The Extraction of PFAS from Produce

Abstract

Per- and polyfluoroalkyl substances (PFAS) are a class of manufactured compounds previously used in many industries because of their favorable properties such as stain resistance and as flame retardants. However, they have been found to cause ill effects on human health. Their widespread use and resistance to degradation has made testing for these compounds important. Because PFAS can bioaccumulate, there is a need to test for these compounds in produce, which, along with water, is a possible avenue of human contamination. Within this work, the EDGE PFAS™ system was used to extract spiked PFAS from cranberries, strawberries, carrots, potatoes, and lettuce. It recovered the spiked compounds within the range proposed by the US FDA Method C-010.01 "Determination of 16 Perfluoroalkyl and Polyfluoroalkyl Substances (PFAS) in Food using Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS)." The EDGE PFAS is an excellent choice for laboratories seeking to automate the extraction of PFAS from produce.

Introduction

Per- and polyfluoroalkyl substances (PFAS) are a class of synthetic compounds. They were previously widely used in many industries and products, including nonstick cookware and firefighting foam, because of their chemical properties, including stain resistance, flame retardance, and temperature stability. These compounds are made of a chain of linked carbon atoms with fluorine atoms branching off this main chain. These carbon-fluorine bonds provide the stability of these compounds, preventing their degradation and earning them the nickname "forever chemicals." With this persistent nature and widespread use, PFAS compounds are found in the environment, easily transmitting through water sources to contaminate soils with limited means to remove them. After migrating into soils, PFAS can go further to contaminate produce where these compounds can bioaccumulate in produce and in turn, accumulate in humans, causing unfavorable effects on human health. These effects include cancer, infertility, and endocrine disruption. Thus, it is critical that PFAS compounds be measured in produce.

In this work, performed in conjunction with Waters Corporation, the EDGE PFAS was utilized to extract PFAS from cranberries, strawberries, carrots, potatoes, and lettuce. These produce types were selected to represent a high acid and high sugar produce, a high sugar only produce, a low water produce, a high starch produce, and a high water produce, respectively.

It was found that the EDGE PFAS could extract PFAS from each of these produce types with high recoveries and favorable standard deviations within the range proposed by the US FDA Method C-010.01 "Determination of 16 Perfluoroalkyl and Polyfluoroalkyl Substances (PFAS) in Food using Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS)." Thus, the EDGE PFAS is an excellent option for PFAS extractions from produce.

Materials and Methods

Carrots, cranberries, lettuce, potatoes, and strawberries were purchased from a local grocery store. HPLC-grade methanol, HPLC-grade water, and ammonium hydroxide were purchased from Sigma. Native PFAS Solution (PFAC30PAR) and Mass-Labelled PFAS Solution (MPFAC-24ES) were purchased from Wellington Laboratories.

The produce was homogenized using a food processor and refrigerated until extraction. Q-Cups were prepared with the S1 Q-Disc® stack. 6 g of sodium sulfate was weighed directly into each Q-Cup®. A portion of 1.75 g of sodium chloride was added above the sodium sulfate layer. Then, 5 g of each type of homogenized produce was added on top of the salts within the Q-Cup. Each produce type was prepared in triplicate. Each sample was then spiked with 5 ng/g of sample with Native PFAS Solution and 10 ng/g of sample with Mass-Labelled PFAS solution on top of the produce sample in the Q-Cup. All Q-Cups, along with polypropylene centrifuge tubes, were loaded into an EDGE rack and the EDGE PFAS was used to extract the samples using the following method:

Cycle 1
Extraction Solvent: 80:20 Methanol:Water + 0.3% NH_4OH
Top Add: 20 mL
Bottom Add: 0 mL
Rinse: 0 mL
Agitation Time: 02:00 (mm:ss)
Temperature: 65 °C
Hold Time: 05:00 (mm:ss)

Cycle 2
Extraction Solvent: 80:20 Methanol:Water + 0.3% NH_4OH
Top Add: 20 mL
Bottom Add: 0 mL
Rinse: 0 mL
Agitation Time: 02:00 (mm:ss)
Temperature: 65 °C
Hold Time: 05:00 (mm:ss)

