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A novel pretreatment method for fluorinated alternatives in soil and sediment using accelerated solvent extraction



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Due to the widespread industrial use and presumed lower toxicity, fluorinated alternatives are increasingly detected in soils and sediments. Nevertheless, their detection remains challenging due to structural diversity, wide variations in physical and chemical properties, and significantly complex matrix effects. A sensitive and cost-effective approach for quantifying fluorinated alternatives in soil and sediment were developed using accelerated solvent extraction combined with ultra-performance liquid chromatography-tandem mass spectrometry. This method showed high sensitivity (LOD/LOQ: 0.14–0.80 ng/g) with satisfactory accuracy (recoveries of 85.4–95.5%) and precision (RSD < 10%), demonstrating strong reliability for environmental analysis. Notably, the optimized pre-treatment approach cost only 23 min per sample reducing processing time by at least two hours compared with the oscillatory or ultrasonic extraction method. Both 6:2 chlorinated polyfluoroalkyl ether sulfonate (6:2 Cl-PFAES) and 6:2 fluorotelomer sulfonate (6:2 FTS) were detected in 37 soil and 23 sediment samples, with concentrations ranging from 0.24 to 749 ng/g.

Fluorinated alternatives are widely used in industrial and consumer applications, with global usage continuing to grow year on year^{1–4}. According to the China Fluorosilicone Organic Materials Industry Association, the annual production of domestic perfluorinated compounds—including both traditional PFASs and certain fluorinated alternatives—is estimated to reach approximately 50,000 to 80,000 tons in 2022. However, initially marketed as more toxicologically favorable than legacy species, accumulating evidence reveals these compounds exhibit environmental and toxicological concerns including potential hepatotoxicity, reproductive toxicity, neurotoxicity and endocrine disrupting effects^{5–9}. Due to their strong sorption affinity to organic matter and mineral surfaces, fluorinated alternatives tend to accumulate in soil and sediment, which act as long-term secondary sources through leaching or bioaccumulation. Therefore, spatially resolved concentration data are essential to model their environmental fate and exposure pathways.

Current pre-treatment techniques for detecting these substances are relatively complex and laborious. In published studies, analytical methods have predominantly employed oscillatory extraction or solvent extraction in

soil and sediment, typically coupled with the Solid Phase Extraction (SPE)^{10–14}. Previous studies research predominantly employed solid-phase extraction (SPE), techniques, including octadecyl-functionalized silica (C₁₈), weak anion exchange (WAX), hydrophilic-lipophilic-balanced (HLB), graphitized non-porous carbon (ENVI-Carb), and primary and secondary amine-functionalized silica (PSA)¹⁵. Nonetheless, these SPE methods were associated with labor-hours cost, particularly when applied to the analysis of environmental samples. For example, the study by Sepulvado et al. involved three extraction cycles, followed by LC-MS/MS analysis¹⁴. Similarly, a patent by Tang et al. describes a segmented multiple extraction process for PFASs in soil and sediment¹⁶, involving oscillation, static standing, ultrasonic extraction in a water bath, and centrifugation to collect the supernatant, which is then transferred to a collection container. The residual solids are retained in the centrifuge tube, with the extraction repeated 1–3 times through additional extractant application and pH adjustment. Such procedures are somewhat cumbersome. Therefore, developing a rapid, straightforward, sensitive, cost-effective, and efficient pre-treatment method is highly urgent for routine detection.

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Fig. 1 | Recoveries of four fluorinated alternatives (6:2 Cl-PFAES, 6:2 FTS, OBS and Et-PFOA) with five extractants (MeOH, ACN, MTBE, mixtures of MeOH:MTBE (1:1) or ACN:MTBE (1:1)) in extraction procedures. The horizontal axis represents four fluorinated alternatives in different five extractants. The vertical coordinate determines the Average Recovery at five different extraction solvents. Black color indicates extraction with methanol solvent, dark blue indicates extraction with acetonitrile solvent, yellow color indicates solvent extraction with methyl tert-butyl ether, light blue color indicates solvent extraction with methanol and methyl tert-butyl ether 1:1 volume ratio, light orange color indicates solvent extraction with acetonitrile and methyl tert-butyl ether 1:1 by volume.

