Perfluoroalkyl acids in dust on residential indoor/outdoor window glass in Chinese cities: occurrence, composition, and toddler exposure

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Abstract
The dust on indoor and outdoor surfaces of the window glasses were collected using sterile cotton balls in 11 cities from China. Two sampling campaigns were conducted with the time interval of 7 days to investigate the accumulation especially during the Spring festival holidays. Twenty-nine perfluoroalkyl acids (PFAA) were quantified to investigate concentration, composition, and toddlers’ exposure. The concentrations of ∑PFAA ranged from no detection (nd) to 43 ng/m² (mean 8.9 ± 10 ng/m²). Perfluorobutanoic acid (PFBA) was detected in 78% samples and accounted for 55 ± 21% of ∑PFAA concentrations. 6:2 fluorotelomer sulfonic acid (6:2 FTSA) and hexafluoropropylene oxide dimer acid (HFPO-DA) were detected in more than 50% samples indicating the use of alternatives. Fluorotelomer carboxylic acid (FTCA) and fluorotelomer unsaturated acid (FTUCA) were found in the dust, implying the degradation of fluorotelomer alcohols (FTOH). The highest concentration of ∑PFAA (43 ng/m²) was found in outdoor dust from Xinzhou, Shanxi Province. Higher ∑PFAA concentrations were found in indoor dust than outdoor in 6 paired samples (3 from Feb. 14 and 3 from Feb. 21). In Tianjin and Handan, the concentrations of ∑PFAA from outdoor surfaces were higher in sampling campaign I (SC I, Feb. 21) than in sampling campaign II (SC II, Feb. 14), implying intensive outdoor release. The exposure of 2-year-old toddlers to PFAA via hand-to-mouth ingestion and dermal absorption was estimated; the mean values of intake were 2.1 and 1.5 pg/kg body weight, respectively, assuming an exposure time of 1 h.

Keywords PFAA · Dust · Window glasses · Toddler exposure

Introduction
Perfluoroalkyl acids (PFAA) are artificial chemicals that possess characteristics of thermal resistance, physical and chemical stability, and repellency of water and oil (Giesy and Kannan 2002; OECD 2018). PFAA have been widely used for more than 60 years in manufacturing industries and commercial products, such as metal plating, fluoropolymer manufacturing, aqueous firefighting foams (AFFF), textile, paper, and water repellent items. Some legacy PFAA, for instance, perfluorooctane sulfonate acid (PFOS) and perfluorooctanoic acid (PFOA), which are named as C₈ compounds, are bioaccumulative and have potential toxicity to biota and humans (Lindstrom et al. 2011). In 2000, the manufacturers began to phase out the production of C₈ analogues (Zhang et al. 2020a). In 2009, PFOS and related compounds were listed in Annex B of the Stockholm Convention on persistent organic pollutants (POPs), and in 2019, PFOA was
added to Annex A (UNEP 2019).Due to the worldwide restriction,short-chained alternatives (C<8) were used, and they have been found in the air, water, sediments, and human body globally (Bao et al. 2017; Guo et al. 2018; Heydebreck et al. 2015). The C4 PFAA, such as perfluorobutanoic acid (PFBA) and perfluorobutane sulfonate acid (PFBS), have been reported low threats to health and high mobility, and they were found in air, surface water, and groundwater in recent studies (Bao et al. 2019; Gao et al. 2020; Yu et al. 2018). Fluorotelomer sulfonate acids (FTSAs), for example, 6:2 FTSA, have been used as substitutes for PFOS in some AFFF and other commercial products, and they have been found in airports and other firefighting training locations (Dauchy et al. 2019; Feng et al. 2020). Hexafluoropropylene oxide dimer acid (HFPO-DA) is an alternative to PFOA in industrial activities, and high concentrations have been found in Chinese fluorochemical industrial parks (Heydebreck et al. 2015, 2016).

Dust is representative for monitoring some POPs, and PFAA have been reported in indoor and outdoor dust all over the world (Besis et al. 2019). Indoor dust in household may reflect the consumption of domestic products, whereas outdoor dust may be influenced by industrial activities (Su et al. 2016). PFOA and PFOS are the predominate compounds reported in many previous studies, with concentrations up to hundreds of nanogram per gram (Fang et al. 2019a; Zheng et al. 2020b). Alternative compounds have been detected in indoor dust at lower concentrations than PFOA and PFOS (de la Torre et al. 2019; Zhang et al. 2020a). Accumulation of dust was different on different surfaces. On the horizontal surface, the accumulation will not stop without interference. While, on the vertical surface, the falling-off and adsorption happen all the time. Therefore, the sampling interval was an important factor to discuss the behavior of PFAA on the vertical surfaces. PFAA in dust are strongly related to the human exposure through ingestion and dermal uptake (Cequier et al. 2014; de la Torre et al. 2019). For children, ingestion of settled dust is comparable to dietary intake, and adverse effects due to the PFAS exposure have been identified (Braun 2017; Egeghy and Lorber 2011; Rappazzo et al. 2017).

