MASTERING MENSTRUUM'S IN HERBAL EXTRACTS

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NUNM

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- Herbalist since 1984, Registered Herbalist with American Herbalist Guild.
- Graduate of NCNM in 1993
- Licensed Naturopathic Doctor since 1993 and currently with the State of Oregon
- Former Associate Professor of Botanical medicine at National University of Natural Medicine in Portland
- Former Assistant Professor at Bastyr University, in Kenmore Washington
- Lead Naturopathic Physician with Herb Pharm and adjunct at NUNM
LEARNING OBJECTIVES

• To understand what is a menstruum and its importance in herbal extractions
• To understand the role of maceration and percolation on the production of herbal extracts
• To understand the importance of the selection and percentage of alcohol in herbal extracts
• To understand the complex botanical matrix of herbal material
• To understand the clinical implications of fresh verses dry herbal extracts and the importance of extraction ratios
• To understand the overview of how to make herbal extracts

WHAT ARE LIQUID EXTRACTS?

• Extracts are hydro-ethanol based which is alcohol and water and herbal extractives
• Extracts can be made from glycerin and water
• Generally, each herb is unique and needs between 25 to 95% EtOH to extract and preserve
• The Extracts are manufactured and sold as dietary supplements and regulated by the FDA
WHY USE LIQUID EXTRACTS

**Advantages:**
- Quickly and easily formulate an individual medicine for each patient using tinctures
- They have a long shelf life requiring no unusual conditions (2-5 years), take up little shelf space, can usually be made palatable, are easy to procure or produce, and easy to combine and dispense
- No difficult preparation on their part is needed, increasing compliance
- Alcohol and water are the best two solvents for most important plant constituents
- Absorbed quickly into the stomach and in blood stream quickly

WHAT FORM: COMPARING QUALITIES OF LIQUID EXTRACTS & CAPSULES & TABLETS

<table>
<thead>
<tr>
<th></th>
<th>Liquid Extracts</th>
<th>Capsules</th>
<th>Tablets</th>
</tr>
</thead>
<tbody>
<tr>
<td>Engage Senses</td>
<td>No taste, smell</td>
<td>No taste, smell</td>
<td>No taste, smell</td>
</tr>
<tr>
<td>Easy to take</td>
<td>Portable (won’t spill)</td>
<td>Portable (won’t spill)</td>
<td>Portable (won’t spill)</td>
</tr>
<tr>
<td>Quick absorption</td>
<td>Must be digested</td>
<td>Must be digested</td>
<td>Must be digested</td>
</tr>
<tr>
<td>Easy to adjust dosage</td>
<td>Dosage fixed</td>
<td>Dosage fixed</td>
<td>Dosage fixed</td>
</tr>
<tr>
<td>Alcohol based (or gly)</td>
<td>Alcohol free (mostly)</td>
<td>Alcohol free</td>
<td>Alcohol free</td>
</tr>
<tr>
<td>No fillers</td>
<td>Often Fillers</td>
<td>Often fillers and binders</td>
<td>Often fillers and binders</td>
</tr>
</tbody>
</table>
WHAT IS A DIETARY SUPPLEMENT?

• A vitamin, mineral, herb or other botanical, amino acid, a dietary substance used to supplement the diet, or is a concentrate, metabolite, constituent, extract or combination thereof
• Orally administrated
• Labeled as a Dietary Supplement
• And is not an antibiotic, drug, or investigational new drug
• May have structure / function claims, cannot treat disease or illness
• Complies with all related regulations
• Dietary Supplement Health and Education Act (DHSEA 1994)
• Herbal Extract are dietary supplements

GMP AND QUALITY

• Good Manufacturing Practices (GMP)
• Quality means that the dietary supplement
  • Consistently meets the established specifications for identity, purity, strength, and composition, and limits on contaminants, and
  • Has been manufactured, packaged, labeled, and held under conditions to prevent adulteration
  • As under section 402(a)(1), (a)(2), (a)(3), and (a)(4) of the act.21 CFR 111.3 c GMPs for Dietary Supplements
TYPE OF HERBAL EXTRACTS
METHODS