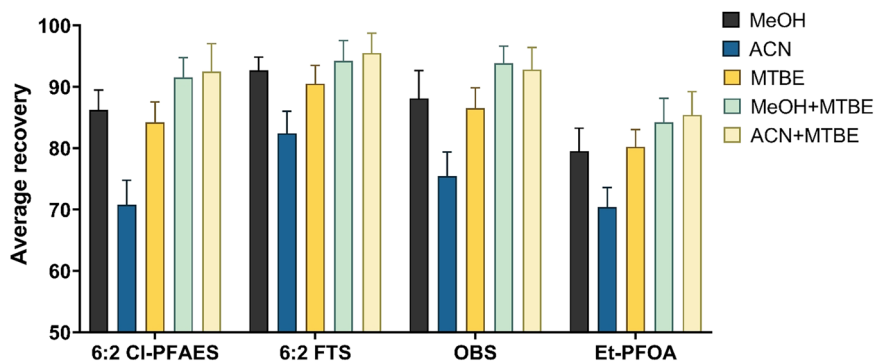
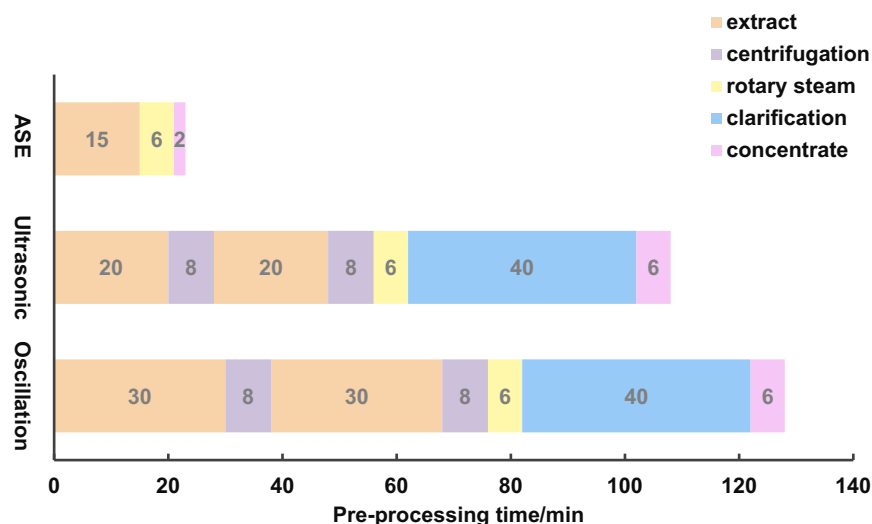


Fig. 2 | Comparison of processing time among three pretreatment methods (ASE, Ultrasonic, Oscillation). The horizontal axis represents three pre-processing time under various processing steps of fluorinated alternatives. The ordinate compares the extraction methods in terms of their consumption time.



Accelerated solvent extraction (ASE) has proven to be a reliable and efficient extraction method and is widely utilized across various media for the extraction of target compounds^{17–19}. Compared to ultrasonic and oscillation extraction methods, ASE offers advantages in terms of time savings, reduced solvent usage and enhanced environmental sustainability. It also demonstrates superiorities of rapid pre-treatment efficiency, a high degree of automation, consistent extraction performance, and reduced processing time. However, this pre-treatment method still presents inherent limitations, a critical challenge originates from the high polarity of methanol and acetonitrile solvents, which indiscriminately extract both target analytes and impurities together. This non-selective extraction solution needs to be cleaned up, which consumes a lot of pre-treatment time.

In this study, we developed a modified accelerated solvent extraction for detecting four fluorinated alternatives with high detection rates in soil and sediment. Through optimization of extraction solvent, the improved selectivity significantly and eliminates the requirement for additional purification steps, enabling direct transition from extraction to instrumental analysis following membrane filtration, greatly streamlining the pre-treatment process and saving time. To validate field applicability, we implemented this method in analyzing 37 soil samples and 23 sediment samples from Zhejiang, China. These findings are crucial for supporting the transition to safer and more sustainable fluorinated alternatives.

