

# Home Extractions of Hemp and CBD Yields

**MACERATION COMPARED TO PERCOLATION**

# Lecture Map



Intro

Maceration

Extractions

Percolation

Maceration  
Time  
Supporting  
Evidence

# Disclosures

I have been a Natural Products, Pharma and *Cannabis* Industry Consultant, for SOPs, GMPs, Regulatory Issues, Pharmacology, Research Initiatives and Formulation



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Contents lists available at ScienceDirect

# Journal of Pharmaceutical and Biomedical Analysis

journal homepage: [www.elsevier.com/locate/jpba](http://www.elsevier.com/locate/jpba)



## Comparison of alkylamide yield in ethanolic extracts prepared from fresh versus dry *Echinacea purpurea* utilizing HPLC–ESI-MS

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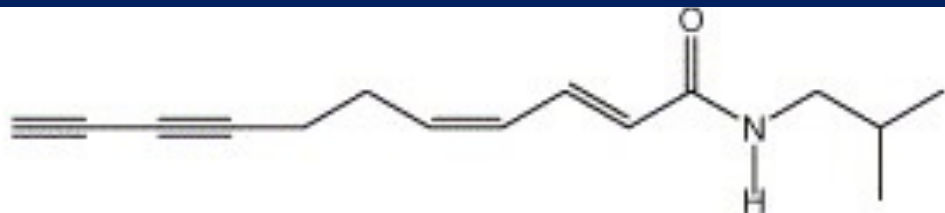
Electrospray

*Echinacea*

### ABSTRACT

*Echinacea purpurea* (L.) Moench, a top selling botanical medicine, is currently of considerable interest due to immunomodulatory, anti-inflammatory, antiviral and cannabinoid receptor 2 (CB2) binding activities of its alkylamide constituents. The purpose of these studies was to comprehensively profile the alkylamide (alkamide) content of *E. purpurea* root, and to compare yields of alkylamide constituents resulting from various ethanolic extraction procedures commonly employed by the dietary supplements industry. To accomplish this goal, a high performance liquid chromatography–electrospray ionization mass spectrometry (HPLC–ESI-MS) method was validated for quantitative analysis of several *E. purpurea* alkylamides. Using this method, at least 15 alkylamides were identified and it was shown that fresh and dry *E. purpurea* extracts prepared from equivalent amounts (dry weight) of roots, with exceptions, exhibited similar yield of specific alkylamides. However, the amount of total dissolved solids in the dry extract

# *Echinacea purpurea*

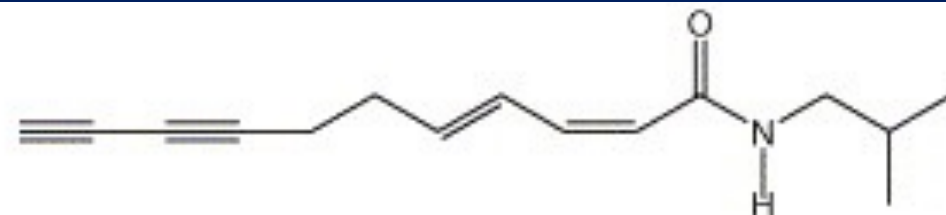


Undeca-2E,4Z-diene-8,10-diynoic acid isobutylamide (3)

$C_{15}H_{19}NO$

Exact Mass: 229.15

Mol. Wt.: 229.32

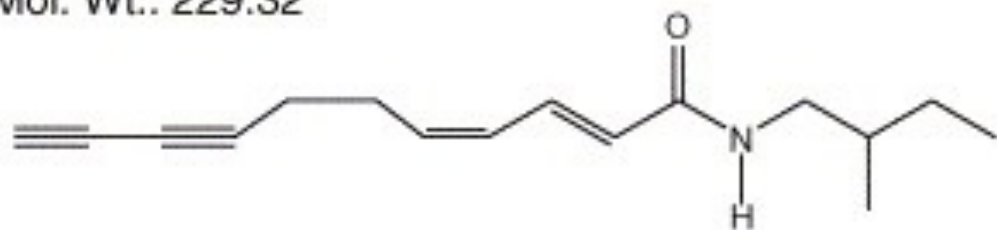


Undeca-2Z,4E-diene-8,10-diynoic acid isobutylamide (4)

$C_{15}H_{19}NO$

Exact Mass: 229.15

Mol. Wt.: 229.32

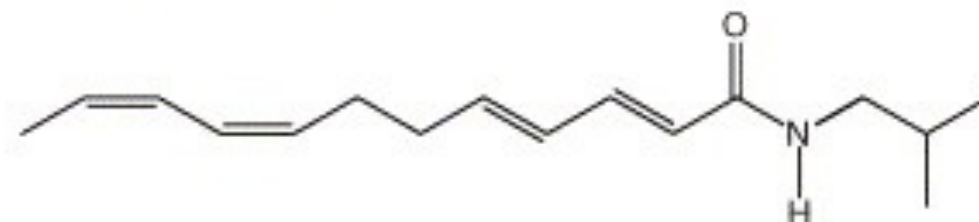


Undeca-2E,4Z-diene-8,10-diynoic acid 2-methylbutylamide (7)

$C_{16}H_{21}NO$

Exact Mass: 243.16

Mol. Wt.: 243.34



Dodeca-2E,4E,8Z,10Z-tetraenoic acid isobutylamide (11)

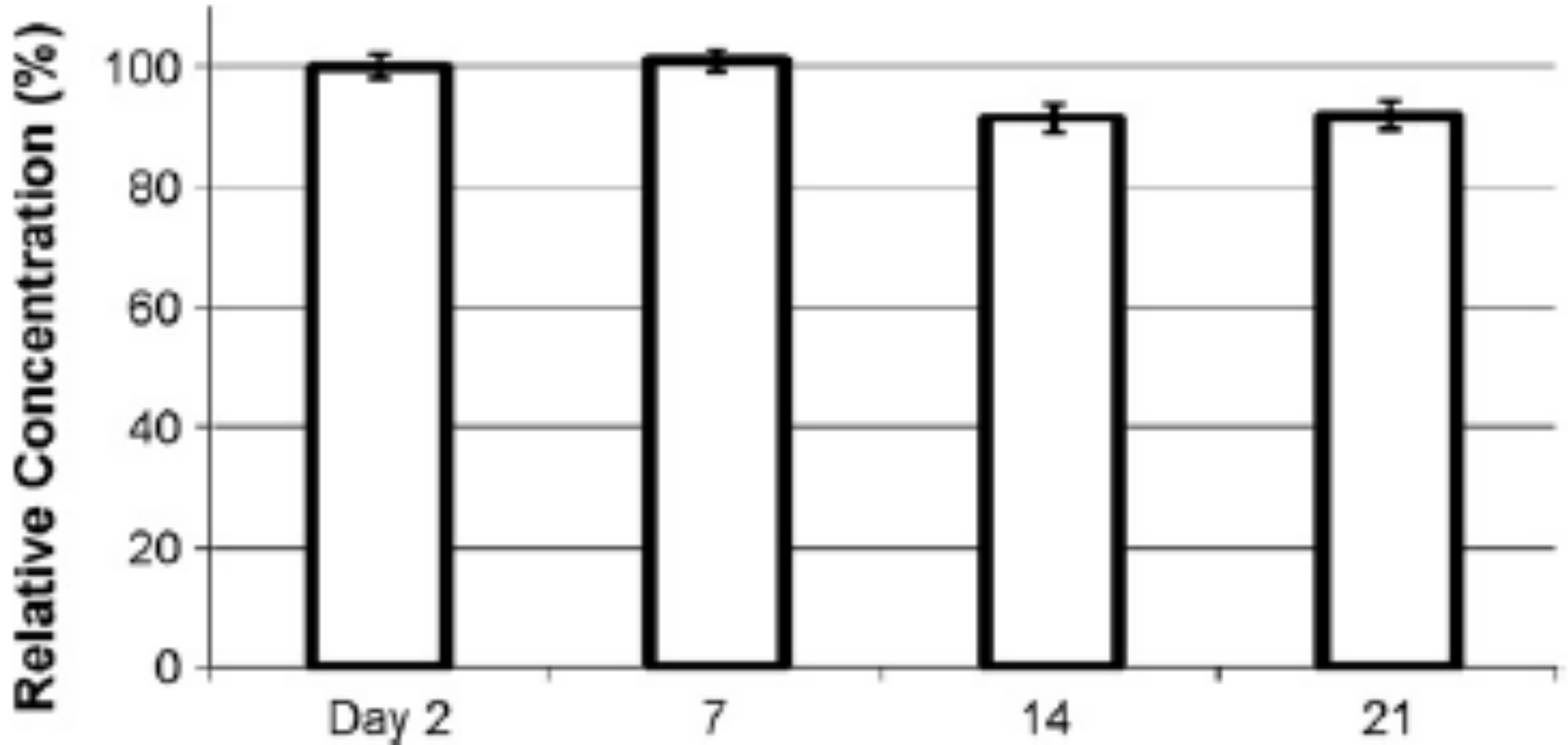
$C_{16}H_{21}NO$

Exact Mass: 247.20

Mol. Wt.: 247.37

# Relative Concentration of Dodeca-tet

Spelman et al., Comparison of alkylamide yield in ethanolic extracts prepared from fresh versus dry *Echinacea purpurea* utilizing HPLC-ESI-MSJPBA 49 (2009) 1141-1149