• Two common methods for making herbal extracts
• The Folk Method (the traditional healer method)
  • Traditional herbalist method
  • Eyeball everything, like good cook
  • Can have inconsistent results but traditional
• The Master Manufacturing Record method (Scientific method)
  • Required for all commercially sold extracts
  • Very exact and reproducible but require equipment, time

HERBAL BUMPER STICKER
JOKE

Herbalism is all Folked Up!
COMMON STEPS FOR MAKING HERBAL EXTRACTS

• Quality Herb, Assay, Testing, Organoleptic's
  • Fresh or Dry and Why?
• Milling and Grinding
  • Type of mill
  • Communion to right size
• Extraction Method
  • Percolation or Maceration
  • Pro and Cons
• Menstruum's and why
  • Alcohol Percentage
  • Ratio
  • Water
  • Glycerin
• Pressing / Filtering / Bottling

NOT ALL HERBS ARE CREATED EQUALLY

Herbal considerations...
Growing & sourcing
Harvest timing & plant part selection
Extraction timing & methods
Identification, purity & potency
Pick Well...
Sensory Evaluation & Microscopy

FOLK AND MMR METHODS

Quality Testing: Limits

- Yeast & mold
- Bacteria
- Pesticides
- Herbicides
- Fungicides
- Heavy metals
- MMR METHOD ONLY
Batch to Batch Constancy

Potency and ID

• MMR METHOD ONLY

FRESH PLANT EXTRACTS

• Made from freshly harvested, undried herbs
• The water present in fresh plants will contribute to the water component of the menstruum
• This must be taken into account when calculating the total volume of menstruum, and the amount of water and alcohol to use
• Fresh herbs must be quickly extracted once harvested
• Fresh herbs rapidly deteriorate unless quickly processed
DRY PLANT EXTRACTS

• Only dry herbs can be used with percolation
• Dry herbs have minimal water (8-12%)
• Can be concentrated to stronger herb / menstruum ratio
• Potential loss of volatiles with drying
• Another step needed with drying

FRESH PLANT EXTRACTS VS DRY HERB
EXTRACTS: SUMMARY

Fresh Plant
• Less processing (no drying)
• Raw material fragile and must be processed quickly
• Contains water
• Good for high essential oils plants
• Fragile plants

Dry Plant
• Raw material shelf stable
• Can be made in a more concentrated herb menstruum ratio
• Good for stable plant parts
WHICH IS BEST FRESH HERB OR DRY HERB?

It all depends on the details and specific Herb. The Answer is Both!

FRESH LEMON BALM (MELISSA OFFICINALIS)

- Fresh Lemon Balm (Leaf and Flower)
- Maceration Method
- 75% EtOH
- Yield: 45% EtOH in final extract
DRY ROOT GOLDENSEAL 
(*HYDRASTIS CANADENSIS*)

- Dry root Goldenseal
- 60% EtOH
- Maceration or Percolation

COMMON STEPS FOR MAKING HERBAL EXTRACTS

- Quality Herb, Assay, Testing, Organoleptic’s
  - Fresh or Dry and Why?
- Milling and Grinding
  - Type of mill
  - Commutation to right size
- Extraction Method
  - Percolation or Maceration
  - Pro and Cons
- Menstruum’s and why
  - Alcohol Percentage
  - Ration
  - Water
  - Glycerin
- Pressing / Filtering / Bottling
**COMMINUTION**

- **Comminution** is the reduction of solid materials from one average particle size to a smaller average particle size, by crushing, grinding, cutting, vibrating, or other processes.
- Objective is to obtain a particular particle diameter: coarse to finely powdered.
- Pharmacopeias use sieve mesh sizes to describe particle size.
- The smaller the particle size, the more rapid the extraction.

