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Evidence of large-scale deposition of airborne emissions of per- and polyfluoroalkyl substances (PFASs) near a fluoropolymer production plant in an urban area

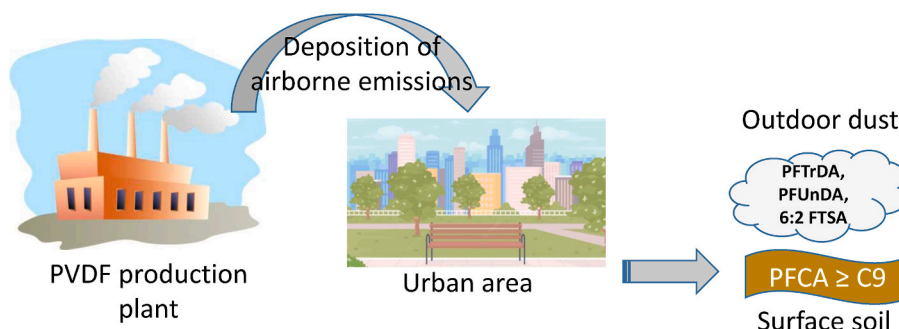
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HIGHLIGHTS

- PFAS soil contamination of an urban area near a fluoropolymer plant.
- PFUnDA and PFTrDA were predominant in surface soil and outdoor dust samples.
- Highest PFUnDA and PFTrDA concentrations in soil were 245 and 75 ng/g dw.
- Highest concentrations were recorded downwind of the PVDF production unit.
- Contamination very likely stems from the use of Surfion® S111 for 14 years.

GRAPHICAL ABSTRACT



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ABSTRACT

Airborne emissions of per- and polyfluoroalkyl substances (PFASs) from fluoropolymer manufacturing facilities—especially those producing polyvinylidene (PVDF)—have rarely been investigated. Once PFASs are released into the air from the facility stacks, they settle in the surrounding environment, contaminating all surfaces. Human beings living in close proximity to these facilities can be exposed through air inhalation and ingestion of contaminated vegetables, drinking water or dust. In this study, we collected nine surface soil and five outdoor settled dust samples within 200 m of the fence line of a PVDF and fluoroelastomer production site near Lyon (France). Samples were collected in an urban area including a sports field. High concentrations of long-chain perfluoroalkyl carboxylic acids (PFCAs) ($C \geq 9$) were found at sampling points downwind of the facility. Perfluoroundecanoic acid (PFUnDA) was the predominant PFAS in surface soil (12–245 ng/g dw), whereas perfluorotridecanoic acid (PFTrDA) was in outdoor dust (<0.5–59 ng/g dw). The PFAS profiles observed in soil and dust samples very likely originate from the processing aids used for PVDF and fluoroelastomer production. To our knowledge, long-chain PFCA concentrations as high as reported herein have never been found outside the perimeter fencing of a fluoropolymer plant. PFAS concentrations in other environmental compartments (such as air, vegetables or groundwater) should be monitored to assess all potential pathways to exposure of nearby residents before carrying out human biomonitoring.