# Maceration Time

**HOW LONG IS NECESSARY FROM A  
CHEMISTRY PERSPECTIVE?**

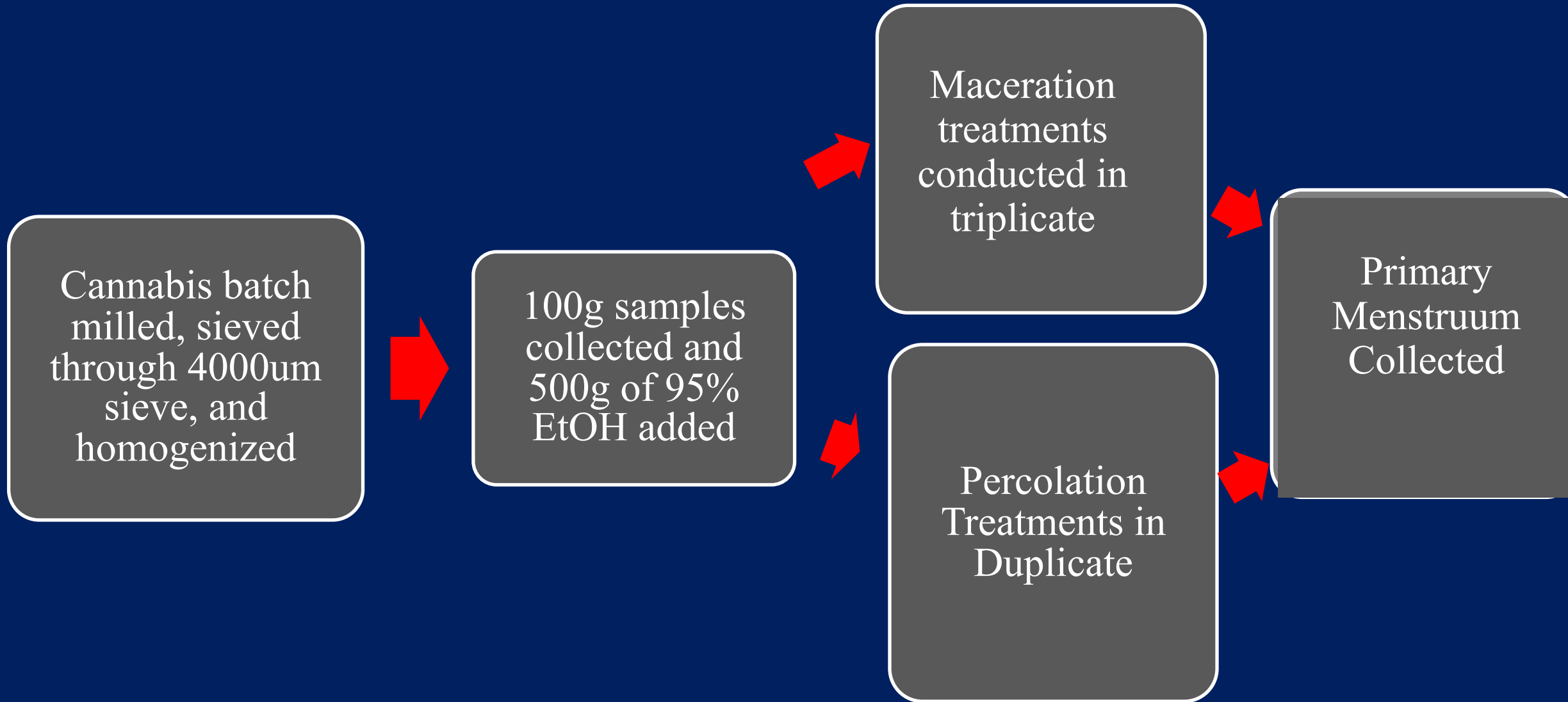
# Hydroethanolic Tincture

<b>% EtOH</b>	<b>THCA</b>	<b>THC</b>
20%	90%	10%
40%	72%	28%
80%	54%	46%



# Applying Maceration Time Studies to Cannabis

# Extraction Flow Chart



# Extraction Flow Chart



# *The Cannabis*



# *Milled Cannabis Flower Potency Characterization*

<b>Biomass Sample</b>	<b>Result</b>
<b>Rep 1</b>	9.36%
<b>Rep 2</b>	8.27%
<b>Rep 3</b>	8.90%
<b>Mean</b>	8.84
<b>STD Deviation</b>	0.55
<b>% RSD</b>	6.19 %
<b>p value</b>	<0.002

# The Grind



# Typical Home Extraction





LABORATORY DOCUMENTATION  
Date: 10/13/18  
Time: 10:00 AM  
Operator: [Name]  
Sample: [Name]  
Location: [Name]  
Equipment: [Name]  
Method: [Name]  
Results: [Name]  
Comments: [Name]  
Signature: [Name]  
Date: [Name]

Normal text = Arial

- 500.08
- 7/30/18
- 8/13/18

RD EX2: 2 weeks

- 100.08
- 500.02
- 7/30/18
- 8/13/18

RD EX3: 1 week

- 100.02
- 500.13
- Start
- End 7/30/18





Ball

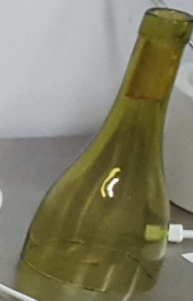
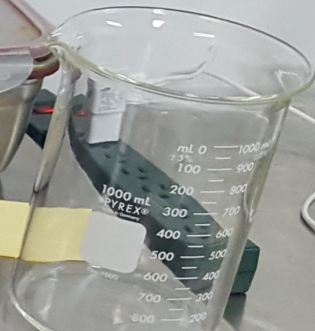
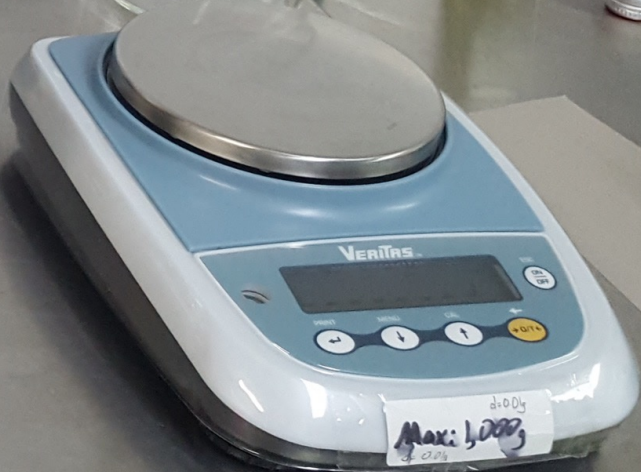
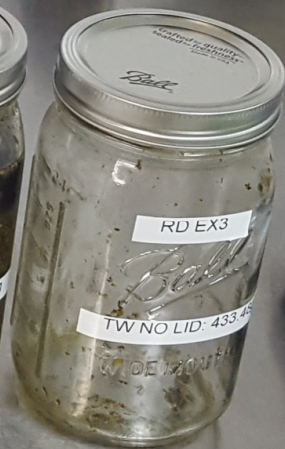
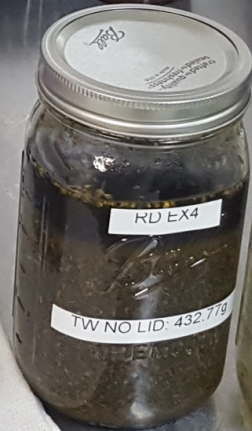
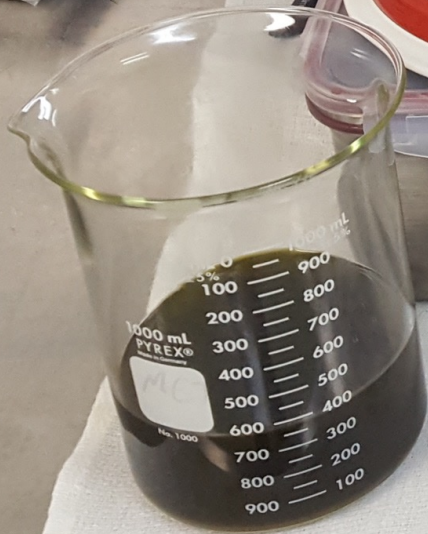
Ball

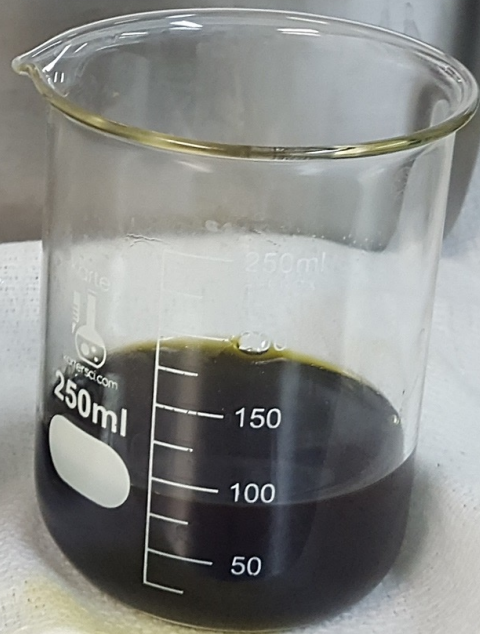
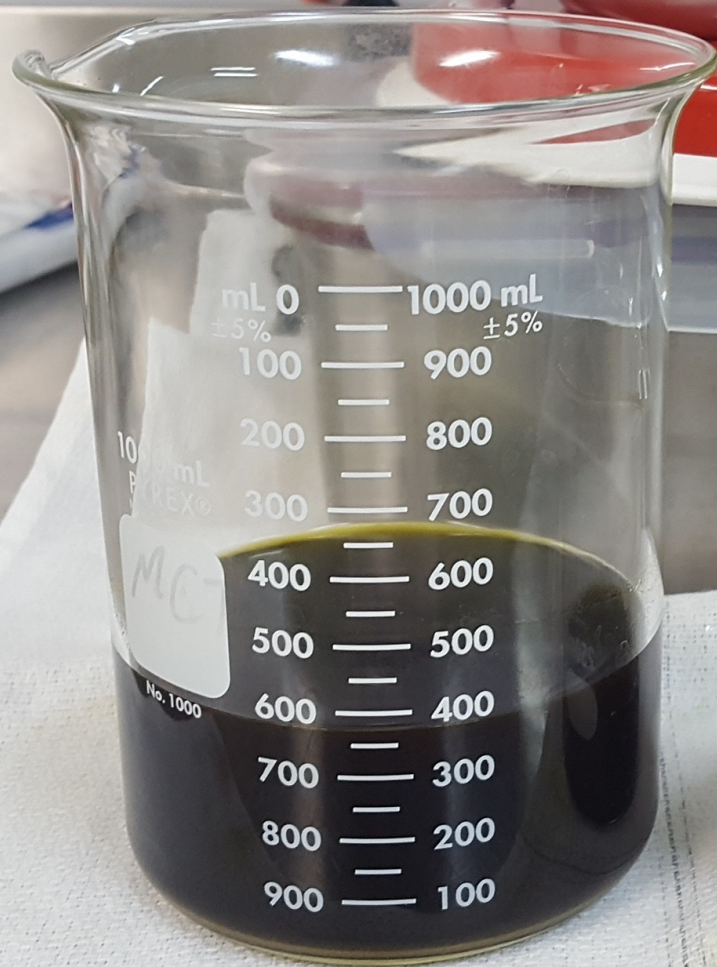
500 mL  
200 mL  
100 mL

Sample ID: \_\_\_\_\_  
Date: \_\_\_\_\_  
Operator: \_\_\_\_\_  
Volume: \_\_\_\_\_  
Concentration: \_\_\_\_\_  
Notes: \_\_\_\_\_