Wash 1
Wash Solvent: Methanol
Wash Volume: 30 mL
Temperature: 65 °C
Hold Time: 00:30 (mm:ss)

Wash 2
Wash Solvent: Methanol
Wash Volume: 30 mL
Temperature: 65 °C
Hold Time: 00:30 (mm:ss)

Wash 3
Wash Solvent: 80:20 Methanol:Water + 0.3% NH_4OH
Wash Volume: 40 mL
Temperature: ---
Hold Time: 00:03 (mm:ss)

Analysis

Analysis was done by Waters Corporation. For the analysis, an ACQUITY™ I-Class PLUS equipped with a Waters PFAS Kit and attached to a Xevo® TQ-XS was used. The compounds were separated using an ACQUITY BEH C18 column (2.1 mm x 100 mm, 1.7 µm). A 10 µL injection was used, and the mobile phases were 2 mM ammonium acetate in water (A) and 2 mM ammonium acetate in methanol (B). The gradient used is indicated in **Table 1**. The source parameters used to monitor the MRM transitions of each compound are in **Table 2**.

Table 1. UPLC Gradient Used for Separation

Time (min)	Flow (mL/min)	%A	%B
0	0.3	95	5
1	0.3	75	25
6	0.3	50	50
13	0.3	15	85
14	0.3	5	95
17	0.3	5	95
18	0.3	95	5
22	0.3	95	5

Table 2. Source Parameters

Parameter	Value
Ion mode	ESI-
Capillary Voltage	0.5 kV
Desolvation Temperature	350 °C
Desolvation Flow	900 L/hr
Cone Flow	150 L/hr

Results

To assess recovery for each sample, pre-spiked samples were compared to post-spiked samples. Spiked recovery was also adjusted using the internal standards. **Figures 1 through 5** show the post spike-adjusted recovery and the internal standard-adjusted recovery for each commodity. The standard deviations for each measurement are highlighted with error bars. For carrot, cranberry, lettuce, potato, and strawberry, all PFAS compounds assessed achieved recoveries for both spiked recoveries and internal standard-adjusted recoveries within the recovery ranges found acceptable by the US FDA Method C-010.01 “Determination of 16 Perfluoroalkyl and Polyfluoroalkyl Substances (PFAS) in Food using Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS).” This range was 40-120%. Also, as shown by narrow error bars, the standard deviations are low, indicating a repeatable extraction.

Conclusion

PFAS compounds were previously used in many industries because of their favorable properties. The stability and widespread nature of PFAS compounds have increased the likelihood of human exposure where they can cause deleterious effects on health and bioacculmulate. Thus, it is important to monitor the PFAS content in produce, a likely source of PFAS contamination. In this work, the EDGE PFAS was used to extract spiked PFAS from cranberries, strawberries, carrots, potatoes, and lettuce. It was found that the EDGE PFAS system was able to extract PFAS from these matrices with high recoveries and good standard deviations. The recovery data were within the range deemed acceptable by the US FDA Method C-010.01 “Determination of 16 Perfluoroalkyl and Polyfluoroalkyl Substances (PFAS) in Food using Liquid Chromatography-Tandem Mass Spectrometry (LC-MS/MS).” Thus, the EDGE PFAS is a great fit for laboratories seeking to extract PFAS from produce samples.

Acknowledgements

Within this work, the extracts were analyzed by Waters Corporation. We would like to thank Waters for their participation in this work and for their ongoing partnership.

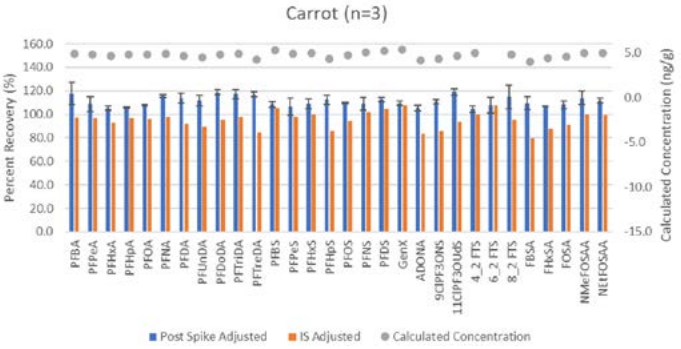

Figure 1. Spiked and Internal Standard-adjusted Recovery Data Found for the Extraction of Carrot