**Commercial Hammer Mill**

**COMMINUTION PROCESS**

- Secondary Inspection of plant material - removal of foreign solids while loading mill.
- Shredding with plant cutters (blades).
- Grinding with hammer mills, pin mills.
- Grinding with vertical cut mixers (VCM).
- Coarse material requires a longer extraction time.
- Folk method uses hands or hand tools and kitchen tools.
- MMR method uses electronic mills and grinders, commercial grade.
COMMINUTION RESULTS

Folk Method

Commercial Mill

COMMON STEPS FOR MAKING HERBAL EXTRACTS

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EXTRACTION PRINCIPLES

• **Rinsing** - dissolving phytochemicals from disintegrated cells on surface of plant powder - fast

• **Diffusion** - permeation of solvent into intact cells, dissolution of phytochemicals in cells, diffusion of mixture out of cells - slow

• **Leaching** - extracting with Alcohol and water as solvent

FACTORS THAT AFFECT EXTRACTION

• Herb : menstruum ratio

• Menstruum composition (alcohol to water ratio)

• Surface area - the finer the powder, the faster the drug is washed off the surface

• Extraction time

• Temperature

• pH (for aqueous systems)

• Interaction between dissolved materials and structural material (cellulose)
FACTORIAL DESIGN OF EXTRACTION

• Hold alcohol and time constant, vary temperature - determine best yield
• Choose temperature of best yield, vary alcohol content - determine best yield
• Choose temperature and alcohol of best yield, vary time - determine best yield
• This gives best menstruum composition, time and temperature for highest yield

HERB : MENSTRUUM RATIO

• The Herb : Menstruum ratio gives one part herb to volume of menstruum, which gives us the relative strength of the extract
• At a ratio of 1 : 5 herb to menstruum
  • 1 gm herb needs 5 ml of menstruum
  • Or a 20% strength
• Then calculate the menstruum components
  (ex. 60 % EtOH + 40% water = Menstruum)
• Common Ratios: 1/1 to 1/5
• Historical ratios from older US Pharmacopeias
COMMON EXTRACTION PROCESS TYPES

MACERATION: TO SOAK

- Maceration - extraction of phytochemicals at room temperature with a solvent, shaking or stirring daily
- Kinetic maceration - extraction at room temperature while stirring continuously
- Digestion – maceration extraction at higher temperature (40 - 50°C)
- Digestion is common with fixed oils extracts
MACERATION KINETICS

- Increase of mixing and/or time increases yield of phytochemicals
- Increase in temperature may increase yield, but may also denature active constituents
- Change in polarity (e.g. adding alcohol) may improve yield
- Addition of acids or bases influences yield (e.g. adding vinegar)

PERCOLATION

- Herb is powdered
- Pre-swelling of shredded herb
- 24 hours maceration in a closed percolator (column)
- Fresh menstruum is dripped onto the column until herb is exhausted
POTENTIAL PROBLEMS WITH PERCOLATION

- Can have pockets of dry herb that are not extracted
- If herb swells too much, occludes the flow
- Mucilage and pectin containing herbs plug column
- If herb is packed unevenly, channels form in column
- Can only use with dry herbs

EXTRACTION METHODS FOR HOME USE

- Percolation
- Maceration
HOME PERCOLATION

- Used white or red wine bottle
- Use special glass cutter to cut off bottom and sand smooth
- Filter paper
- Rubber cork and stopcock

COMMON STEPS FOR MAKING HERBAL EXTRACTS

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WHAT IS A MENSTRUUM?

**Herbal**

- A menstruum is a liquid solvent (or solvent combination) that one uses to extract plant constituents from a herb.
- Solvents most often used in herbal medicine to make tinctures or extracts are: Water, alcohol and glycerin.

