR (**d**). There is a clear separation between the two groups, with preen oil samples being much more clustered together, whilst preen gland samples appear to be more spread. *Ellipses* drawn around individual samples show a 95% concentration of points

birds and similar between liver (41.43 ng/g) and preen gland (44.11 ng/g) in dead birds, though a strong correlation was seen between both sets of sampling techniques: preen oil vs feathers (0.73) and liver vs preen gland (0.96). All samples were dominated by high molecular weight components, suggesting an accumulation of such congeners and potential metabolising of low molecular weight congeners. Concentrations in all matrices, apart from preen oil exceed the EcoQO for ∑ PCB, which is 20 ng/g in eggs (Dittmann et al. 2012). Research on Foster's Terns (*Sterna forsteri*) suggests that three PCB congeners (−126, −77 and −105) might contribute to 90% of toxicity in eggs (Kubiak et al. 1989). Laboratory experiments that involved the injection of PCB 126 in common tern eggs showed that all three different dosage levels given from 44 to 434 ng/g caused significant mortality (27–53%) after a week of treatment. The median lethal dose (LD_{50}) for PCB 126 in common tern eggs, based on hatching success of said study, is approximately 104 ng/g (Hoffman et al. 1998). Deformities in bills (crosses and shortened) increased with higher doses (Hoffman et al. 1998). PCBs 126 and −77 were not detected in common terns from Rockabill colony. PCB 105, however, was detected in all matrices, but at low levels (0.08–2.00 ng/g ww). The lowest observed adverse effects level (LOAEL) in common terns affected reproduction

and is reported to be 8 mg/kg (= 8000 ng/g) (Bosveld and Van den Berg 1994; Su et al. 2014).

Total PAH concentrations were threefold higher in feathers (30.01 ng/g ww) when compared to preen oil (10.52 ng/g ww), while mean concentrations in preen gland (46.65 ng/g ww) were nearly twice as high than in liver (27.64 ng/g ww). The contribution profile of congeners differs highly between preen oil and feathers, but it shows two of the same congeners for liver and preen gland. In general, PAH levels were comparable to values found in livers of Bulwer's petrels (*Bulweria bulwerii*) in the Atlantic Ocean (range 17.2–66.2 ng/g) (Roscales et al. 2011). It has been reported that PAH levels in the tissues of birds far from industrialised areas and non-contaminated sites tend to be low (Hall and Coon 1988). Additional studies have also found higher levels of PAH in tissues of birds that feed on lower trophic prey, such as invertebrates, rather than higher trophic prey, such as pelagic fish (Broman et al. 1990; Custer et al. 2001), which is the main common tern prey (Cabot and Nisbet 2013). This is possibly due to the fact that PAH tend to accumulate mostly in sediments and have been shown to have low bio-magnification properties (MacRae and Hall 1998; Nfon et al. 2008; Perugini et al. 2007; Wan et al. 2007).

Total OCP mean concentrations were fourfold higher in feathers (13.36 ng/g ww) than in preen oil (3.69 ng/g ww). Mean concentrations in liver (15.15 ng/g ww) and preen gland (13.48 ng/g ww) were similar. Heptachlor highly contributed to the burden in preen oil and feathers, whilst for liver and preen gland, dieldrin and DDE isomers were the common contributors. HCB was present in all matrices. Mean concentrations did not exceed the EcoQO of 2 ng/g for eggs, with mean values between 0.07 ng/g preen gland and 1.46 ng/g liver. ∑DDT was below the EcoQO (10 ng/g) for eggs in all matrices, with the highest mean in liver (6.55 ng/g). ∑HCH was above the EcoQO (2 ng/g) for eggs in all matrices, but preen oil, with the lowest mean at 2.54 ng/g in liver and the highest at 3.11 ng/g in preen gland. PCBs, DDT and DDE were previously associated with abnormalities in chicks. Hays and Risebrough (1972) recorded various deformities in bill, eye and foot in common and roseate terns unhatched and chicks up to a few days old. Premature feather losses (PFL) were also recorded in young chicks, sometimes preventing them from fledging. These abnormalities were similar to the chick edema disease in poultry, associated with the toxic compound chlorinated dibenzo-*p*-dioxin, a substance that has been reported to contaminate commercial PCB mixtures (Barron et al. 1995). Sublethal effects in adult birds include reduced parental attentiveness and abnormal reproductive behaviour (Barron et al. 1995).

