The Extraction of PFAS Molecules from Spiked Soil

Abstract

Per- and polyfluoroalkyl substances (PFAS) are a class of manmade chemicals used in various industries because of their favorable properties for goods such as nonstick cookware and firefighting foam. Their stability and widespread use have contributed to their accumulation in the environment, and the lack of remediation techniques for their removal has allowed for their bioaccumulation in humans and animals. PFAS have been shown to cause health issues in humans, such as cancer, endocrine disruption, and infertility. Thus, their monitoring is critical. The EDGE®, an automated extraction system, was used to extract spiked PFAS from soil samples at both low and high spike levels. The EDGE extraction was less than 10 minutes and yielded excellent recoveries and RSDs without carryover in the system. The EDGE is an ideal option for laboratories that want to automate their PFAS extractions.

Introduction

Per- and polyfluoroalkyl substances (PFAS) are a group of manufactured chemicals that are used in a wide variety of industries because of their resistance to stains, grease, and high temperatures. PFAS possess a chain of linked carbon atoms with fluorine atoms branching off of the main chain. The presence of the strong carbon-fluorine bond contributes to the stability of these compounds, earning them the nickname “forever chemicals.” PFAS are used in products such as nonstick cookware, firefighting foam, and stain-resistant carpets.

Because of their persistent nature and their widespread use, this group of substances has leached into the environment with limited methods of remediation. Furthermore, these compounds have been found to bioaccumulate in animals and humans, and exposure in humans has been shown to cause adverse health outcomes, including cancer, infertility, and endocrine disruption. Thus, the assessment of the levels of PFAS in the environment is important to the health and safety of humans.

The EDGE, an automated solvent extraction system, was used to extract a subset of PFAS molecules from spiked soil samples. The EDGE was able to extract the soil samples in less than 10 minutes. The extraction yielded excellent recoveries and standard deviations. Furthermore, there was no carryover found between samples. The EDGE is an excellent choice for laboratories seeking to automate their PFAS extraction.

Materials and Methods

Reagents

Clean sandy loam was purchased from Millipore Sigma. A PFAS standard containing 24 different compounds (Part number 99207) was purchased from Absolute Standards, Inc. HPLC-grade methanol, HPLC-grade water, HPLC-grade formic acid, and ammonium hydroxide were purchased from Fisher Scientific.

Sample Preparation

Five grams of clean sandy loam was weighed directly into a Q-Cup® containing the S1 Q-Disc® stack (C9+G1+C9 sandwich). Each sample was spiked with 2 ng or 200 ng of standard in HPLC-grade methanol, resulting in low spike and high spike samples, respectively. Each set of spikes was done in triplicate. The sample was extracted with the EDGE using methanol/water (80:20) with 0.3% ammonium hydroxide using the method provided. Each set of spikes were extracted on the EDGE, and then, a blank extraction of the system was done with a Q-Cup containing the S1 Q-Disc stack (C9+G1+C9 sandwich) to assess the level of carryover. Each extraction was collected in a polypropylene conical tube using an EDGE rack. The sample was brought up to a final volume of 20 mL using methanol/water (80:20) with 0.3% ammonium hydroxide, and 20 µL of formic acid was added to each sample to neutralize the sample. The samples were then analyzed by Pace Analytical.

EDGE Method for PFAS from Soil

Q-Disc: S1 Q-Disc stack (C9+G1+C9 sandwich)

Cycle 1
Extraction Solvent: Methanol/Water (80:20) with 0.3% ammonium hydroxide
Top Add: 10 mL
Bottom Add: 0 mL
Rinse: 0 mL
Temperature: 65 °C
Hold Time: 03:00 (mm:ss)

Cycle 2
Extraction Solvent: Methanol/Water (80:20) with 0.3% ammonium hydroxide
Top Add: 10 mL
Bottom Add: 0 mL
Rinse: 0 mL
Temperature: 65 °C
Hold Time: 04:00 (mm:ss)

Wash 1
Wash Solvent: Methanol
Wash Volume: 10 mL
Temperature: 50 °C
Hold: 00:03 (mm:ss)