Vacuum cleaners (Eriksson and Karlman 2015; Jogsten et al. 2012; Zheng et al. 2020b), air conditioners (Besis et al. 2019), and precleaned brushes (Su et al. 2016; Zhang et al. 2020a) have been used to collect dust settled on floors, carpets, and furniture in households, classrooms, offices, and cars. Wipes such as sterile gauze pad soaked in isopropyl alcohol were used to investigate flame-retardants (FRs), chlorinated paraffins (CPs), and organophosphate esters (OPEs) on household and office surfaces (Hammel et al. 2020; Liu et al. 2018; Watkins et al. 2013; Yuan et al. 2020). Poothong et al. (2020) used wipes to collect poly- and perfluoralkyl substances (PFAS) from hands to evaluate dermal exposure (Poothong et al. 2020). Sterile cotton balls have been used to study polycyclic aromatic hydrocarbons (PAHs) and microencapsulated cyfluthrin (Fleming and Ashley 2013; Stout and Leidy 2000) and could be useful to collect PFAA in dust. Vertical surfaces were studied less than horizontal surfaces. Wipes such as sterile gauze pad soaked in isopropyl alcohol were used to investigate flame-retardants (FRs), chlorinated paraffins (CPs), and organophosphate esters (OPEs) on household and office surfaces (Hammel et al. 2020; Liu et al. 2018; Watkins et al. 2013; Yuan et al. 2020). Poothong et al. (2020) used wipes to collect poly- and perfluoralkyl substances (PFAS) from hands to evaluate dermal exposure (Poothong et al. 2020). Sterile cotton balls have been used to study polycyclic aromatic hydrocarbons (PAHs) and microencapsulated cyfluthrin (Fleming and Ashley 2013; Stout and Leidy 2000) and could be useful to collect PFAA in dust. Vertical surfaces were studied less than horizontal surfaces. Glass was used in structures to allow sunlight inside, reduce weight, or ornament buildings. Dust on window glass could not accumulate in thick layers (Wang et al. 2021). The heterogeneous conditions may be beneficial for the degradation of PFAA precursors, especially due to sunlight conditions (Kutsuna et al. 2006; Sun et al. 2015; Taniyasu et al. 2013).

The aims of this study were to investigate the levels and composition of novel and legacy PFAA in dust on window glass, to reveal the distribution of PFAA in dust in different Chinese cities, and to estimate toddlers’ hand-to-mouth exposure to PFAA.

### Materials and methods

#### Chemicals and standards

Twenty-nine PFAAs were targeted including perfluoroalkyl carboxylic acids (PFCAs) with carbon chain lengths of 4-12, i.e., PFBA, perfluoropentanoic acid (PFPeA), perfluorohexanoic acid (PFHxA), perfluoroheptanoic acid (PFHpA), PFODA, perfluorononanoic acid (PFN), perfluorodecanoic acid (PFDA), perfluoroundecanoic acid (PFUnA), and perfluorododecanoic acid (PFDoDA), perfluoroalkane sulfonic acids (PFSA)s with carbon chain lengths of 4, 6, and 8, i.e., PFBS, perfluorohexanesulfonic acid (PFHxS), and PFOS, 6:2 and 8:2 fluorotelomer unsaturated carboxylic acids (FTUCAs), 3:3, 5:3, 6:2, 8:2, and 7:3 fluorotelomer carboxylic acids (FTCAs), 4:2, 6:2, 8:2, and 10:2 fluorotelomer sulfonic acids (FTSAs), 6:2 and 8:2 chlorinated polyfluorinated ether sulfonic acid (6:2 and 8:2 Cl-PFESA), ammonium 4,8-dioxa-3H-perfluorononanoate (ADONA), HFPO-DA, and perfluoro-4-ethylcyclohexanesulfonate (FECHEs). Thirteen mass labeled compounds were used as internal standards (IS), i.e., $^{13}$C$_4$-PFBA, $^{13}$C$_3$-PFPeA, $^{13}$C$_2$-PFHxA, $^{13}$C$_4$-PFHpA, $^{13}$C$_2$-PFODA, $^{13}$C$_2$-PFUnDA, $^{13}$C$_2$-PFDoA, $^{18}$O$_2$-PFHxS, $^{13}$C$_4$-PFOS, $^{13}$C$_2$-6:2 FDSA, and $^{13}$C$_2$-8:2 FDSA. All the standards were purchased from Wellington Laboratories Inc. (Ontario, Canada). Chromatographic grade methanol and ammonium hydroxide (v/v, 25%) were purchased from Merck company (Darmstadt, Germany). The Envi-Carb cartridge (3 mL, 250 mg) was purchased from Supelco (Bellefonte, USA). Nylon membrane filters (13 mm, 0.22 μm) were purchased from Pall Co. (New York, USA).
Sampling campaign

February 16, 2018 was the Spring Festival of China, and before the festival, all the Chinese families would clean their house as a tradition more than 4000 years. This might be an efficient way to eliminate the potential health threat from indoor dust. We collected the dust on the window glasses inside and outside of 11 flats on February 14 (Sampling Campaign I, SC I) and February 21, 2018 (Sampling Campaign II, SC II). The sampling sites were in cities of China from north to south: Harbin (Heilongjiang Province), Ordos (Inner Mongolia), Tianjin, Xinzhou (Shanxi Province), Handan and Shijiazhuang (Hebei Province), Nanmenxia and Nanyang (Henan Province), Jiujiang (Jiangxi Province), Sanya (Hainan Province), and Hong Kong. In Shijiazhuang and Sanya, the dust on the outside glass surface was not collected for safety. The kitchen and bathroom windows were not considered. The details of the sampling information were presented in Table S2. The medical sterilizing cotton ball (0.5 g) was used to collect the dust on the window glasses. The soft and fluffy cotton balls were more effective to collect and keep the dust than gauze or brush. Methanol or other organic solvent was not used when cleaning the glasses, and bulk film was not collected (Diamond et al. 2000). The areas for cleaning ranged from 4 to 7 m² (for Hong Kong 0.6 m²). Dust in the 2-cm range from the window frame were not collected. The numbers of the balls were fixed in each city. Cotton balls with dust were wrapped in aluminum foil, then sealed in polyethylene bags, and stored at −20 °C before treatment in laboratory.