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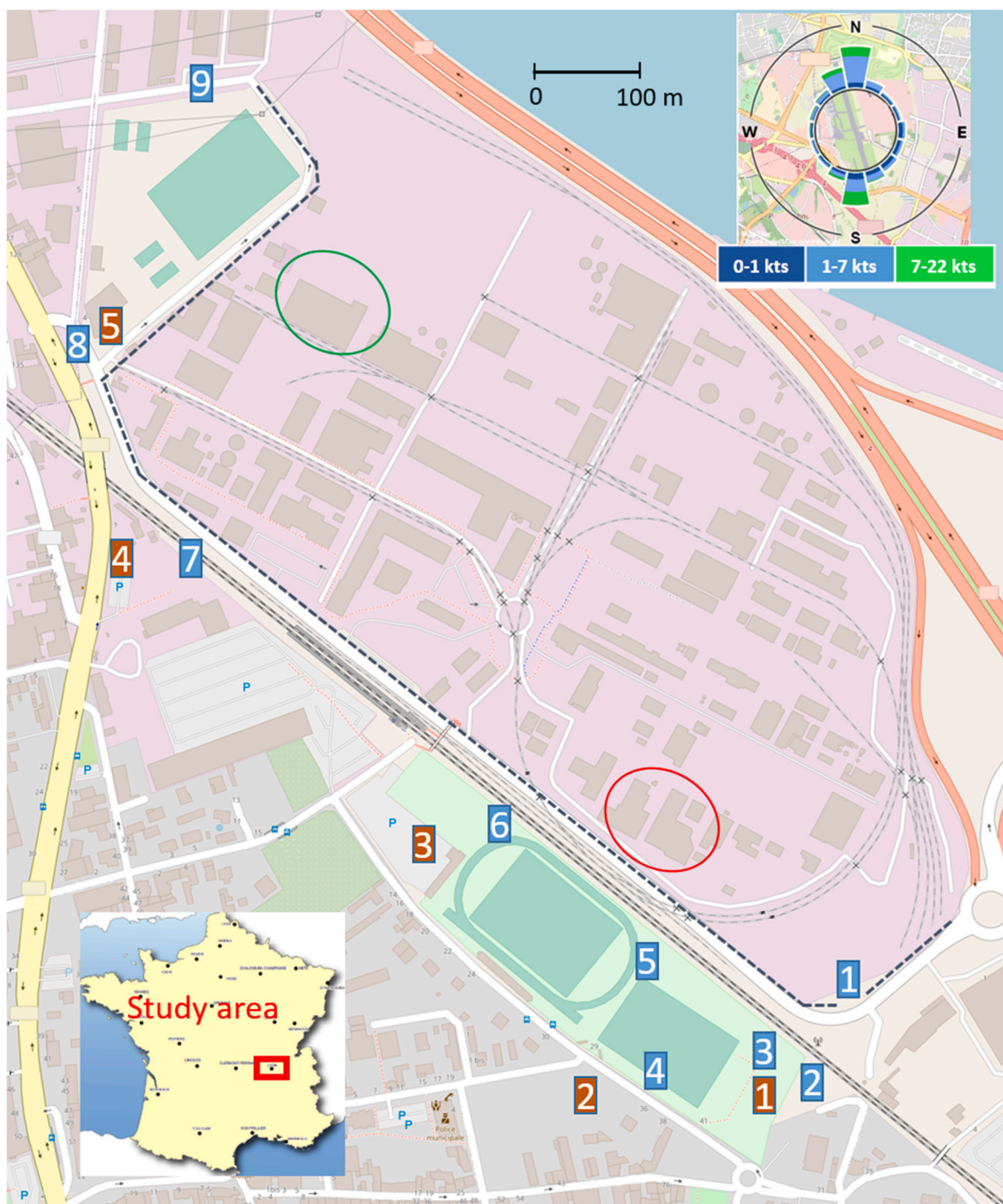


Fig. 1. Soil (blue squares) and dust (orange squares) sampling locations. The wind rose at the top right represents the annual average in knots (kts) for prevailing wind based on data from a measuring station 9.3 km east of the industrial park. The PVDF production unit is indicated by a red circle, the fluoroelastomer unit by a green one, and the industrial park's perimeter fence by a black dotted line.

1. Introduction

Per- and polyfluoroalkyl substances (PFASs) are anthropogenic organic chemicals that contain at least one carbon atom on which all the hydrogen atoms have been replaced by fluorine atoms (Buck et al., 2011; OECD, 2021). Production and use of PFASs have been steadily scaled up since the 1950s (Prevedouros et al., 2006; Wang et al., 2014). One of their first large-scale applications was as processing aids for manufacturing fluoropolymers such as polytetrafluoroethylene (PTFE) and polyvinylidene (PVDF) (Lohmann et al., 2020; Prevedouros et al., 2006). Perfluorooctanoic acid (PFOA) and perfluorononanoic acid

(PFNA) were the most widely used PFASs during emulsion polymerization of PTFE and PVDF, respectively (Lohmann et al., 2020; Prevedouros et al., 2006). Nowadays, these legacy long-chain PFASs have been replaced by new PFASs such as 6:2 fluorotelomer sulfonic acid (6:2FTSA) or per- and polyfluoroalkylether carboxylic acids (PFECAs) (Bach et al., 2017; Gebbink et al., 2017; Strynar et al., 2015). These processing aids were released into the environment in large quantities during fluoropolymer production, with several studies reporting significant pollution of water bodies around these facilities (Bach et al., 2017; Dauchy et al., 2012; Gebbink and van Leeuwen, 2020; Shi et al., 2015). However, detection of PFASs in soil samples collected several kilometers

from different plants suggests airborne transport in addition to PFAS emissions through wastewater effluents discharged into receiving rivers or industrial waste disposed of in landfills (Davis et al., 2007; Galloway et al., 2020; Gebbink and van Leeuwen, 2020; Hangen et al., 2010; Washington et al., 2020). Global emissions of PFOA and PFNA from 1951 to 2002 from fluoropolymer manufacture ranged from 2400 to 5400 tonnes (Prevedouros et al., 2006; Wang et al., 2014).