# EJWOX 0.53 Gallon Stainless Steel Soft Fruit Wine Juice Press Cheese Making Press Tincture Press Herbal Press

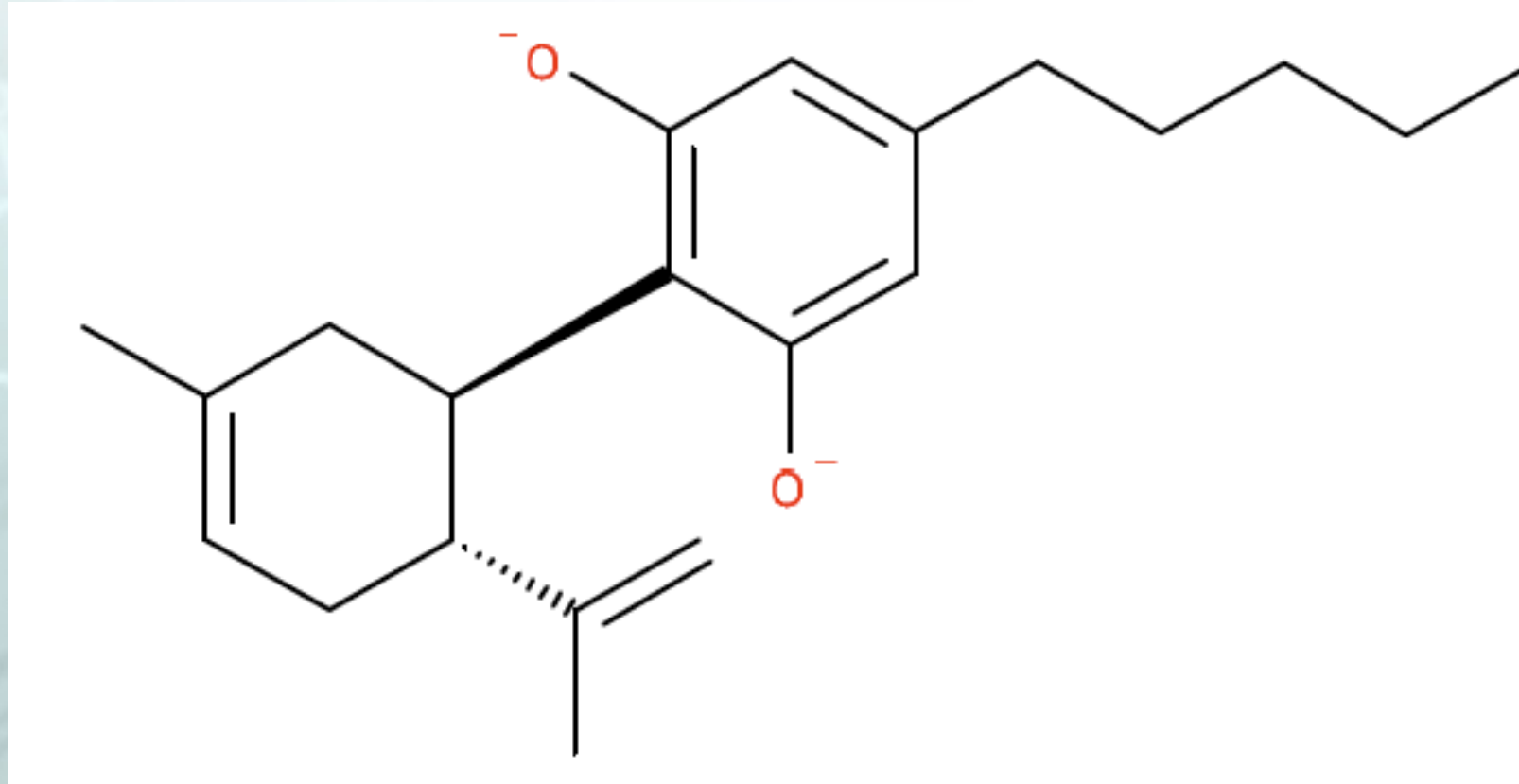




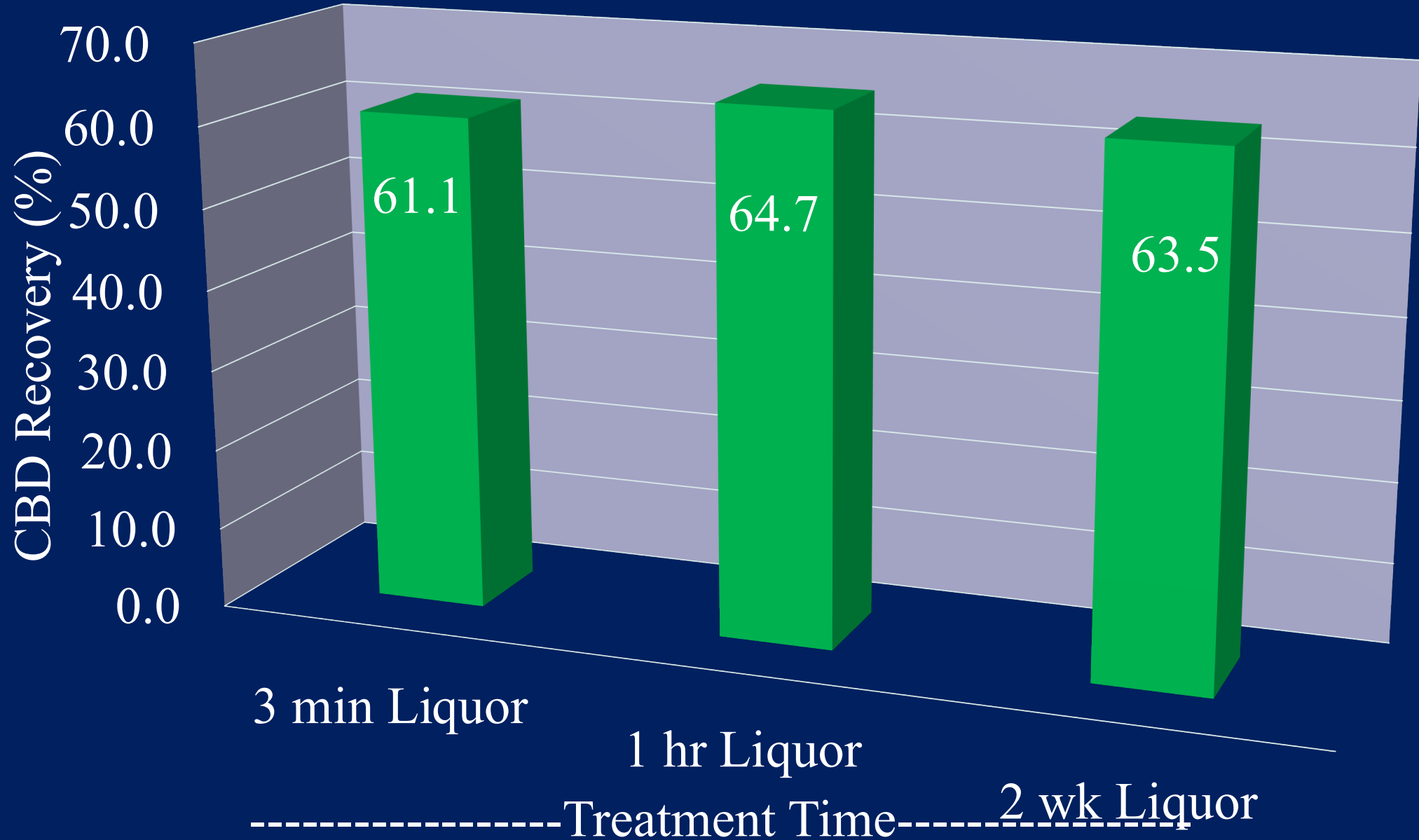


# Cannabidiol (CBD)

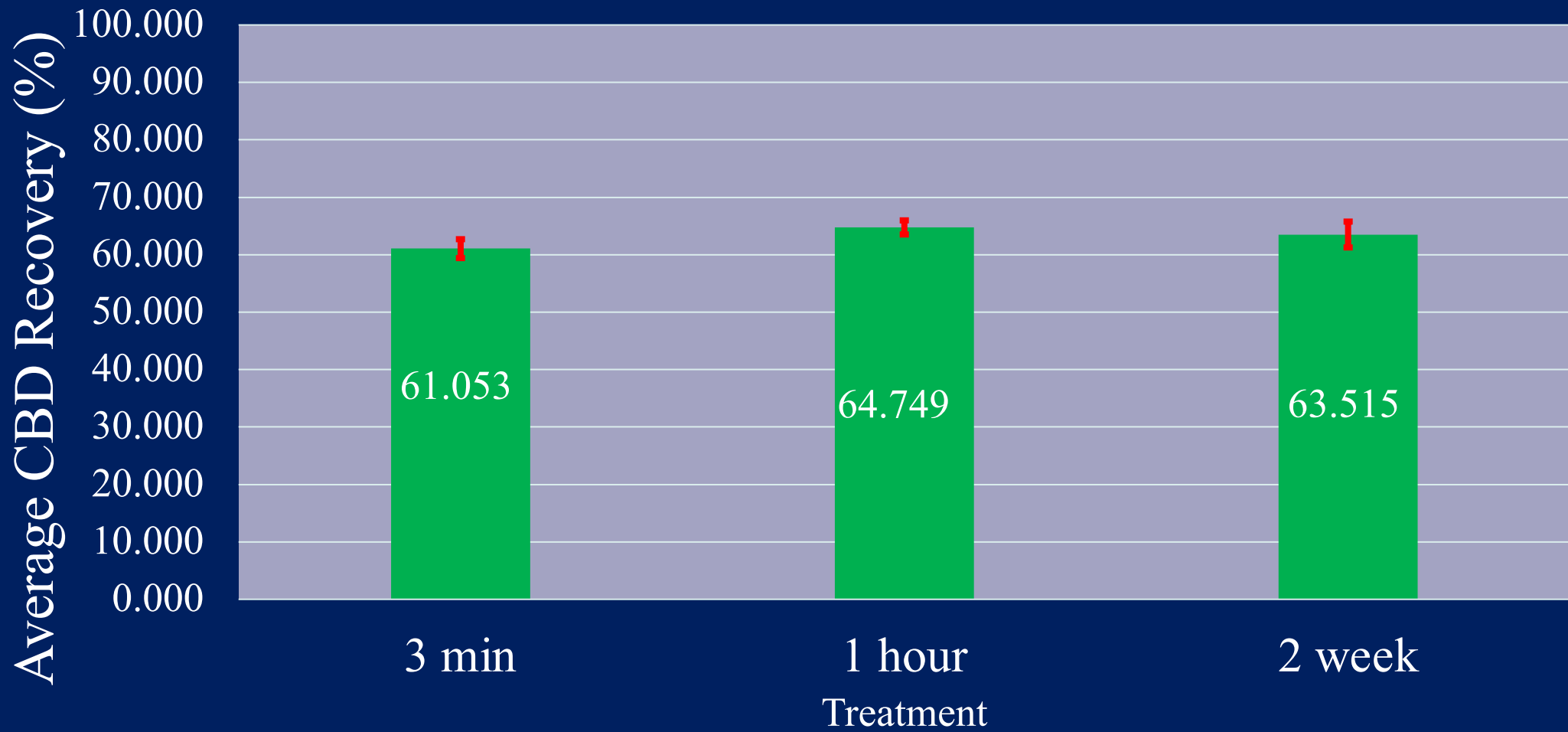
2-[(1R,6R)-6-isopropenyl-3-methyl-cyclohex-3-en-1-yl]-5-pentyl-benzene-1,3-diolate