Total BFR mean concentrations were similar between matrices for both preen oil (1.86 ng/g ww) and feathers (1.98 ng/g ww), and liver (5.07 ng/g ww) and preen gland (4.37 ng/g ww). Feathers and liver appear to have a higher contribution from high molecular weight congeners, whilst preen oil and preen gland appear to have lower molecular weight congeners. Common tern carcasses in the north Atlantic have reported a much higher ∑BFR concentration (121 ± 25 ng/g lipid weight) (Jenssen et al. 2007) compared to values from this study in liver and preen gland. The same is true for the Arctic tern (*S. paradisaea*) (95.4 ± 36 and 40.9 ± 8.4 ng/g lipid weight) (Jenssen et al. 2007). BFRs from our study were just above the level of quantification (LOQ). BFRs are applied in industry to combustible materials to meet safety regulations (Jenssen et al. 2007). Such additives can leach out of products in certain conditions and have become of environmental importance due to their persistent and toxic nature. In experimental conditions, BFRs have been shown to leach out of plastic products 20–50 times more in stomach and fish oil than in seawater (Tanaka et al. 2015). Due to the ubiquity of plastic pollution at sea, BFR dispersal and bioaccumulation has become of greater concern (Derraik 2002).

In general, feathers have demonstrated more similar concentrations to internal organs than did preen oil. That could be explained by the fact that feathers tend to carry a higher burden due to the various sources of contaminant input: the blood stream when feathers are grown, external contamination (although that has been claimed to be irrelevant by Jaspers et al. 2008) and additionally, preen oil, due to the constant act of preening of the feathers. In the case of common terns, they undergo a post-breeding moult (Ginn and Melville 1983), which means that in the case of these samples, collected during the breeding season, birds would still be carrying contaminants acquired during winter and southern migration. Preen oil on the other hand is constantly produced and is more likely to reflect local contamination (Jacob and Ziswiler 1982), like eggs in the case of income breeders (Arnold et al. 2004; Janke et al. 2015).

Pollutant concentrations in seabirds depend on a variety of factors. Moulting influences the uptake of contaminants onto feathers by the blood stream (Jaspers et al. 2006; Van den Steen et al. 2007). Migration can alter contaminant burden in two ways: by exposing the birds to more or less contaminated areas and by the mobilisation of lipids to cope with energy expenditure. Such mobilisation affects contaminant load in starving birds in the same way (Barron et al. 1995; Jaspers et al. 2008). Breeding affects the burden of female birds, which are known to pass from 4 to 45% (45% in Arctic terns) of their burden to their eggs (Lemmetyinen et al. 1982; Tanabe et al. 1984), contaminating unborn chicks. Variation in contaminant load and different congener profiles can be attributed to species specific metabolism and elimination and congener-specific toxicokinetics (Barron et al. 1995; Brunström et al. 1990; Hoffman et al. 1998; Hoffman et al. 1996; Smith et al. 1990).

Results from the PCA analysis between dead and live common terns revealed that the utility of organs (e.g. dead birds) for POP monitoring might bring biased results due to great variation among individuals. If death is accidental, birds might have recently experienced starvation, migration, moulting or

even intoxication. These unknown factors result in great individual variation.

POPs in common terns in Ireland are not at toxicological levels to cause embryonic deformities, or reproductive failure. However, some levels are higher than recommended by European policy, such as OSPAR's EcoQO in eggs (OSPAR 2010). In reality, effects of certain compounds are difficult to properly quantify as biota and environmental media is pre-contaminated with various pollutants, thus it is recommended to keep periodic monitoring of concentrations and potential effects.

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