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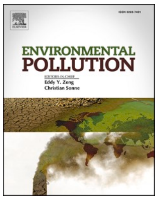

A comparative analysis of PFAS in archive and fresh soil samples in England and implications for large-scale surveys☆

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ABSTRACT

This research addresses a knowledge gap in the detectability, concentrations, and types of per and polyfluoroalkyl substances (PFAS) present in shallow soils in England, UK. While the water environment has been, and continues to be, the subject of significant testing and research for these chemicals, soils are still poorly understood. Estimates of anthropogenic background concentrations of PFAS helps with the assessment of potential contamination scenarios, setting benchmark levels and risk evaluation. This research comprised an assessment of the suitability of using archive soil samples to support contemporary samples to give a preliminary indication of likely anthropogenic background concentrations of PFAS in shallow soils in England. We retrieved 25 archive samples and collected nine contemporary samples from the same geographical location as their paired archive counterpart. Samples were analysed for 53 PFAS with limits of quantification between 0.1 and 0.5 μg/kg dry weight (dw). The results showed that archive samples contained lower concentrations (mean $\sum$PFAS-53, 1 μg/kg dw) than paired contemporary soil samples (mean $\sum$PFAS-53, 3 μg/kg dw). The concentrations reported by this study were similar to other surveys at international, national and regional scales. Our study showed that PFAS were detected in both archive and contemporary field samples. Based on the current availability of archive samples in England, contemporary field samples should be used as the primary sample type in future research on background concentrations of PFAS in soils. Archive samples have the potential provide complimentary temporal and compositional insights to support the derivation of background concentrations.

1. Introduction

The aim of our research was to address knowledge gaps in the detectability, concentrations, and types of per and polyfluoroalkyl substances (PFAS) present in shallow soils in England, UK. The research was designed to give a preliminary indication of PFAS in shallow soils in England to support future national surveying to determine anthropogenic background concentrations, informed by archive and contemporary samples. The potential benefits of using archive compared to fresh field samples include cost savings, simplified logistics, and avoidance of the environmental impact of sample collection. Conversely, archive samples carried a risk that results may not be representative of present-day PFAS concentrations , either as the result of sample collection or aging or due to changes in the pattern of contamination over time. Our research objectives were: i) assess the availability of archive shallow soils collected and stored by the British Geological Survey (BGS); ii) select, collect, and prepare for analysis a sub-set of archive and paired contemporary field samples; iii) analyse both archive and contemporary field samples for a suite of $>$50 individual PFAS and evaluate the results.

PFAS are a large family of manufactured fluorinated organic chemicals, comprising thousands of individual compounds. The most widely studied PFAS are non-polymer perfluoroalkyl acids (PFAA) due to their long-term use beginning in the 1940–50s, abundance in the environment, and relative ease of analysis. PFAS are often distinguished by the length of their perfluoroalkyl chain. For example, long-chain PFAA subgroup perfluoroalkyl carboxylic acids (PFCA) usually comprise $\geq$8 carbon alkyl chains e.g. perfluorooctanoic acid (PFOA); and long-chain PFAA subgroup perfluoroalkyl sulphonic acids (PFSA) usually comprise $\geq$6 alkyl chains e.g. perfluorooctanesulfonic acid (PFOS). For further information about when PFAS were first synthesised, introduced into

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industrial products and became widespread in their application in home, commercial, and industrial products refer to Buck et al. (2011) and ITRC (2022).

Some PFAS have been shown to pose risks of harm to human and environmental health. PFOA was recently reported as carcinogenic to humans (Group 1) and PFOS as possibly carcinogenic to humans (Group 2B) by the International Agency for Cancer Research (IARC) (Zahm et al., 2024). The United States Environmental Protection Agency (USEPA) reported that scientific literature on exposure to some PFAS may lead to a range of adverse health effects in humans including decreased fertility and increased blood pressure in women, development delays in children, increased risk of cancer (prostate, kidney, testicular), reduced immunity, interference with hormone levels, and increased cholesterol and/or obesity (USEPA, 2024). These health effects lead to need to understand the spatial distribution, concentration, and type of PFAS present in the environment and the impacts on people and ecosystems. While the water environment has been - and continues to be - the subject of significant testing and research for these chemicals, UK soils are still poorly understood. An understanding of background concentrations of PFAS in soil helps with the assessment of potential contamination exposure scenarios, setting benchmark levels and risk evaluation. To date there have been no systematic studies of PFAS in soils in the UK.

PFAS have been shown to accumulate in shallow unsaturated soils at some locations as a result of firefighting activities, contaminated biosolids amendments to land, and atmospheric deposition (Sharifan et al., 2021). Reported concentrations of PFOS in soils from a literature review for fire-training areas in Europe and North America ranged from 750 to 55,000 μg/kg dry weight (dw) and for biosolid applications to land from 1 to 500 μg/kg dw (Sharifan et al., 2021). Background concentrations in soils can be expected to be much lower than primary or secondary sources (Rankin et al., 2016).

Anthropogenic background concentrations of persistent organic pollutants (POPs) in shallow soil assumes a distribution initially from anthropogenic point sources that over time has become diffuse. Background concentrations of legacy POPs vary dependant on type and source. Global background concentrations of polychlorinated biphenyls (PCBs), a family of 209 synthesised chlorinated organic compounds, in shallow soils were reported to range from 26 to 97,000 ng/kg dw (Meijer et al., 2003). Polycyclic aromatic hydrocarbons (PAH), a family of 1000s of naturally occurring and synthesised chemicals, where reported an order of magnitude higher with concentrations between <1 and 52 mg/kg dw (Nam et al., 2009; Vane et al., 2014).