Wash 2
Wash Solvent: Methanol/Water (80:20) with 0.3% ammonium hydroxide
Wash Volume: 10 mL
Temperature: - - -
Hold: - -:- -

Results and Discussion

The recovery data in **Table 1** (page 3) from the low and high spikes indicated that the samples were extracted with high efficiency, with recoveries ranging from 63% to 101%. The resulting RSD values were also low, indicating the recovery data were reproducible. The extraction data from the empty Q-Cup indicated that there was no carryover of the spiked compounds within the system, indicating the wash was aggressive enough to remove any residual PFAS compounds.

Conclusion

The analytical assessment of PFAS compounds is critical because of their widespread nature, increased stability, and adverse health effects. The EDGE was able to rapidly and efficiently extract spiked soil samples with excellent recoveries and RSD values. The EDGE also saw no carryover after extractions into the subsequent extraction. The EDGE is an excellent extraction tool for laboratories seeking to automate their PFAS extractions with great efficiency.

Table 1. Average Recovery Results from the Low Spike and High Spike Soil Samples

Compound	Low Spike	RSD (n=3)	High Spike	RSD (n=3)
1H, 1H, 2H, 2H-perfluorodecane sulfonic acid (8:2 FTS)	83.00%	7.81%	87.00%	7.00%
1H, 1H, 2H, 2H-perfluorooctane sulfonic acid (6:2 FTS)	80.00%	3.00%	87.67%	7.09%
1H,1H,2H,2H-perfluorohexane sulfonic acid (4:2 FTS)	86.67%	10.02%	87.67%	5.13%
N-ethylperfluoro-1-octanesulfonamidoacetic acid (EtFOSAA)	85.67%	6.66%	86.00%	5.57%
N-methylperfluoro-1-octanesulfonamidoacetic acid (MeFOSAA)	78.67%	1.15%	81.33%	4.16%
Perfluoro-1-butanesulfonic acid (PFBS)	78.00%	3.00%	86.00%	1.73%
Perfluoro-1-decanesulfonic acid (PFDS)	77.00%	6.56%	84.33%	0.58%
Perfluoro-1-heptanesulfonic acid (PFHpS)	81.00%	2.65%	87.00%	2.65%
Perfluoro-1-nonanesulfonic acid (PFNS)	76.00%	6.56%	84.67%	2.89%
Perfluoro-1-octanesulfonamide (PFOSA)	81.00%	8.00%	86.67%	5.03%
Perfluoro-1-pentanesulfonic acid (PFPeS)	78.00%	4.36%	86.33%	0.58%
Perfluorohexanesulfonic acid (PFHxS)	98.33%	2.89%	89.67%	3.79%
Perfluoro-n-butanoic acid (PFBA)	85.65%	4.01%	88.17%	2.20%
Perfluoro-n-decanoic acid (PFDA)	101.33%	7.51%	93.33%	2.89%
Perfluoro-n-dodecanoic acid (PFDoA)	79.67%	7.37%	79.33%	2.08%
Perfluoro-n-heptanoic acid (PFHpA)	90.67%	9.02%	81.67%	3.21%
Perfluoro-n-hexanoic acid (PFHxA)	100.67%	9.02%	91.08%	6.64%
Perfluoro-n-nonanoic acid (PFNA)	96.33%	3.21%	92.67%	2.52%
Perfluoro-n-octanoic acid (PFOA)	82.77%	3.87%	85.53%	2.25%
Perfluoro-n-pentanoic acid (PFPeA)	79.50%	0.71%	85.67%	4.04%
Perfluoro-n-tetradecanoic acid (PFTeDA)	86.67%	4.73%	88.67%	1.15%
Perfluoro-n-tridecanoic acid (PFTrDA)	63.00%	3.46%	68.33%	3.51%
Perfluoro-n-u0ecanoic acid (PFUdA)	76.33%	1.53%	79.67%	3.51%
Perfluorooctanesulfonic acid (PFOS)	84.00%	4.00%	79.60%	0.53%

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