Results and discussion

Optimization of the extraction and UHPLC-MS/MS

Terrestrial environments constitute the primary context for human production and daily activities, with soil ecosystems often serving as significant

receptors for anthropogenic pollutants²⁰. To optimize extraction efficiency, MeOH, ACN, MTBE, and mixtures of MeOH:MTBE (1:1) or ACN:MTBE (1:1) were selected as the extractants. Previous studies predominantly utilized MeOH and ACN as extractants^{21–25}. This study, however, incorporated MTBE and its mixtures with MeOH or ACN will reduce the extraction of impurities. The recovery rates of each fluorinated alternative by using these extractants were illustrated in Fig. 1. Notably, ACN exhibited the lowest recovery rates (70.4%–82.4%), whereas ACN:MTBE (1:1) demonstrated the highest recovery rates (85.4%–95.5%). Although MeOH has been commonly employed as an extractant in numerous studies^{14,22,23}, this study observed recovery rates of 79.5%–92.7% for MeOH. Consequently, ACN:MTBE (1:1) emerged as the most effective extractant for the extraction of fluorinated alternatives from soils.

The aforementioned methods have been hindered by time-consuming pre-treatment processes, extended extraction times, and the high cost associated with SPE. In contrast, accelerated solvent extraction (ASE) utilized glass fiber filter membranes and purified siliceous earth, offering a more cost-effective alternative. As illustrated in Fig. 2, ASE shortened pre-treatment time by a minimum of two hours compared to both ultrasonic or oscillatory extraction approaches. Additionally, fully automated sampling capabilities of ASE significantly enhanced sample handling efficiency. Fig. 3 shows that ASE provides superior recovery ranged from 79.5 to 92.7% compared to other methods, likely due to its enhanced extraction efficiency under high temperature and pressure conditions. Consequently, ASE emerged as a more economical, rapid, and effective approach.

The ESI-negative mode was employed, with the deprotonated molecular ion ($M + H$)[−] selected as the precursor ion for each fluorinated

alternative. Table 1 details the optimized UHPLC–MS/MS acquisition parameters, along with the LOD and LOQ. Following the optimization of parameters, the most abundant product ions were chosen for quantification via MRM. Fig. 4 illustrates the MRM chromatogram of four fluorinated alternatives under optimized conditions. The results demonstrated smooth peak shapes with short retention times (RT) and enhanced separation for each fluorinated alternative. The RT ranged from 8.527 to 9.988 min, with a total run time under 12 min. Although GC/MS has been recognized for its superior separation technique and reduced instrument-related pollution for PFAS analysis¹⁵, UPLC–MS/MS is more commonly used than GC/MS for the analysis of PFASs^{15,26}.

Method linearity and sensitivity

The standard curves for four fluorinated alternatives, with concentrations of 2, 5, 10, 20, 50, and 100 ng/mL, are presented in Supplementary Table 1. The correlation coefficients for both the matrix-matched and blank solvent standard curves exceeded 0.9990, demonstrating a strong linear relationship.

The LOD and LOQ for these fluorinated alternatives ranged from 0.14 to 0.20 ng/g and 0.56 to 0.80 ng/g, respectively, as shown in Table 1. Powley et al. (2005) reported that the LOQ for PFCAs was 1.0 ng/g in soil, sediment, and sludge samples¹⁴. In Semerád et al. (2020) study, the LOD were between 0.01 and 0.25 ng/g, the LOQ were 0.1–9.6 ng/g for PFASs and 0.3–0.7 ng/g for fluorinated alternatives in sludge samples²⁷. Overall, the present study exhibits high sensitivity and is suitable for analyzing the concentrations of fluorinated alternatives in soil and sediment matrices.