Diffuse sources of PFAS include aerial deposition as well as contaminated organic-derived materials applied to land for agricultural benefit (e.g., biosolids, composts, and digestates) and wastewater irrigation. Our literature search for background PFAS in soils showed that a range of different types accumulate in shallow unsaturated soils. We used Scopus and a set of search terms to return articles on background concentrations of PFAS in soils (Supporting Information: Section 1). Our comparison of different studies of background PFAS in soils was challenging due to the differences in types, limits of quantification, land uses selected, and the contribution of of point sources in the concentrations determined. These challenges highlight the need for and importance of countries like the UK to conduct their own survey work. A summary of the studies we reviewed are summarised by geographical scale.

1.1. International background surveys

Brusseau et al. (2020) conducted a review of PFOS and PFOA concentrations from over 1400 sites around the world including 30,000 samples from contaminated locations and 5700 samples from background locations without a known PFAS source in the immediate vicinity. Maximum PFOS concentrations recorded by the surveys reviewed ranged from 0.003 to 162 μg/kg dw and the maximum PFOA concentrations ranged from 0.01 to 124 μg/kg dw. Rankin et al. (2016) sampled 62 shallow soil samples from 22 countries and analysed them for 32 PFAS. $\sum$PFCA concentrations for those detected ranged between 0.3 and 1.4 μg/kg dw and for $\sum$PFSAs between the limit of quantification (LoQ) and 3.3 μg/kg dw. Washington et al. (2019) used Rankin et al.'s (2016) PFAS data ($n = 62$) to estimate global surface soil background concentrations, reporting a global mean PFOS concentration of 0.056 μg/kg dw. Strynar et al. (2012) developed a laboratory method for measuring PFAS concentrations in shallow soil as part of early method development work conducted by the US Environmental Protection Agency. The authors collected 60 soil samples from six countries. Eleven PFAS were detected at concentrations ranging between 0.5 and 79 μg/kg dw.

1.2. National background surveys

Sörengård et al. (2022) sampled 27 remote forest soils in Sweden and measured 28 PFAS. The maximum concentration of PFOS was 1.5 μg/kg dw and for PFOA was 0.27 μg/kg dw. These authors used the concentration data to estimate PFAS loads along transects from north to south and east to west of Sweden, finding that loads were much higher in remote areas in the urbanised south compared to the north. Sim et al. (2021) sampled 57 soils from a variety of land uses in South Korea including urban, industrial and remote locations. Concentrations differed marginally between them with PFOS reported at a maximum of 2.95 μg/kg dw and PFOA at 1.36 μg/kg dw (both from industrial locations). Kotthoff et al. (2020) and Göckener et al. (2021) reported PFAS in seven archived soil samples in Germany collected between 2014 and 2017; reported average concentrations 3 μg/kg dw and 1.5 μg/kg dw for PFOS and PFOA respectively. The Public Waste Agency of Flanders (OVAM) reported background concentrations of PFAS in Flanders for PFOS, PFOA, and PFBA from 50 samples (OVAM, 2021a,b,c,d). Background based on the 90th percentile was reported for PFOS at 1.5 μg/kg dw, PFOA at 0.96 μg/kg dw, and PFBA at 1.25 μg/kg dw. The National Institute for Public Health and the Environment (RIVM), Netherlands published temporary background values for PFOA at 0.8 μg/kg dw PFOS at 0.9 μg/kg dw (95th percentile) (RIVM, 2019). Wintersen et al. (2020) conducted a second survey to build on the initial results in the RIVM report comprising a further 100 samples. Using the additional data and the same methodology used by RIVM (2019), revised background values for the Netherlands would be 1.4 μg/kg dw and 1.9 μg/kg dw for PFOA and PFOS, respectively.

1.3. Regional (in country) surveys

Cabrerizo et al. (2018) collected and analysed 34 soils for 16 PFAS from a remote high arctic island environment in Canada. Despite the remote location, the study reported relatively high concentrations of PFAS e.g. PFOS at 7.5 μg/kg dw, recording all 11 commonly measured PFAS. Zhu et al. (2022) published a Vermont, US state-wide survey comprising 66 shallow soils sampled based on a grided sampling strategy. Analysis showed that the total PFAS concentration was dominated by PFOS (0.1–4 μg/kg dw), PFOA (0.1–5 μg/kg dw) and PFNA (0.05–0.4 μg/kg dw) with a total of 17 PFAS detected more or less ubiquitously across the state. Higher PFAS concentrations were detected in the north of the state, which correlated with high population centres and the proximity to more industrialised land uses.

2. Materials and methods

For this study, 25 G-BASE archive samples were selected, from which a sub-set of 9 samples were chosen for paired contemporary field sampling. We used targeted sampling of the national archive to ensure that soils of interest and most likely to contain measurable PFAS were selected, favouring more recent samples but avoiding known source sites.

2.1. Sample selection

The BGS National Geological Repository (NGR) soil archive comprises over 44,000 shallow soil samples collected in England over the past 30 years as part of the geochemical baseline survey of the environment (G-BASE).

2.1.1. Archive soil samples

The geographical locations of 25 archive samples selected for PFAS analysis were in southern England and have been anonymised to protect the identity of the land owners which gave permission to collect the original samples. The overarching interest in background concentrations of PFAS led to samples being selected from the rural areas rather than urban ones where multiple point sources were more likely. The selection of the samples was based on criteria agreed by the project team (Supporting Information: Section 2).

