OilExTech
Oil Extraction Technologies

EssenEx™-100 extraction techniques

D. Hackleman 9/5/15

Non-Specific Plant materials

Abstract:
While many plant materials have been the subject of essential oil extraction in the EssenEx™-100, far more have not been studied or little data has been reported. This note is to offer a starting point and mechanism to develop an optimal extraction scheme for any plant material.

Method:
The process to discover the quantity and types of essential oils that can be extracted from any botanical material are relatively straightforward. The EssenEx-100 is capable of extracting the essential oils from any botanical material in which the essential oils have adequate volatility and concentration to enable the process known as "Steam Distillation". Steam Distillation is not actually a true distillation of the material as the mass transfer of the essential oil is performed at near the temperature of Steam (~ 100C) which can often be far lower than the boiling point of the specific essential oils extracted. This means the process is very "gentle" and minimizes possible reconfiguration (isomer racemization, for example) of the specific essential oil molecular structures which can happen at more elevated temperatures. In general; between 10 and approximately 50 grams of 'available water" need be within the plant material for this extraction process to be effective. This paper offers a process to help determine the quantity of botanical material which may be placed in the reaction chamber and result in an optimal extraction in the EssenEx-100. Such an optimal process results in a modest amount of un-melted ice remaining attached to the ice suspender at the end of the process.
A good estimate of the amount of water which can be removed from a botanical material is offered in Appendix "A". The estimate is that roughly 50 to 200 grams of most "moist but not very wet" botanicals can be the subject of an essential oil extraction, depending on the amount of water contained.

The process outlined below is essentially a means to optimize the extraction of the essential oils and involves a starting point as well as adjustment method which may be used to find the optimal conditions. Whenever a different essential oil botanical material is employed, it may be necessary to adjust the extraction time somewhat depending on a combination of the type of material and its water content. Botanical materials tend to have considerable variance in water content as they age and/or dry under normal ambient conditions.

We welcome you to report your individual accomplishments to share with others!

Initial (first) runs:
(Some essential oil should be recovered even with the first run, but it probably will not be the optimal extraction conditions.)

The approximate parameters for the first experiment are as follows:

1. Prepare ~50 Grams (2 ounces) of botanical of interest.
    a. If the essential oil is embedded within the material, it may be necessary to chop and/or grind the material for optimal extraction. Visit literature references on the particular type of material if uncertain of the location of the essential oil in the botanical material, or if desired, go ahead and experiment from the onset by assuming that oil may be recovered from the outer surfaces of the material.
        i. A secondary possible option is to freeze the prepared botanical prior to extraction as this often both disrupts the cellular barriers that may retain the essential oil and embeds as well as releases free water from the botanical for use in the process.
            1. *In the case of materials with attending long chain waxes and resins, often freezing the botanical prior to chopping/grinding is superior to performing the function at room temperature as this will cause the resins to be below their "glass transition temperature" and become nicely "brittle".*

    b. Best results take place if the botanical is placed in the reactor in a manner that as steam is generated from the water in the material, it can escape the matrix and pass to the ice core. This means one should not tightly pack the botanical material, but rather have it loosely deposited in the reactor in the appropriate region (the annulus cylinder of volume external to the shield enclosing the condensate container [beaker] and internal to the outer wall of the reactor vessel. An example, moderately chopped peppermint leaves totaling up to 50 grams with a few portions of the stem of the plants would suffice. (In the case of peppermint, the oil is in "oil sacs" on the outer top surface of the leaves, and little if any is on the stems.)
        i. *If a compound with resins is the target for extraction, the placement of a disk of paper (such as filter paper) below the botanical inside the reactor will result in far easier cleaning after the extraction!*

2. Operate Microwave on "High", Duration 6 Minutes using the standard method as described in the operation manual.
    a. Note: The Duration is based on use of a home Microwave oven with an approximate power usage of 1000 Watts. Should the specific unit in use be dramatically different than this value, adjust the time such that the product of time and power is roughly 6000 Watt-Minutes (corresponding to roughly 360 KJ).

3. Prior to removal of the reactor from the microwave oven, let it cool for a minimum of 2 minutes. (5 minutes is actually better for reproducibility of the experiment runs.) In any event, plan to remove all of the experiment runs after roughly the same time duration to keep results more reproducible.

