Multiresidue Analysis of Pesticides in Cannabis-Infused Edibles by Extraction and Enhanced Matrix Removal-Lipid Cleanup

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Introduction

A cannabis edible, also called cannabis-infused food, is a food product that contains cannabinoids, especially THC and CBD. Cannabis edibles are consumed for both medical and recreational purposes.

Because cannabinoids are soluble in lipids and alcohols, cannabis must be mixed with one of these two substances in order to infuse the cannabinoids into the food. The oil-solubility of cannabis extracts has been known since ancient times. Since the infused cannabinoids are a concentrate it is important to monitor chemical residues in the concentrate, specifically pesticides. However, it can be difficult to analyze for pesticide residues in high lipid content matrix. Lipids can cause both analysis and instrument issues over time. QuEChERS or organic extraction is a commonly used sample preparation method for the analysis of pesticides from food products however the dispersive SPE containing C18 which is used for fatty matrix is insufficient at selectively removing lipids. A novel sorbent Enhanced Matrix Removal-Lipid (EMR-Lipid) specific for the removal of lipids has shown to be very effective at retaining lipids without compromising recovery of the pesticides. The application will show the matrix removal capabilities of new EMR-Lipid formulation for high lipid content cannabis-infused food products and the analysis of pesticides.

Experimental

Sample Preparation

Chocolate Chip Cannabis Cookie:

2 g of homogenized matrix was added to a 50 mL centrifuge tube, spiked with pesticide mix (OAR 33-07-044) at 25, 50 and 100 ppb and TPP at 50 ppb as (IS), vortexed.

10 mL of ACN (0.1% FA) was added, 2 ceramic homogenizers, vortexed 5 min, GenoGrinder for 10 min, centrifuged at 5000 rpm, 5 min.

Cascadia Cannabis Tincture:

0.5 g of oil matrix was added to a 15 mL centrifuge tube, spiked with pesticide mix (OAR 33-07-044) at 25, 50 and 100 ppb and TPP at 50 ppb as (IS), vortexed.

2.5 mL of ACN (0.1%FA) was added, 2 ceramic homogenizers, vortexed 5 min, GenoGrinder for 10 min, centrifuges at 5000 rpm, 5 min.

Experimental

New EMR-Lipid Formulation Cleanup: High Lipid Cannabis-Infused Products

To 2 mL of the ACN extract 0.5 mL of water is added, vortexed.

The extract is passed through a new EMR-Lipid formulation cartridge, 300 mg/3 mL by gravity, pull vacuum at end to evacuate the cartridge.

The eluted extract is analyzed by LC/MS/MS.

LC-MS/MS Instrument Conditions

LC-MS/MS: Agilent 1290 Infinity LC, 6490 Triple Quadrupole LC/MS, Jet Stream ESI
Ionization: Positive

Table 1. Source Parameters.

Parameter	Value (+)
Gas Temp (°C)	120
Gas Flow (l/min)	20
Nebulizer (psi)	50
Sheath Gas Heater	325
Sheath Gas Flow	12
Capillary (v)	3500
V Charging	300

Column: Poroshell C18-EC, 2.1 x 150 mm, 2.7 µm
Injection volume: 5 µL
Mobile Phase:
A: Water, 5 mM Formate, 0.1% FA
B: Methanol, 5 mM Formate, 0.1% FA
Flow Rate: 0.5 mL/min

Table 2. Timetable.

	Time, min	A	B
1	0.00	95.0%	5.0%
2	0.80	60.0%	40.0%
3	3.50	60.0%	60.0%
4	5.00	40.0%	60.0%
5	6.00	40.0%	60.0%
6	12.00	35.0%	65.0%
7	17.00	0.0%	100.0%

Results and Discussion

EMR sorbent technology effectively traps lipids through two mechanisms:

- **Size exclusion** – Unbranched hydrocarbon chains (lipids) enter the sorbent; bulky analytes do not.
- **Sorbent chemistry** – Lipid chains that enter the sorbent are trapped by hydrophobic interactions.

