Final Report
Essential Oil Distillation for Boa Vista do Acará

Photo taken from Debora Leal "Organic Producers System in Acara, Amazon"

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EBS 170C
Submitted to Professor Ruihong Zhang
June 14, 2017

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Acknowledgement

Executive Project Summary

Our three objectives for this school year were to develop a final prototype (1), test our oil quantity and quality (2), and to implement our project in the community this summer (3). We have successfully completed all of our objectives and are traveling to Boa Vista do Acara this summer from July 14, 2017 to August 14, 2017.

Fall and Winter quarter were largely devoted to planning and research. Project accomplishments can be evaluated through the design approach, chemical approach, mass transfer model, steam distillation procedure, testing methodology, safety protocols, economic analysis, and testing results listed in this report. The design approach describes the design decision-making process which explains the reasoning behind the four major design decisions pertaining to this project. The chemical approach describes plant and oil interactions involved in oil distillation on a molecular level. The mass transfer model uses engineering principles to determine the amount of oil that will be collected per thickness of herb with respect to time. The steam distillation procedure was developed based on similar published procedures and the constraints of Boa Vista. The testing methodology outlines the materials and methods for our testing methods to determine the peroxide value, acid value, refractive index, and relative density. The safety protocols describe the safest practices for both oil distillation and peroxide value and acid value testing. This section also includes a summary of the design constraints and dimension justification for the final design.

This last quarter was dedicated to development of prototypes one and two and the testing of our oil and hydrosol products. Prototype one developed by augmenting a purchased moonshine distiller, we used this prototype to distil peppermint and ginger oil. We tested our product by determining the acid and peroxide values and the specific gravity. Prototype one also served as a small scale model for our final prototype. For our final prototype our team utilized materials that would be locally available in general cooking and hardware stores. The final prototype consisted of a lobster pot, copper coil, plastic bucket, bike tire, shower hose, common NPT unions/valves, and a metal strainer. Overall the final prototype ran to approximately $140.00 in materials. Over the course of the summer our team will be improving our final prototype up until our departure to Boa Vista.

Problem Statement

Boa Vista do Acará is a small agricultural community in Northern Brazil that consistently faces financial instability. This interferes with their access to basic social services such as healthcare and education. Currently, Boa Vista harvests 50 tons of herbs per year, however, rather than distilling essential oil themselves, they sell their herbs as wholesale raw material to the Natura company. If Natura were to ever pull out of their contract, Boa Vista would be in a financial crisis. Therefore the ability to distill essential oils independently would allow Boa Vista Do Acará residents to stabilize their income source.

Currently, our team is partnering with with the Association of Organic Farmers of Boa Vista do Acará (APOBV) to build an oil distillation device specifically to suit the needs of this community. Due to the remote location of Boa Vista, the community does not have access to electricity, so the oil distiller must operate in humid conditions with fire as the primary energy source. In addition, the distiller must be practical and safe because it will be introduced to a community without a technical background. The essential oil produced must also be high quality in a high quantity to sell to tourists in local markets. With all of these parameters in mind, our team must design an essential oil distillation device that can produce essential oils using the local tools and materials.

Background Materials /Research

Steam Distillation

Essential oils can be produced by a number of chemical means such as solvent extraction or carbon dioxide extraction. These methods are not suitable for this community due to their cost, waste, and complexity. This method must be simple and not require electricity because of the location constraint. The primary artisanal method for essential oil production is steam-based oil distillation.
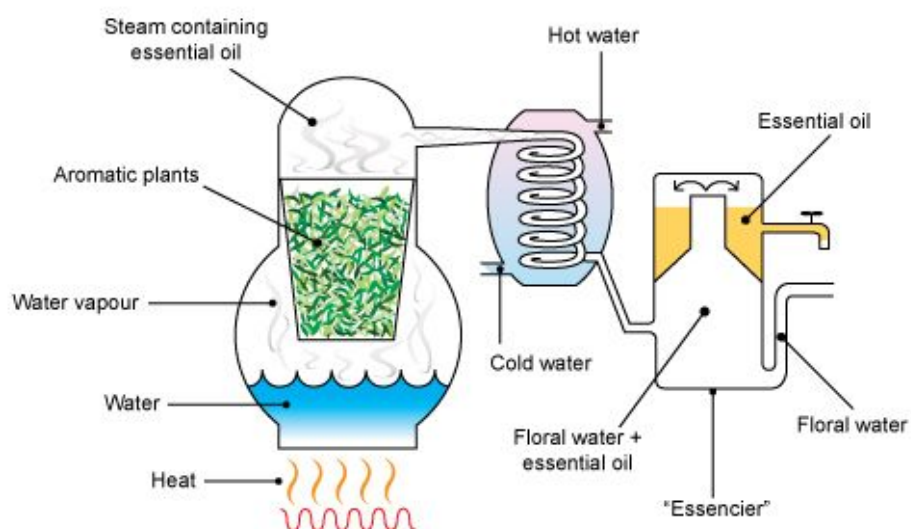

Figure 1. Essential Oil Distillation Process (Mulvaney, 2012)

Steam distillation is a three part process that utilizes steam to break down volatile compounds from plant material. As depicted in Figure 1, the plant material is elevated above the boiling water. The steam is first pushed through the plant in the boiling chamber, where it rises with the volatile compounds. Then, the steam flows through the condenser coil where cool water condenses the steam. The water and essential oil are directed into a container where they separate naturally, due to their differences in density This is the most commonly used method because steam is injected through the material at higher pressures and temperatures compared to other methods. It also produces clear hydrosols and oil with a light and delicate aroma. This method is also faster and the separation of the oil from the hydrosol is more pronounced. (Mulvaney, 2012).

Research revealed that glass, copper, and stainless steel were the three most common materials used for essential oil distillers. However stainless steel is the best choice in terms of building material for the distiller because it is considered non-reactive. This means that the

material will not interfere with chemical structure of the oil or hydrosol in a way that would make it unsafe for human use (Mohamed, 2004).

Plant Chemistry

The primary goal of plants is to grow and reproduce. To achieve this goal, most of plant metabolites give the plants structure and function. Plants also produce very small amounts of *secondary metabolites*, compounds that are not directly related to growth or reproduction. Secondary metabolites play an important role in protecting the plant against consumption and other threats. Many of these secondary metabolites are commercially valuable and make up important compounds in essential oil (NMT,2017).