PFASs in vapor and particulate phases are emitted from facilities manufacturing fluoropolymers, transported by the wind and removed from the atmosphere through wet or dry deposition on surface soil (Barton et al., 2006, 2007, 2010; Davis et al., 2007). Data on soil and outdoor dust contamination near PFAS facilities are scarce. Investigations have occasionally been carried out around mega fluorochemical industrial parks with massive production and/or use of PFASs for several purposes (Chen et al., 2018; Su et al., 2016; Wang et al., 2010, 2021). Studies around fluoropolymer manufacturing facilities have been carried out in the USA (D'Ambro et al., 2021; Davis et al., 2007; Galloway et al., 2020; Washington et al., 2020), the Netherlands (Brandsma et al., 2019; Gebbink and van Leeuwen, 2020), and Germany (Hangen et al., 2010). Different PFASs have been monitored (e.g., PFOA, hexafluoropropylene oxide dimer acid (HFPO-DA)) at distances from the facilities ranging from a few 100 m to several kilometers, but rarely both in soil and outdoor dust samples. One study (Washington et al., 2020) focused on a PVDF production facility, but only soil samples were collected and the closest sampling point was 1.7 km from the facility.

This study was designed to characterize PFAS contamination in surface soil and outdoor dust following atmospheric deposition within 200 m of a fluoropolymer plant's perimeter fence. To our knowledge, this is the first sampling campaign to have been conducted so close to such a facility's fence line. This choice appears fully justified since the facility is located in an urban area with a sports field within 200 m of the fence, downwind of the source.

2. Material and methods

2.1. Sampling sites

The fluoropolymer plant is inside the 2.3-km-long fence line of an industrial park covering about 260,000 m². This park is surrounded by an urban area except to the north, where it is bordered by a highway and a river (Fig. 1). It is located in Pierre-Bénite, a suburb in the southern part of Lyon (Fig. 1).

The fluoropolymer plant, belonging to ARKEMA, used PFOA to manufacture PTFE between 1960 and 1986. The PTFE production volumes ranged from 500 to 1000 tonnes per year. Setting a PFOA use rate at 2 kg/tonne of PTFE (Wang et al., 2014), 26–52 tonnes of PFOA were used during this period.

PVDF production started in 1973. Depending on the PVDF grade, different PFASs were used as processing aids. PFNA was used between 2003 and 2017 under the trade name Surflon® S111, a mixture of perfluoroalkyl carboxylic acids (PFCAs) containing 74 wt (wt)% of PFNA, 20 wt% of perfluoroundecanoic acid (PFUnDA), 5 wt% of perfluorotridecanoic acid (PFTrDA), and 1 wt% of PFOA (Prevedouros et al., 2006). Based on production volumes and Surflon® S111 use rate, 19–36 tonnes of Surflon® S111 were consumed for PVDF during this period. The facility had also been using 6:2 FTSA for another PVDF production since 1973. Based on production volumes and use rates, 44–117 tonnes of 6:2 FTSA were consumed from 1973 to 2017.

A fluoroelastomer plant belonging to DAIKIN was established at the same industrial park in 2002. PFOA was used as a processing aid until 2008, when it was replaced by perfluorohexanoic acid (PFHxA). Volume productions and use rates are unknown. Based on the air emission factor published for similar productions, it could be estimated that 11–22 tonnes of PFOA and 4.7–11.5 tonnes of PFNA were released into the air (USEPA, 2006; Wang et al., 2014).

2.2. Dust and surface soil sampling

Nine surface soil samples (S-1 to S-9) and five outdoor dust samples (D-1 to D-5) were collected in May 2022 within 200 m of the industrial park's fence (see Fig. 1). Prevailing wind patterns were taken into account when selecting sample collection sites. After removing grass, the surface soil was sampled using methanol-washed stainless-steel shovels to a depth of roughly 0–3 cm. Dust samples were collected on asphalt surfaces (typically parking spaces and one skate park) with a hand brush, carefully cleaned between each sample. Samples were stored in precleaned high-density polyethylene bottles and kept in the dark at 4 °C until analysis.

