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Standard Operating Procedure

Procedure: Steam distillation of biomass to extract essential oils

Department: BEI

Building/ Room Number: Biorenewables Education Laboratory (BRL) 1114

Supervisor: Jacqulyn Baughman

Procedure Overview: An all-glass, vertical steam distillation unit, consisting of a hot plate, boiling flask, biomass flask, still head, condenser and receiver, is used for "dry steam" distillation of plant material. Steam produced in the boiling flask travels upward into the biomass flask where essential oils and water-soluble compounds are removed into the vapor stream. This vapor stream travels through the still head, condenses in the water-cooled condenser, and collects in the receiver, where the essential oil layer phase separates.

Health and safety information for materials used: The glassware must be handled with care during assembly, dismantling and cleaning to prevent breaks and cuts. The hot plate and boiling water/steam can cause burns.

Hazard Control Measures:

- safety glasses
- lab coat
- latex or nitrile gloves for handling biomass
- heat resistant gloves for handling hot equipment

Waste Disposal Procedures: Biomass can be put into regular, non-hazardous garbage
Decontamination Procedures: none

Spill containment and clean up procedures: Biomass can be swept up or wiped up with a wet cloth and disposed of in the garbage.

Using substances requiring special procedures? No

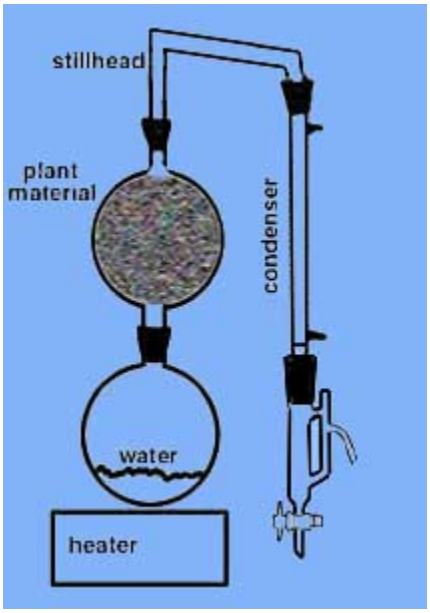
Written By: Catie Brewer **Date:** 5/24/11

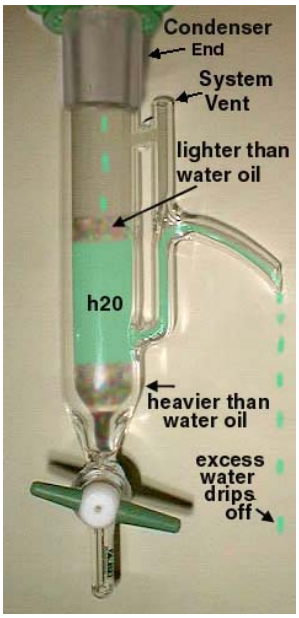
Approved By: **Date:**

Detailed procedures, instrument operation and maintenance, emergency contact information and a list of those trained for this procedure are attached.

Equipment Description

This all-glass, vertical steam distillation unit, consisting of a hot plate, boiling flask, biomass flask, still head, condenser and receiver, is used for "dry steam" distillation of plant material (see equipment diagram below). Steam is produced in the boiling flask by heating distilled water with the hot plate. This steam travels upward into the biomass flask where essential oils and water-soluble plant compounds are removed into the vapor stream. The vapor stream travels through the still head, condenses in the water-cooled condenser, and collects in the receiver, where the essential oil layer phase separates. The receiver is specially designed to retain both heavier-than-water oils and lighter-than-water oils, while allowing excess water, containing the water-soluble compounds, to be drained out and collected separately (see diagram below). In this way, the essential oils are condensed. Dry steam distillation is different from "wet" steam distillation because the biomass does not directly contact the water and the vertical design allows any water that condensed on the biomass to drain back into the boiling flask.