It was considered likely that a substantial proportion of analyses might yield PFAS concentrations below the LoQ. For this reason, there was a need to ensure that archive soils contained detectable levels of PFAS. This was addressed by focussing on samples originally collected in 2014, the most recent survey year. These younger samples were assumed most likely to contain the largest accumulation of PFAS and least likely to be affected by changes due to storage and aging.

Data held by the Environment Agency was used to determine whether each sample location was situated on or close to a likely point source and the number of such sources that were within 1 km of the sample location. PFAS point sources considered included fire training grounds, airfields, and industries likely to have used significant quantities of PFAS such as carpet manufacturers and electro-metal plating works. Two samples were selected on the basis of having more than two potential point sources within 1 km to maximise the likelihood of detecting background PFAS concentrations, while also avoiding direct contact with source sites.

Although most selected archive samples were originally collected in 2014, we examined the relationships between the measured PFAS concentrations and the age of the sample. To examine the role of sample age, we selected and analysed three samples collected in 1999 and two in 2007, which were located geographically close to the 2014 samples.

We also took two archive samples from Royal Parks located in London to account for urban locations that had not been directly impacted by development. These were selected because large public open spaces tended to show lower concentrations of anthropogenic contamination including PAH compared to developed urban areas (Vane et al., 2014).

Our archive sampling strategy was also designed to investigate whether soil texture and soil organic matter content influenced the detection and concentrations of PFAS. Data describing these factors were divided into three classes, with one sample randomly selected from each. The texture classes were the 'sand clay ratio' classes described below. Soil organic matter classes were based on loss on ignition (LOI) measurements. We excluded high organic content LOI>20% samples because they presented a unique set of analytical challenges that could have led to erroneous or misleading data.

2.1.2. Contemporary paired field soil samples

The selection of nine paired contemporary soil samples was based on a sub-set of the archive samples selected for analysis. Landowners were identified for each archive locationand twenty-three letters were sent to ask permission to resample these sites. Seven landowners agreed to resampling, covering 9 individual rural sites.

2.2. Sample collection

2.2.1. Archive sample collection

G-BASE archive soil samples were collected by hand from the BGS NGR. A sub-sample of approximately 15 g was taken from each plastic zip-fastening bag using a pre-cleaned stainless-steel spatula and transferred to new plastic bag.

2.2.2. Paired field sample collection

Paired contemporary field samples were collected in January 2023 using the G-BASE procedures for grid based composite sampling of shallow soils (Johnson, 2005). G-BASE sampling methods were not originally designed for PFAS sampling. We conducted a review of the procedures against current guidelines and concluded that the materials and approaches, while not optimised for PFAS, were suitable for this study and minimised any differences between archive and contemporary data that may have been introduced by the sampling.

One contemporary field sample and one replicate ($n = 2$) was taken from each location by field sampling staff. Each site location was photographed. Land use of the sites varied and included: arable land, parkland, unimproved grassland, and a nature reserve. A handheld global positioning system (GPS) was used to geolocate the centroid of the sampling point as close as possible to the historical G-BASE sample site (± 5 m). Unique sample reference numbers were assigned including an identifier linking to the original G-BASE sample reference.

Two paper Kraft™ sample bags measuring 12.5 × 27 cm were labelled using a ballpoint biro pen, with the sample number, sample type, date, sample depth and the organisation (BGS). Uncoated Kraft™ bags constructed from paper using a high-density weave which is naturally water repellent.

Each sample was collected manually using a Dutch auger. . Prior to sampling, the auger was pre-cleaned using a wire brush, then rinsed with PFAS free water , and finally by collecting and discarding one auger flight of soil. The samplers wore powderless nitrile gloves (the original G-BASE protocol was to use bare hands, but this was not considered appropriate for this project due to health and safety reasons). The samplers took care to avoid wearing make-up, suncream, clothes containing Gore-Tex or other suspected potential PFAS sources.

Each sample comprised a composite of material taken from five vertical auger holes: one at each corner and then the centre of a 20 m × 20 m square. The grid reference was recorded at the centre point of the sampling square and the edges paced out on foot. Where it was not possible to take samples from a 20 m × 20 m square due to space constraints, the sampler spread the sub-samples out as much as possible.

The auger was twisted into the ground vertically to ensure the correct depth of 20 cm was reached. The full flight was carefully removed from the hole, and the top 5 cm, containing grass and roots discarded. The remaining sample was carefully placed into the Kraft™ bag. The four other samples were collected in the same way and placed into the same sample bag. The sampler recorded the soil texture, the sample colour, the likely organic content, and the soil clast lithology.

All field samples were placed in secondary zip lock bags and then into a cool box containing pre-frozen ice packs (also placed in zip lock bags). Samples were transported back to the BGS and stored at minus 20 °C until sample preparation at BGS and/or onward preparation and analysis.

2.3. Sample analysis and quality control

RPS-Tetra Tech Laboratories in the UK were used to analyse soil PFAS samples ($n = 50$). The samples analysed included G-BASE archive soils ($n = 25$), paired fresh field samples ($n = 9$), replicate archive samples ($n = 5$), replicate fresh field samples ($n = 5$), unextracted BGS reference soil samples ($n = 1$), extracted BGS reference soil samples that were spiked with an analytical standard containing 25 PFAS (Supporting Information: Section 2) ($n = 5$). The in-house BGS PFAS spiked-reference soil was created to aid assessing the precision and accuracy of the analytical data. The reference soil was a sandy loam from the Balderton Sand and Gravel Member (river terrace deposits) collected near Newark, Nottinghamshire, UK in January 2023.