One of the key building blocks of secondary metabolites is a 5 carbon molecule called isoprene.
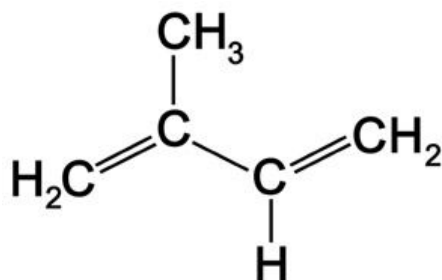
Figure 2: Isoprene

Isoprene, displayed in Figure 2, is used by plants to produce *terpenes and terpenoids*. Essential oils are filled with these compounds because terpenes and terpenoids are responsible for plant smells and flavors (Kumari, 2014).
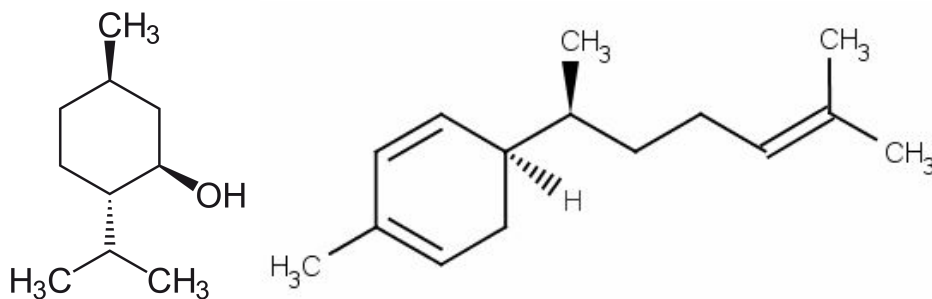
Figure 3: Methanol and zingiberene.

Because isoprene has 5 carbon molecules and terpenes are produced from isoprenes, most terpenes contains multiples of five carbon atoms. For example, menthol, the major constituent of peppermint oil, has the formula $C_{10}H_{18}O$, and zingiberene, the major constituent of ginger oil, has the chemical formula $C_{15}H_{24}$. These terpenes are displayed in Figure 3.

Oil Quality Chemistry
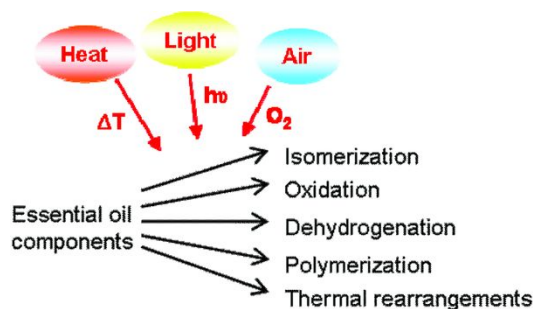
Figure 4: Possible reactions in essential oils.

Essential oils are known to lose their quality through the five processes shown in Figure 4; isomerization, oxidation, dehydrogenation, polymerization, and thermal rearrangements. These processes occur when the oil is exposed to heat, light, or air (Alankar, 2009).
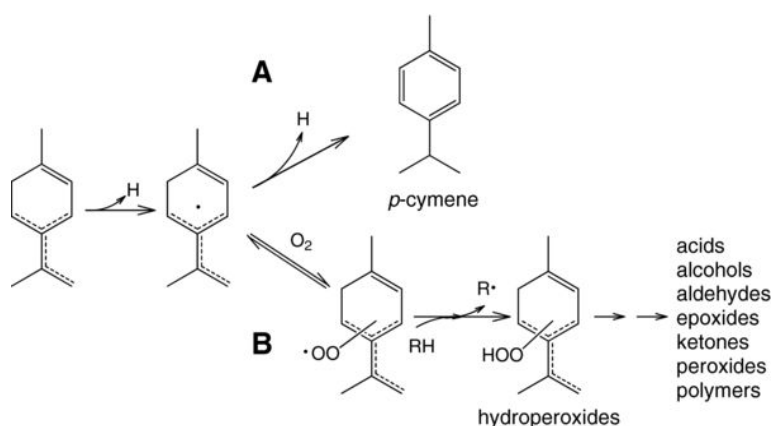
Figure 5. Terpenoid dehydrogenation (A) and oxidation (B), processes that degrade the quality of essential oils.

When essential oil terpenoids are exposed to the five degradation processes, their color, flavor, or consistency may be effected. To avoid quality loss, proper essential oil handling and storage is critical (Kumari, 2014). Proper storage entails dark-colored, glass, and air-tight containers (Alankar, 2009). The oils must also be kept at room-temperature because too cold or too warm conditions will induce thermal rearrangements. Two of the chemical reactions that should be avoided are shown in Figure 5, the dehydrogenation (A) and oxidation (B) of terpenoids (Kumari, 2014). Peroxides and acids are produced through the terpenoids oxidation process. Our team will measure the peroxide value and acid value of the oils to understand their degradation behavior over time.

Mass Transfer Model

   The goal of this mass transfer model is to find the yield of oil that will be produced based on the amount of herbs, the time of distillation, and the diffusion coefficient of that specific herb. In steam distillation the steam molecules impact the herbs and mix with the oil from the herbs. The mixture will vaporize when the sum of the pure vapor pressures of the oil and water is equal to the atmospheric pressure.

$$P_{atm} = P_{water} + P_{oil}$$

This leads to a mixture of the two liquids boiling at a temperature lower than the normal boiling point of either component. $P_{water} \gg P_{oil}$ so the mixture will boil at a temperature slightly less than the boiling point of water. This means the oil will vaporize under very mild conditions. Then if we know the molecular weight (M) of the oil, we can estimate the mass (m) of the oil (NMT, 2017).

$$\frac{n_{oil}}{n_{water}} = \frac{P_{oil}}{P_{water}} \qquad \frac{m_{oil}}{m_{water}} = \frac{P_{oil} \times M_{oil}}{P_{water} \times M_{water}}$$

Mass Transfer Assumptions:
1. The system is isothermal and isobaric
2. The bed of herbs is considered a batch
3. The porous bed formed by the plant is stable. No changes in form or disposition took place during the process
4. Vapor phase inside of the distillation vessel is considered to be perfectly mixed, with constant flow rate of steam
5. All oil inside herbs was extracted during the process
6. Condensed water and essential oil were completely immiscible
7. The steam flowing through herb bed is completely oil free (Cerpa, 2008)

   This model simulates steam distillation using the equation from Fick's Law. This equation solves for the amount of oil that will be collected per thickness of herb with respect to time.

$$\frac{d^2 C_A}{dx^2} = \frac{1}{D} \frac{dC_A}{dt} \text{ in } 0 \leq x \leq L \text{ (Eq. 1)}$$

Where L is the thickness of the herb, D is the diffusion coefficient, initial conditions: $C_A = C_{A_0}$ in $t = 0$, the original concentration of oil, and the boundary conditions: $C_A = 0$ in $x = 0$, $C_A = 0$ in $x = L$. If we solve this using the separation variable technique then we get

$$C_A(x,t) = \Sigma \frac{4 C_{A_0}}{n \pi} \bullet sin(\frac{n\pi}{L} \bullet x) e^{-D(\frac{n\pi}{L})^2 t} \text{ (Eq. 2)}$$ This equation will give us the amount of oil collected given the amount of herb and length of time of distillation (Cassel, 2009).

Goals/Objectives of Engineering Design

Over the course of this year our team had three main objectives. The first and main objective was to develop a final working prototype. The second was to test our product in terms of both quantity and quality. The third and last objective was to implement our project into Boa Vista do Acara.