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HYDRO-ETHANOLIC MENSTRUUM BASICS

- Determine Best Ratio of Alcohol, Water and any Adjuncts
- What type of Alcohol
- What strength of Alcohol (Proof)
- What type of Water: Distilled, Filtered, Spring
- Any adjuncts: Glycerin, Vinegar, others
- Always pre mix menstruum solution and stir well.
WATER CHOICES FOR MENSTRUUM'S

<table>
<thead>
<tr>
<th>Distilled</th>
<th>Filtered</th>
<th>Spring</th>
</tr>
</thead>
<tbody>
<tr>
<td>Commercial use</td>
<td>Easily available</td>
<td>Source from springs</td>
</tr>
<tr>
<td>100% water</td>
<td>No chlorine</td>
<td>High affinity for minerals</td>
</tr>
<tr>
<td>No minerals</td>
<td>May or may not be pure</td>
<td>Highly variable</td>
</tr>
<tr>
<td>Fully able to extract</td>
<td>Various mineral content</td>
<td>Adds local energy and spiritual aspect</td>
</tr>
<tr>
<td>because only H20</td>
<td></td>
<td>May not be pure</td>
</tr>
</tbody>
</table>

THE ALCOHOL DIVIDE

- Recreational Use / Abuse
  - Heavily Taxed
  - Feds: $16 gallon
  - Oregon: $22.72 gallon (2nd Highest in nation)
  - Perceived as recreation
  - Proof varies widely

- Herbal Supplement Use
  - Taxed
  - Perceived as a Solvent to extract herbs
  - Not recreational
  - 190 proof only, then diluted
ALL SPIRITS ARE DISTILLED

- Highly purified
- No source of original plant parts left
- Commonly from cane sugar, grain sugars, grape sugars, or other starches.
- Distillation is highly regulated and illegal for home production.
- Wine, Beer, Cider and Mead are fermented but not distilled

VAPOR PRESSURE CURVE

- Ethanol is lighter and vaporizes at a lower temperature than water
- This is the reason we can have distilled spirits
- Ethanol 173 F
- Water 212 F
COMMERCIAL EXTRACTS

- USP Grain, Cane Sugar or Grape Sugar
- 190 proof, 95% EtOH (Highest proof)
- Allows for dilution from 95% to 25%
- Mix with Distilled water for ideal % EtOH

HOME MADE HERBAL EXTRACTS

- You can adjust the type of alcohol based on Proof and because of sourcing, flavor.
- Most distilled spirits are 40 to 60% EtOH
- Cannot be made stronger only diluted
- Sourcing wide variety
TYPES AND SOURCE OF ALCOHOL FOR HOME USE

<table>
<thead>
<tr>
<th>Alcohol</th>
<th>Source</th>
<th>Flavor</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vodka</td>
<td>Grains, Potato</td>
<td>Neutral</td>
</tr>
<tr>
<td>Brandy</td>
<td>Grapes (wine)</td>
<td>Fruity</td>
</tr>
<tr>
<td>Rum</td>
<td>Sugar Cane</td>
<td>Sweet, Carmel</td>
</tr>
<tr>
<td>Gin</td>
<td>Various</td>
<td>Botanical, Juniper</td>
</tr>
<tr>
<td>Tequila</td>
<td>Blue Agave</td>
<td>Fruity, Smokey, herbal</td>
</tr>
<tr>
<td>Whiskey</td>
<td>Wheat, Barley, Corn</td>
<td>Smokey, Bold, Tannic</td>
</tr>
</tbody>
</table>
SUGGEST RANGE OF ALCOHOL
FOR HERBAL EXTRACTION

Low Ethanol  Medium Ethanol  High Ethanol
20-40%  40-60%  60-95%

Reishi Mushroom  Burdock  Cayenne fruit
Althea Root  Dandelion  Propolis
Licorice Root  Astragalus  Myrrh
Willow bark  Almost every herb that is not in low or high ethanol category  Cotton Wood buds
American Ginseng  60-95%  Black Cohosh