Sample preparation and instrumental analysis

The extraction method was referred to the treatment of air filters with some modification (Zhao et al. 2020). Cotton balls with dust were extracted in polypropylene tube by ultrasound equipment. Before extraction, 5 ng IS mixture was spiked. About 15 mL 0.1% ammonia methanol was added in the tube and then extracted for 15 min. After being centrifuged at a speed of 3500 r/min for 10 min, the superficial liquid was collected. Cotton balls with dust were extracted twice. All extract was collected and then concentrated to less than 0.5 mL under a gentle stream of high purity nitrogen. Envi-carb cartridges (100 mg, 1 mL) and nylon member filters which were preconditioned by 3 mL methanol were used to remove the impurity. The final volume of each sample was reduced to 200 μL.

The instrument analysis employed Agilent 1290 series liquid chromatograph interfaced with an Agilent 6460 triple quadrupole mass spectrometer (HPLC-MS/MS, Agilent Technologies, USA). The detailed information of instrument analysis could be found in previous studies (Fang et al. 2018; Zhao et al. 2020).

Equations for uptake exposure

According to the questionnaire, toddlers played near the bay or floor-to-ceiling windows to get more sunlight under the safety supervision. The uptakes via dust ingestion and dermal absorption for toddlers were calculated considering their touch on the window glasses. The exposure time (ET) was set as 1 h. The equations were shown below referring previous studies (COPCC 2003; Liu et al. 2018).

Dust ingestion

\[
\text{Dust ingestion} = \frac{C_s \times FTSS1 \times SA1 \times F \times TE \times ET \times 1000}{BW}
\]

Dermal absorption

\[
\text{Dermal absorption} = \frac{C_s \times FTSS2 \times SA2 \times ABS \times ET \times 1000}{BW}
\]

Quality assurance and quality control

To quantify the background of cotton balls, ten balls were picked at random and extracted as real samples. During sampling, one field blank sample was prepared at each site on each day, and the ball number was consistent to the real sample. The blank samples did not contact the window glasses. PFBA (1.17±0.70 ng/m²), PFHpA (7.96±1.43 ng/m²), and PFHxA (9.6±1.77 ng/m²) could be detected in blank samples possibly due to the background of the sampling materials. All the tools, i.e., stainless steel tweezers, which could contact the samples were precleaned by ethanol three times. Glass material was avoided through the entire treatment processes to prevent the adsorption (Sorengard et al. 2020). The signal-to-noise ratio of
3 determines the instrument quantification limit (IQL). The method quantification limit (MQL) was defined using a signal-to-noise ratio of 10 for compounds with concentrations below the IQLs in blank samples. For PFBA, PFHpA, and PFHxA, the MQLs were calculated as 3 times the standard deviation plus the mean value in the blanks. Table S3 lists the IQL and MQL of each compound. The concentrations below the MQLs were expressed as “not detected (nd).” The calibration gradient of the standard curve is 0, 0.5, 1, 2, 5, 10, 20, 50, 100, and 200 μg/L. The linear correlation coefficients of the individual calibration curves were all greater than 0.991. The recoveries of IS ranged from 58% ± 21% (13C₄-PFBA) to 95% ± 7% (13C₄-PFNA). The concentrations were corrected by average levels of blanks.

Statistical analysis

Spearman relationship was tested for individual compound to examine the associations. For the concentrations below the MQL, the MQL/√2 were assigned. Statistical analysis was performed using SPSS statistics 24 (Spss INC IBM USA).

Results and discussion

Concentrations and compositions of PFAA

Twenty-two PFAA were detected in 40 samples of dust from glass, with ΣPFAA concentrations ranging from nd to 43 ng/m² (8.94±10.2 ng/m²). The three most frequently detected compounds were PFDA (45% in indoor and 35% in outdoor samples), PFBA (45% indoor and 33% outdoor), and PFNA (45% indoor and 35% outdoor). The detection frequencies of PFOA (20% indoor and 23% outdoor) and PFOS (5% for both indoor and outdoor) were lower than those of short-chained(C<8) substitutes, i.e., PFBA (45% indoor and 32% outdoor) and PFBS (35% indoor and 28% outdoor). New alternatives, i.e., HFPO-DA and 6:2 FTSA, were detected in 53% (30% indoor and 23% outdoor) and 68% (40% indoor and 28% outdoor) of samples, respectively, levels that were similar to those of C₄ substitutes. PFECHS and ADONA had low detection frequency (8%). PFPNa, PFDS, 4:2 FTSA, 7:3 FTCA, 8:2 FTCA, 6:2 Cl-PFESA, and 8:2 Cl-PFESA were not detected (<MQL) in any samples.