For spring quarter our main objective was to develop a final working prototype. To become familiar with the process, our team first practiced on the eight liter testing prototype (Prototype #1) that was purchased in winter quarter. With this device we practiced distilling peppermint essential oil. Based on our experience with this device, we had a better idea of the factors which affect the yield and quality of oil. It should be noted that the testing prototype can only be used to test peppermint because its volume capacity does not allow for enough water to distill ginger essential oil. However the experience with this device will be valuable as the base for the development of our final prototype (Prototype #2).

The final prototype was developed based on the data and experience gathered from trials of the testing prototype. We also developed a final distillation procedure specific to the final prototype. The final design is larger in volume in order to produce more oil per batch and to accommodate the longer distillation time for ginger oil. Throughout the second half of spring quarter, we continuously tested and improved our second prototype. The improvements helped us reach our goal of creating a testable prototype capable of producing essential oil in Boa Vista.

From the testing prototype we produced both peppermint and ginger oil, these herbs were chosen in order to give our team experience with both a root and leaf based herb. By determining the acid value, peroxide value, and specific gravity of our product we were able to access the product quality. The procedures and safety protocols for these tests are listed in the Appendix sections E, F, and G. While distilling our team recorded the product yield with respect to time in order to ensure our product quantity followed reasonable standards.

In order to ensure the implementation of this project in Boa Vista, our team secured funding through the Blum Grant awarded by the UC Davis Blum Center for Developing Economics. Our team also received funding from Professors Kurt Kornbluth and Fadi Fathallah to cover the rest of the costs.

Description and Specifications of Final Design Project

Figure 6. Breakdown of Components 1-5 of the Final Prototype

Final Prototype Fabrication

Before constructing the final prototype an approximated SolidWorks design was developed, the schematics of this design are available in Appendix B and C. The Final Prototype consists of five main components as labeled above in Figure 6.

Component 1 is the main chamber which is where the water will be boiled to produce steam which latches onto the volatile compounds from the herbs. The main chamber vessel is an altered stainless steel five gallon lobster pot. In order to ensure no steam loss the lid and rim were insulated using a bike tire that was glued to the pot rim using a heat resistant caulk. In order to divert the steam out of the main vessel, a shower head hose (Component 2) is attached to the lid of the main chamber. To attach the hose, a Green Leaf ½" diameter MPT Nylon Bulkhead Union was attached to the lid by drilling a ½" hole into the stainless steel lid. To drill this hole, a

smaller ⅛" pilot hole was first drilled into the lid, followed by a ½" hole. The hole was sanded and then the union piece which consists of a male and female piece was screwed together with the lid between the two pieces. Two gaskets also bordered the lid between the male and female part in order to seal the union and avoid steam leaks.

Component 2, was used in order to give the device some flexibility in placement of the heat exchanger (Components 3 and 4). Component 2 is attached to the copper coil (Component 3) by Elkhart ½" x ½" Copper Male Adapter that was soldered onto the end of copper coil. The copper coil was nine feet total that was bent into four coils by bending by hand. At the other end of the coil there is a ½". Copper CSST x MIPT Male Adapter that was also soldered and then positioned out a ½" hole in Component 4 (the 5 gallon plastic bucket). The hole in Component 4 was also drilled using a ½" drill bit with a ⅛" pilot hole. This hole is where the end of the copper coil with the copper male adapter and the ½" a ½" female copper adapter meet, this is where the hydrosol and essential oil will exit at the end of the process. Component 4 is also adorned with a ½" Hose Bibb Valve that will be used to empty the bucket of the warm water that accumulates due to the heat exchange between the steam in the copper coil and the water in the bucket. This valve was also installed by drilling a ⅛" pilot hole followed by ½" drill bit for the final hole.

Component 5 is the grate that will go into the main chamber to elevate the herbs above the boiling water. The black grate was altered by cutting and then bending the steel in order to decrease the diameter so it fit into the pot. Larger holes were then drilled into four of the pre-existing holes in order to install the legs. The legs are six inch long male screw threads that were measured and then cut using a hand saw. These legs were placed in the larger drilled holes and then secured with two nuts on either side of each leg.

All of the components used in the development of the final prototype can be purchased in either the city of Belem or in local markets surrounding Boa Vista. The tools required for construction include a power drill, hand saw, and soldering equipment all of which are either available in the community or for purchase in a general hardware store. The skills required for handling the drill and hand saw were taught in the the EBS 170A Lab in the construction of the widget. While soldering was taught by Professor Kornbluth in D-Lab.

Economic Analysis

The final prototype was developed through the allocation of common cookware and hardware that can be bought in general stores. Each component of prototype one was broken down and then substituted for common parts that are cheap and readily available. All of our fittings and piping are ½" inner diameter with NPT threading in order to maximize the availability of workable materials for our design. Table 1 provides a comprehensive list of components and substituted parts that were used in the final prototype and Figure 7 displays the cost percentage of each component relative to the total cost.

Table 1. Breakdown of the cost of components

| Components	Substitutions	Dimensions	Cost
Boiling Chamber	5 Gallon Stainless Steel Lobster Pot	15" x 14" x 14"	$27.00
Grate	Cooking Strainer	15" x 15" x 2"	$3.00
Insulation	Bike Tire	14" Circumference	$5.00
Steam Transport Tube	Stainless Steel Shower Head Hose	60" Long	$10.00
Heat Exchanger	Copper Tubes	⅜" OD x 9'	$40.00
Heat Exchanger Vessel	5 Gallon Plastic Bucket	11.75" x 11.7" x 14.5"	$20.00
Fittings	Valves, Adapters, & Unions	• 1/2in Hose Bibb Valve • Elkhart 1/2in x 1/2in Copper Male Adapter • Green Leaf 1/2 in. Dia. MPT Nylon Bulkhead Union • 1/2 in. Copper CSST x MIPT Male and Female Adapter	$32.00
Total			**$137.00**
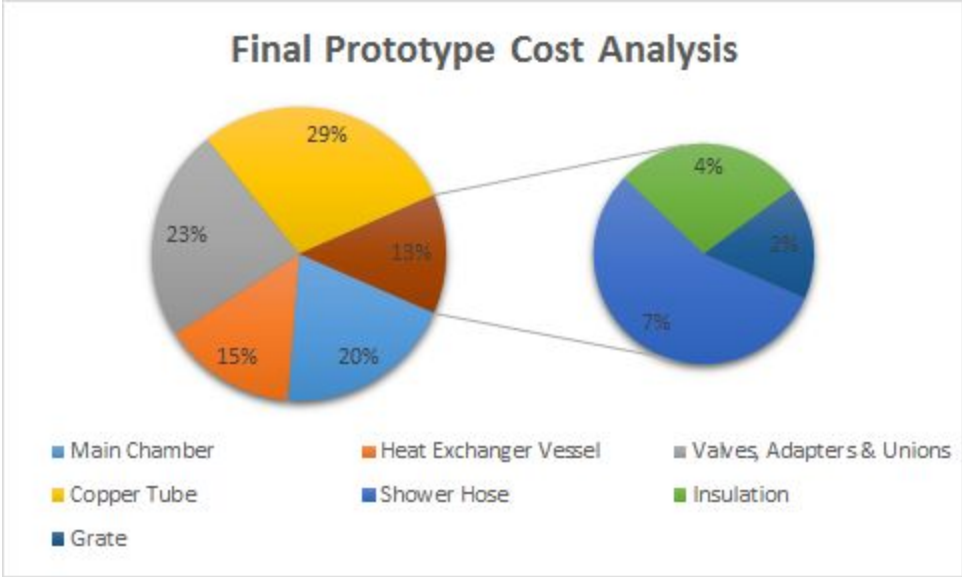
Figure 7. Pie Graph displaying Final Prototype Cost Breakdown

Performance Evaluation of Final Design Project

To evaluate the success of the prototype, both the essential oil quality and quantity were measured.