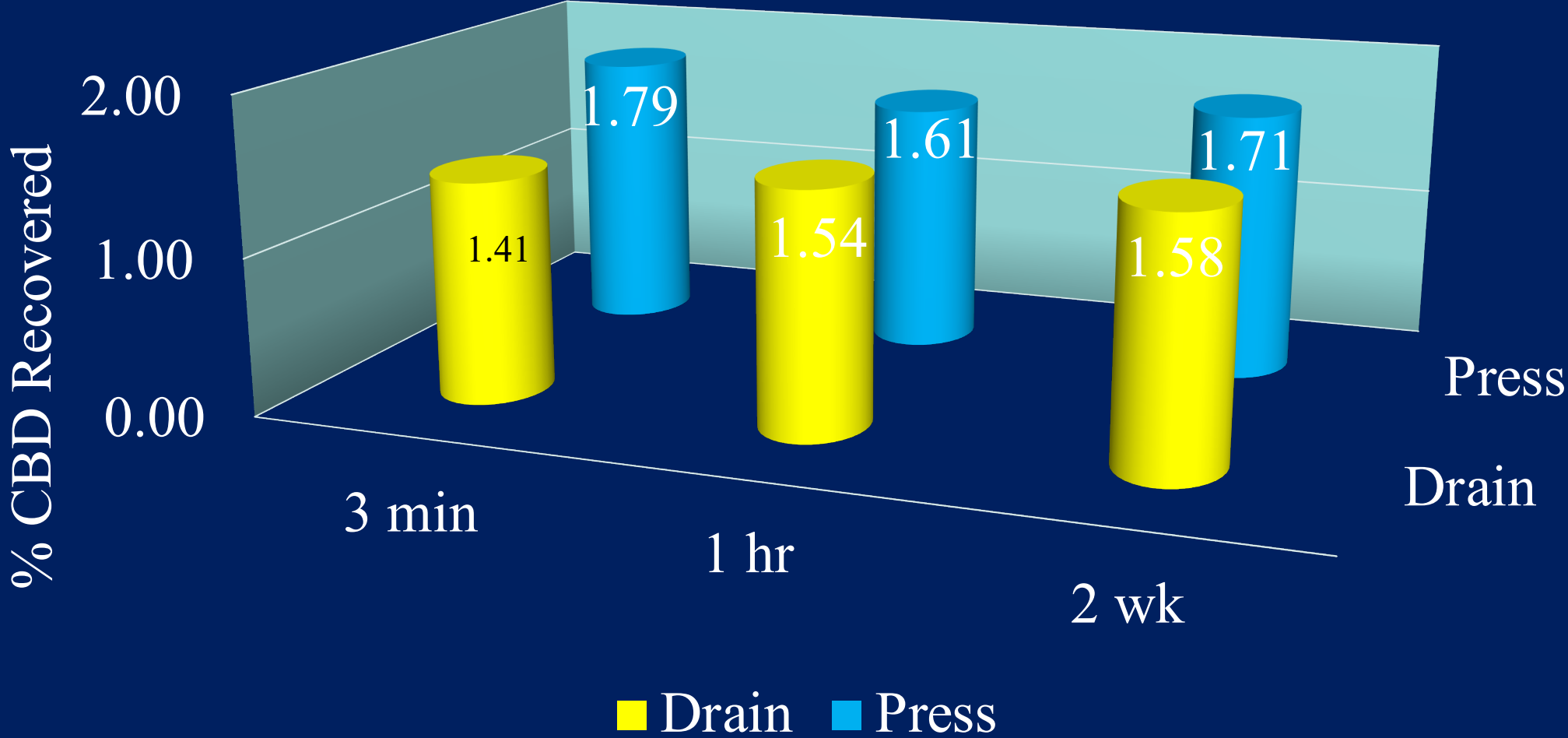
# CBD in Liquor



# Average Percent CBD Recovery in Total Recovered Liquor per Treatment

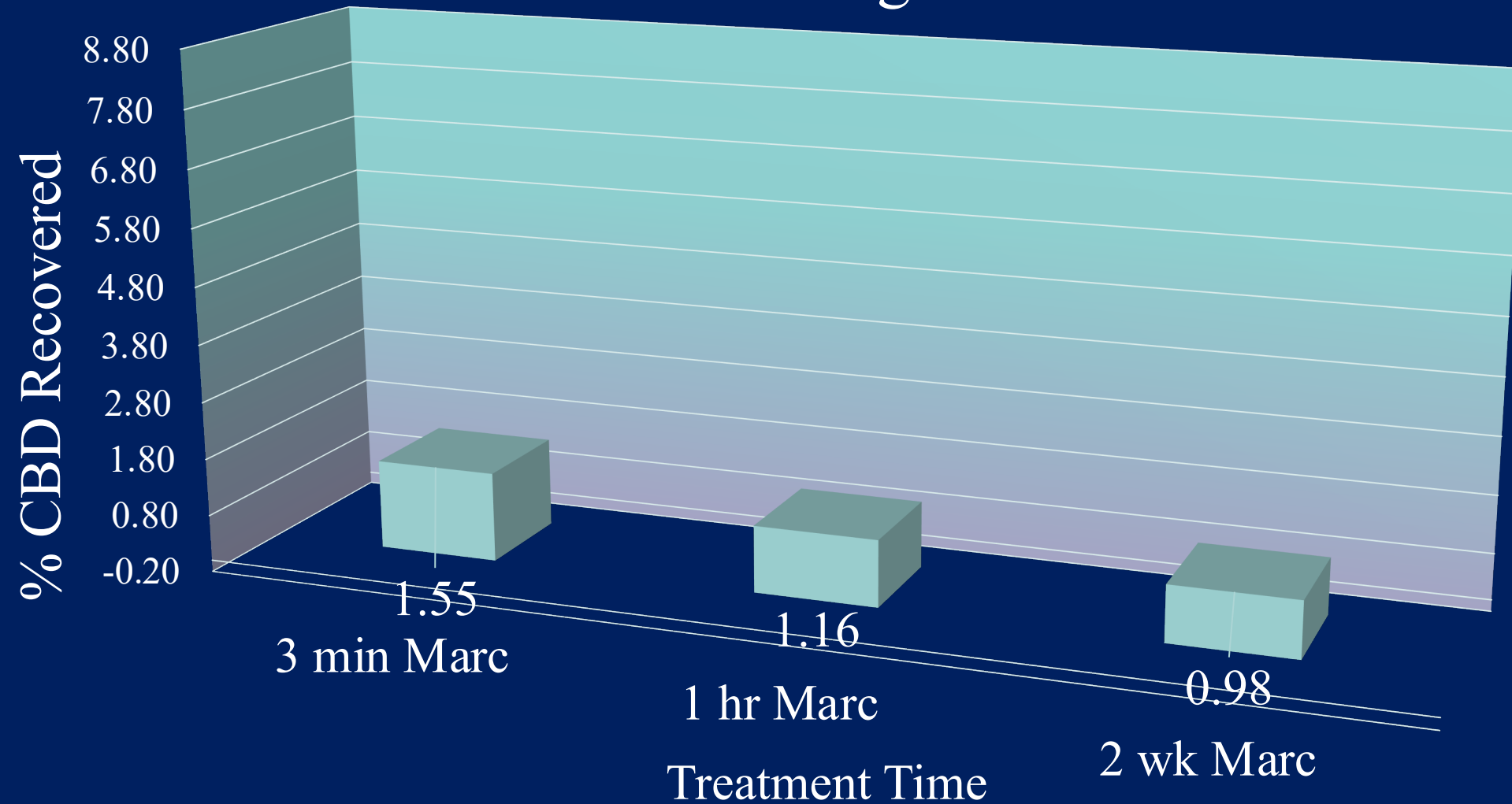


# Drain vs Press CBD Potency (%)





# CBD Remaining in Marc



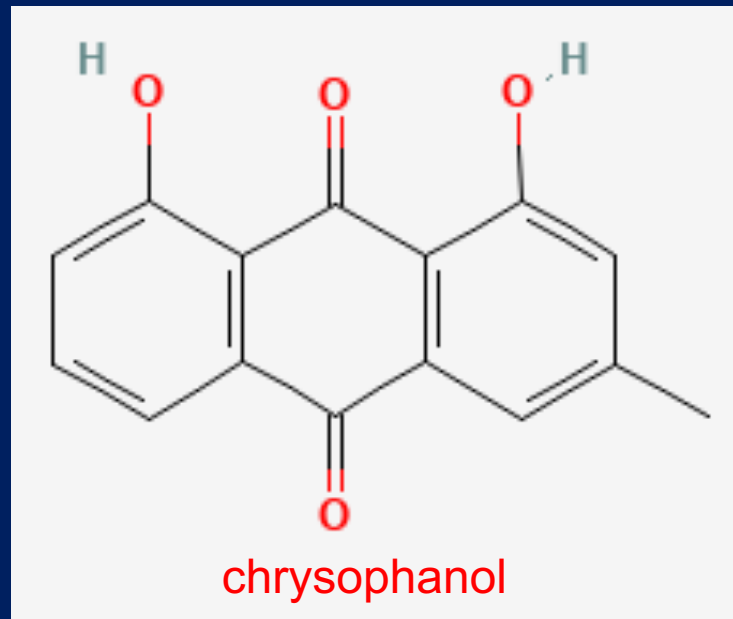
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**Are These Results Supported with Other Data?**

# One hr vs. 8 hr

*Senna* spp.