2.3. Laboratory analysis

Prior to PFAS analysis, soil and dust samples were air-dried and sieved (1-mm stainless-steel sieve). The extraction method used (detailed in Appendix A) is based on previous methods (Boiteux et al., 2016). The targeted PFASs were analyzed with a Waters Acquity ultra-performance liquid chromatograph in tandem with a Waters Xevo TQ-XS triple-quadrupole mass spectrometer (see Appendix A).

2.4. Quality assurance and quality control (QA/QC)

Appendix A provides information on relative standard deviations of replicates, blanks, spiking recoveries, labeled standards used and method performances.

3. Results and discussion

3.1. PFASs in surface soil samples

Among the 28 PFASs investigated, only ten were quantified in at least one sample (Table S4). Only one sample (S-7) had quantified levels for these ten PFASs. The sum of PFASs ranged from 42 (S-8) to 347 ng/g dw (S-4) (Table S4).

Low concentrations of perfluorooctane sulfonic acid (PFOS) (<0.5–1.8 ng/g dw), perfluoroheptanoic acid (PFHpA) (<0.5–1.2 ng/g dw), and PFOA (<0.5–0.7 ng/g dw) were recorded. There is no known use of PFOS on this industrial site and the concentration range was similar to those commonly measured in soils far from obvious human activity (Brusseau et al., 2020; Rankin et al., 2016; Söregård et al., 2022). To our knowledge, there is no industrial application for PFHpA. It is an end-stage breakdown product of several fluorotelomers that has already been found at similar concentrations in background soils (Rankin et al., 2016; Söregård et al., 2022). As PFOA was intensively used from 1960 to 2008, two hypotheses may explain the low concentrations recorded. Dispersion of PFOA in air up to several kilometers away from fluoropolymer plants has already been demonstrated (Barton et al., 2010; Davis et al., 2007; Galloway et al., 2020; Hangen et al., 2010). Washington et al. (2020) recorded the highest PFOA concentrations in soil 9 and 12 km downwind of a PVDF production facility, so the sampling area may be too close to the industrial park. More likely, once on the surface soil, PFOA can migrate downward with precipitation (Davis et al., 2007).

PFHxA and 6:2 FTSA, currently used in the industrial park, were quantified in several soil samples. Data on 6:2 FTSA concentrations are scarce in background soils. The levels we recorded are similar to those reported by Söregård et al. (2022), but the highest concentrations were recorded around the PVDF production area and in the prevailing wind direction (samples S-3 to S-6). Similarly, the highest PFHxA concentrations were observed near the fluoroelastomer production area where it is used (samples S-7 to S-9). They were above those reported in background soils (Rankin et al., 2016; Söregård et al., 2022).

PFCAs with a chain length between C9 (PFNA) and C13 (PFTrDA) were quantified in all soil samples. PFUnDA and PFTrDA were the