The PFAS-53 suite by substance class, sub-class, group sub-order, alkyl chain carbon number, and chain length is presented in the

Supporting Information (Section 3). Just under half of the PFAS-53 were perfluoroalkyl substances ($n = 35$) comprising PFCA ($n = 13$) and (PFSA) ($n = 11$). Other perfluoroalkyl substances were perfluoroalkyl ether carboxylic acids (PFECA) ($n = 6$), perfluoroalkyl ether sulphonic acids (PFESA) ($n = 1$) and perfluoroalkyl sulfonamides (PFASA) ($n = 3$). The remaining PFAS were polyfluoroalkyl substances ($n = 19$) comprising fluorotelomer carboxylic acids (FTCA) ($n = 3$), fluorotelomer sulfonic acids (FTSA) ($n = 4$), PFECA ($n = 1$), PFESA ($n = 2$), PFASA ($n = 8$), and PAP ($n = 1$). Alkyl chain lengths ranged from 4 to 18 carbons.

2.3.1. *Sample preparation*

At the time of sampling, archive samples were oven dried and sieved to <2 mm after collection as part of the original G-BASE sample preparation method and prior to storage in the NGR. The mean average ambient temperature of the G-BASE sample storage area in the NGR is 18.4 °C and has a relative humidity of 37.2 %. While not optimised for PFAS analysis these procedures were considered sufficient for this study.

Contemporary paired field samples collected during January 2023, were oven dried at BGS (30 °C, 48 h), gently disaggregated with a pestle & mortar and sieved using 2 mm mesh-size brass sieve. This method was selected because it was consistent with the original method used for the archive samples. The dried and sieved samples were stored in the dark in polyethylene zip lock bags.

For this research, a sub-sample of up to 20 g was collected for each respective archive and field sample and transferred to polyethylene zip lock bag and sent to RPS-Tetra Tech Laboratories for analysis.

2.3.2. *Soil sample analysis by liquid chromatography mass spectrometry*

Fifty-one soil samples comprising of archive, paired contemporary field and QC samples, were received by the laboratory for analysis of PFAS-53 (Supporting Information: Section 3). LoQ ranged from 0.1 to 0.5 μg/kg dw.

Extraction and analysis of PFAS-53 were performed by RPS-Tetra Tech Laboratories using ultra high-performance liquid chromatography (UHPLC) coupled to a triple quadrupole tandem mass spectrometer (LC-MS/MS). The dried soil was accurately weighed (5 g) into a vessel and spiked with surrogate recovery standards and deuterated/carbon-labelled internal PFAS standards at 2.5 μg/kg DW. The internal standards were used to compensate for matrix/ recovery effects. A methanol-based solvent was added to the soil, which was then shaken and centrifuged. A sample of resulting supernatant was subject to clean-up to remove interfering compounds by proprietary solid phase extraction (SPE). This extract was concentrated to 0.5 mL prior to analysis by LC-MS/MS using negative ion electrospray ionisation (ESI) set to dynamic multiple reaction monitoring (ERM).

2.3.3. *Laboratory quality control*

Analytical quality control samples used by RPS-Tetra Tech Laboratories in each LC-MS/MS batch included a spiked solvent blanks and their own internal manufactured reference soil.

3. Results and discussion

Archive and field samples were collected from southern England. Accurate sample locations are confidential to protect the anonymity of the landowners that gave permissions to sample. PFAS-53 concentrations, quality control data, and certificates are presented in the Supporting Information: Section 4.

3.1. *G-BASE archive samples*

PFAS were detected in all 25 archive samples with 11 PFAS detected > LoQ (Supporting Information: Section 4). ∑PFAS-53 based on PFAS detected ranged from 0.15 to 6.32 μg/kg dw, with a mean for all samples of 1 μg/kg dw.

The PFAS detected in the archive samples were mostly PFCA (62 %) and PFSA (29 %), the exception was 6:2 (polyfluoroalkyl phosphate diester) DiPAP (6 %). Four of the 11 recorded PFAS were at detection frequencies ≥30 % (10 samples with all five). In order of most commonly detected, these were: PFOA ($n = 24$; mean 0.27 μg/kg), PFBA ($n = 22$ 0.26 μg/kg), PFOS ($n = 21$; mean 0.63 μg/kg), and 6:2 diPAP ($n = 10$; mean 0.1 μg/kg) (Supporting Information: Section 5). Non-detects were included as zero in the calculation of mean values.

Analytical uncertainties for PFBA and 6:2 DiPAP were identified. Both PFAS were detected in the laboratory analytical blanks and PFBA showed a high Coefficient of Variation in archive replicate samples (Supporting Information: Section 5). The cause of these anomalies may be related to analytical carry-over between samples or cross-contamination somewhere else in the sample collection, preparation or analysis procedures. 6:2 DiPAP was not recorded in the contemporary field samples.

3.2. *Contemporary paired field samples*

PFAS were detected in all nine contemporary paired field samples, nine were detected > LoQ. ∑PFAS-53 based on PFAS detected ranged from 0.68 to 8.96 μg/kg dw, with a mean of 3 μg/kg dw (Supporting Information: Section 4). The PFAS detected were PFCA (77 %) and PFSA (23 %). Eight out of the nine PFAS were recorded at detection frequencies ≥40 % (8 samples). In order of most commonly detected, these were: PFOA ($n = 9$; mean 0.60 μg/kg dw), PFBA ($n = 9$; mean 0.60 μg/kg dw), PFOS ($n = 8$; mean 0.72 μg/kg dw), PFHpA ($n = 7$; mean 0.31 μg/kg dw), PFHxA ($n = 7$; mean 0.31 μg/kg dw), PFPeA ($n = 6$; mean 0.34 μg/kg dw), ($n = 4$; mean 0.31 μg/kg dw), and PFNA ($n = 4$; mean 0.14 μg/kg dw) (Fig. 2).