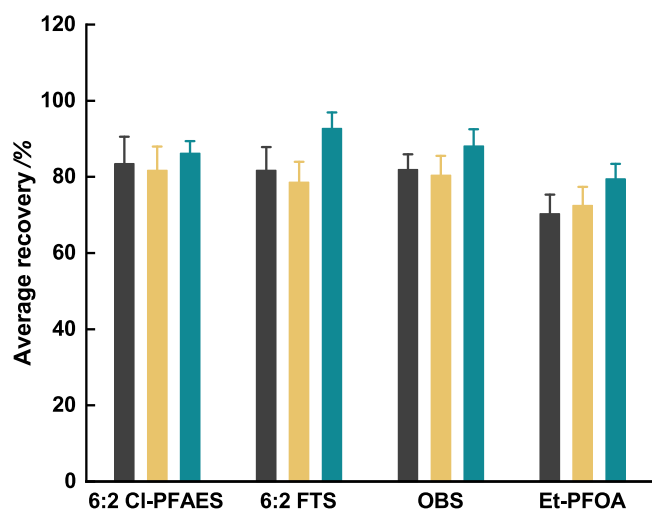


Fig. 3 | Comparison results of extraction effects for different sample pretreatment methods in four fluorinated alternatives. The horizontal axis represents three extraction methods in four fluorinated alternatives, the vertical coordinate expresses the degree of goodness of the Average Recovery Rate. Black color indicates ultrasonic extraction, orange indicates oscillation extraction, blue indicates ASE extraction.

Specificity and matrix effect

Validation analyses were conducted using purified siliceous earth blank samples, which contained no detectable trace of fluorinated alternatives ($n = 3$). The results demonstrated the absence of blank matrix peaks in the chromatograms, and confirming the method's applicability to the four fluorinated alternatives. The matrix effects (ME) for four fluorinated alternatives were presented in Supplementary Table 1, with values ranging from -26.29% to $+9.47\%$. These findings indicated that 6:2 FTS was exhibited ionic amplification ($+9.47\%$), while the other alternatives experienced ion suppression (ranging from -26.29% to -16.23%).

Method precision and accuracy

To verify the repeatability of the instrument and method, quantification was conducted over three consecutive days utilizing matrix-matched standards and spiked matrices, with each quantification was repeated three times per day. The inter- and intra-day precision of the four fluorinated alternatives in the matrix-matched standards and spiked matrices is detailed in Table 2. The RSD for method repeatability ranged from 3.5% to 8.2% for both inter- and intra-day analyses. Additionally, the RSD for instrument repeatability ranged from 4.8% to 10% for inter- and intra-day measurements. These results indicate that the study demonstrates satisfactory repeatability of both the instrument and the method.

Analysis of soil and sediment samples

The method was used to analyze the concentration of fluorinated alternatives in 37 soil and 23 sediment samples in Zhejiang. The summary statistics of fluorinated alternatives levels in soil and sediment samples were given in Table 3. Et-PFOA was not detected in any samples, 6:2 Cl-PFAES and 6:2 FTS had the 100% detection rate (DR), followed by OBS (35%). Also, the concentration of 6:2 Cl-PFAES were ranged from 0.17 to 749 ng/g. The minimum concentration of 6:2 FTS and OBS were 0.24, 1.14 ng/g, as well as the maximum concentration were 98.8, 26.4 ng/g. The average concentration of 6:2 Cl-PFAES, 6:2 FTS and OBS were 24.3 ± 121 , 5.14 ± 16.3 , 8.87 ± 8.34 ng/g. The concentration of fluorinated alternatives in soil and sediment samples were individually given in Supplementary Table 2 and Supplementary Table 3. In soil samples, only Et-PFOA was not detected, others substances remained in the soil. Moreover, only 6:2 Cl-PFAES were detected in sediment samples, others were all below the LOD.

A previous study in Changshu, China, the concentration of total fluorinated alternatives in soil samples were $1.10\text{--}43.0$ ng/g²⁸. And in indoor dust from office environment in south China, PFASs were detected with high concentration ($51.6\text{--}219$ ng/g), fluorinated alternatives were comprising 34.2% of the total PFASs concentrations²⁹. In addition, fluorinated alternatives and legacy PFASs had the same source. In north China, agricultural soils were affected by fluorinated alternatives and legacy PFASs, 6:2 fluorotelomer sulfonic acid and OBS were main alternatives of PFOS³⁰. Therefore, fluorinated alternatives may gradually replace traditional PFASs in China. In this study, we found that the concentrations in soil and sediment were higher than in other area. Therefore, we should pay more attention to PFASs in Zhejiang, China.