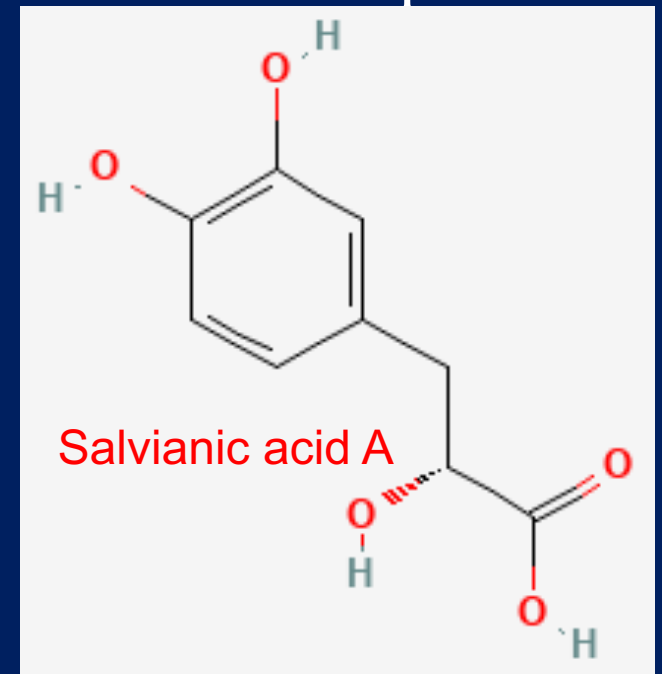
- No significant difference in macerations of seed between the 1 hr and 8 hr time points for the yield of chrysophanol



# One hr vs. 12 hr

## *Salvia miltiorrhiza*

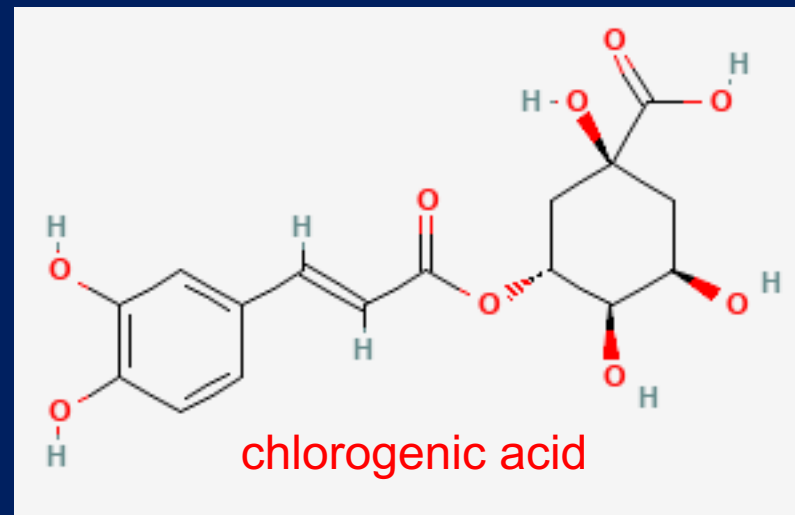
- No significant difference between 1 hr and 12 h maceration for the yield of salvianic acid A in extracts of in both cut and sift herb and powder forms



## 0.5 hr vs. 8 hr

*Lonicera* spp. (honeysuckle)

- 30 minute extraction was no different than an 8 hr extraction for chlorogenic acid yield





## Extraction and HPLC Analysis of Sage (*Salvia officinalis*) Plant

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<sup>\*</sup>**Corresponding author:** Fatma Ebru K, Aksuvital R and D Center, Kavakli Mah, Kuzey Cad, Beylikdüzü, İstanbul, Turkey, Tel: +90 212 671 55 47; E-mail: e.koc@aksuvital.com.tr

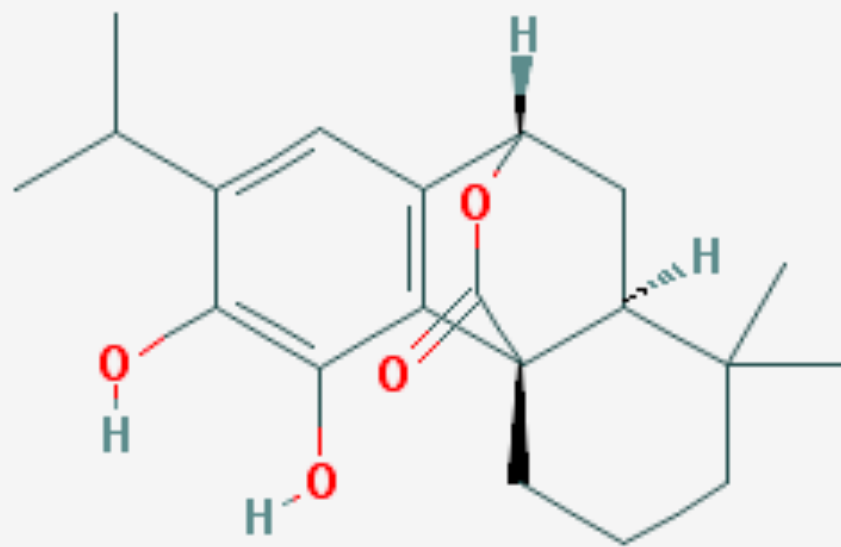
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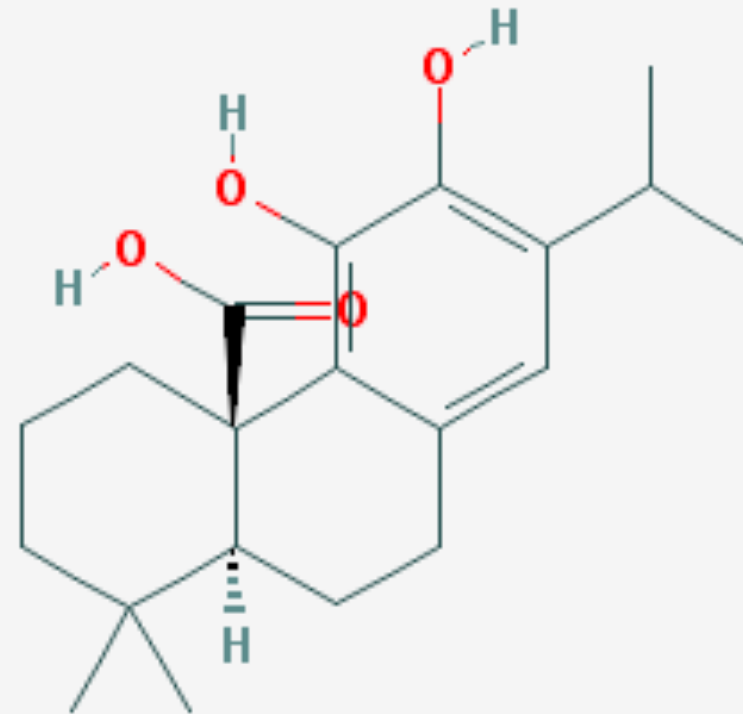
### Abstract

In this study, the sage (*Salvia officinalis*) plant collected from the Mediterranean region was extracted with different solvents and methods. The extract yields were compared. The quantities of rosmarinic acid, carnosic acid and carnosol, which are responsible for the antioxidant capacity of the sage plant, were analyzed qualitatively and quantitatively by an HPLC.

Ground sage plant were sieved with 70 mesh sieves. Maceration were done for powder sage plant at 45°C with 70% ethyl alcohol in "1:6" (w/v) ratio at different time (3; 6; 8; 10 hours); 100% methanol and 100% ethanol extractions; Soxhlet extraction efficiencies were compared. The proportions of rosmarinic acid and the amounts of carnosic acid and carnosol were analyzed in the extracted at UV detector and 280 nm wavelength by a Thermo Scientific Ultimate HPLC instrument. As a result of the experiments, a maceration method with 70% ethanol with 25%



Carnosol



Carnosic acid

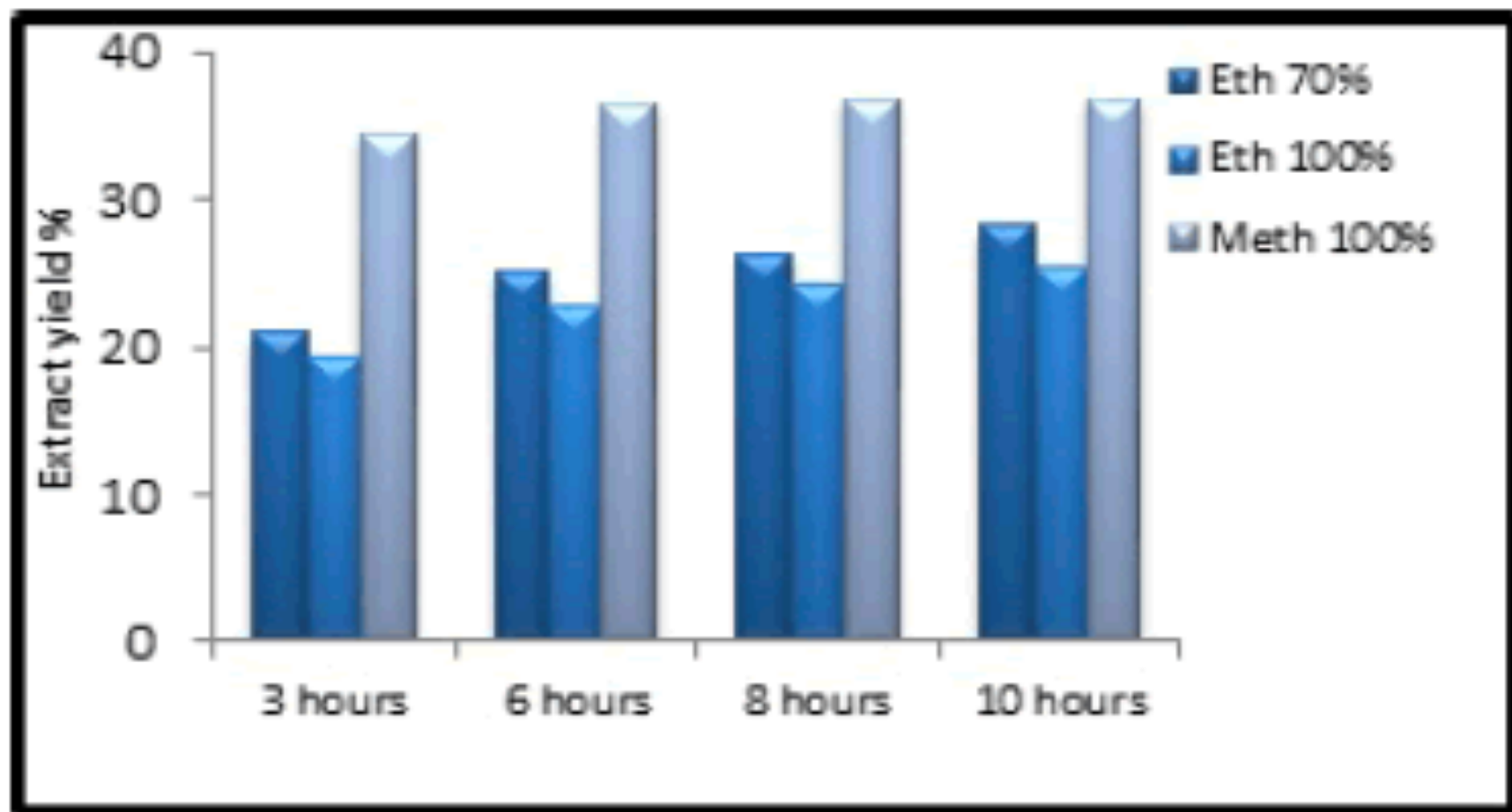


Figure 1: Change of extract yield (%) for maceration.