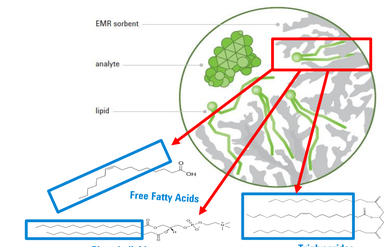

Figure 1. Chocolate Chip Cannabis Cookies and Tincture used in the study

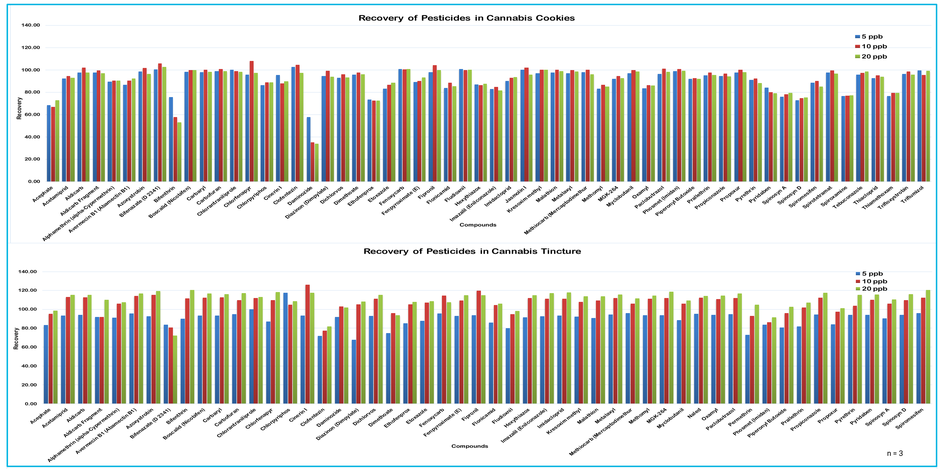

Figure 4. Pesticide recovery data in Cannabis Cookies and Tincture, at 5, 10 and 20 ppb, n=3.

Results and Discussion

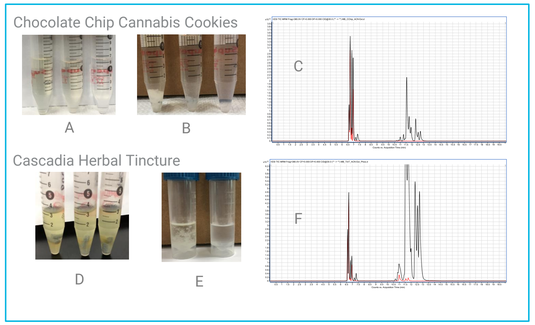

Figure 2. Extracts after d-SPE (C18/PSA) and new EMR-Lipid formulation cleanup

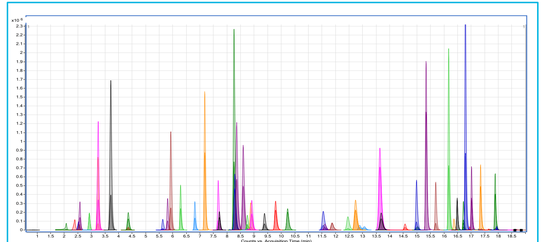

Figure 3. LC/MSMS Chromatogram for Fifty Seven Pesticides in the Study from OAR 33-07-044 (Oregon Pesticide List in Cannabis), 20 ppb.

Figure 2 is a picture of the extracts. Photo A. after ACN extraction of the cookie material (left), after cleanup d-SPE (C18/PSA) (middle) or new EMR-Lipid formulation cleanup (right). A yellow color is still present after d-SPE (C18/PSA) but not observed after EMR-Lipid. Photo B. after freezing the extracts for 2 hours at minus 20 degrees, lipids are observed falling out of solution in the ACN extract (left), and after d-SPE (C18/PSA) cleanup (middle) but not after new EMR-Lipid formulation cleanup (right), showing more complete lipid removal. Chromatogram C. overlay chromatogram of ACN extract (black), after EMR-Lipid cleanup (red).

Photo D. after ACN extraction of the tincture. Photo E. after freezing the extracts for 2 hours at minus 20 degrees, lipids are observed falling out of solution in the ACN extract (left) but not after new EMR-Lipid formulation cleanup (right), showing more complete lipid removal. Chromatogram F. overlay of chromatogram of ACN extract (black), after EMR-Lipid cleanup (red).

Conclusions

- The NEW EMR-Lipid formulation was extremely effective and efficient at removing lipids from a high fat cannabis edibles with an easy pass through cartridge format without the need for conditioning or washing of the sorbent.
- Chocolate Chip Cookie had 96% of the pesticide recoveries within 70-120% and RSD ≤ 20%; The Cascadia Tincture had 100% of the pesticide recoveries within 70-120% and RSD ≤ 20%.
- Calibration curves were linear from 0.5-100 ppb for pesticides in both Chocolate Chip Cookies and Cascadia Tincture; > 0.99 for 95% of the pesticides.

Agilent products and solutions are intended to be used for cannabis quality control and safety testing in laboratories where such use is permitted under state/country law