Quality:

In order to measure the quality of the oil, the peroxide and acid values were measured. The peroxide value (PV) is a parameter used to quantify the extent to which an oil sample has undergone oxidation. The acid value (AV) represents the amount of acidity of oil, and both parameters indicate the oil quality (Paudyal, 2012). Specific gravity was also measured using a refractometer as another indication of oil quality. Ideal values for the AV,PV, and specific gravity analyses are represented by the standard values.
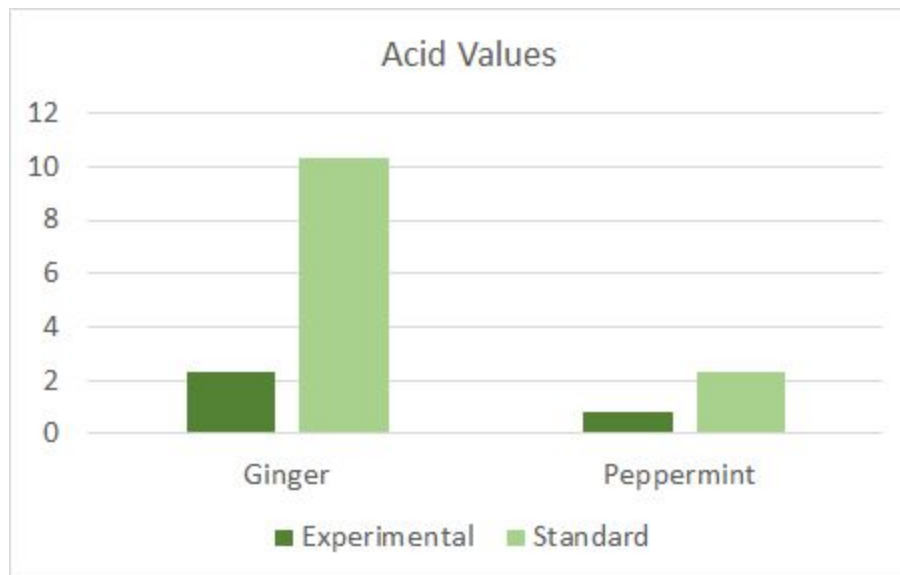

Figure 8: Acid values for ginger and peppermint, comparing the experimental and standard results.
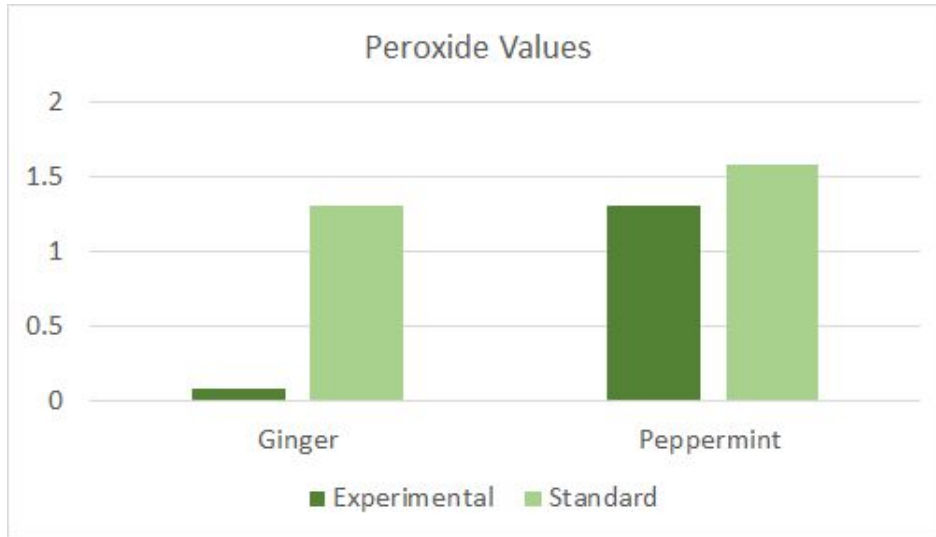
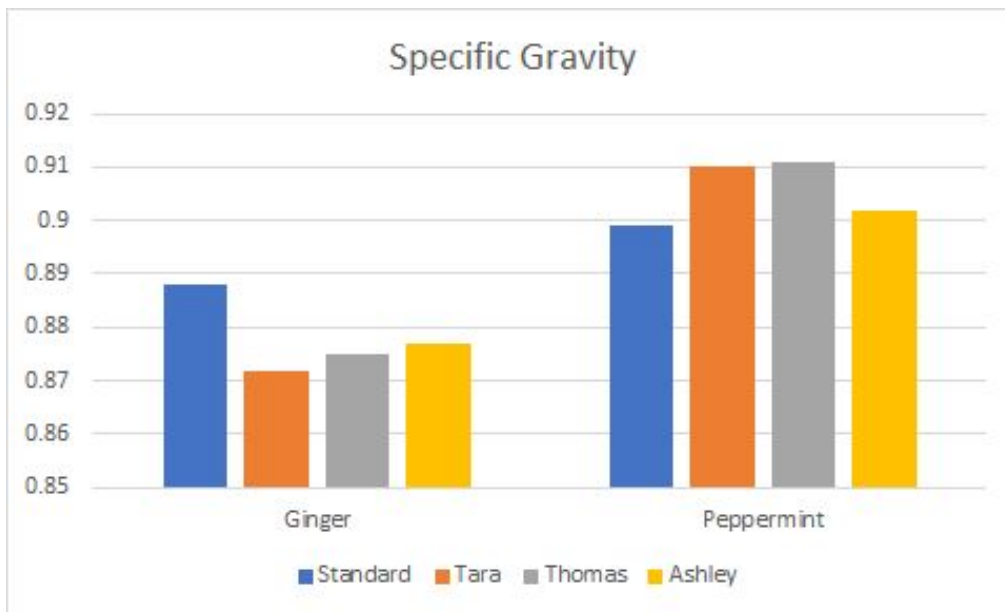**Figure 9**: Peroxide values for ginger and peppermint, comparing the experimental and standard results.