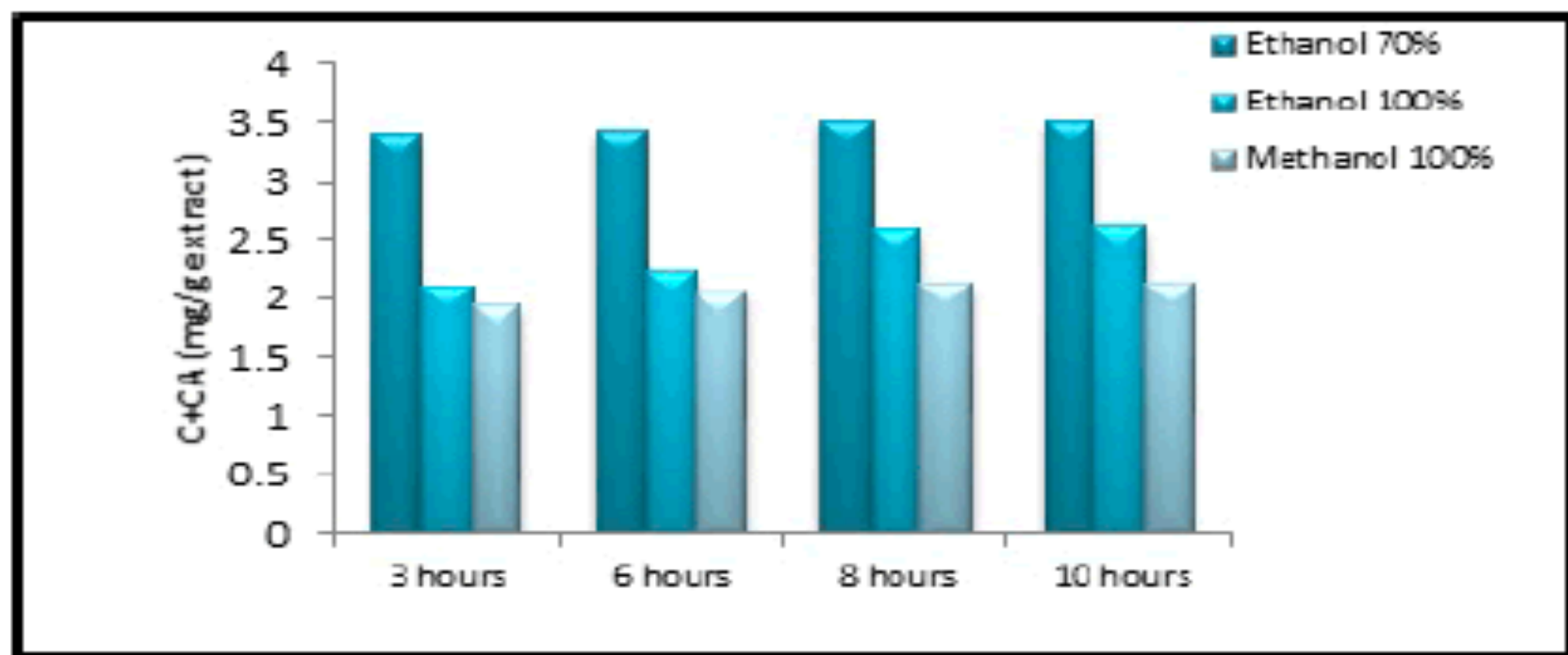
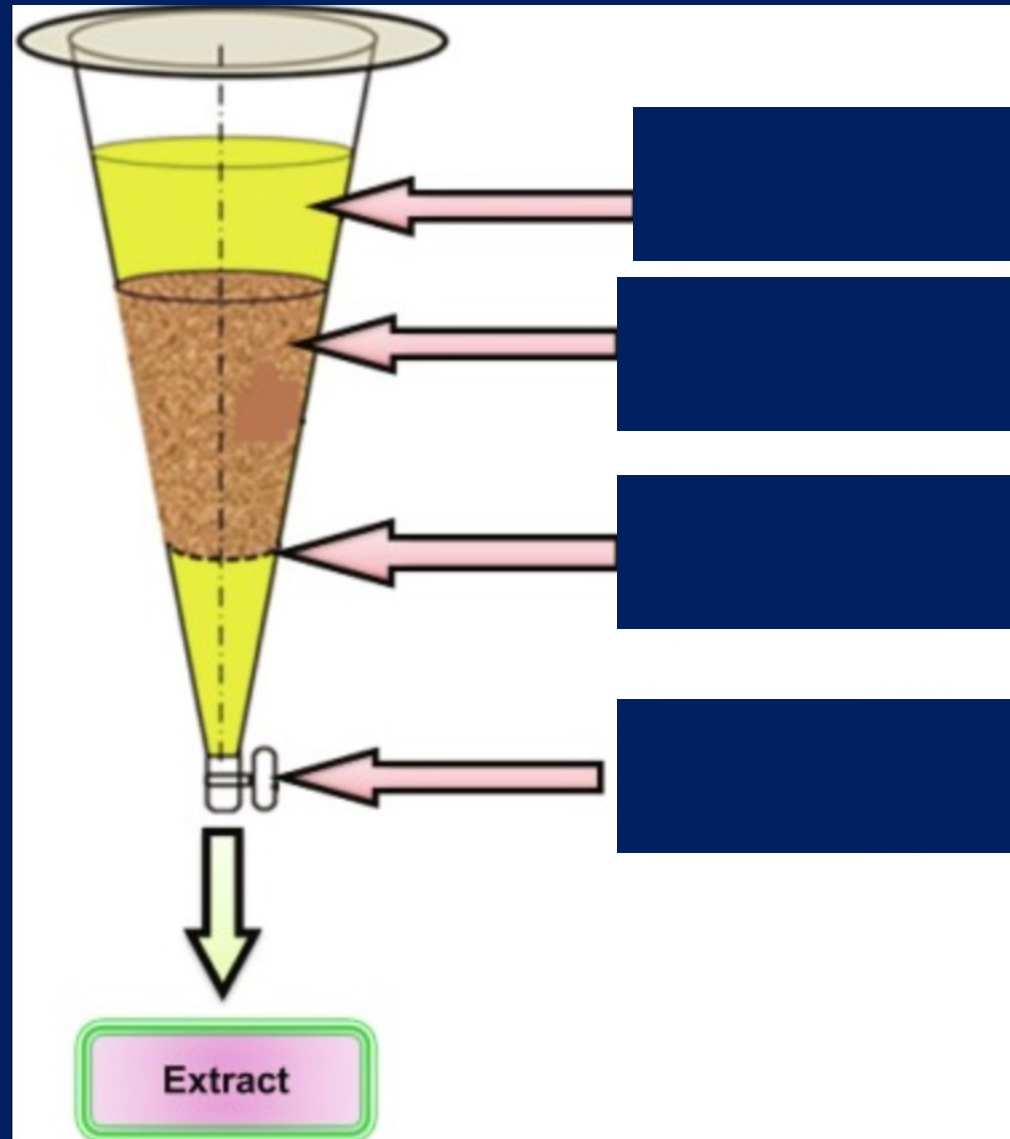


Figure 4: Change of “Carnosol+Carnosic acid” (mg/g ext.) for maceration method.

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# Percolation vs Maceration

Percolation  
Cone



# A Percolation Cone Disguised as a Bottle of Water





**Rhone**



**Burgundy**



**Alsace**



**Bordeaux**



**Sparkling**



Coarse grind of biomass

Moist but  
not Muddy



# The Pack

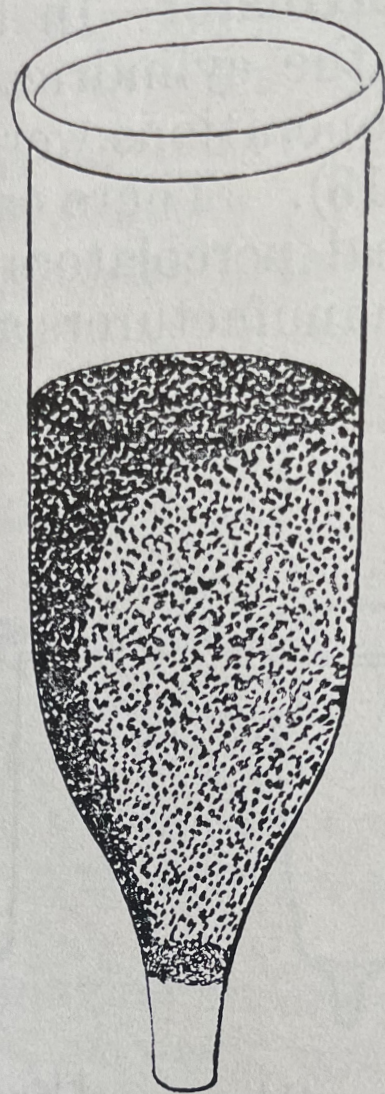


Fig. 423—Improperly packed percolator.