Table 1 | The optimized UPLC–MS/MS acquisition parameters, LOD and LOQ of the method for fluorinated alternatives

Fluorinated alternatives	Precursor ions (m/z)	Productions (m/z)	Collision energy (v)	Retention times (min)	LOD (ng/g)	LOQ (ng/g)
6:2 Cl-PFAES	531	351	27	9.278	0.16	0.64
6:2 FTS	427	407	23	8.527	0.20	0.80
OBS	603	172	41	9.713	0.16	0.64
Et-PFOA	413	369	10	8.567	0.14	0.56
¹³ C ₄ -PFOA	417	372	51	/	/	/
¹³ C ₄ -PFOS	503	80	27	/	/	/
¹³ C ₂ -PFOA	415	370	10	/	/	/

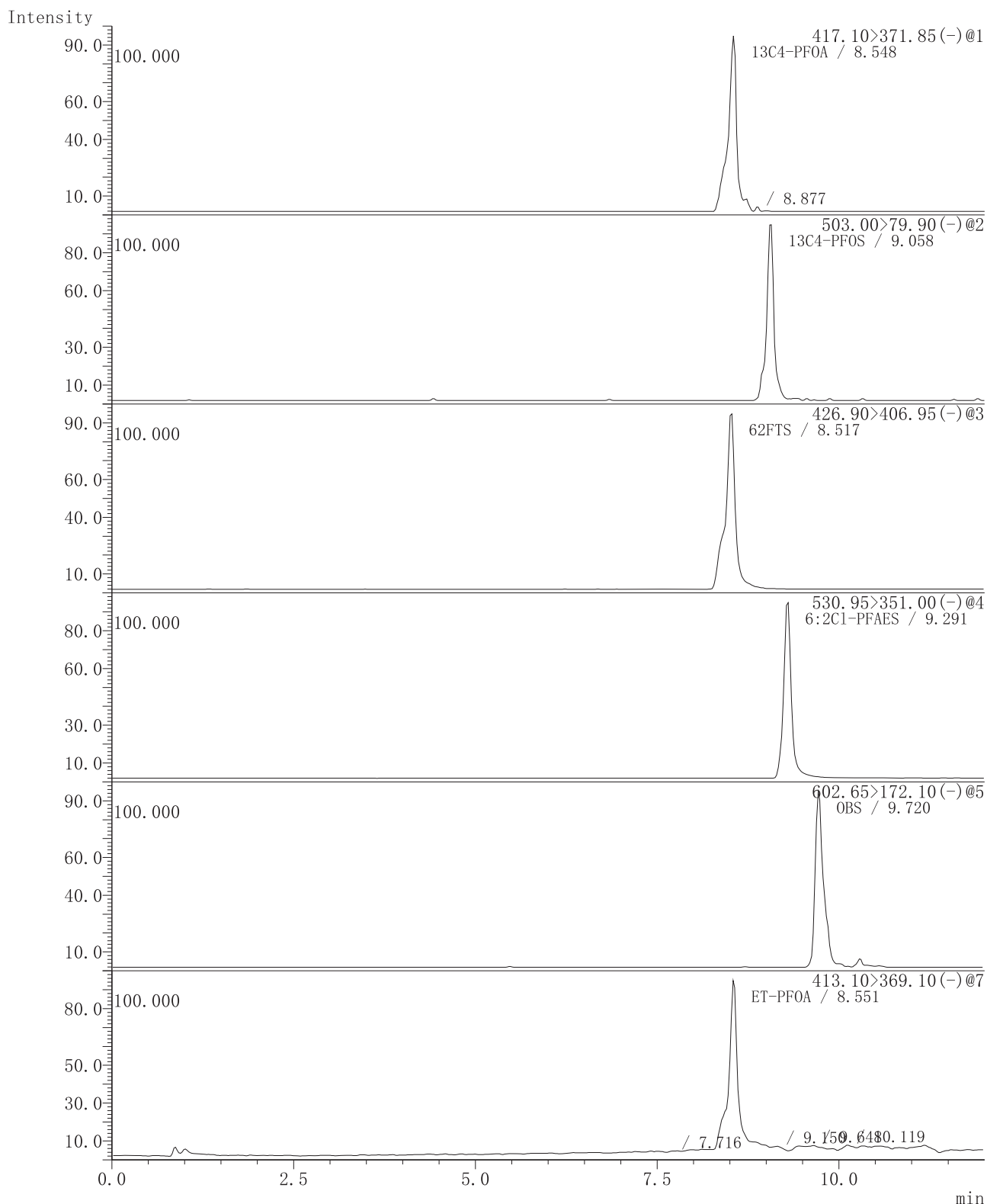


Fig. 4 | The multi-reaction monitoring chromatogram of four fluorinated alternatives and two tracer standards. The order reflected in the diagram is $^{13}\text{C}_4\text{-PFOA}$, $^{13}\text{C}_4\text{-PFOS}$, 6:2 FTS, 6:2 Cl-PFAES, OBS, Et-PFOA.