There was a potential uncertainty with PFBA because it was also detected in the analytical blanks, the cause of which could not be determined.

3.3. *Comparison of archive and contemporary field samples*

Sunburst plots show that in archive soils: PFOS, PFBA and PFOA dominated the profile (Figs. 1 and 2), with concentrations >2 μg/kg dw in some samples. The PFAS profile for contemporary paired samples was spread between a wider range of PFAS but still with PFOS providing the

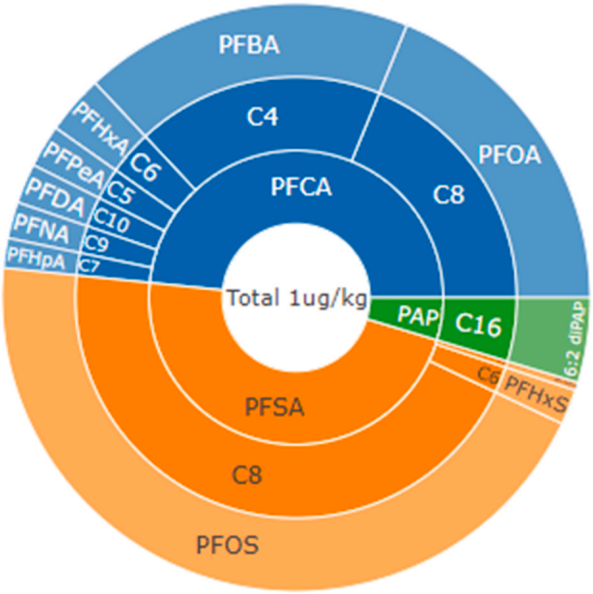

Fig. 1. Sunburst plot showing the mean PFAS contributions recorded in archive samples and ∑PFAS-53.

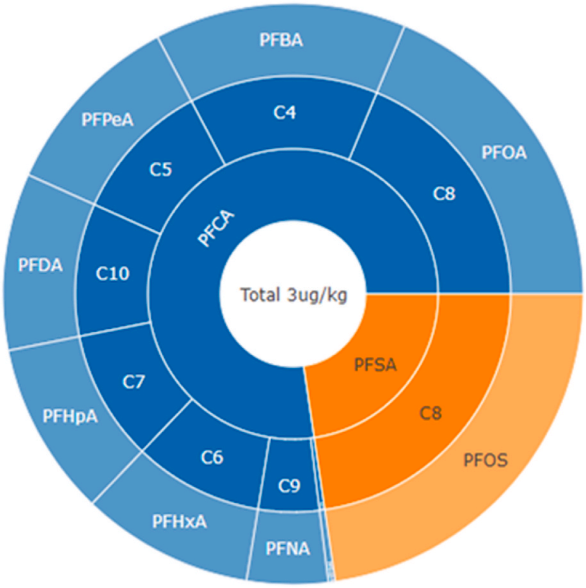

Fig. 2. Sunburst plot showing plot showing the mean PFAS contributions recorded in contemporary paired field samples and the mean $\sum$PFAS-53.

greatest contribution. This finding suggests that either: (1) PFOS dominated older emission profiles reflecting its recent phasing out; and/or (2) PFOS proved more resistant to degradation in archive samples than other PFAS. Further research is needed to investigate the cause and the most likely reason.

Concentrations and contributions of PFAS-53 for both archive and contemporary paired field samples are presented as stacked bar charts in Figs. 3 and 4. The figures show that paired contemporary samples generally had higher detection frequencies and concentrations of PFAS than those reported in archive samples. The charts emphasise that for commonly observed individual PFAS in this study, paired field samples tended to have higher concentrations than archive samples and that the difference between archive and contemporary field replicates varied between locations for many of the samples. Again, this could be due to older PFAS emission profiles in the archive samples, the durability of PFAS in these older samples or changes in the PFAS mixtures used in contemporary sources. The differences between PFBA in archive and contemporary samples could have been influenced by sampling (materials used, cross-contamination) or analytical factors. The presence of 6:2 DiPAP in the archive samples may have been due to similar factors. Studies have shown that PFBA has been recorded in laboratory equipment and consumables (Denly et al., 2019; Wanzek et al., 2024) and DiPAP in waterproofing and other additives in paper (Morris, 2024). The bar charts also show that some of the archive and contemporary field samples present similar PFAS profiles (e.g. sample 10, 16, and 20), whereas for others (e.g. sample 1 and 4) they were different. Whilst the causes of these observations were not clear, they could reflect differences in PFAS use and emissions over time.

A comparison of the concentrations of commonly detected PFAS in the paired archive and contemporary field samples shows that the contemporary field samples tended to have higher concentrations than archive samples (Supporting Information: Section 4). This shows that the concentrations of PFAS in archive and field replicates varied between locations for some samples. These differences could also have been due to older PFAS emission profiles, the durability of PFAS in older samples or changes in the PFAS mixtures used in contemporary sources. The relationship between PFBA in archive and contemporary field samples could have been influenced by analytical uncertainties. The possibility of a one-to-one relationship biased by high PFAS concentrations in contemporary samples should not be entirely discounted due to the small sample size for this study.