4. After removal of the reactor (prior to removal of the lid), place the reactor on a cloth (Towel) adjacent to the microwave oven or in a suitable place for analysis ease. (This author prefers to place the reactor on a table of comfortable height to perform the remainder of the operations while seated.)

5. Allow the reactor to further cool for 3-5 minutes.

6. Remove the lid, noting whether any vapors are emitted. If there is a wisp of steam, then note that effect and plan to wait longer prior to removing the lid for future runs. Ideally, no wisp of steam / vapor will be observed, but under high humidity and low temperature ambient conditions, a modest wisp of this nature may take place and would be inconsequential.

7. Observe the ice core. There should be some remaining and no ice should be floating in the collection vessel.

    a. If there is ice in the vessel, see Appendix "B" regarding ice fracture issues.
    b. If there is no ice remaining, the duration of the microwave excitation may have been too great, or the last residues may have melted during the "cool down" period.

8. Carefully collect the extracted oil in the separator (volumetric flask) using the method described in the instructions.

9. **Before removing the oil from the separation vial measure the amount collected**:

a. Instead of an exact volume or mass, one can utilize a ruler and measure the mm length of the column of oil in the separator (volumetric flask).
    i. Place a ruler with mm graduations adjacent to the neck of the flask and measure the distance between the two menisci (the lower one is the water and the upper is the top of the essential oil.
        1. If the layer is very small, this may not be easy to perform and a qualitative statement of "trace" may work.
        2. One can take an image of the layers and then measure the image either directly on the camera (or smart phone) or by printing the image and using a ruler.
            a. Retention of this image may be useful in experimental work toward optimization of extraction conditions!

b. To convert this measurement into volume, perform the following calibration:
    i. Fill the separator with water.
    ii. Draw out a portion into one of the pipettes, measuring the change in the column height with the same ruler used in "a" above.
    iii. Weigh the amount of water drawn into the pipette with a postage (or other) scale capable of measurement to at least 1 gram, but preferably 0.1 gram, or count the number of drops of water that were drawn into the pipette, then count the number of drops from the pipette that represent a minimum of 10 times the minimum resolution of any balance available. This will allow calculation of the volume (mass) of one drop of water from the pipette. (One ml of water weighs one gram within a reasonable degree of accuracy.) From this calibration, the volume of oil extracted can be calculated from the length of the column of oil observed in the separator flask neck.
c. If desired, one can convert the volume of oil measured in this manner to a mass. The conversion is to use the density of the oil. Many essential oils have published densities, but if such is not available, then one can measure the mass of a known volume of the oil, possibly collected from more than one extraction, and compute the density.

2. Note the approximate mass of remaining ice on the ice holder
    a. This is best accomplished by weighing the ice and holder (and probably include the lid) before and after the extraction. The difference is the mass of ice which was used in the condensing operation. By weighing the lid with an ice core support screw and knob, one can also determine the amount of ice which remained at the end of the extraction.

3. Note whether or not the Ice departed from the ice holder.
    a. If so, see Appendix B for means to produce a superior Ice core.

4. If ice remained after the run:
    a. add 10% to the microwave excitation duration time and run another batch
    b. Note results and determine if more oil was extracted.

5. If no ice remained after the run:
    a. Reduce the duration by 10% and run another batch.
    b. Note results and determine if more oil was extracted.

Once a minimum of 3 runs are accomplished with the same parameters, the data taking element is adequate for initial calculations. Plot the data and determine the optimal conditions for essential oil extraction from this particular plant species. All data information should be stored and a functional plot of the data produced with appropriate error "whiskers".

Use of the "standard operating conditions" found will usually be adequate to get effective yield even with new crops of the botanical material and often even for other species of the same or similar plant. All users are welcome to send in information regarding the conditions under which a particular botanical material was used and if known, the resulting chemical compounds.

It is through our individual experimentation that we can learn more about the opportunities for essential oils from the flora that surround us on this planet!

Thank you for your interest. It is hoped that this note helps a bit in your quest!