About 70% of PFASs in soil were found to originate from fluor-chemical manufacturing and metal processing, with atmospheric deposition and groundwater irrigation as important input pathways²⁸. The actual production, use, and releases of all fluorinated alternatives were unknown, and knowledge on the degradability, bioaccumulation potential and

ecotoxicity was still largely lacking³¹. Thus, continuous attention was needed for these industries.

This study successfully developed and validated a method that integrates ASE coupled with UPLC-MS/MS for the analysis of four fluorinated alternatives in soil and sediment. The LOD and LOQ of the method ranged

Table 2 | The interday and intraday precision for fluorinated alternatives in matrix-matched standards and spiked matrix

Fluorinated alternatives	Standard(ng/g)	Instrument repeatability (RSD%) (<i>n</i> = 6)		Spiked level(ng/g)	Method repeatability (RSD%) (<i>n</i> = 6)	
		Intraday	Interday		Intraday	Interday
6:2 Cl-PFAES	1	6.4	4.8	1	5.2	4.3
	5	12	7.4	5	7.3	5.1
	9	10	8.5	9	6.7	7.4
6:2 FTS	1	6.1	6.5	1	3.5	7.8
	5	10	7.1	5	7.8	6.9
	9	9.0	10	9	7.5	5.4
OBS	1	10	8.9	1	3.9	5.8
	5	5.4	7.7	5	8.2	3.8
	9	9.8	7.0	9	5.6	6.4
Et-PFOA	1	5.3	6.4	1	5.4	7.3
	5	6.6	5.7	5	4.2	4.6
	9	6.4	7.9	9	4.3	6.0

RSD relative standard deviation.

Table 3 | The summary statistics of fluorinated alternatives levels in soil and sediment samples

Fluorinated alternatives	DR (%)	Min (ng/g)	Max (ng/g)	Average (ng/g)	SD (ng/g)
6:2 Cl-PFAES	100	0.17	749	24.3	121
6:2 FTS	100	0.24	98.8	5.14	16.3
OBS	35	1.14	26.4	8.87	8.34
Et-PFOA	/	/	/	/	/

DR detection rate, SD standard deviation, ND not detection.

from 0.14 to 0.20 ng/g and 0.56 to 0.80 ng/g, the relative standard deviations (RSDs) were less than 10%, and the recovery of the method ranged from 85.4 to 95.5%. The method exhibited high sensitivity, reproducibility, and accuracy, rendering it suitable for environmental monitoring. Compared to alternative methods, this approach not only achieves substantial time savings in sample pretreatment but also provides reliable and stable recoveries. Application of this method to soil and sediment samples from Zhejiang, China, revealed extensive contamination, particularly by 6:2 Cl-PFAES and 6:2 FTS. These findings underscore the increasing environmental prevalence of fluorinated alternatives as replacements for legacy PFASs. In light of the potential ecological and health risks associated with these compounds, ongoing monitoring and further investigation into their environmental behavior and toxicological effects are essential.

Methods

Chemicals and reagents

Four fluorinated alternatives, including 6:2 chlorinated polyfluoroalkyl ether sulfonate (6:2 Cl-PFAES, 50 µg/mL), 6:2 fluorotelomer sulfonate (6:2 FTS, 100 µg/mL), Sodium p-perfluorooctanoic acid ethyl ester (OBS, purity>97.0%), perfluorooctanoic acid ethyl ester (Et-PFOA, purity>97.0%), purified siliceous earth (500 g, Analytical Reagent) were all purchased from Macklin Biochemical Technology Co., Ltd (Shanghai, China). Internal standard (¹³C₂-PFOA, purity>99.9%), two tracer standards (¹³C₄-PFOA, 2 µg/mL and ¹³C₄-PFOS, 2 µg/mL) were all purchased from Wellington Laboratories (Guelph, Canada). HPLC grade acetonitrile (ACN), methanol (MeOH), and methyl tertiary butyl ether (MTBE), and ammonium acetate were purchased from J&K Scientific (Beijing, China).