The concentrations and compositions of the PFAA were shown in Table 1 and Figure 1. The predominant compound was PFBA on both indoor and outdoor surface with concentrations ranging from 0.57 to 25 ng/m² (average of 5.8 ng/m² in 78% samples), indicating the replacement of C₈ legacy compounds by short-chained ones. Zheng et al. (2020a) reported that concentrations of PFBA were higher than those of other PFCA in settled dust in the USA, which was consistent with the present study (Zheng et al. 2020b). In China, PFBA was identified as the major compound in PM₁₀ collected from Beijing and Jinzhou, and high concentrations of about 100 pg/m³ accounting for more than 20% of ΣPFAA concentrations were reported in Tianjin and Yantai (Yu et al. 2018). The enrichment of PFBA in dust indicated the replacement of C₈ legacy compounds by short-chained ones. Including the direct sources, PFBA was the product compound of photolysis from other fluorinated compounds, i.e., fluorotelomer alcohols (FTOH) (Taniyasu et al. 2013), N-methyl perfluorobutane sulfonamidoethanol (NMeFBSE) (D’Eon et al. 2006), and PFOA (Zhang et al. 2016). The window glass provides surface for precursors’ adsorption. With the irradiation of the sunlight and the proper temperature, the degradations could result in the accumulation of PFBA, which is a persistent final product. The occurrence of the intermediates FTCAs and FTUCAs in this study was the evidence for degradation from precursors (D’Eon et al. 2006, Ellis et al. 2004). The average concentration of 6:2 FTSA was 1.4 ng/m² in 58% of samples, with the highest concentration at 16 ng/m². 6:2 FTSA has been widely detected in water, sediments, and indoor dust due to its use as a substitute for PFBA (Eriksson and Karrman 2015). Zheng et al. (2020) reported high proportions of 6:2 FTSA and 8:2 FTSA in indoor dust from childcare facilities in the USA, possibly released from carpets, paints, waxes, polishes, and floor cleaners, implying that regular cleaning may increase indoor exposure (Field and Seow 2017; Herzke et al. 2012; Zheng et al. 2020a). Wu et al. (2020) found that 6:2 FTSA was one of the predominate compounds in indoor dust and carpet suggesting its occurrence in textiles (Wu et al. 2020). An alternative to PFOA, HFPO-DA showed similar average concentration (1.6 ng/m²) to that of PFOA (1.1 ng/m²) in 38% samples. PFECHS was detected in 18% of samples (Tables S4-1 and S4-2), with the highest concentrations of up to 9.2 ng/m². PFECH is used as an erosion inhibitor in aircraft hydraulic fluids (Wang et al. 2016), and it is also an impurity in (AFFF) (MacInnis et al. 2017). The low detection frequency was consistent to a previous study (8%) conducted by De Silva et al. (2012) (De Silva et al. 2012). The average concentrations of PFNA in 70% of samples and PFDA in 80% of samples were 1.0 and 0.18 ng/m², respectively. Long-chained PFAA (C > 8) have stronger affinity to particles/dust than do shorter ones, which may explain the high detection frequencies (Higgins and Luthy 2006). Photodegradation may also occur on the window glass. 8:2 FTOH could be photodegraded to PFOA and PFNA (Ellis et al. 2004). Young et al. (2007) investigated the composition of PFAA in Arctic snow and provided some fingerprint ratios to identify the degradation from FTOH, which were the major precursors of PFAA in China (Li et al. 2011; Young et al. 2007). (Ellis et al. 2004) In field samples, the PFOA/PFNA ratio of 1.5 ± 0.8 was found as the reference for degradation in the environment. In this study, four samples presented PFOA/PFNA
ratios in this range. 10:2 FTOH could degrade to PFDA and PFUnDA with a fingerprint ratio of 0.9 ± 0.8 in field samples; eight samples presented such ratios. The composition of PFAA in indoor dust in the present study showed different patterns from previous studies. Jian et al. (2017) reported that PFOA was the predominant