3.4. *Effect of sample selection factors*

The selection of the archive samples was based on criteria agreed by the project team (Supporting Information: Section 1). The concentrations of $\sum$PFAS-53, PFBA, PFOA and total PFOS were compared against these criteria (age of sample, proximity to source sites, soil texture proxy, and organic matter proxy) (Supporting Information: Section 4). The data showed no obvious relationship between PFAS concentrations and any of the selection factors. Given the small number of samples ($n = 25$) and the range of confounding factors likely to have influenced the PFAS concentrations (e.g. the presence/absence of influencing original

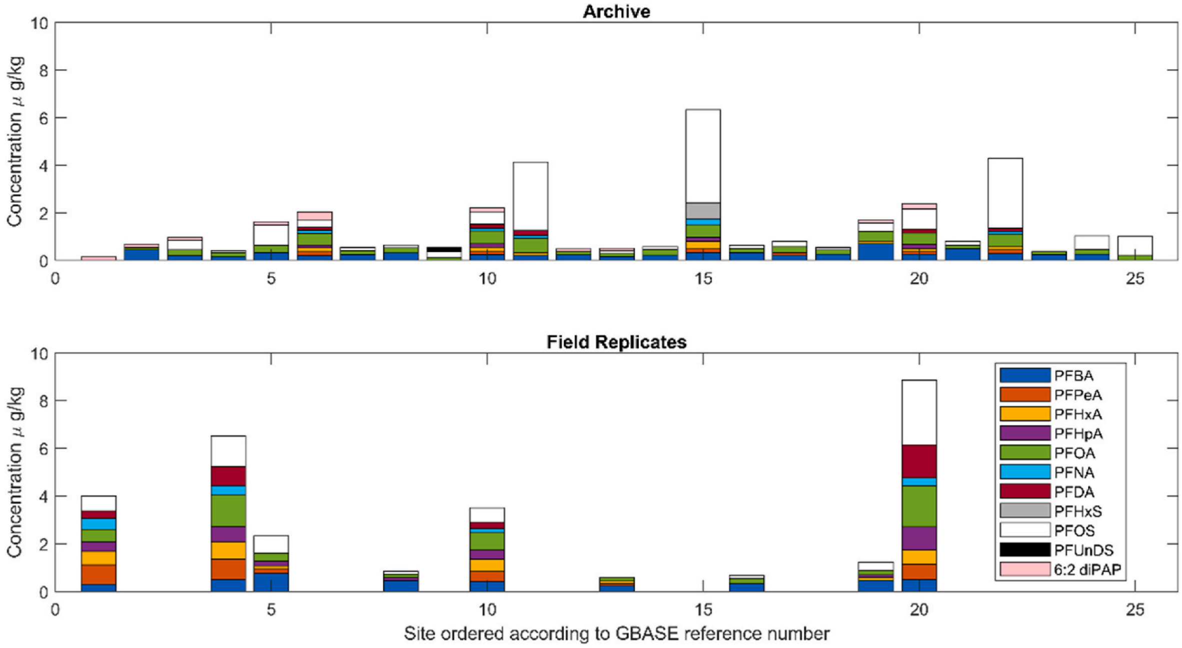

Fig. 3. Stacked bar graph of concentration of constituents of $\sum$PFAS-53 measured in BGS archive samples and at corresponding contemporary paired field sites.

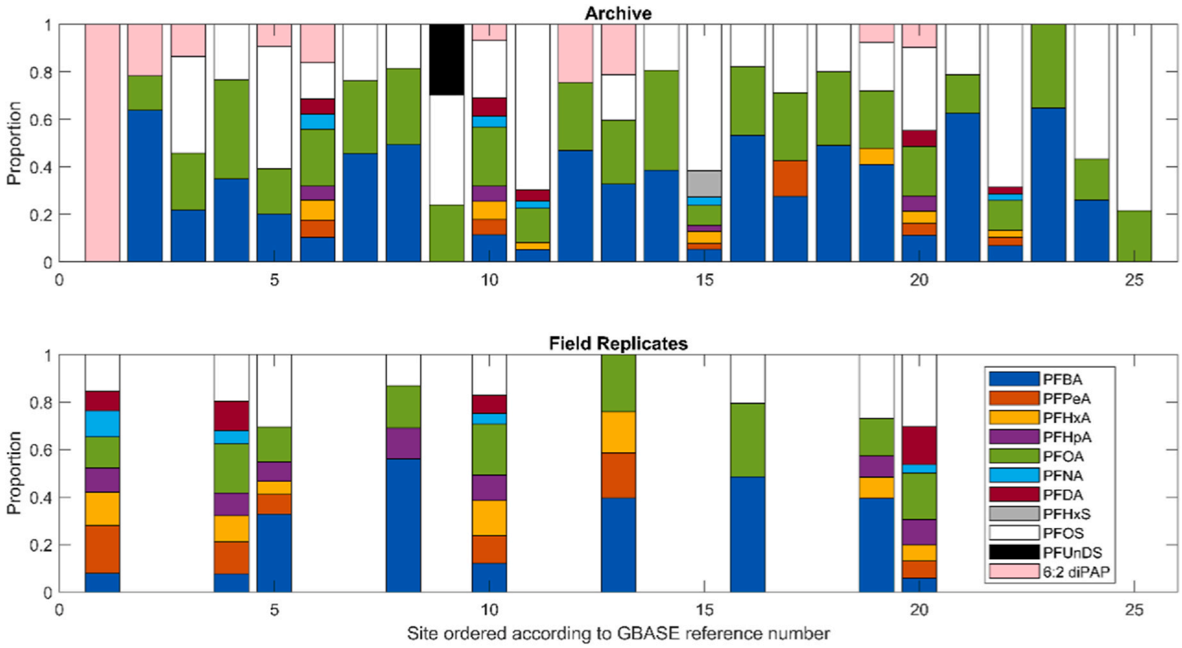

Fig. 4. Stacked bar graph of proportion of constituents of ∑PFAS-53 concentration measured in BGS archive samples and at corresponding contemporary paired field sites.

point sources, soil properties, prevailing weather conditions and land use), it was not unsurprising that no clear relationships were evident.