The solvent of each standard solution was prepared in ACN, and the concentration of fluorinated-alternative standard solutions was 1 µg/mL.

Also, the concentration of two tracer standards and internal standard solutions was 0.2 µg/mL. All standard solutions were stored at 4 °C^{32–34}.

Soil and sediment samples

The collection of soil and sediment samples adhered to the guidelines outlined in the Technical Specification for Soil Environmental Monitoring (HJ/T 166), as established by the Ministry of Ecology and Environment of the People's Republic of China. Following collection, all samples were promptly frozen at −4 °C, sealed to prevent contamination, shielded from light exposure, and subsequently analyzed within a 28-day period.

Sample preparation and analysis

All samples must undergo freeze-drying for a duration of 72 h, followed by grinding and sieving through a 60-mesh sieve. Subsequently, 10 grams of the dried sample were combined with 20 grams of purified siliceous earth within the sample cell of an Accelerated Solvent Extractor (ASE), and 50 µL of two tracer standards (0.2 µg/mL) were added. A solution of ACN and MTBE in a 1:1 ratio was employed as the extraction solvent. Approximately 60 mL of the extract was transferred to a rotary evaporator and evaporated to dryness. The resultant residue was then dissolved in 2 mL of the ACN:MTBE (1:1) solution, and 50 µL of internal standards (0.2 µg/mL) were introduced into the solution. The solution was subsequently filtered through a 0.22 µm membrane into a 1.5 mL HPLC vial and analyzed using UPLC-MS/MS. Sample pre-processing flowchart of four fluorinated alternatives were illustrated in Fig. 5.

Instrumental parameters

The ASE was Dionex ASE 350 (Thermo Scientific, USA). The extraction pressure was 1500 psi, extraction time was 5 min, temperature set was 60–70 °C, pump fresh solvent was 60% of the sample cell volume, purge time was 60 s, and number of static rinse cycles was 2.

The UPLC-MS/MS was SCIEX Triple Quad 7500 (AB Sciex Pte.Ltd, USA), and the analytical column was the Kinetex F5 (100 mm × 3 mm, 2.6 µm, Phenomenex, USA). The injection volume was 5 µL, the flow rate was 0.3 mL/min, and the column temperature was 35 °C. The A mobile phases were methanol and B was 2 mmol/L ammonium acetate in water. The elution gradient program of UPLC was shown in Table 4, the total run time was 12 min. MS/MS was operated in electrospray ionization (ESI) negative ion mode by multiple reaction monitoring (MRM). The transitions for quantification were shown in Table 1.

Validation procedure of the calibration curve

Mixed standard solutions containing four fluorinated alternatives at different concentration gradients were prepared using MEOH as solvent. The

Fig. 5 | Sample pre-processing flowchart of four fluorinated alternatives Freeze-drying and Accelerated solvent extraction.