Steam distillation of biomass generally yields two products: a relatively high purity essential oil and an aqueous condensate called a hydrosol. The oil consist of hydrophobic, often aromatic compounds that are produced in very small concentrations as secondary metabolites in the plants. The hydrosol also contains secondary metabolites but these compounds are more hydrophilic. Plant parts that can be used to produce essential oil are berries (anise, juniper), seeds (almond, nutmeg, cumin), bark (cinnamon, sassafras), wood (cedar, rosewood, sandalwood), rhizome (ginger), leaves (basil, bay leaf, sage, eucalyptus, oregano, peppermint, pine, rosemary, spearmint, tea tree, thyme, wintergreen, lemon grass), resin (frankincense, myrrh), flowers (chamomile, clove, geranium, hops, jasmine, lavender, marjoram, rose), peels (orange, lemon, lime, grapefruit), and roots (valerian).

Pre-Distillation Checklist

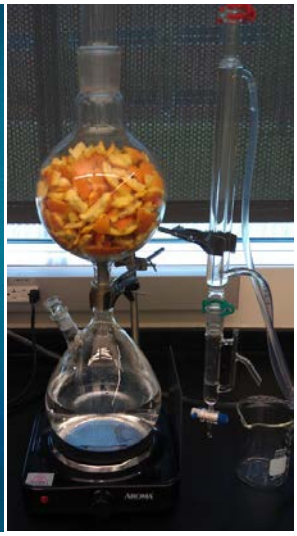
The following checklist is to be performed each time before setting up the distillation.

- Cold water for the condenser(s) is available at the sink and the proper faucet adapter is installed.
- Hot plate surface is clean and dry. Power cord is in good condition.
- Biomass is available and prepared. Essential oils are best extracted from fresh biomass shortly after harvesting. If fresh biomass needs to be stored between harvest and extraction, store in a labeled plastic bag or sealed container in the refrigerator. Dried biomass can also be used. To prepare, remove as much of the non-oil containing plant parts as possible (steams, fruit pieces, etc.) and break/cut biomass to be extracted into ~1" wide pieces. In general, the smaller and thinner the biomass pieces (as long as it is not powder), the more efficient the extraction is.
- All the distiller pieces (boiling flask, boiling flask stopper, biomass flask, metal screen, still head, condenser, condenser tubing, receiver, large (red) and small (green) plastic glassware connector clamps, metal vertical support rod, beaker to collect hydrosol, and two metal tube clamps) are present and clean.
- Silicon joint lubricant is on hand for distiller assembly.

Distiller Set-Up

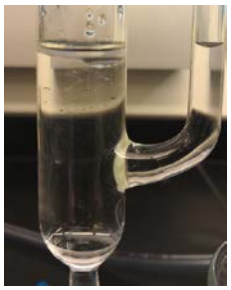
1. Check all glassware for chips or cracks and discard any broken pieces in broken glass container. Even small cracks can become big problems when heated. See lab supervisors for replacements.
2. Fill boiling flask with approximately 1.25 L of distilled water such that flask is between 1/2 and 2/3 full. Place boiling flask on top of hot plate and secure in place with tube clamp around the neck of the flask. (Tube clamps should already be in the correct orientations and therefore should not need to be adjusted much aside from closing and opening the "pincher".)
3. Coat boiling flask stopper ground glass joint with thin layer of silicon lubricant and place in boiling flask. (Keeping ground glass joints lubricated is critical to preventing joints from "freezing" and breaking during disassembly.)
4. Secure condenser in place using second metal tube clamp such that the male end is facing downwards and tube clamp is attached slightly above water inlet connection (water tubes will likely already be connected to inlet and outlet). Coat male end of condenser with thin layer of silicon lubricant.
5. Attach receiver to bottom of condenser and secure in place with green plastic connector clamp. (Condenser height may need to be adjusted to accommodate receiver).
6. Make sure stopcock on receiver is in closed position and fill receiver with distilled water such that the water level is at least as high as the top of the lower outlet (see above diagram of how receiver works). The metal heat shield should be between receiver and hot plate.
7. Carefully insert metal screen (folded part ups) into the male end of the biomass flask such that bottom (open part) of screen is flush with the bottom of the ground glass joint.
8. Fill biomass flask through the top with prepared biomass. Coat male end ground glass joint of biomass flask with thin layer of silicon lubricant and set flask into top of boiling flask.

