A Rapid, Simple, and Efficient Automated Method for the Extraction of Pesticides from Difficult Food Matrices

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Abstract

In the modern world, consumers increasingly want to know what is in their food and that the substances they are putting in their body are safe. This, along with stringent regulatory requirements, is leading the call for improved extraction of food contaminants such as pesticides. The QuEChERS method has been shown to be practical for pesticide analysis on a number of different sample types. While the QuEChERS method is relatively quick compared to other methods, it is also a manual and tedious process. Herein, a rapid, simple and efficient automated method for the extraction of difficult food matrices that improves the pesticide extraction process is being reviewed. The extraction of some difficult food matrices such as lemons and potatoes is performed via extraction on the EDGE and compared to the QuEChERS method. The EDGE methods utilizes the same solvent and clean up sorbents as the QuEChERS method, making it a simple transition for labs currently running QuEChERS. The EDGE extraction allows for extraction, cleanup, and filtration in less than ten minutes. Filtration sub 0.3 µm is possible on the EDGE allowing for direct injection of the extract for UPLC analysis. The EDGE extraction method is applicable to many different types of difficult matrices including dry commodities such tea and spices. No matter the matrix on the EDGE the same rapid, simple and efficient automated method is sufficient leading to good pesticide recovery data. The extraction and determination of multiple pesticide residues from these difficult matrices will be described showing improved recoveries and workflow in comparison to QuEChERS.

EDGE Extraction

1. Assemble Q-Cup sample holder with S1 Q-Disc (allows for 0.3µm filtration)
2. Weigh 2.5g of Q-Matrix Hydra directly into the Q-Cup
3. For lemon only weigh 1g sodium citrate dihydrate and 0.5g of sodium citrate sesquihydrate directly into the Q-Cup
4. Weigh 10g of homogenized lemon or potato directly into Q-Cup
5. Spike samples with 300µL of 10,000 ng/mL of Restek Multiresidue LC Pesticide mix 6 and 9
6. Carefully mix contents of the Q-Cup with a glass rod
7. Wipe glass rod with small piece of Kimwipe, drop into Q-Cup
8. Place prepared Q-Cup into the EDGE rack alongside 50 mL centrifuge tube for extract collection
9. Run EDGE extraction
10. Confirm extract volume of 40 mL
11. Transfer to vial for concurrent analysis
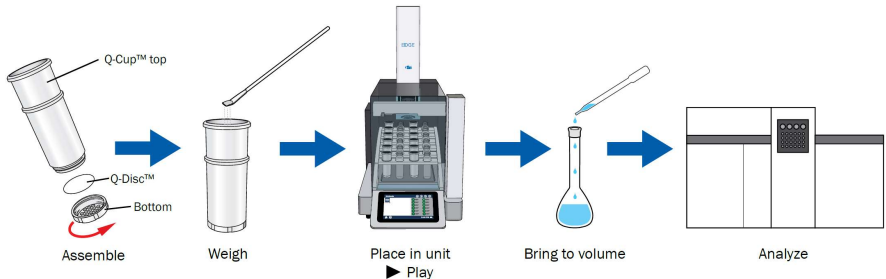
EDGE Method

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

Cycle 1 Extraction Solvent: Acetonitrile w/ 1.0% Acetic Acid (v/v) Agitation: 01:00 (mm:ss) Top Add: 20 mL Bottom Add: 0 mL Rinse: 10 mL Temperature: 40 °C Hold Time: 02:00 (mm:ss)

Cycle 2 (Rinse Only) Extraction Solvent: Acetonitrile w/ 1.0% Acetic Acid (v/v) Top Add: 0 mL Bottom Add: 0 mL Rinse: 10 mL Temperature: - - - Hold Time: - -:- -

Wash 1 Wash Solvent: Acetonitrile w/ 1.0% Acetic Acid (v/v) Wash Volume: 15 mL Temperature: 40 °C Hold: 0:03 (mm:ss)