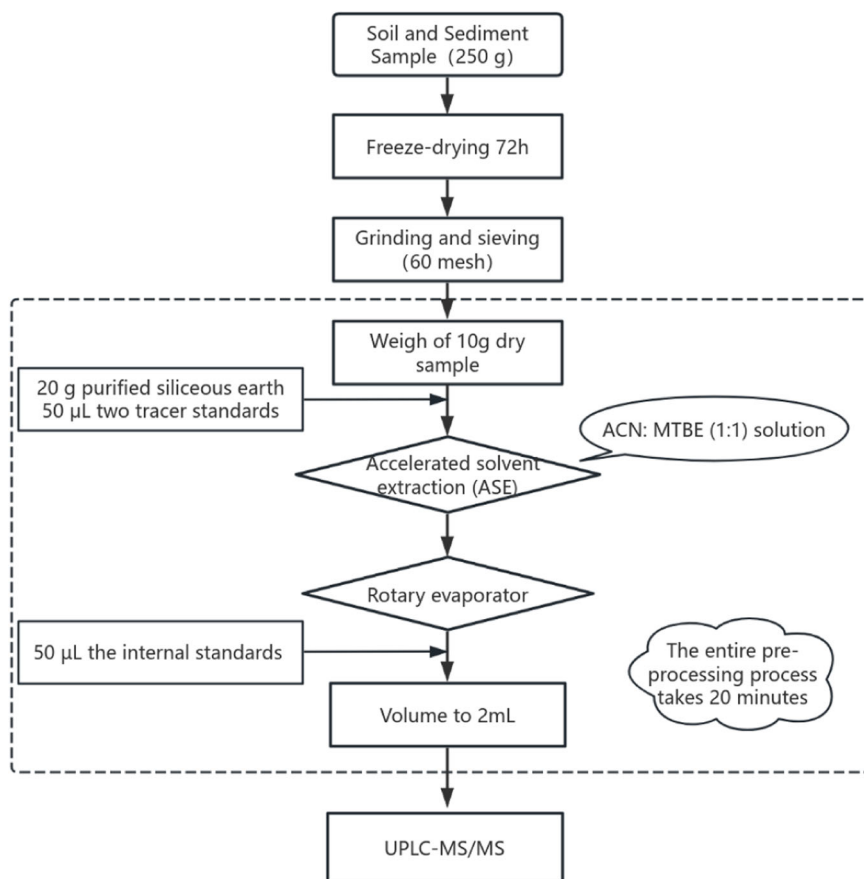


Table 4 | The elution gradient program of UPLC-MS/MS

Time(min)	Methanol(%)	Ammonium acetate solution(%)
0	30	70
5	65	35
9	95	5
11	95	5
11.1	30	70
12	30	70

purified siliceous earth sample without any fluorinated alternative was used as the blank soil matrix, and the calibration curve was plotted by transferring the standard mixing solution into the blank soil matrix extract, and this calibration curve was used for quantification. The calibration concentration gradients were 2, 5, 10, 20, 50, and 100 ng/mL for each fluorinated alternative. A certain amount of standard (5 mL, 1 µg/mL) was added to 7 purified siliceous earth blank samples (10 g). The limit of detection (LOD) was calculated from the standard deviation of 7 repeated experiments, and the equation was followed as:

$$LOD = t(n - 1, \alpha = 0.99) \times S \quad (1)$$

where t was the student's t -value for a degree of freedom of $n - 1$, n was the number of spiked samples for repeated experiment, α was the confidence level. The limit of quantification (LOQ) was 4 times LOD.

Method recovery

Two concentrations of fluorinated alternatives were spiked in the blank soil matrix (1 ng/g and 5 ng/g). The same concentrations of fluorinated

alternatives were added to the blank soil matrix extract repeated three times at each concentration.

Method precision

To ensure instrumental and methodological reproducibility, the matrix-matched standards (1 ng/g, 5 ng/g, and 9 ng/g) were replicated three times for each level on the same day (intraday), and the analysis was replicated three consecutive days (interday). Precision was measured by the interday and intraday variability of the relative standard deviation (RSD).

Methodological matrix effect

In the quantification process, the matrix effect (ME) can either enhance or suppress the analyte response. To assess this, blank solvent and blank matrix extract were each spiked with three concentrations of fluorinated alternatives standard solutions. The ME was then quantified using the following equation:

$$ME = \left(\frac{A}{B} - 1 \right) \times 100\% \quad (2)$$

where A is the slope of the matrix-matched calibration curve and B is the slope of the blank solvent calibration curve.

A negative value of ME indicated that the ion suppression of targeted PFAS and, correspondingly, a positive value indicated ionic amplification^{27,35}.

Statistical analyses

Statistical analysis was using GraphPad Prism (2018), and Microsoft Excel 2019, IBM® SPSS Statistics 26. T test was applied to verify the recovery between different extractants, and fluorinated alternatives concentrations between different soil and sediment samples. The significance level was set at 0.05.

Data availability

The datasets generated and analyzed during the current study are available from the corresponding author on request.

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Author contributions

R.W.: Investigation, Data curation, Writing—original draft. Y.W.: Material preparation, Writing—review & editing. Z.H.: Investigation, Data curation. X.D.: Investigation. S.H.: Conceptualization, Writing—review & editing. H.Z.: Data curation. J.L.: review & editing, Supervision. Q.Z.: Conceptualization, Methodology, Writing—review & editing, Supervision. All authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

Competing interests

The authors declare no competing interests.

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Additional information

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