Figure 10: Specific gravity for ginger and peppermint, comparing the each team members' observation to the standard results.

Quantity:

Oil quantity is an important measure of distillation success. An inadequate yield of oil is an indicator of in error either plant material preparation or distillation.
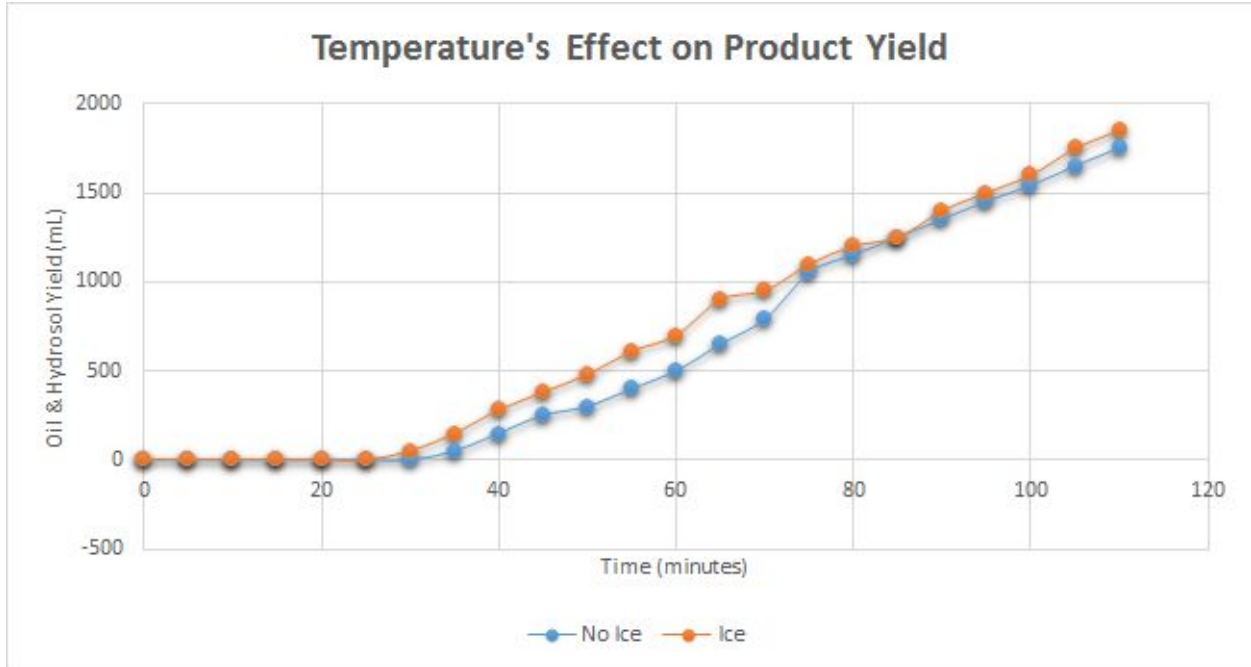
Figure 11: The quantity of oil and hydrosol produced in two trials comparing the effect of ice in the heat exchanger.

The acid value and peroxide value analysis revealed that the distilled ginger oil was poor quality. However, after a literature review, our team discovered that copper (the heat exchanger material) is reactive with volatile ginger compounds. Because the herbs in Boa Vista are unreactive with copper, the discrepancy is not significant. With regards to specific gravity, our oil had the appropriate density and was close to the standard values. The volumes of oil and hydrosol provided confidence in the prototype design because it revealed that there were no leaks in our system.

Our team was unable to test prototype 2, however, we are confident that its design is sufficient because of mass balance calculations. The heat exchanger coil length was justified using the calculations below:

$$Q \ = \ U \ \times A \times \frac{T_{entering} - T_{leaving}}{ln(\frac{T_{entering}}{T_{leaving}})}$$

$$U = \frac{1}{\frac{1}{h_1} + \frac{\Delta r}{k} + \frac{1}{h_2}}$$

$(h_1 = .0188 \ W/m^2K, \ h_2 = .58 \ W/m^2K, \ \Delta r = .001016 \ m, \ k = 400 \ W/mK)$

$$A = L \times (\pi \times 2r)$$

$(r = .015875 \ m)$

$(T_{entering} = 373.15 \ K, \ T_{leaving} = 368.15 \ K)$

$(Q = 1500 \ W)$

$$1500 \ W = 1014.56571 \ m/W \times L$$

$$L = 1.478 \ m = 4.85 \ ft$$

Our heat exchanger was 6 feet long so we conclude it is long enough to condense the steam into oil and hydrosol.

Description of Engineering Design and Product Development Process

Due to the remote location of Boa Vista, the community does not have access to electricity or reliable transportation. The device will need to rely on artisanal methods to produce essential oil and fire will be the primary energy source.

Essential oils can be produced by a number of chemical means such as solvent extraction or carbon dioxide extraction. These methods are not suitable for our project due to their cost, waste, and complexity. Our method must be simple and not require electricity because of our location constraint. The primary artisanal method for essential oil production is steam based oil distillation. There are two main methods of steam based distillation that differ only in the contact between the water and plant material.

The two approaches include:
1. Hydrodistillation
   In hydrodistillation the plant material is placed directly into the boiling water. This distillation process is overall slower and occurs at a lower temperature. The oil produced has a complex aroma and the hydrosol appears milky. This method is commonly used for flowered plants such as roses and orange blossoms. (Mulvaney, 2012)
2. Steam Distillation
   In steam distillation the plant material is elevated above the boiling water. This is the most commonly used method since steam is injected through the material usually at higher pressures and temperatures then the other methods. It also produces clear hydrosols and oil with a light and delicate aroma. This method is also faster and the separation of the oil from the hydrosol is more pronounced. (Mulvaney, 2012)

Table 2. KT analysis for distillation methodology.

Criteria	Weight	Rating (Hydro)	Rating (Steam)	Hydro-distillation	Steam Distillation
Oil Yield (%)	1	1	2	1	2
Oil Quality	1	1	2	1	2
Practicality	2	2	2	4	4
Affordability	3	2	2	6	6
Ergonomics/Safety	3	2	2	6	6
Feasibility for Plant Material	2	1	2	2	4
			Totals	20	**24**

The total ratings for the KT analysis were determined by multiplying the weight of each criteria by the rating for each device. The weight and rating is on a one to three scale; one being the lowest priority and three being the highest. The weight of each factor was then multiplied by the rating given to each method, and these numbers were added for the overall score. The ratings were assigned with the Boa Vista parameters in mind. The highest overall score is the indicator for the best methodology. Based on this KT analysis, steam distillation is the best method for the project.