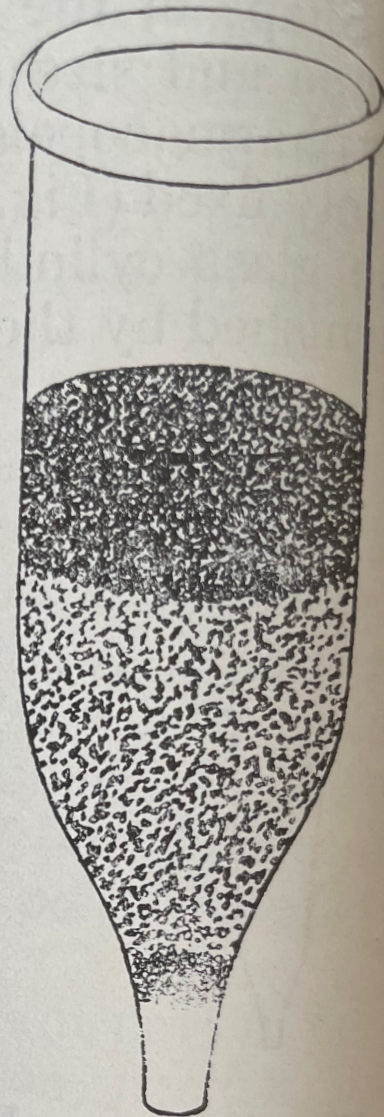
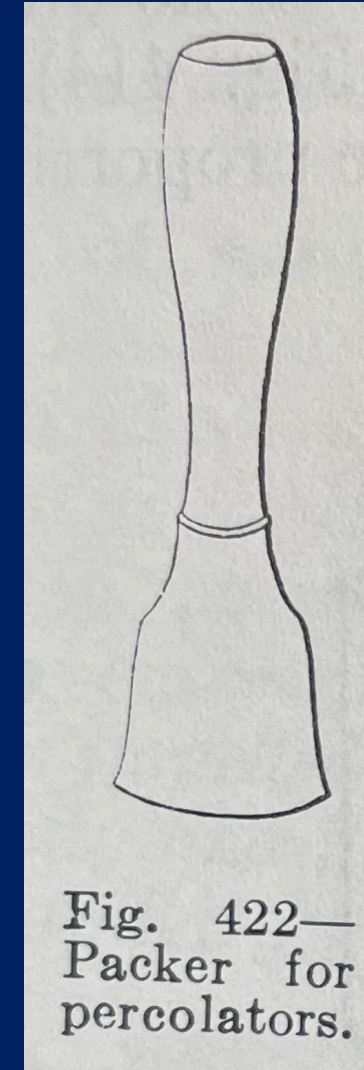
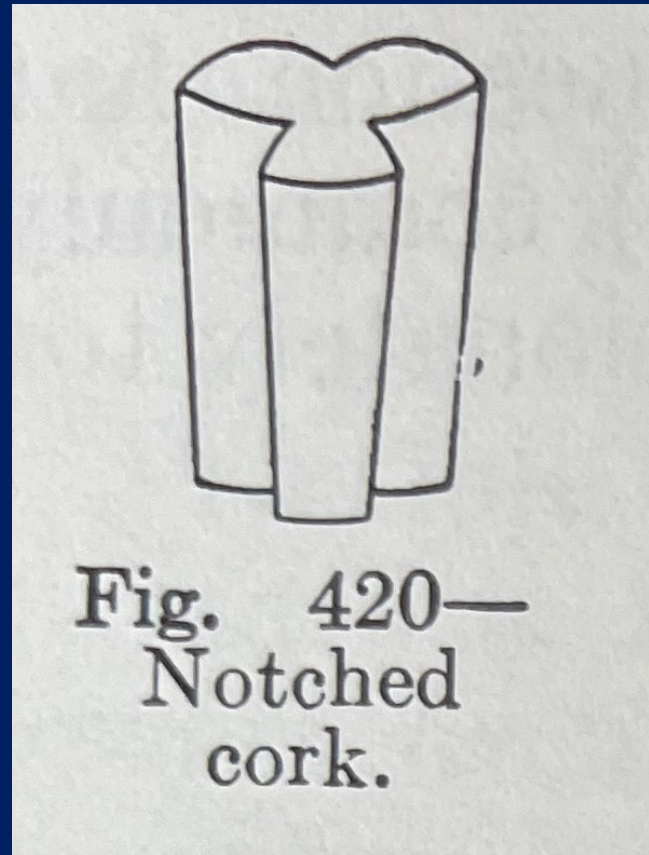
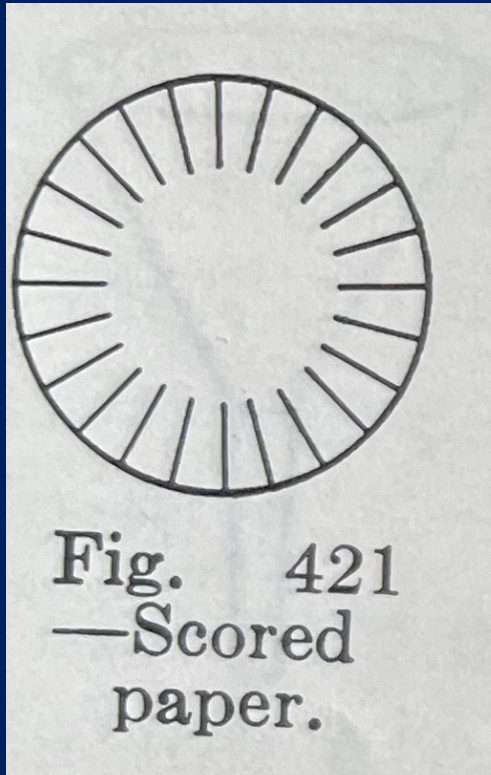


Fig. 424—Properly packed percolator.



# Tools to Consider









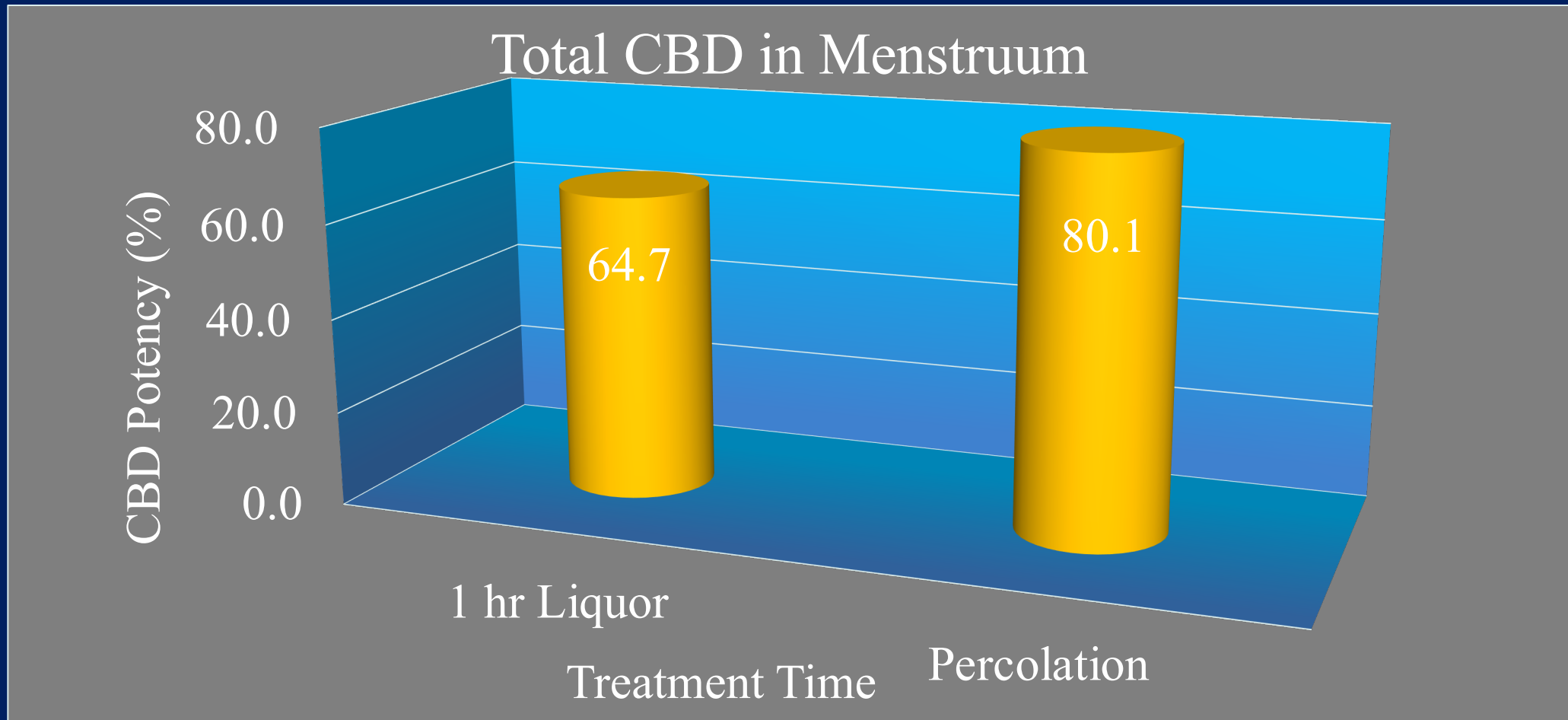
# CBD Recovery Efficiency (% Recovery) per Extraction Treatment

Treatment	Starting Solvent Mass (g)	Drain Liquor Mass (g)	Press Liquor Mass (g)	Total Liquor Mass (g)	Liquor Recovery %
Perc - 1	269.000	205.000	0.000	205.000	76.208
Perc - 2	269.000	199.502	0.000	199.502	74.164

# CBD Recovery Efficiency (% Recovery) per Extraction Treatment

Treatment	Total Liquor CBD Recovery (%)	Drain CBD Recovery (%)	Press CBD Recovery (%)
Perc -	79.277	79.277	0.000
Perc - 2	80.925	80.925	0.000

# Macerations Vs. Percolation



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**Are These Results Supported with Other Data?**