| City                          | Sample label | PFBA (ng/m²) | PFNA (ng/m²) | PFDA (ng/m²) | PFUnDA (ng/m²) | PFBS (ng/m²) | 6:2 FTSA (ng/m²) | HFPO-DA (ng/m²) | ∑PFAA (ng/m²) |
|-------------------------------|--------------|--------------|--------------|--------------|----------------|--------------|-------------------|----------------|--------------|
| Harbin, Heilongjiang          | I-HRB-0214   | 1.9          | 0.16         | 0.14         | 0.02           | 0.01         | 0.16              | 0.50           | 3.2           |
|                               | I-HRB-0221   | 1.0          | 0.16         | 0.17         | 0.04           | 0.02         | 0.19              | nd             | 2.1           |
|                               | O-HRB-0214   | 0.94         | nd           | 0.15         | nd             | nd           | 1.87              | 1.13           | 3.7           |
|                               | O-HRB-0221   | 2.3          | 0.13         | 0.14         | 0.01           | nd           | nd                | 1.11           | 4.3           |
| Ordos, Inner Mongolia Municipality | I-ORDS-0214 | 1.2          | 0.10         | nd           | 0.02           | nd           | nd                | nd             | 1.3           |
|                               | I-ORDS-0221  | 0.85         | 0.12         | nd           | nd             | nd           | 0.26              | 0.47           | 0.47          |
|                               | O-ORDS-0214  | nd           | nd           | nd           | nd             | nd           | nd                | 0.30           | 0.52          |
| Tianjin                       | I-TJ-0214    | 3.8          | 0.75         | 0.11         | nd             | 0.11         | 0.13              | 6.4            |
|                               | I-TJ-0221    | 0.69         | 4.8          | 0.13         | 0.03           | 0.04         | nd                | 7.1            |
|                               | O-TJ-0214    | 2.3          | 0.08         | 0.05         | nd             | 0.05         | 0.10              | 2.7            |
|                               | O-TJ-0221    | 5.8          | 2.0          | 0.05         | 0.02           | 0.07         | 0.10              | 13             |
| XinZhou, Shanxi               | I-XZ-0214    | 3.4          | 0.25         | 0.02         | 0.02           | 0.04         | 0.28              | 4.3            |
|                               | I-XZ-0221    | 3.4          | 0.24         | 0.02         | nd             | 0.33         | 2.9               | 7.7            |
|                               | O-XZ-0214    | 24           | 2.9          | 0.32         | 0.03           | 0.16         | 3.0               | 8.6            |
|                               | O-XZ-0221    | 2.6          | 0.32         | nd           | nd             | 0.04         | 0.24              | 0.33           |
| Handan, Hebei                 | I-HD-0214    | 5.6          | 0.80         | 0.13         | 0.04           | 0.06         | 0.47              | 3.5            |
|                               | I-HD-0221    | 2.3          | 0.15         | 0.05         | 0.02           | 0.19         | 0.73              | 3.4            |
|                               | O-HD-0214    | 1.2          | nd           | 0.02         | nd             | 0.23         | nd                | 1.5            |
|                               | O-HD-0221    | 0.96         | 0.13         | 0.13         | 0.01           | 0.09         | 0.53              | 4.7            |
| Sanmenxia, Henan              | I-SMX-0214   | 25           | 1.9          | 0.27         | 0.01           | 0.13         | 1.4               | 1.4            |
|                               | I-SMX-0221   | 3.4          | 1.1          | 0.23         | 0.02           | 0.04         | 0.40              | 0.52           |
|                               | O-SMX-0214   | 22           | 2.0          | 0.26         | 0.04           | 0.19         | 1.1               | 28             |
|                               | O-SMX-0221   | 3.7          | 0.38         | 0.13         | 0.02           | 0.02         | 0.87              | 5.5            |
| Nanyang, Henan                | I-NY-0214    | nd           | nd           | 0.02         | nd             | 0.65         | nd                | 0.76           |
|                               | I-NY-0221    | nd           | nd           | nd           | nd             | 0.02         | 0.27              | 0.90           |
|                               | O-NY-0214    | nd           | nd           | nd           | nd             | nd           | nd                | 0.12           |
|                               | O-NY-0221    | nd           | 0.11         | 0.02         | nd             | nd           | nd                | 0.12           |
| Jiujiang, Jiangxi             | I-JJ-0214    | 1.8          | 0.16         | 1.06         | nd             | nd           | nd                | 10             |
|                               | I-JJ-0221    | nd           | nd           | 0.53         | nd             | nd           | nd                | 3.4            |
|                               | O-JJ-0214    | 6.0          | 0.80         | 0.69         | nd             | nd           | nd                | 18             |
|                               | O-JJ-0221    | nd           | nd           | 0.29         | nd             | nd           | nd                | 1.5            |
| Hongkong                      | I-HK-0214    | 11           | 1.5          | 0.15         | 0.11           | 1.2          | 1.1               | 16             |
|                               | I-HK-0221    | 8.4          | 1.5          | 0.49         | 0.10           | 0.10         | 0.88              | 0.34           |
|                               | O-HK-0214    | 14           | 6.0          | 1.6          | 0.08           | 0.65         | 2.6               | 1.6            |
|                               | O-HK-0221    | 5.5          | 1.1          | 0.17         | 0.12           | 0.08         | 0.28              | 7.4            |
| Shijiazhuang, Hebei           | I-SIZ-0214   | 0.57         | 0.12         | 0.02         | nd             | nd           | nd                | 1.0            |
|                               | I-SIZ-0221   | nd           | nd           | nd           | nd             | nd           | nd                | 0.28           |
| Sanya, Hainan                 | I-SY-0214    | 2.5          | 0.10         | 0.05         | 0.09           | nd           | nd                | 3.3            |
|                               | I-SY-0221    | 4.9          | 0.42         | 0.09         | 0.04           | 0.01         | 0.29              | 1.1            |

∑PFAA: The sigma concentrations of 22 PFAA which could be detected in this study

nd: not detected
compound in most European countries (Jian et al. 2017). This pattern was consistent with their precursor attribution in indoor air, i.e., FTOHs and FOSA/Es were at high levels. Similarly, in Norway, PFOA was the main compound in home dust to which humans were exposed (Poothong et al. 2020). This phenomenon can also be found in home dust in Belgium, Italy, and Spain (Fang et al. 2019b). Conversely, in Irish households, PFBS had a higher detection frequency (81%) and average concentration (17 ng/g) than other compounds (Harrad et al. 2019). In China, near a mega fluorochemical industrial park, the percentages of PFOA for $\sum_{12}$PFAA were more than 60% in 2014. In another study monitoring the dust in Chinese households between 2015 and 2017, PFHxS concentrations were 4 to 40 times higher than those of other PFAA, which differed from both the fluorochemical industrial park and European countries, possibly due to the application of its precursor perfluorohexanesulfonyle fluoride (PFHxSF)(Ao et al. 2019). PFBA and Cl-6:2 PFESA were detected in dust from student dormitories and communal flats in Guangzhou China, but the contribution was lower than that of PFBS, which was the major compound (Zhang et al. 2020a).

