Optimization Study of Supercritical Fluid Extraction of Cannabinoids from *Cannabis sativa*

**Eric Kawka** | Founder
**Cattis Scientific**
How They **REALLY** Made the Periodic Table...
Appreciating the Complexity of Cannabis Sativa

100 + Cannabinoids

200 + Terpenes

Endocannabinoid system
300 Receptors

Each receptor can accept multiple compounds

\[ nCr = \frac{n!}{r!(n - R)!} \]
The California Cruiser

Cannabinoids

- THC (tetrahydrocannabinolic acid)
- THCA (tetrahydrocannabinolic acid)
- CBD (cannabidiol)
- CBDA (cannabidiolic acid)
- CBG (cannabigerolic acid)
- CBC (cannabichromenic acid)
- CBGVA (cannabigerovarinic acid)

Terpenes

- Δ9-THC (tetrahydrocannabinol)
- Δ8-THC (tetrahydrocannabinolic acid)
- β-Caryophyllene
- β-Pinene
- α-Pinene
- Limonene
- Myrcene
- Ocimene
- Pinene
- Linalool
- Geraniol
- Camphene
- Terpinolene
- Carvacrol
- Terpinen-4-ol
- Pinocarveol
- Sabinene

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Overview

• Review of select prior work on cannabinoid extraction utilizing Supercritical SC-CO₂ and co-solvents
• Extending the boundaries of cannabinoid extraction with SC-CO₂ by altering CO₂ flow
• Future optimization work
Scale-up Study of Supercritical Fluid Extraction Process for Clove and Sugarcane Residue

- Presents overall extraction curves (OEC’s) for clove and sugar cane at different pressures.
- Optimize a scale up of clove and sugarcane from 250mL to 5.15 L by maintaining S/F ratio.

Fig. 1. OECs for clove (a) and sugarcane residue (b) at LS 1 (○), LS 2 (○), PS 1 (○) and PS 4 (○).

J.M. Prado et al. / J. of Supercritical Fluids 56 (2011) 231–237
Cannabinoid Solubility Data in SC-CO$_2$

- CBD >> CBDa
- CBDa >> THCa
- THC >> THCa

H. Perrotin-Brunel et al.
J. of Supercritical Fluids
55 (2010) 603–608
The use of ethanol as a co-solvent was investigated with two different approaches:

1. constant co-solvent flow
2. pulses of ethanol at different times though the extraction procedure

Process extraction efficiency as high as 92% was achieved.
Terpenes and Cannabinoids are Synthesized and Stored in Trichomes
SC-CO$_2$ Efficiency Curve

- Mechanical region
- Diffusion limited region

$\text{g COI}$ vs. time

- $\text{CO}_2 + \text{COI}$
- $\text{CO}_2 + \text{COI}$
Mass Transfer – CO$_2$ Flow Rate

Low CO$_2$ Flow  

Higher CO$_2$ Flow  

rate, g/min COI

time

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Like a Fine Cup of Java.....

Soak for 4 minutes and repeat

**Static / dynamic approach** to boost efficiency in the diffusion limited region of the efficiency curve
Instruments Used in Optimization Study

Water’s Acquity UPC2

Water’s Bio Botanical Extractor (BBES)
Dynamic / Static Experimental Set Up

SFE Conditions
1. 250 Bar, 50C, **Dynamic Run**
2. 250 Bar, 50C, **Dynamic/Static**
   time constant
3. 250 Bar, 50C, **Dynamic/Static**
   S/F constant

BoAx → Homogenized 25 pounds → 5 Liter Vessel

13.47 % CBDa
<table>
<thead>
<tr>
<th>Dynamic/Static</th>
<th>Step</th>
<th>Co-Solvent Flow (g/min)</th>
<th>CO2 Flow (g/min)</th>
<th>Extraction Vessel 1 Temp (°C)</th>
<th>Cyclone Vessel 1 Temp (°C)</th>
<th>Cyclone Vessel 2 Temp (°C)</th>
<th>Cyclone Vessel 3 Temp (°C)</th>
<th>Inline Heater Temp (°C)</th>
<th>Inline Collection Heater Temp</th>
<th>Extraction Pressure (bar)</th>
<th>Dynamic Duration 1 (min)</th>
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## Experiment 1: Dynamic Versus Dynamic/Static

<table>
<thead>
<tr>
<th>Ext. #</th>
<th>Description</th>
<th>g CBD avail. (total potential)</th>
<th>time, min.</th>
<th>g CO₂</th>
<th>s/f</th>
<th>g CO₂ / g CBD</th>
<th>g CBD extracted (total potential)</th>
<th>Extraction Efficiency %</th>
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<tbody>
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<td>215</td>
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## Table

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<td>%CBDa TOP</td>
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<td>%CBDa MID</td>
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Dynamic

Dynamic/Static, time constant

Dynamic/Static, S/F constant

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## Extraction Economics

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<tr>
<th>Extraction</th>
<th>g CBD remaining in raffinate</th>
<th>$ remaining in raffinate</th>
<th>$ normalized to 90% efficiency</th>
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<td>25</td>
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$1,470 / 5 L extraction run X 10 L /day X 5 days = $14,700 / week
A: Grams CBD/CBD-A yielded over time
B: Rate of extraction; total potential CBD /minute

15 min Soak

[Graph showing the extraction process over time with different markers for CBD and CBDA]
### Adjust Soak Time While Maintaining S/F

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#### Extraction time:
650 min

#### Grams CO₂:
107,625

#### Starting mass:
1820g

#### S/F:
59.13

#### Efficiency 90 %
Future Experiment: Beyond 5 Liters
Conclusion

A 30% efficiency boost is observed with a static/dynamic approach in the diffusion limited region of the efficiency curve.
Thanks to:

• Andy Aubin
• Shawn Helmueller
• John A. MacKay, PhD
• Emerald Conference
Thanks to:

YOU!