3.5. *Implications for national surveys of background PFAS in soils*

Our research was designed to address knowledge gaps on anthropogenic background PFAS in English soils which included an assessment of the suitability of archive samples to support contemporary samples in national surveys. The research has been able to provide a preliminary indication of the type and concentration of PFAS in shallow soils and has highlighted some questions around sampling and analytical factors affecting PFBA and 6:2DiPAP.

We conducted a literature search of various scales of background soil surveys. The search showed that median values of the maximum concentrations in μg/kg dw for commonly measured PFAS reported, in descending order include: PFOA (2.7) > PFOS (1.63) > PFDA (0.68) > PFNA (0.62) > PFUnDA (0.49) > PFHpA (0.42) > PFHxA (0.22) > PFDoDA (0.10) > PFBS (0.05) > PFHxS (0.01) > PFDS (0) (Supporting Information Table S5).

We found that international surveys, while providing useful information, are either based on very few samples distributed across a small number of countries (Strynar et al., 2012; Rankin et al., 2016) or they bring together literature data from a range of surveys conducted by other authors (Washington et al., 2019; Brusseau et al., 2020). Each type of international study presents its own limitations which suggests a combination of country and regional surveys provide a better proxy to designing and implemented national scale surveys for the UK. In addition, each study was designed to select samples from a range of land uses with differing associations to point sources and to perform analysis based on lab-specific LoQs and PFAS suites. These factors make comparing results to our research challenging and calls for the need for a national survey of England to determine anthropogenic background concentrations of PFAS in shallow soils rather than relying on secondary data. In spite of these challenges we found that the central tendency of background PFAS concentrations for national and regional shallow soil surveys for commonly detected substances range from <0.01 to 3 μg/kg dw (Table 1). These concentrations are broadly consistent with concentrations for our mean archive (1 μg/kg dw) and contemporary field samples (3 μg/kg dw).

The literature on background PFAS in shallow soils showed that shallow soil samples tend to be fresh and sampled from between 1 and 20 cm below ground level. However, two studies of reviewed used archived rather than contemporary shallow soil samples collected from the same depths and stored in a national archive (Kotthoff et al., 2020; Göckener et al., 2021). With regard to depth, it has been reported that transport of PFAS through the soil profile sometimes leading to increases at depth (Gerardu et al., 2023). Sample numbers collected for analysis for the studies reviewed ranged from 30 to 100. Sample preparation prior to analysis, where reported, was by drying and sieving to ≤2 mm. Analysis was exclusively by LC-MS/MS and very few surveys used supplementary enhanced testing such as TOP analysis (Göckener et al., 2021; Shojaei et al., 2022) or mass balance techniques such as total organic chlorine (Bugsel et al., 2022; Zweigle et al., 2022).

Taking into consideration wider published literature, there is a gap in the literature for a national survey of soils in England and the UK to improve on the design of existing large-scale surveys. The use of statistical power of analysis methods should be used for sample selection, including consideration of land use types, soil types and known PFAS sources. There is potential value in using screening techniques such as the TOP assay, total organic fluorine, or high-resolution analytical techniques to provide an indicator of potential future PFAS burden and risk based on a wider range of PFAS that may be present than those being detected with standard suites and possible substance transformation (by oxidation or similar processes) over time. We show that while archive samples tend to have lower concentrations of PFAS than contemporary, our study indicates that temporal and spatial variation in concentration and type may be present if sample numbers were increased. It would also be useful to identify other surveys where multiple co-located samples have been collected over time and to examine deeper samples to understand the how PFAS behaves in different parts of the soil profile. We therefore assert that archive samples can provide useful supporting data rather than the primary sample type for national background surveys.

Future national surveys should also ensure and report how sampling procedures have been optimised to reflect good practice and relevant literature on PFAS analysis. These factors include an assessment of the influence on the accuracy and repeatability of PFAS analysis from: background PFAS in field/laboratory materials and equipment, cross-contamination during sample collection, the effects of drying and

Table 1
Summary of maximum concentrations and selected metadata for commonly recorded PFAS in large scale soil surveys (μg/kg).