Sincerely,

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OilExTech
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Appendix A:

Calculation of the Steam condensation capacity

of the

EssenEx™-100

D. Hackleman, 14 October, 2014

Assumptions:

Ice core size: Approximately 200 Grams

The condensation capacity of the ice core can be calculated as follows:

$$M_{water} \times ((\Delta T_{cs} \times c_p^{water}) + \Delta H_v^{water}) \leq M_{ice} \times ((\Delta T_{ci} \times c_p^{water}) + \Delta H_f^{water}) \quad (1)$$

Re-writing:

$$M_{water} \leq M_{ice} \times ((\Delta T_{ci} \times c_p^{water}) + \Delta H_f^{water}) / ((\Delta T_{cs} \times c_p^{water}) + \Delta H_v^{water}) \quad (2)$$

Where:

ΔT_{ci} Difference between Condensate Temperature and 0C (ice), in degrees C (Generally, the condensate is ~60C, so this value is usually 60C)

ΔT_{cs} Difference between Condensate Temperature and 100C (steam), in degrees C (Generally, the condensate is ~60C, so this value is usually 40C)

M_{ice} Mass of Ice, in grams g (~ 200 g)

M_{water} Mass of water condensed from steam, in grams g (This water is from the botanical material plus any added water to that material)

c_p^{water} Heat capacity of water (4.184 J/g-K at ~60C)

ΔH_v^{water} Heat of Vaporization of water (2258 J/g at 100C)

ΔH_f^{water} Heat of fusion of water (333.55 J/g at 0C)

Result, inserting the values above:

$$M_{water} \leq (M_{ice}) \times ((60 \times 4.184) + 333.55) / ((40 \times 4.184) + 2258)$$

$$M_{water} \leq (M_{ice}) \times (584.59) / (2425.36)$$

$$M_{water} \leq (M_{ice}) \times 0.24$$

Hence, maximum capacity is about 50 grams of water from botanical and any added water.

Note: This is only an estimate and assumes no loss of steam during the process as well as no melting of the ice core by ambient conditions. (These actually affect each side of the equation; likely mean that the estimate is a bit conservative.)

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Appendix B:

Ice Fracture Issues:

Some freezing processes and freezers have significantly different characteristics than others. Experimentation at OilExTech, LLC., have led us to understand these issues and we are in the process of offering accessories for improvement of the ice freezing process independent of freezer device. However, in the interest of expediency, here are some guidelines for your own experimentation:

The reason fractures may form in ice cores:
Water releases a great deal of energy in the process of undergoing a phase transition to ice. Depending on the convective air circulation in the freezer, the location on the ice core mold which will commence ice formation first can sometimes be the top and tip of the water column instead of just the tip of the mold. When this happens, then as the ice is formed, and since water expands in volume upon freezing, there will be semi-crystalline segments of ice forming from both the top and bottom of the mold. These will then meet and one will observe a fracture plane between pieces of ice in the final ice core.

How to avoid this issue:
To circumvent this issue, we have found the placement of an insulator (such as a 2 cm thick piece of Styrofoam or similar such substance) on the top of the plastic screw support disk can lead to an ice core with no such fracture plane. When done carefully, a practically clear (no fracture) ice core will be produced. It is a bit of an art, but even if some modest starburst or other pattern is observed in the finished ice core, likely it still be far superior to one with the built-in fracture plane as mentioned previously.

Experiment and discover an even better method!

Appendix C:
Cleaning the reactor after a resinous deposit:

Frankincense, Juniper, Cedar, and other resinous compounds can indeed leave a very sticky, difficult to remove layer inside the reactor.

Easy method:

The easiest method of dealing with this substance has been found to utilize another essential oil, limonene (Orange Oil). Extracting limonene from citrus peel (the outer portion of the skin has the majority of the essential oil) is very easy and also has the advantage that one has citrus fruit to consume! Wiping the inside of the reactor with orange oil will dissolve the majority of these resins.

Alternatively:

- Take all internal parts of the reactor (Beaker and Shields)
- Add a layer of water that covers sap and place in microwave. Run for 5 minutes.
- Using heat resistant gloves take out and scrape the bottom freeing all the material stuck while still warm and hence less solid. Caution: Do not use metal scraping implements as they can remove the protective coating from the shields. Remember, the shields and parts WILL BE HOT! Empty contents into a disposable container to allow cooling.
- When done running trials for the day, put all parts into the reactor and mix soap, degreasing fluid, and water into the container and soak. Scrub, rinse thoroughly, and dry.

For any question please contact us at Info@oilextech.com or fill out our Customer Feedback at our website http://www.oilextech.com/contact-us/