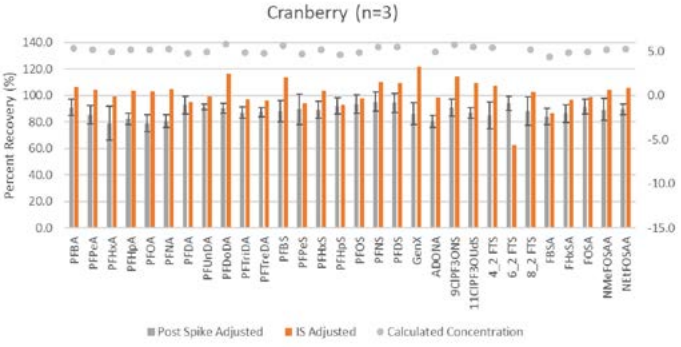

Figure 2. Spiked and Internal Standard-adjusted Recovery Data Found for the Extraction of Cranberry

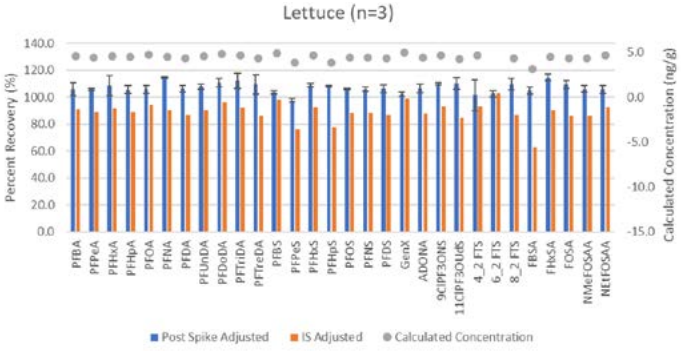

Figure 3. Spiked and Internal Standard-adjusted Recovery Data Found for the Extraction of Lettuce

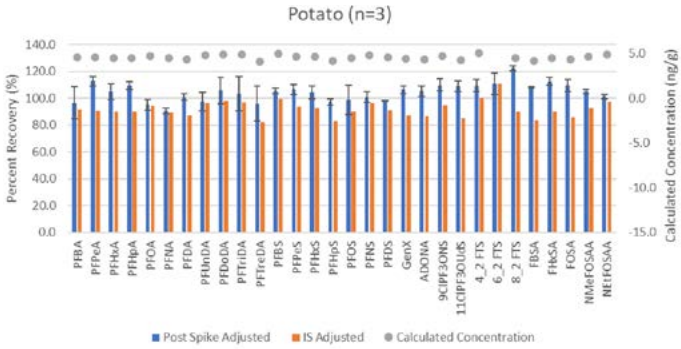

Figure 4. Spiked and Internal Standard-adjusted Recovery Data Found for the Extraction of Potato

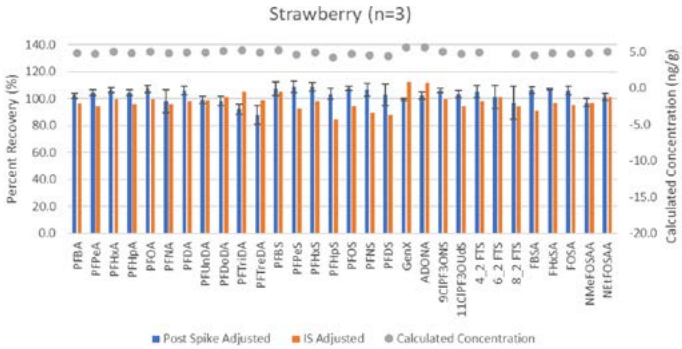

Figure 5. Spiked and Internal Standard-adjusted Recovery Data Found for the Extraction of Strawberry

Find a Local Contact

cem.com/contact