9. Coat both ground glass joints of still head with thin layer of silicon lubricant. Simultaneously connect still head to top of biomass flask and condenser. If the height/orientation of the condenser needs be adjusted, set the still head on the counter before making the adjustments to prevent dropping or knocking over any of the glassware (another set of hands is useful here). Secure still head to condenser using red plastic glassware connection clamp.
10. Verify that condenser tubing is connected. Cold water should enter in the bottom and exit out of the top.
11. Connect inlet condenser tubing to sink cold water tap using cream-colored faucet adapter. Make sure that outlet condenser tubing is drained into the sink, or connected to inlet of second condenser if running multiple distillations in a daisy-chain set-up.
12. Turn on cold water such that there is a gentle flow of water through the condenser. The outlet temperature of this water will be monitored throughout the distillation to make adjustments to the flow rate.
13. Place beaker (400 ml or larger) underneath overflow spout on receiver to catch hydrosol.

Distillation Procedure

1. Double-check all ground glass connections to make sure there is a good seal and that joints are lubricated.
2. Plug in hot plate and turn on to high (this setting will be used throughout the distillation).
3. Water in boiling flask should gradually begin to boil and travel upward into biomass flask. Steam will appear and start to condense in the still head after about 30 minutes of heating. Some water will also condense in biomass flask and flow back down into boiling flask which may cause the water in the boiling flask to change color; this is normal (think tea-making).
4. Make note of the time when the first drops run down the condenser and into the receiver.
5. Begin periodically checking the temperature of the water draining out of the condenser outlet tube into the sink. Water should be cool and at most, lukewarm; if water is warm, gradually increase the flow rate of the cold water until the outlet water is cool.
6. Watch liquid continue to collect in the receiver. An oil layer should begin to form on top of the hydrosol. Most of essential oil will collect in the first 10-20 minutes, but more will come off in the next 1-2 hours.



7. Continue the distillation until essential oil layer has not grown in the last ½ hour or until new hydrosol collecting in the receiver no longer has any smell (this can be checked by collecting a couple drops from the overflow spout in a small beaker). When the distillation is finished, the biomass should look "spent".
8. To stop the distillation, turn off and unplug the hot plate. Let the entire set-up cool until the biomass flask is cool enough to handle (takes about 30-40 minutes).
9. Disconnect receiver from condenser and use stopcock to carefully drain remaining hydrosol into overflow beaker, leaving the essential oil layer. If desired, save a sample of the hydrosol in a labeled vial.
10. Wait about 5 minutes and then drain out the essential oil layer into a vial. Close and label vial. If the essential oil has a slightly "burnt" smell, it contains some hydrophobic phenolic compounds. This smell can be removed by letting the essential oil vial sit uncovered for a day or two; during this time, the phenolic compounds should evaporate off and the essential oil should smell better.

Disassembly and Clean-up

1. Partially fill Rubbermaid tub with warm soapy water for washing glassware. Glassware should be washed in this tub rather than in an unlined sink to prevent breaking the glassware against the hard sides of the sink.
2. Set empty receiver into tub to soak.
3. Turn off water flowing to condenser.
4. Disconnect still head from condenser and carefully lift off condenser and biomass flask. Set still head into tub.
5. Left off biomass flask and carefully empty contents through top opening into non-hazardous waste can. If biomass gets stuck, gently pull it out and/or tap flask. Bits clinging to the side can be rinsed out with very small amount bit of water into garbage can. **Make sure to save metal screen.** Set biomass flask and metal screen into tub.
6. Using hot gloves, unclamp boiling flask and set on bench to cool further. Dump water left in boiling flask down the sink.
7. Wash all glassware except for condenser. Rinse thoroughly with tap water then distilled water before leaving to dry.
8. Disconnect condenser from stand (water inlet and outlet tubes do not need to be disconnected from condenser). Rinse condenser with warm tap water then distilled water and reconnect to stand to dry.

Emergency Contacts

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Approved Trainers:
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Training Sign-Off

Trainee	Date	Trainer