After thoroughly researching the two methods we chose to design a prototype that utilizes steam distillation. Table 2 summarizes the findings and final decision. Steam distillation is preferable to hydrodistillation for this project because it is faster and used for herbs rather than flowers (Mulvaney, 2012). Steam distillation is a three part process that utilizes high temperature water and steam to break down plant material and release its volatile compounds. The steam is first pushed through the plant in the boiling chamber, where it rises with the volatile compounds. Then, the steam and compounds flow through the condenser coil where a coolant comes in contact with the coil and condenses the solution. The water and essential oil are directed to a container where they separate naturally, oil on top and water on bottom, due to their differences in density. (Mulvaney, 2012)

After choosing our distillation method, our team researched the following three criteria:
1. To determine the ideal material for the distillation device.
2. To evaluate the best input state for the raw plant material.
3. To determine the best dimensions for our final prototype.

1. Distillation Device Material

Research on different types of materials showed us that glass, copper, and stainless steel were the three most common materials used for essential oil distillers. However stainless steel is the best choice in terms of building material for the distiller because it is considered non-reactive. This means that the material will not interfere with chemical structure of the oil or hydrosol in a way that would make it unsafe for human use (Mohamed, 2004).

2. Input State of Plant Material

The client's ideal choice of herbs for distillation included two native herbs tansy (herb) and priprioca (root). Because these herbs are native to Brazil, they are not accessible for our project. We choose to narrow down our testing herbs to plants that are easily accessible and would give our team the best experience for distillation; peppermint (herb), and ginger (root).

Table 3. KT analysis for state of peppermint.

Function	Weight	Rating (Whole, Dry)	Rating (Chop, Dry)	Whole, Dry	Chop, Dry
Oil Yield (%)	3	3	2	9	6
Oil Quality	3	3	2	9	6

Practicality	2	2	2	4	4
Affordability	1	2	1	2	1
			Totals	**24**	17

Table 3. KT analysis for state of ginger.

Function	Weight	Rating (Chop, Dry)	Rating (Ground, Dry)	Chop, Dry	Ground, Dry
Oil Yield (%)	3	1	2	3	6
Oil Quality	3	3	1	9	3
Practicality	2	2	2	4	4
Affordability	2	2	2	4	4
			Totals	**20**	17

As depicted in Table 3, the ideal input state of peppermint is dried and whole. According to a multitude of journals, the method most commonly used to distil peppermint oil is with whole-dried herbs (IFRJ, 2014). The reasoning behind this choice is that it would take less time to release the volatile compounds of peppermint when it is dried. The only time peppermint was ever dried-chopped was during hydrodistillation (AJC, 2011). However the yield of peppermint oil for a dried-whole peppermint for steam distillation was higher than the yield for dried-chopped peppermint for hydrodistillation, where the yield percentages were 1.0% and 0.64% of the dry weight respectively (Alankar, 2009). Based on this analysis, we determined that dried-whole peppermint is the best state of the herb for steam distillation.

As depicted in Table 3, the ideal input state of ginger is dried-chopped. According to a study published by The 4th Annual Seminar of National Science, dried-chopped ginger is the best state to use during steam distillation. Although the yield of the oil was slightly lower when using dried-chopped rather than dried-ground, the quality of the oil was higher when using dried-chopped ginger. This had to do with how the high temperatures can overheat the ginger and cause it to decompose thus degrading the quality of the oil (Mohamed, 2004). Based on this analysis, we determined that dried-chopped ginger is the best state of the root for steam distillation.

3. Prototype Dimensions

To determine the dimensions of our device we primarily considered oil yield. As a process, essential oil distillation produces low yields of oil from fresh herbs. To distill approximately 5 grams of oil, 1.5 pounds of fresh peppermint are needed for a 1.0% yield, and 1 pound of fresh ginger is needed at a 1.4 % yield (Alankar, 2009)(Mohamed, 2004) . Based on

these ratios and the needs of Boa Vista, our final design will be substantially larger than our testing prototype which has an eight liter capacity. Currently our final design consists of a cylindrical boiling chamber with approximately a forty-seven liter capacity, with a height of 31.5 cm and a radius of 21.875 cm. The oil yield calculations, water volume requirements, and plant mass requirements for the final design are calculated in the Appendix A. The Solidworks schematic for this final design is provided in Appendices B and C.

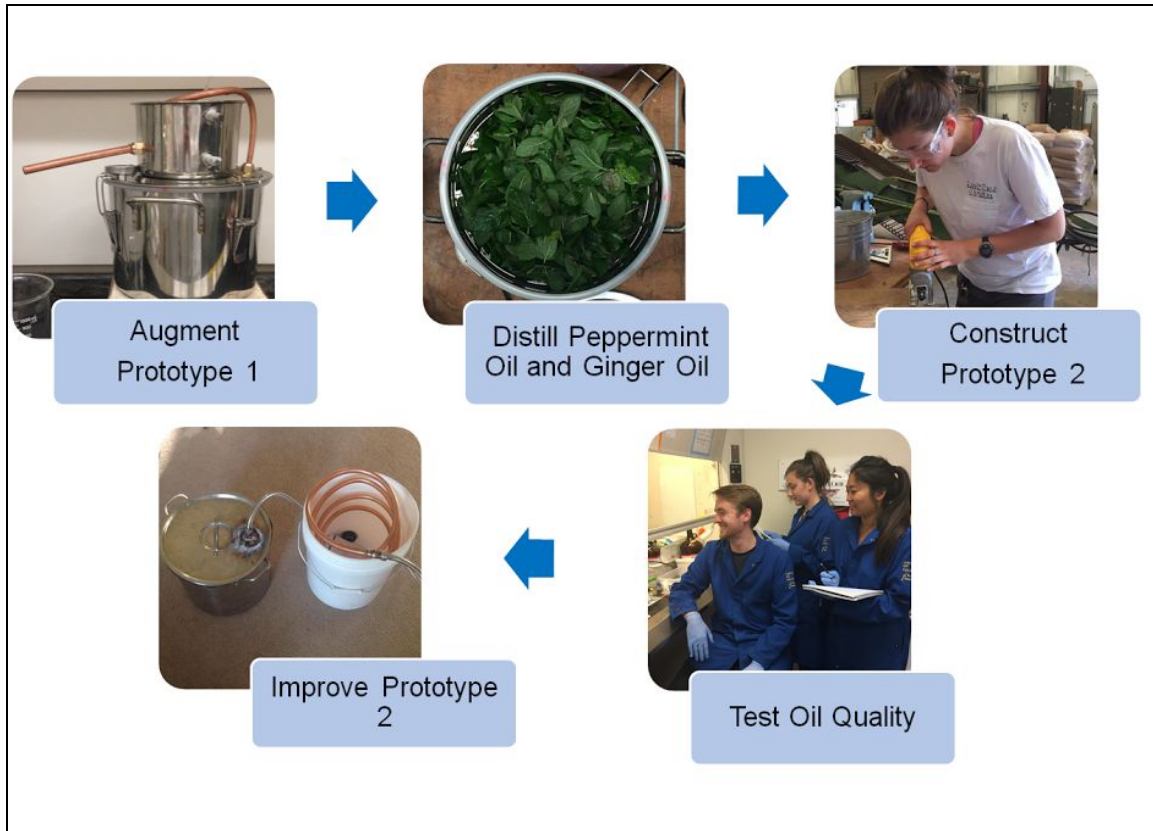

Figure 12: Design approach.