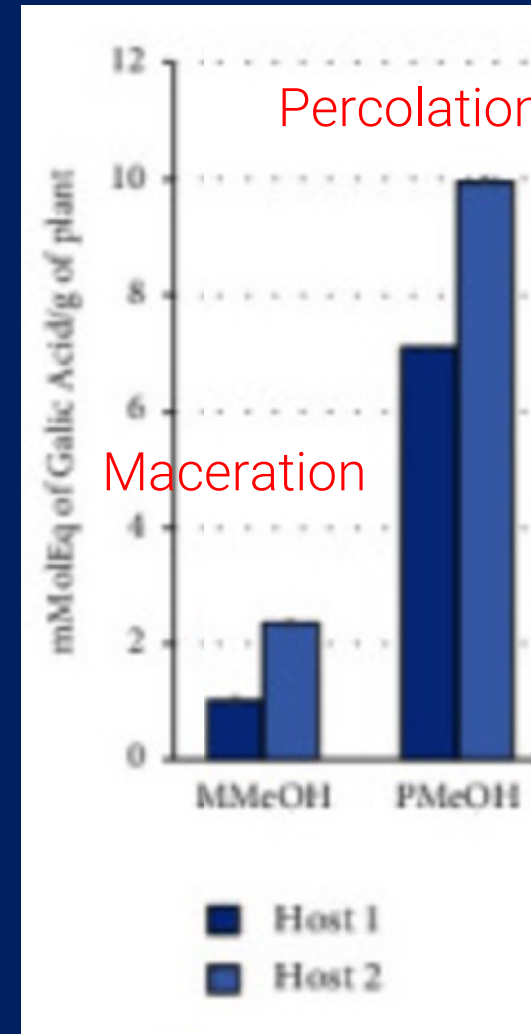
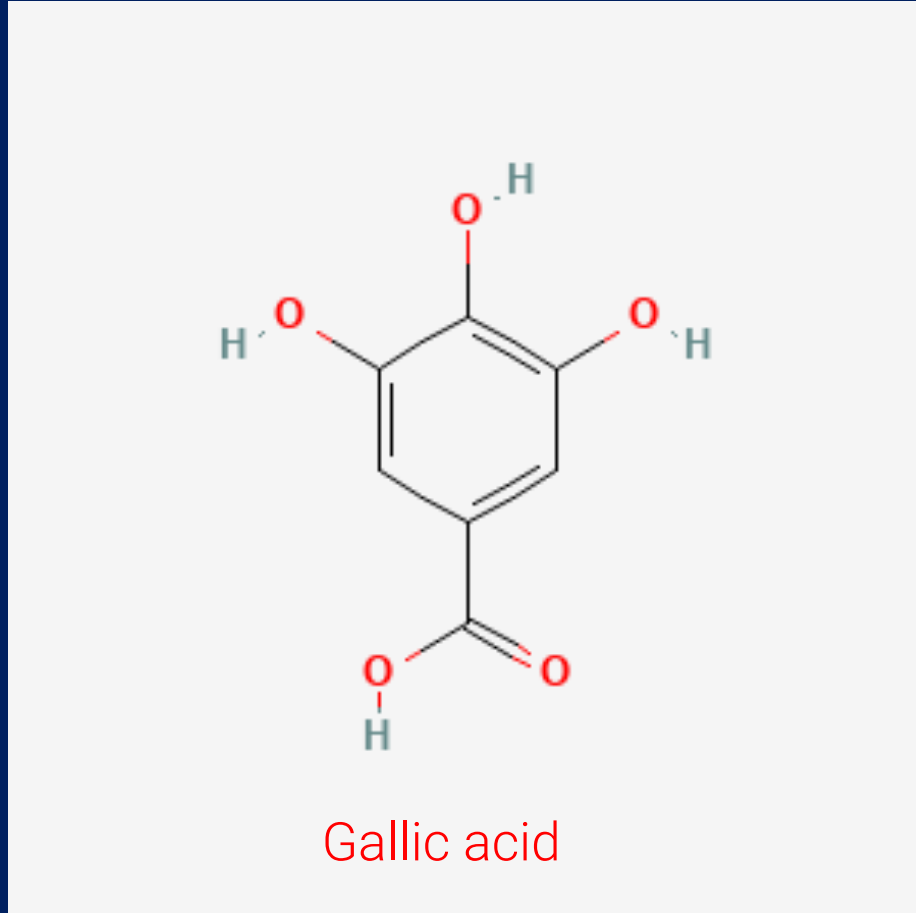
Hindawi  
Evidence-Based Complementary and Alternative Medicine  
Volume 2022, Article ID 9679739, 9 pages  
<https://doi.org/10.1155/2022/9679739>

*Research Article*

# **In Vitro Inhibitory and Proliferative Cellular Effects of Different Extracts of *Struthanthus quercicola*: A Preliminary Study**

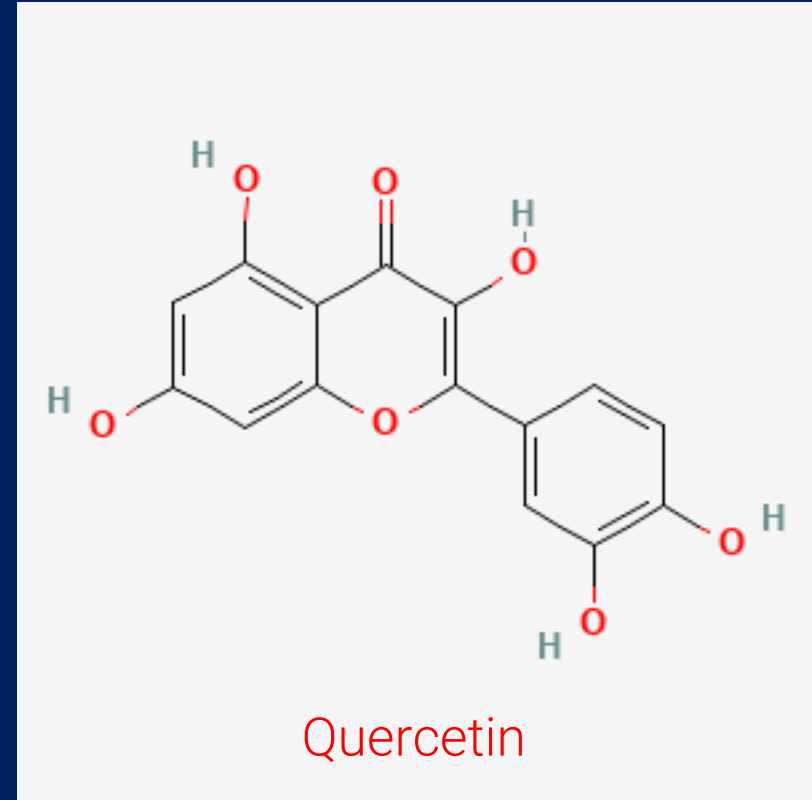
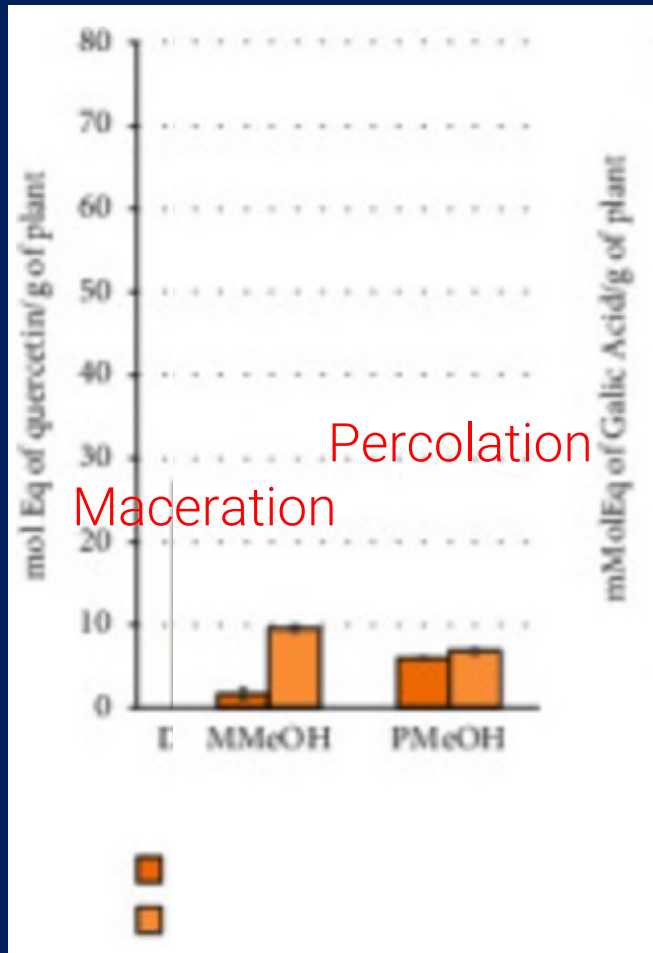
**Luz Eugenia Alcántara-Quintana,<sup>1</sup> Carely Arjona-Ruiz,<sup>2</sup> Denisse de Loera,<sup>2</sup>  
Rubí Gamboa-León,<sup>3</sup> and Yolanda Terán-Figueroa <sup>4</sup>**

# *Struthanthus quercicola*: Total Phenolic Extraction



Alcántara-Quintana LE, *et al.* In Vitro Inhibitory and Proliferative Cellular Effects of Different Extracts of *Struthanthus quercicola*: A Preliminary Study. *Evid Based Complement Alternat Med.* 2022 Apr 13;2022:9679739.

# *Struthanthus quercicola*: Total Flavonoid Extraction



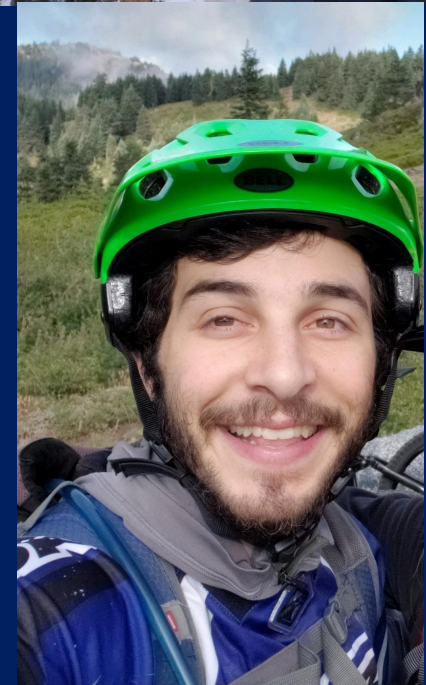
Alcántara-Quintana LE, *et al.* In Vitro Inhibitory and Proliferative Cellular Effects of Different Extracts of *Struthanthus quercicola*: A Preliminary Study. *Evid Based Complement Alternat Med.* 2022 Apr 13;2022:9679739.

# Future Directions

- Quick wash comparison to short maceration
- Percent Ethanol
  - 70% vs 100%
  - Fresh plant vs Dry plant
- Hot EtOH vs Cold EtOH vs RT EtOH
  - In Percolation
  - In Maceration
  - In Quick Wash

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- Research Team
- Jason Wilson, MS - Chemical Analysis
  - From U Miss
- Travis Simpson, BS - Extraction
  - From Bastyr University





Thank You for your attention!

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