For outdoor environments, studies have usually focused on the particles in air, and the concentrations and composition have reportedly been influenced by many factors, e.g., location, relative humidity, and daily precipitation (Fang et al. 2019b). In the Canadian Arctic, from 2006 to 2015, the most frequently detected compound was PFBA, and the concentrations were higher than those of C$_8$ homologues, which was consistent to this study (Wong et al. 2018). Urban studies presented spatial differences in previous studies. In Beijing, China, PFBA and PFOA were found at comparable levels in the particle phase in Nov. 2013 (Wu et al. 2019). In Chengdu, China, PFOA contributed more than 80% of the $\sum_{12}$PFAA concentrations indicating that legacy compounds were still the main contaminants in air particles collected from 2016 to 2017 (Fang et al. 2019b). Spearman’s rank correlation coefficients between PFBA and 6:2 FTSA ($r = 0.807$, $p < 0.01$) and between PFBA and HFPO-DA ($r = 0.498$, $p < 0.05$) were positive and significant, suggesting the common sources of these alternatives for PFOA and PFOS. PFDA and PFNA were significantly related ($r = 0.689$, $p < 0.01$). All were long-chained (C > 8) PFAA which have stronger affinities to dust than short-chained compounds.

**Spatial comparison**

The distribution of $\sum$PFAA was shown in Figure 2. Relatively high levels of $\sum$PFAA were detected SC I. The highest concentration of $\sum$PFAA was detected in Xinzhou on the outdoor surface (43 ng/m$^2$, O-XZ-0214). Before SC I, the dust on the window glass had accumulated for 1 year, which was one reason for high concentration. Xinzhou is a city of Shanxi Province. Air pollutions have been investigated in this province, and the dust was harmful with high heavy metal contents (Han et al. 2020; Su et al. 2020; Zhang et al. 2020b). PFDA concentration in Xinzhou was not reported in previous studies. The occurrence and high concentration in dust suggested that PFDA contamination in other environment medias may exist, and more studies were needed in the future. In Ordos, another city famous for textile manufacturing, the outdoor $\sum$PFAA
concentration was also at a high level of 28 ng/m² (O-ORDS-0214). In Hong Kong, \( \sum \) PFAA concentration of one sample collected on the outdoor surface in SC I was 29 ng/m² (O-HK-0214), higher than three other samples from the same city, and last cleaning of the window was done 18 months ago. No intensive fluorine-related industry distributed in this city. In previous studies, PFAA were reported in water, sediments, and biotas around the airports suggesting the release from aviation-related activities (Awad et al. 2011; Lie et al. 2014).

In Sanmenxia, \( \sum \) PFAA concentrations were comparably high at 33 ng/m² (I-SMX-0214) indoors and 28 ng/m² (O-SMX-0214) outdoors. PFAA transport from the air to the earth surface via atmospheric deposition (Galloway et al. 2020). Considering one important drinking water source located in this city, PFAA in the dust should be concerned since the concentration in the drinking water source could be elevate due to the atmospheric deposition. Samples in Nanyang and Shijiazhuang presented low concentrations (nd—1.0 ng/m²). Window cleaning had been conducted in both cities 7–10 days before SC I.

**Indoor/outdoor comparison**

The indoor and outdoor concentrations of \( \sum \) PFAA were shown in Figure 3a. In SC I, samples from 4 cities, i.e., Ordos, Xinzhou, Jiujiang, Hongkong, showed higher concentrations of \( \sum \) PFAA on outdoor surfaces. In SC II, the concentrations in the same four cities were higher for indoor surfaces. During the Spring Festival, industrial activities slowed down, and indoor exposure from domestic sources was the major threat to human health. In Sanmenxia, samples collected on the same days presented comparable concentrations on both indoor and outdoor glass surfaces. Exchange of air between indoors and outdoors was weak in winter due to the low temperatures resulting in windows remaining closed. In Nanyang, indoor \( \sum \) PFAA concentrations were at the same levels, although the samples were collected on different days, and concentrations were all slightly higher than that in outdoor samples. This indicated that the exposure risk in this city was mainly from indoor sources. In Tianjin and Handan, indoor concentrations of \( \sum \) PFAA were higher outdoor in SC I, whereas the opposite was found in SC II.

For PFBA, the average concentration in SC I was 6.7 ng/m² on indoor glass surfaces, which was lower than that outdoor (10 ng/m²) on the same day (8 cities, Shijiazhuang and Sanya were excluded due to the lack of outdoor samples). Higher concentrations were found on indoor surfaces in 5 cities, but not in Xinzhou, Jiujiang, and Hong Kong indicating intensive outdoor release in these three cities, probably from industries and airports (Fig. 3b). In SC II, the average concentrations on indoor and outdoor surfaces were 2.9 and 3.5 ng/m², respectively. In Tianjin, concentration (5.8 ng/m²) outdoors was higher than indoors (0.7 ng/m²). For 6:2 FTSA, higher concentrations were detected on outdoor surfaces in Ordos (indoors: nd, outdoors: 16 ng/m²) and Xinzhou (indoors: 0.28 ng/m², outdoors: 2.9 ng/m²) in SC I. Similarly, PFNA concentrations were higher outdoors in Xinzhou (indoors: 0.25 ng/m², outdoors: 2.9 ng/m²) and Hongkong (indoors: 1.5 ng/m², outdoors: 6.0 ng/m²) in SC I. For other long-chained PFAA, i.e., PFDA, PFUnDA, and PFDoDA, no differences between indoor and outdoor samples were found.