As shown in Figure 12, the design approach involved two prototypes. The first prototype was purchased and customized for essential oil distillation. Using the knowledge gained from the first prototype, prototype 2 was built to meet the parameters of Boa Vista. This prototype was built using materials and techniques that will be available to the team in Brazil.

Conclusions/Recommendations for Future Development

Although significant progress was made on this project, we conclude that projects aimed at alleviating poverty in foreign countries need more time to be completed. There were many instances where our design was insufficient due to a lack of communication with our client or because we were unfamiliar with the parameters of the community. Ideally, the community understanding and relationship would be fully developed long before the prototyping process. We also conclude that joint mentorship, like our team had with the Department of Biological and Agricultural Engineering and D-Lab, was extremely conducive to a successful engineering design project.

Our team has three major recommendations for future development. We recommend improving the heat exchanger material, the technique of oil/hydrosol separation, and the building/device costs. The heat exchanger material should ideally be made out of a material that is not copper because copper is reactive with some volatile plants compounds. However, copper was also an ideal material because through soldering (a technique unique to copper) we created the seals of the heat exchanger. For now, we suggest keeping copper as the material, but doing thorough research into the plant that will be distilled to ensure it will not react. As a final design, a stainless steel heat exchanger pipe is preferred.

Our second recommendation is to improve the method of separation between the oil and the hydrosol. Our team only distilled oils with Prototype 1, which was too small to produce enough product to separate. We suggest distilling with distillation devices larger than five gallons, or investigating chemical or filtration methods to extract the oil is using a smaller device. In our experience, stopcock and buret techniques were not sufficient for separation.

The third and final recommendation was to reduce the cost of the distillation device. Our device cost was within ten dollars of distillation devices found online. Although our device has the advantage of being fixed with easily replaced parts, our team would still like to see a reduction in cost. Because a high quality boiling chamber is prioritized, we suggest investigating a cheaper way to transfer the water in and out of the heat exchanger.

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APPENDIX A: Calculations.

Calculation of plant material required per distillation batch:

Our team would like our prototype 2 to produce five, 10 mL bottles per distillation batch. Considering that only 8 mL of oil should be placed into each 10 mL bottle, and the density of oil is 0.898 g oil per mL (Alankar, 2009):

$$5 \text{ bottles } * \frac{8 \text{ mL oil}}{1 \text{ bottle}} * \frac{0.898 \text{ g oil}}{1 \text{ mL}} = 35.92 \text{ g oil}$$

While peppermint has a yield of 1% w/w and ginger has a yield of 1.5% w/w , our team will consider the conservation peppermint oil yield (Alankar, 2009)(Mohamed, 2004):

$$35.92 \text{ g oil} * \frac{100 \text{ g plant material}}{1 \text{ g oil}} = 3592 \text{ g plant material}$$
$$3592 \text{ g plant material} * \frac{1 \text{ lb}}{453.592 \text{ g}} = 7.92 \text{ lb plant material}$$

Calculation for water required per distillation batch:

To have adequate water using a standard stove-top, or a heating plate which emits 1.5 kW (Nuwayhid, 2003):

$$m'[kg/hr] = \frac{power \text{ } [kJ/s]}{enthalpy \text{ } of \text{ } water \text{ } vaporization \text{ } at \text{ } 20C \text{ } [kJ/kg \text{ } H20]} * 3600 \text{ } [s/hr]$$

$$m' = \frac{1.5 \text{ } kJ/s}{2260 \text{ } kJ/kg \text{ } H20} * 3600 \text{ } s/hr * \frac{L \text{ } water}{kg \text{ } water} = 2.39 \text{ } \frac{L \text{ } water}{hr}$$
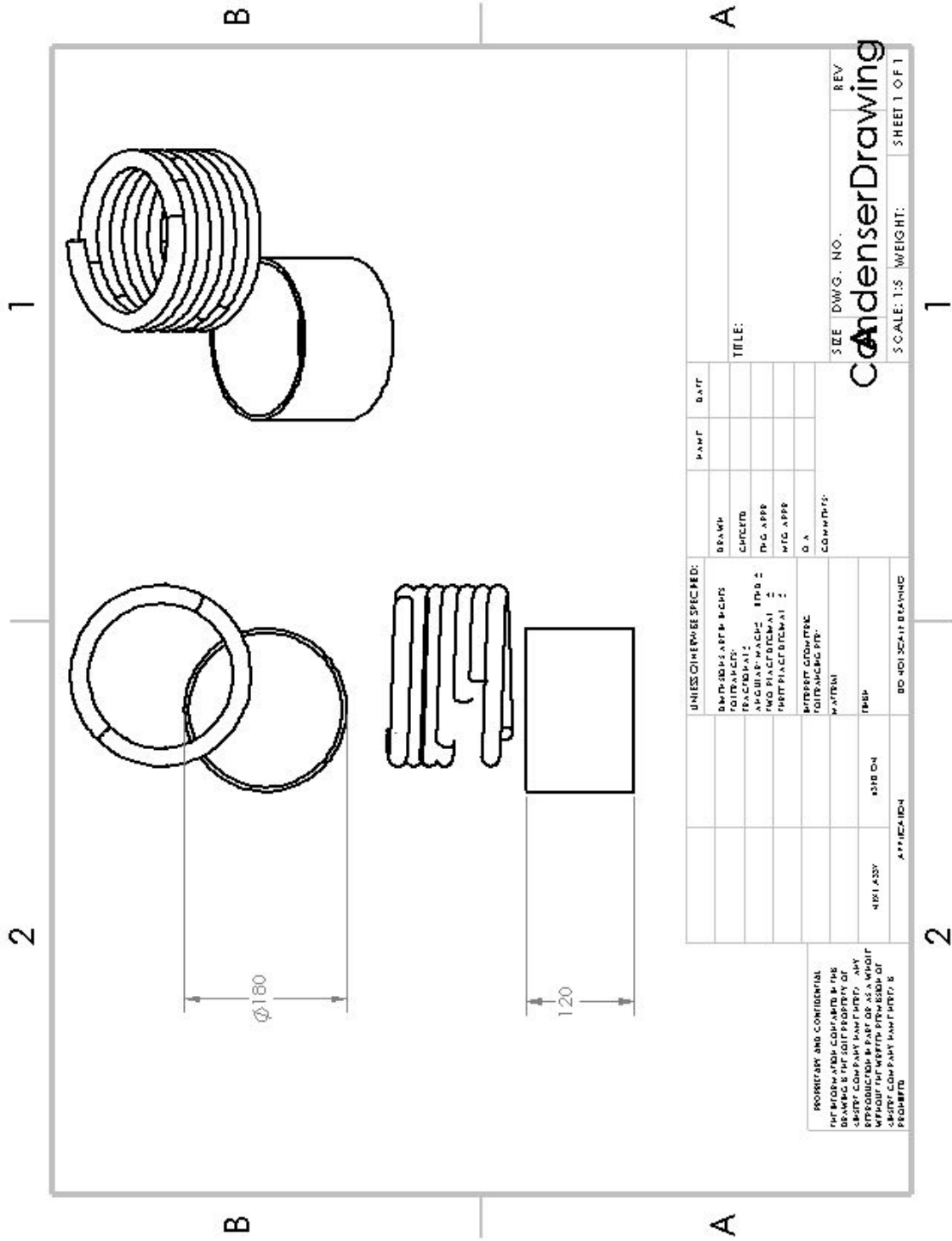
Based on previous literature, volatile compounds are released from peppermint after two hours and after four hours for sliced ginger (Alankar, 2009)(Mohamed, 2004).