QuEChERS Extraction

1. 10g homogenized sample spiked with 300 µL of 10,000 ng/mL Restek Multiresidue LC Pesticide mix 6 and 9
2. Add 10 mL 1% acetic acid in acetonitrile,
3. Shake 1 min
4. For lemon add 6g MgSO$_4$, 1g NaCl, 1g Na citrate dihydrate, 0.5g citrate sesquihydrate
5. For Potato add MgSO$_4$, 1g NaCl
6. Shake 1 min
7. Centrifuge at 6000 rpm for 5 min

UPLC MS/MS Method

- Waters Acquity H Class with Xevo TQD
- Restek ARC-18, 2.7 µm, 100 x 2.1 mm column
- 10 µl injection in acetonitrile
- A: Water with 4 mM Ammonium Formate and 0.1% Formic Acid
- B: Methanol 4 mM Ammonium Formate and 0.1% Formic Acid
- Gradient

Time (min)	Flow (mL/min)	%A	%B
Initial	0.25	95	5
2	0.25	40	60
4	0.25	25	75
14.50	0.25	0	100
15.50	0.25	0	100
15.51	0.25	5	95
18.00	0.25	95	5

Results

% Recoveries of Pesticides from Potatoes

Average % Recoveries
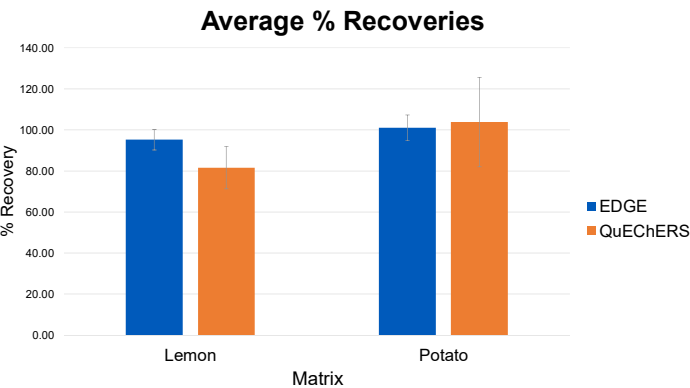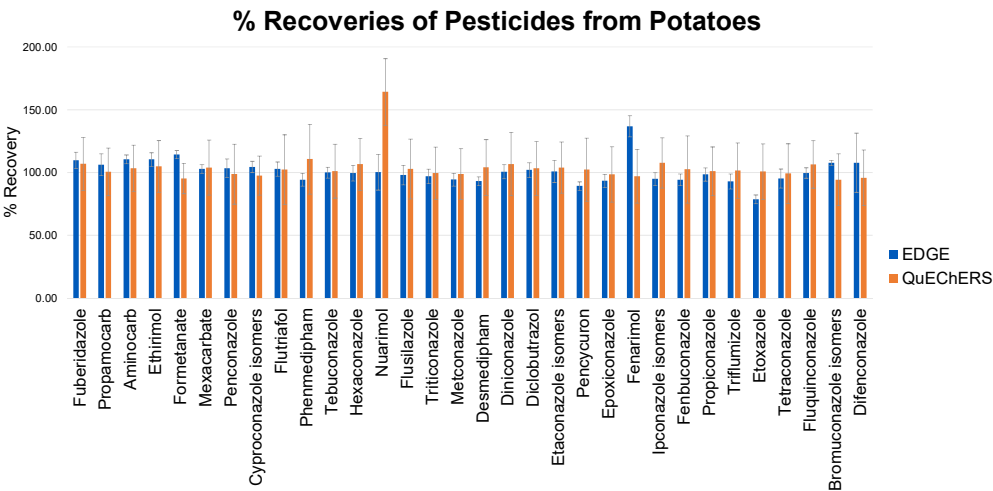
Conclusion

The extraction of lemon and potato using the EDGE has proven to be a reliable efficient and robust technique when applied to the various food commodities. With its simple pesticide extraction method, it can be adapted for any properly prepared food commodity. The incorporation of Q-Matrix Hydra when preparing the sample helps to support the sample, improve dispersion through mixing, and remove water. In addition, comparable or better recoveries and RSD values were obtained when compared to the traditional QuEChERS method.