**Comparison of the two sampling campaigns**

The comparison between the SC I and SC II was shown in Figure S1. Seven cities (Harbin, Ordos, Tianjin, Shijiazhuang, Nanyang, Hong Kong, and Sanya) had comparable concentrations of \( \sum \) PFAA on indoor surfaces in SC I and SC II. The concentrations of \( \sum \) PFAA were 1.3 ng/m² (I-ORDS-0214) and 1.3 ng/m² (I-ORDS-0221) for Ordos and 0.77 ng/m² (I-NY-0214) and 0.90 ng/m² (I-NY-0214) for Nanyang. The last cleaning was 7 days before SC I, but in additional 7 days of accumulation, concentrations did not change much in SC II. The concentrations of \( \sum \) PFAA were 16 ng/m² (I-HK-0214) and 12 ng/m² (I-HK-0221) for Hong Kong and 6.2 ng/m² (I-SY-0214) and 7.0 ng/m² (I-SY-0221) for Sanya, where the last cleaning before the SC I had been more than 1 year prior. In Harbin, Tianjin, and Shijiazhuang, where the last cleaning before SC I had happened more than 7 days but less than 1 year prior, the concentrations of \( \sum \) PFAA did not change significantly (< 25%) after 7 days of accumulation (i.e., from SC I to SC II). This implied that at least 7 days for equilibrium on horizontal glass in 7 cities mentioned above. Lower
concentrations of $\sum$PFAA were found in SC II than SC I in the other 4 cities, i.e., Xinzhou (I-XZ-0214:11 ng/m$^2$, I-XZ-0221:7.7 ng/m$^2$), Handan (I-HD-0214:15 ng/m$^2$, I-HD-0221:3.4 ng/m$^2$), Sanmenxia (I-SMX-0214:33 ng/m$^2$, I-SMX-0221:6.2 ng/m$^2$), and Jiujiang (I-JJ-0214:10 ng/m$^2$, I-JJ-0221:3.4 ng/m$^2$), suggesting a longer equilibrium time than 7 days. For outdoor surfaces, concentrations were higher in SC II than in SC I in Tianjin (O-TJ-0214:2.7 ng/m$^2$, O-TJ-0221:13 ng/m$^2$) and Handan (O-HD-0214:1.5 ng/m$^2$, O-HD-0221:4.7 ng/m$^2$), suggesting intensive outdoor sources. In Harbin and Nanyang, comparable concentrations were found in SC I and SC II, but the increasing trend was not negligible (O-HRB-0214:3.7 ng/m$^2$, O-HRB-0221:4.3 ng/m$^2$; O-NY-0214: nd, O-NY-0221:0.12 ng/m$^2$). In other 5 cities, i.e., Ordos, Xinzhou, Sanmenxia, Jiujiang, and Hong Kong, concentrations were lower in SC II than SC I (see Figure S1).

Indoor surfaces might be influenced by human activities which could result in particles falling off, while outdoor surfaces might be highly influenced by wet deposition (Wu et al. 2008). Unlike other semi-volatile organic compounds, PFAA are hydrophilic and mainly distributed in particle phase (Fang et al. 2018). PFAA could adsorb on glass materials, which is a unique behavior. Moreover, when degradation occurs on glass surfaces, concentrations of the compounds were not related to the content of organic carbon (Li et al. 2010). Future studies should be conducted to elucidate the behaviors of PFAA on the window glass surfaces.

**Toddlers’ uptake estimations**

The uptake of PFAA via hand-to-mouth dust ingestion and dermal absorption were estimated by methods presented in previous studies (COPCC 2003; Liu et al. 2018; Poothong et al. 2019). Adults touched window glass occasionally, and the daily exposure time was much less than 1 h. Additionally, adults washed their hands frequently and seldom lick their fingers. Children could be attracted by the view outside when they are playing near windows and thus touch the glass, and the finger licking always happens. In this study, an average body weight of 12 kg for 2-year-old Chinese toddlers was applied when calculating intake. The exposure time was set as 1 h/day according to the questionnaire. Older children may...
also touch windows, but the exposure values likely decrease due to decreasing exposure time and increasing body weight. The summary statistics of intakes of individual PFNA for which detection frequencies were higher than 50% and that of ∑PFNA were shown in Table 2. The average estimated ingestion of ∑PFNA (2.1 pg/kg body weight [bw]) via dust on window glass was higher than that for dermal absorption (both hands, 1.5 pg/kg bw). Ingestion intakes and dermal absorption of PFBA were higher than for other compounds. Estimated mean exposure values of 6:2 FTSA and HFPO-DA were all lower than 0.5 pg/kg bw. No safety thresholds were available for PFBA, 6:2 FTSA and HFPO-DA for toddlers, and more studies should be conducted in the future. Poothong et al. (2019) calculated the exposure of adults to PFAS through hand-to-mouth contact and dermal absorption by measuring PFAS collected directly from hands using hand wipes (Poothong et al. 2019). PFOA and PFHpS were the two major compounds, with mean exposure values 2.0 and 6.2 pg/kg dw d, respectively, for hand-to-mouth contact, and 1.2 and 3.7 pg/kg dw d, respectively, for dermal absorption, which differed from this study. When adults touch glass, the exposure to alternative compounds should be considered. The criteria developed by the European Food Safety Authority (EFSA) in 2008 was used to assess PFOA (1.5 μg/kg bw d) and PFOS (150 ng/kg bw d) daily exposure (EFSA 2008). Assuming the daily exposure time was 1 h per day, the highest PFOA and PFOS intakes were 0.26 ng/kg bw d and 0.82 ng/kg bw d, values that are much lower than the risk criteria. For toddlers, the PFNA exposure scenarios might differ from those of adults due to their daily activity, living habits, and low body weight. Moreover, besides windows, the other horizontal surfaces in the houses or flats should be considered, especially for toddlers between 1 and 2 years old, since they would likely touch these surfaces when learning to walk.