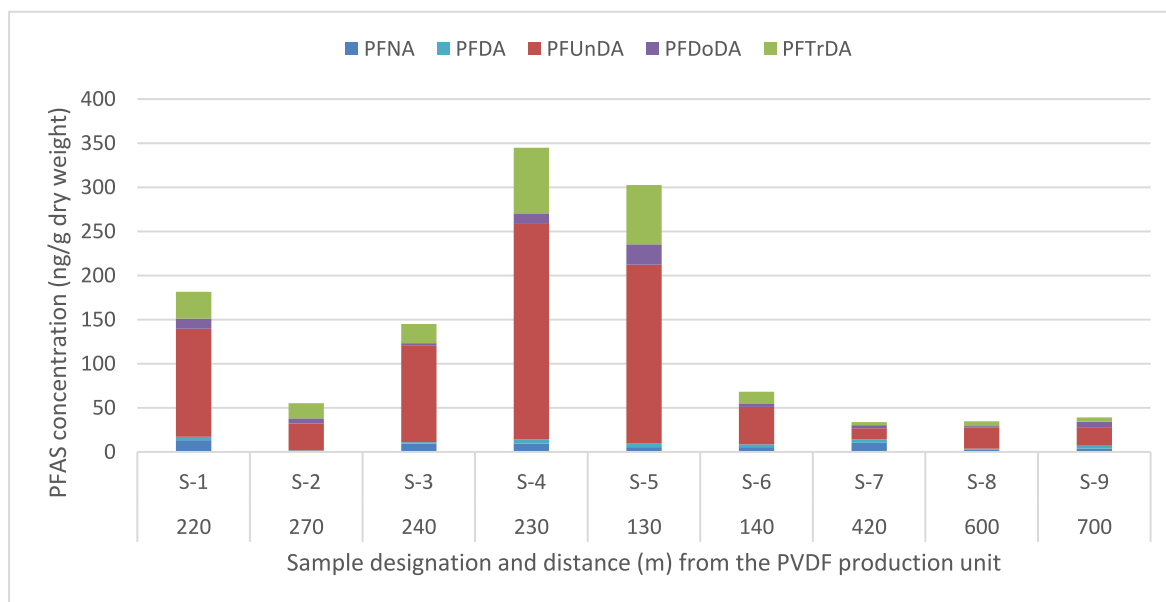


Fig. 2. Concentrations (ng/g dry weight) of PFCAs with a carbon length ≥ 9 (i.e., PFNA, PFDA, PFUnDA, PFDoDA, and PFTrDA) in surface soil samples.

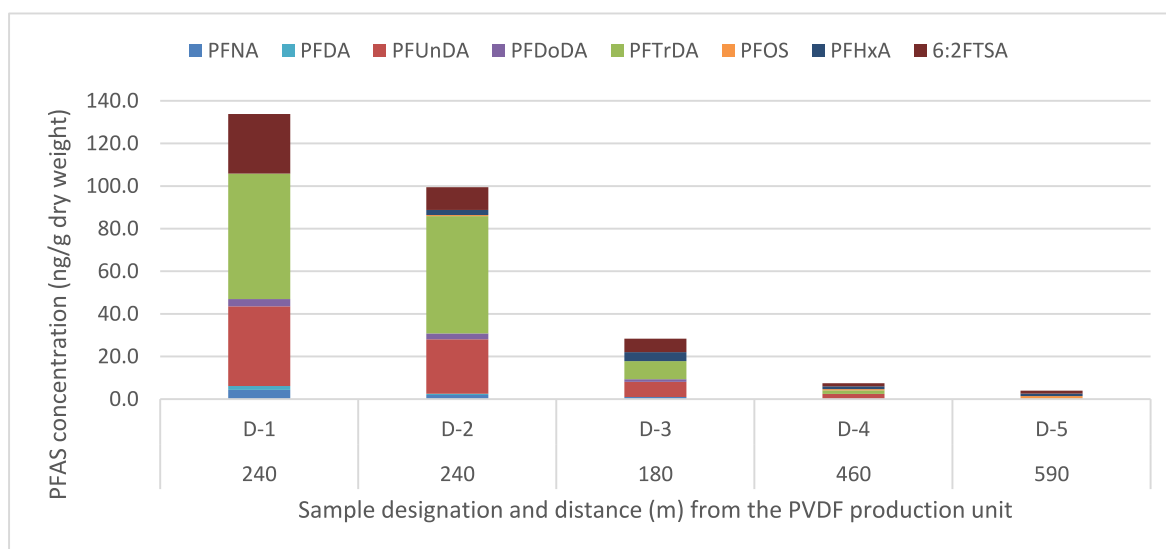


Fig. 3. Concentrations (ng/g dry weight) of quantified PFASs in outdoor settled dust samples.

predominant PFASs in soil with concentrations ranging from 12.4 to 245 and 3.6–74.5 ng/g dw, respectively (Fig. 2). To our knowledge and compared with similar facilities (Washington et al., 2020), these are the highest concentrations ever reported for these PFASs. Soil samples collected closest to the PVDF production area and in the prevailing wind direction (S-5 and S-6) had the highest concentrations. Furthermore, since PFUnDA and PFTrDA are two known major impurities of Surflon® S111, used between 2003 and 2017, it is very likely that soil contamination stems from this usage. The long-chain PFCA profile patterns also showed the systematic presence of perfluorodecanoic acid (PFDA) and perfluorododecanoic (PFDoDA), not known as Surflon® S111 impurities or having industrial applications (Fig. 2). However, similar PFCA profiles have been reported around a PVDF production facility where Surflon® S111 was used from 1985 to 2010 (Washington et al., 2020) (Fig. S2). Surprisingly, concentrations of PFNA—the major ingredient of Surflon® S111—were not the highest recorded. Like for PFOA, we can speculate that the atmospheric deposition range for PFNA was greater than for PFUnDA, PFDoDA, and PFTrDA. Indeed, Washington et al.