PERCENT ALCOHOL IN MENSTRUUM

• The Alcohol % is based on the best ratio for total extraction of medicinal agents
• When in doubt use dilute alcohol (50%)
• Polysaccharides or starches are best extracted in water (infusion or decoction)
• Alkaloids are best extracted in a variable percentage of alcohol (25%-95%)
• Glycosides are best extracted in a combination of alcohol & water (60% - 85%)
• Tannins are best extracted in a combination (60%-85%) Low alcohol will extract, use glycerin
• Resins are best extracted in alcohol (88%-95%)
• Essential oils are best extracted in alcohol (80%-95%)
EXAMPLES OF ALCOHOL CONTENT FOR CERTAIN HERBS

• For plants that are high in resins and volatile oils you will need more alcohol:
  • Example: dried cottonwood buds (Populus balsamifera) 95%
  • Example: dried juniper berries (Juniperus spp.) 1:5, 75% alcohol
  • Example: dried biscuitroot (Lomatium dissectum) 1:5, 70% alcohol

• For plants high in starches and polysaccharides you will need more water:
  • Example: dried burdock root (Arctium lappa) 1:5, 60% alcohol
  • Example: dried gingko leaves (Gingko biloba) 1:5, 60% alcohol
  • See the back of Sharol Tilgner’s Book (Herbal Medicine for the heart of the Earth) for tincture strengths and % alcohol
  • Look at Commercial Extracts for Ratios and % EtOH
MACERATION TIMES: TO SOAK AND STIR

- Every herb extract is unique
- 14-21 days average range of soaking
- Daily stirring or shaking very important
- Longer maceration does not make it better, once fully extracted the marc should be removed

HERBAL EXTRACT INCOMPATIBILITIES: OVERVIEW

- Polarity
- pH
- Precipitation
- Temperature
- Solvent Used
- Solvent Percentage
PROBLEMS WITH DISPENSING: MIXING EXTRACTS

- Mix ability and Miscibility
- Tinctures alcoholic %
- Water and alcohol are miscible but it will lower the alcohol % leading to precipitation.
- Like dissolves like.
- Keep alcohol content similar + or - 20%

PRECIPITATION

- General problems:
  - Alcohol of different strengths
  - Tannins
  - Mucilage’s
  - Essential oils
- Mixing precipitates, or suspending them.
INCOMPATIBILITY: ALKALOIDS

- Alkaloids are precipitated by alkaline substances, tannic acids or salicylates.
- Some Alkaloids are more water soluble at acid ph and oil soluble at alkaline ph.
- The alkaloidal salts may precipitate to the bottom of the tincture bottle. Concentrating the alkaloids at the bottom of the tincture bottle.
- Take Home: Shake your tinctures well before using.

INCOMPATIBILITY: ESSENTIAL OILS

- Essential oils do not mix with water and will float to the top.
- Essential oils can be dispensed in polar agents by emulsification with gums.
- Essential oils can be miscible in partially polar and non polar agents like alcohol and vegetable oil.
INCOMPATIBILITY: MUCILAGE'S AND POLYSACCHARIDES

- Carbohydrates are in general quite polar and water soluble.
- Monosaccharides, disaccharides, Soluble fiber, insoluble fiber.
- These will generally begin to fall out of solution in alcohol levels over 25%-50%

POLARITY

- Polar: a separation of electric charge across a molecule
- Polar solvent is Water, then Glycerin,
- Fixed oils are non polar.

<table>
<thead>
<tr>
<th>Solvent</th>
<th>Relative Polarity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>1.00</td>
</tr>
<tr>
<td>Glycerol (glycerin)</td>
<td>0.812</td>
</tr>
<tr>
<td>Ethanol</td>
<td>0.654</td>
</tr>
<tr>
<td>Acetic Acid</td>
<td>0.648</td>
</tr>
<tr>
<td>Acetone</td>
<td>0.355</td>
</tr>
<tr>
<td>Ethyl acetate</td>
<td>0.228</td>
</tr>
<tr>
<td>Diethyl ether</td>
<td>0.117</td>
</tr>
<tr>
<td>Hexane</td>
<td>0.009</td>
</tr>
<tr>
<td>Olive oil</td>
<td>~ 0</td>
</tr>
</tbody>
</table>

A water molecule, The two charges are present with a negative charge in the middle (red shade), and a positive charge at the ends (blue shade).
GLYCERIN

- Technically a alcohol with 3 hydroxyl groups
- Tastes sweet, is humectant
- Produced commercially by saponification of fixed oil with Na OH
- Half the solvent power of alcohol
- Can mix with water and or alcohol
- Need 60% glycerine in water for preservation
- Holds tannins from precipitation.