Table 2 The estimated intake (pg/kg body weight) through hand-to-mouth dust ingestion and dermal absorption.

|       | PFBA | PFNA | PFDA | PFUnDA | PFBS | 6:2 FTSA | HFPO-DA | ∑PFNA |
|-------|------|------|------|--------|------|----------|---------|-------|
| Ingestion intake |      |      |      |        |      |          |         |       |
| Max   | 7.3  | 1.4  | 0.46 | 0.03   | 0.35 | 0.43     | 1.3     | 9.7   |
| Min   | 0.17 | 0.03 | 0.005| 0.004  | 0.003| 0.04     | 0.08    | 0.08  |
| Mean  | 1.4  | 0.26 | 0.07 | 0.01   | 0.04 | 0.14     | 0.48    | 2.1   |
| Median| 0.87 | 0.10 | 0.04 | 0.01   | 0.01 | 0.09     | 0.28    | 1.9   |
| Dermal absorption |      |      |      |        |      |          |         |       |
| Max   | 5.1  | 1.0  | 0.32 | 0.02   | 0.25 | 0.30     | 0.88    | 6.76  |
| Min   | 0.12 | 0.02 | 0.003| 0.003  | 0.002| 0.03     | 0.06    | 0.06  |
| Mean  | 0.94 | 0.18 | 0.05 | 0.01   | 0.03 | 0.10     | 0.33    | 1.5   |
| Median| 0.61 | 0.07 | 0.03 | 0.01   | 0.01 | 0.06     | 0.19    | 1.3   |

∑PFNA: The intakes of ∑PFNA were calculated using the ∑PFNA concentrations in Table 1 which included 22 compounds.

Implications and limitations

The dust on window glass has not been investigated frequently in previous studies. The conditions are unique due to the sunlight, heterogeneous medium, and glass surface, which could influence the degradation of precursors and partitioning between gas and particle phases. The dust could be disturbed when opening and closing windows, thereby increasing the exposure risk for humans. Glass walls are widely used in apartments, offices, schools, hospitals, and laboratories to provide more sunlight and save energy. Ingestion and dermal intake should be concerned due to the fine particle size of dust and compounds found in dust and on glass. To avoid possible misevaluation, vertical surfaces in indoor and outdoor environments should not be ignored when collecting dust. In this study, sterile cotton balls were employed. They are easy to acquire and use and can be applied for monitoring.

There are several limitations in this study. Hotspot cities and background regions were not included, and the sampling intensity was low to provide a comprehensive understanding of distribution and human exposure. The detection limits were high for PFHpA and PFOA, which affected the discussion of sources. The cotton balls were not soaked in isopropyl alcohol or any other solvent; thus, PFNA absorbed on the glass may not be collected, which results in partial loss of the bulk film.

Conclusion

Twenty-two PFNA were detected in dust collected from window glass from 11 Chinese cities. Three alternatives, PFBA, 6:2 FTSA, and HFPO-DA, were found in more than 50% samples suggesting the use of novel PFNA. PFBA was the predominant compound, indicating the shift to short-chained compound and its precursors. The two sampling campaigns were conducted before and after Chinese Spring Festival, in
winter, when most people were off work and stayed at home, which may increase exposure time. Higher concentrations of PFAA were found on outdoor surfaces in 5 paired samples. In Tianjin and Handan, higher concentrations were found in outdoor dust after a further 7 days of accumulation (Feb. 14 to 21). Exposure via touching window glass touching may not be important for adults, but it should be considered for toddlers in view of their unique behaviors. Moreover, PFAA in dust from horizontal surfaces and impervious urban surfaces should be considered in future studies.

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Author contribution ZZ: conceptualization, methodology, software, writing—review & editing, data curation; LY: data curation, investigation; HQ: writing—original draft preparation, data curation; YL: data curation, investigation; XC: data curation, writing—reviewing and editing; XH: visualization, investigation, data curation; TL: resources, funding acquisition, writing—reviewing and editing, project administration; QL: conceptualization, investigation, writing—reviewing and editing, supervision; HS: resources, project administration.

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Data availability All data and materials were provided in supplementary material and open to other researchers.

Declarations

Ethics approval The samples collected in this study were dust in households. No human or animal tissue, organs, or cells were involved in the experiment. The sampling campaign used cotton balls which was not harmful to human subjects, and the sampling processes did not use harmful solvent. The ethical committee approved the sampling campaign and the experiments.

Consent to participate The households in whose flats the samples were collected all agree to participate in this study.

Consent for publication The participants and all the authors all agreed to publish the results in a public journal.

Competing interests The authors declare no competing interests.

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