(2020) observed an increase in PFNA-PFDA contributions with distance from the PVDF production facility (Fig. S2), and speculated that sorting by mass could explain why several of the largest congeners expected for a new class of PFAS were not detected. Furthermore, PFNA has a much higher mobility than PFUnDA and PFTrDA in soils (Washington et al., 2020), which could also help explain its relatively low concentration in surface soil samples. Kim et al. (2015) have shown that volatility and water solubility of PFCA homologs decrease as their carbon chain length increases, and Chen et al. (2018) suggested that short-chain PFCAs have a longer atmospheric retention time.

3.2. PFASs in outdoor settled dust samples

Among the 28 PFASs investigated, only eight were quantified in at least one sample (Table S4). The dust samples collected downwind of the PVDF production area (D-1 and D-2) had the highest sum of PFASs (134 and 99 ng/g dw, respectively). The predominant PFASs were PFTrDA > PFUnDA > 6:2 FTSA (Fig. 3), a very specific profile never previously

encountered in similar studies near fluorochemical industrial parks (Chen et al., 2018; Su et al., 2016; Wang et al., 2010). Since PFUnDA and PFTrDA are known impurities of Surflon® S111, and 6:2 FTSA is currently used for PVDF production, this unique profile clearly stems from this activity. The total concentration of PFASs was lower than those observed in other studies. Chen et al. (2018) reported total concentrations exceeding 500 ng/g in five out of 12 samples and Su et al. (2016) in five out of 17 samples. In the latter study, the mean total concentration of PFASs 5 km from the facility was 319 ng/g.

3.3. Comparison between soil and outdoor dust samples

Comparing percent distribution of PFCAs with a carbon length ≥ 9 (i. e., PFNA, PFDA, PFUnDA, PFDoDA, and PFTrDA) in surface soil with outdoor settled dust samples (Figs. S3 and S4) showed a clear difference. PFTrDA contributions were significantly higher ($p < 0.01$) in outdoor dust samples, whereas PFUnDA was ($p < 0.05$) in surface soil samples (Fig. S5). Chen et al. (2018) also recorded a difference in PFCA profiles between soil and outdoor dust. Of all the Surflon® S111 ingredients, PFTrDA has the highest hydrophobicity and greatest affinity for the particle phase. It is thus reasonable to hypothesize that once on the surface soil, PFTrDA remains strongly adsorbed on its particles, which are then transported as dust from soil erosion (runoff and wind). It is also likely that PFTrDA is mainly associated with fine particles at facility stack outlets, since the particle-phase proportion increased along with carbon chain length (Chen et al., 2018). PFTrDA would therefore be more prone to dry deposits than PFCAs with a shorter chain length.

4. Conclusion and prospects

To our knowledge, this is the highest soil contamination by long-chain PFCAs ever reported outside the fence line of a fluoropolymer manufacturing site. Not surprisingly, the primary direction of transport for Surflon® S111 ingredients and its replacement (6:2 FTSA) was that of the prevailing wind. One of the ultimate environmental reservoirs for long-chain PFCAs such as PFUnDA and PFTrDA is likely to be soil and dust. Before the phase-out of Surflon® S111 air emissions (2017), the distribution of its ingredients between soil and dust was probably different from today. Indeed, at that time, the atmosphere around the industrial park was likely to be tainted, with PFAS deposition ongoing and constantly added to by both dry and wet depositions, leading to a different balance between Surflon® S111 ingredients in soil and dust.

The present study only provides a first insight into the consequences of these PFAS air emissions. Further research is needed to define the boundaries of the impact zone. Several studies have shown that PFAS concentrations in air, and thus amounts deposited on surface soil, decline with increasing distance from the emission source (Barton et al., 2006; Chen et al., 2018; D'Ambro et al., 2021; Davis et al., 2007; Hangen et al., 2010; Washington et al., 2019). The distance and magnitude of atmospheric transport probably depend on the physicochemical properties of each PFAS. Moreover, these properties affect their fate and behavior once on the ground. The competitive rates of soil deposition and losses via leaching may explain the findings of the present study and modify human exposure over time, since short-chain PFCAs tend to migrate downward with precipitation.

Moreover, other environmental compartments (air, urban runoff water, indoor dust, vegetables, eggs, livestock, and groundwater) should be monitored to better understand PFAS transfer mechanisms between these compartments, along with their momentum. By doing this, the main routes of PFAS exposure for the general population living nearby can be determined.

Credit author statement

Xavier Dauchy: Conceptualization, Resources, Data Curation, Investigation, Writing - Original Draft, Visualization, Writing - Review &

Editing.

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.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.chemosphere.2023.139407>.

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