ALCOHOL VERSUS GLYCERIN

- Best Solvent for extracting secondary plant constituents
- Best preservative and sanitizer
- Minimum need to preserve is 20-25%
- Safety Issues: use with alcoholics and certain illnesses
- Natural sourced
- Alcoholic taste

- Fatty alcohol from the saponification of fat
- Commonly from Palm oil or other vegetable oils
- Fair solvent, good preservative
- Need 60% glycerine to stabilize extract
- Low glycemic index
- Sweet taste
- Best for alcohol sensitive persons
OIL AS EXTRACTING AGENT

• Fixed oil - does not evaporate, lighter than water, does not mix with water
• Olive oil, or any vegetable oils are fixed oils
• Good solvent for resins, oleo-resins, essential oils, flavonoids
• Decomposes on exposure to heat, air, moisture, light, bacteria - rancid smell & taste
• With fresh herbs must remove water from the bottom of macerate.

COMMON STEPS FOR MAKING HERBAL EXTRACTS

• Quality Herb, Assay, Testing, Organoleptic's
  • Fresh or Dry and Why?
• Milling and Grinding
  • Type of mill
  • Communion to right size
• Extraction Method
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  • Pro and Cons
• Menstruum's and why
  • Alcohol Percentage
  • Ration
  • Water
  • Glycerin
• Pressing / Filtering / Bottling
PRESSING

• Recovery of absorbed extract from spent plant material
• Recovery of juice from crushed, fresh plants
• Ancient tradition - pressing grapes for wine production
• The higher the pressure the more complete the yield

BATCH PRESS

• Basket press or wine press: lined sieve basket filled with macerate, contents are compressed against a header, extract runs out on the bottom
• Pack presses: bagged macerate between perforated sieves, compressed as a stack
• Up to 10,000 psi
BATCH PRESS

ADJUSTING FLAVOR

- General Rules
- Classic carminatives taste good and support healthy digestion
- Can be added to formulas at various amounts
- Classic forms of corrigents
  - Mint Carminative herbs
  - Saponin Digestive Supportive Herbs
CARMINATIVE CORRIGENTS

• Classically used as food spices
• Naturally anti-nausea and gas
• Support healthy digestion

• Peppermint /Spearmint
• Chamomile
• Catnip
• Cardamom
• Cloves
• Cinnamon
• Lavender
• Ginger
• Clove
• Nutmeg

SAPONIN DIGESTIVE SUPPORTIVE CORRIGENTS

• Natural high in plant saponins
• Saponins are naturally occurring soap like triterpenoid (C-30) or steroidal(C - 27) glycosides
• Saponins help absorption and support healthy digestion
• Classically used to support fat digestion and absorption
• Often can be considered an alterative or adaptogenic
• Licorice (Glycyrrhiza spp.) is classic example

• Examples
• Licorice Glycyrrhiza
• Wild Yam
• Angelica
• Chinese Angelica
• Eleuthero
• Ginseng
• Smilax
• Nettle root
CUSTOM PATIENT FORMULAS
POURING YOUR OWN

• There is a long history of custom pouring for clients
• Once seal is opened then, practitioner takes all liability, not the manufacturer
• GMP's do apply to practitioners, but currently not enforced
• Use common sense
• Keep pour log with batch numbers and expiration dates
• Create database to track sales by herb name
• Searchable database for a product recall.

THANK YOU!

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