Author	Scale	Countries	Samples	Per										
				PFCA							PFSA			
				PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUnDA	PFDoDA	PFBS	PFHxS	PFOS	PFDS
Rankin et al. (2016)	I	Twenty-three (see notes)	62	7.54	3.75	3.44	1.06	0.96	1.51	0.79	NM	0.01	3.13	0.13
Strynar et al. (2012)	I	Six (see notes)	60	12.4	79.1	31.7	0.61	2.03	3.3	3.94	0	3.23	10.1	0
Sim et al. (2021)	N	S. Korea	16	0.16	0.23	0.99	0.74	0.3	0.49	0.22	0.11	0	1.05	0
Kotthoff et al. (2020)	N	Germany	7	0.85	1.42	3.23	1.79	0.69	0.66	0	0	0	6.17	0
Göckener et al. (2021)	N	Germany	6	0	0	1.09	0.62	0	0	0	0	0	NM	0
Sörengård et al. (2022)	N	Sweden	27	0	1.9	0.57	0.7	0.68	0.76	0.33	0.96	0.40	1.70	0
Naile et al. (2013)	R	Korea (west coast)	13	0	0	3.40	0	0	0	0	0	0	1.70	0
Tan et al. (2014)	R	Nepal (Koshi River)	14	0.22	0.78	0.26	0	0	0	0.25	0.38	0	0.13	0
Meng et al. (2015)	R	China (Liaoning, Hebei, Shandong, Tianjin)	37	0.10	0.10	0.67	0.45	0.84	1.01	0.47	0.06	0	0.46	0
Chen et al. (2016)	R	China (Eastern)	55	1.08	0.59	3.48	3.19	3.85	2.29	0.98	0.06	0.03	1.56	0.02
Cabrerizo et al. (2018)	R	Canada (high arctic Islands)	34	0.18	0.24	0.40	0.90	0.11	0.33	0.04	0.01	0.11	7.50	0.004
Zhang et al. (2019)	R	China (Shandong)	7	4.80	0.75	8.75	1.93	1.03	0	0	0	0.25	0.12	NM
Cao et al. (2019)	R	China (Tianjin)	17	0.19	0.42	2.70	0.17	0.76	0.95	0.15	0.06	0	0.25	0
Santangelo et al. (2022)	R	United States (New Hampshire)	100	1.10	1.30	4.10	7.20	3.20	2.40	1.80	0.82	0.22	5.40	0
Zhu et al. (2022)	R	United States (Vermont)	66	1.50	0.90	4.9	0.40	0.43	0.2	0.10	1.60	0.44	4.40	0.38
Liu et al. (2022)	R	China (Qiantang)	14	0.94	0	2.80	0.97	1.22	1.33	0	0	0.83	1.10	0
Wang et al. (2022)	R	China (Three-North Shelter Forest Region)	8	0.01	0.10	0.16	0.02	0.09	0.05	0.04	0.04	0.01	0.04	0
Sanborn Head & Associates (2022)	R	United States (Maine)	32	13.70	0.24	0.57	0.27	0.16	0.20	0	0	0	5.32	0
Mean of study maximums			32	2.49	5.10	4.07	1.17	0.91	0.86	0.51	0.26	0.31	2.95	0.03
Median of study maximums			17	0.22	0.42	2.70	0.62	0.68	0.49	0.10	0.05	0.01	1.63	0.00

NOTES: Scale: I = International; N = national; R = regional (sub-national); NM = not measured by study; Rankin et al. (2016) countries: United States, Canada, Mexico, Puerto Rico, Denmark, Estonia, Ireland, Norway, Germany, Russia, China, Japan, Jordan, Nigeria, South Africa, Cameroon, Uganda, Australia, New Zealand, Chile, Uruguay, Chile, Antarctica; Stryner et al. (2012) countries: United States, China, Japan, Norway, Greece, and Mexico; Where the value zero is reported, this reflects less than the LoQ and has been used in to calculate the mean and median PFAS concentration.

comminution (Beriro et al., 2014) including the potential defluorination during ball milling (Yang et al., 2025), analysis, and archiving/storage, and the use of suitable reference materials created from natural contaminated soils.

4. Conclusions

National and regional studies of background concentrations of PFAS in shallow soils have been reported in the literature, but it is often challenging to interpret these across borders due to the study design including but not limited to limits of detection and the PFAS selected for analysis. Countries such as England are therefore considering whether they should carry out their own national or regional surveys. One option to reduce the potential costs of such sampling exercises is to consider the role that archive soil samples might play. This study assessed the role of using archived soil samples from historical large-scale surveys to support contemporary field sampling to estimate the anthropogenic background concentrations of PFAS in English soils. In doing so we have provided the first dataset for background PFAS for shallow soils in England.

PFAS concentrations in the contemporary paired field samples were generally higher than the archive samples, although there was no clear relationship between the counterparts was established. In addition, there were also no obvious spatial relationships in PFAS concentrations or between the factors used to select the samples (sampling year, soil properties, proximity to sources). While archive samples tend to have lower concentrations of PFAS than contemporary samples. Our study indicates that temporal and spatial variation in concentration and type may be present if sample numbers were increased, and we consider that archive samples could play a supporting role in future research and development of anthropogenic background concentrations of PFAS in shallow soils.

Future large-scale surveys should aim to optimise consistency with similar surveys in terms of the PFAS types, sampling depth, and sample preparation to aid with comparisons between studies to enable comparisons between studies to be made. Improvements that could be made to future large scale surveys include sample selection, collection and analysis, sample depth, temporal effects. Improvements could also made by optimising sample collection and preparation procedures to minimise contamination, PFAS types detected, non-target analysis, limits of quantification, data handling including reporting and non-detects, and the use of reference soils to aid with quality control.

CRediT authorship contribution statement

D.J. Beriro: Writing – review & editing, Writing – original draft, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Data curation, Conceptualization. **J.M. Bearcock:** Writing – review & editing, Writing – original draft, Resources, Methodology, Investigation, Conceptualization. **C.H. Vane:** Writing – review & editing, Resources, Methodology, Funding acquisition, Conceptualization. **B. Marchant:** Writing – review & editing, Writing – original draft, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **I. Martin:** Writing – review & editing, Methodology, Funding acquisition, Conceptualization. **A. Haslam:** Writing – review & editing, Methodology, Funding acquisition, Conceptualization. **H. Pickering:** Writing – review & editing, Project administration, Methodology, Funding acquisition, Conceptualization. **M. Hughes:** Writing – review & editing, Project administration, Methodology, Funding acquisition, Conceptualization. **A. James:** Writing – review & editing, Project administration, Methodology, Funding acquisition, Conceptualization.

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.

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Appendix A. Supplementary data

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Data availability

No data was used for the research described in the article.

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