$$Water \text{ } required \text{ } for \text{ } peppermint = 2.39 \text{ } \frac{L \text{ } water}{hr} * 2 \text{ } hr = 4.78 \text{ } L \text{ } water$$
$$Water \text{ } required \text{ } for \text{ } ginger = 2.39 \text{ } \frac{L \text{ } water}{hr} * 4 \text{ } hr = 9.56 \text{ } L \text{ } water$$

Therefore, prototype 1 (volume 8 L) has an adequate volume for testing peppermint but not for testing ginger. Prototype 2 has been designed to handle a sufficient amount of water to distill both herb types that the team may encounter, leaves and roots.

APPENDIX B: Schematic of main vessel.

APPENDIX C: Schematic of condenser vessel.

APPENDIX D: Steam Distillation Procedure, Prototype 1

Materials
- Plant material
- Heat source
- Distilled water (~15 C)
- OLizee 2 gallon stainless steel distiller
- 1 L stopcock
- 1 L beaker
- Amber-glass containers

Methods

Preparation of plant materials:

Mint leaves

The yield of peppermint essential oil is roughly 1% (w/w). This approximation is from literature we read about the yield of peppermint oil. (Mohamed, 2004) (Alankar, 2009). To produce ~5 grams of oil, 1.5 pounds of peppermint leaves with an initial moisture content 0.85% w.b. will be needed (Colack, 2008). Record the moisture content of the root in triplicate using a laboratory oven.

Ginger

After calculations displayed in Appendix A, prototype 1 is not large enough to contain water for four hours of distillation. This section of the protocol will be used for the prototype 2 procedure.
The yield of ginger essential oil is roughly 1.4%. This approximation is from literature we read about the yield of ginger oil (Mohamed, 2004) (Alankar, 2009). To produce ~5 grams of oil, 1.0 pounds of ginger slices at an initial moisture content of 0.69% (w.b.) will be needed (Waheed, 2014). Once the ginger is obtained, chop the root into thin slices. Record the moisture content of the root in triplicate using a laboratory oven.

Distillation:

Once the herb/root have been obtained and moisture contents recorded, distillation may occur. Fill the boiling chamber with room temperature water until the water level is just below the grate (~5L). Once the grate is in place, add the peppermint leaves. Secure the lid on the boiling chamber using the clamps. Fill the condenser vessel with cool water, approximately 15°C. Place a sterile 1 L beaker beneath the condenser output to collect the hydrosol and oil mixture. Apply heat to the bottom of the boiling chamber using electric or wood burning sources for two hours for peppermint and four hours for ginger (Mohamed, 2004) (Alankar, 2009). The extraction process is continued until no more essential oils are obtained.

Separation:

Place the hydrosol and oil solution into a 1 L stopcock. Filter the hydrosol liquid from the oil, and place the oil into amber-glass bottles for further testing. Label all samples and store in a dark, room-temperature location.

APPENDIX: Oil Quantity Testing Protocol

Oil quantity is an important measure of distillation success. An inadequate yield of oil is an indicator of in error either plant material preparation or distillation.

Materials:
Stop cock
50 mL Graduated Cylinder

Methods:
1. Once distillation has concluded, drain the hydrosol from the stopcock.
2. Drain the separated oil into a sterile graduated cylinder.
3. Record volume, and compare to literature values.
4. Store the oil in amber glass bottles for further testing.

APPENDIX F: Oil Quality Testing Protocol

Peroxide Value (PV)
The peroxide value (PV) is a parameter used to quantify the extent to which an oil sample has undergone oxidation. It shows the amount of peroxides in a substance by finding the amount of oxygen milli-equivalents (mEq) per kilogram of fat. Peroxides of organic compounds can be skin irritants and can cause allergic reactions depending on the molecule. Although the PV does not directly measure the aromatic quality of the oil it can provide valuable safety information for human use (Wabner, 2002). The procedure was from Professor Pan's lab, when graduate student Bei Wang walked us through the procedure on February 2, 2017.

Materials:
- Mass Balance
- Metrohm 848 Titrino plus
- Pipets
- Stop Watch
- 50 mL Erlenmeyer Flask

Reagents:
- Potassium Iodide
- Acetic Acid
- Isooctane
- 0.025 and 0.1M $Na_2S_2O_5SH_2O$
- Distilled Water

Methods:
1. Weigh 2.0 +/- 0.01 grams oil sample into 50 mL flask
2. Add 20 mL of a 3:2 solution of Acetic Acid to Isooctane
3. Add 0.5 mL of Potassium Iodide with a volumetric pipet
4. Allow the solution to stand with occasional mixing for exactly one minute
5. Immediately add 15 mL of distilled water
6. Titrate with 0.025 M $Na_2S_2O_5SH_2O$ until the yellow iodine color has almost disappeared
7. Conduct a blank determination of the reagents daily the blank titration must not exceed 0.1 mL of 0.1 M $Na_2S_2O_5SH_2O$

Calculation:
$$\text{Peroxide Value (mEq)} = \frac{(V-x)(0.25M)(1000)}{\text{mass of sample (g)}}$$
Where V is the reading from the Metrohm 848 Titrino plus in the main test , and where x is the reading in the blank test. The peroxide values for peppermint and ginger oil will be taken from the ISO oil standards (ISO 856: 2006(en) & ISO 16928:2014(en)).

Acid Value (AV)
The acid value (AV) represents the amount of acidity of oil by measuring the amount of Sodium Hydroxide (NaOH) required to neutralize organic acid groups in one gram of oil. Free fatty acids contribute to distinctive smells that affect the quality of oil (Paudyal, 2012)

Materials:
- Mass Balance
- Metrohm 848 Titrino plus
- Pipets
- 50 mL Erlenmeyer Flask

Reagents:
- Ethanol
- Diethyl Ether
- Phenolphthalein indicator
- Sodium Hydroxide

Methods:
1. Weigh 1.0 gram sample of peppermint of ginger essential oil into a 50 mL flask
2. Add 20 mL of a solution 1:1 of ethanol to diethyl ether
3. Add 1-2 drops of phenolphthalein indicator
4. Titrate with 0.1 M of Sodium Hydroxide

$$\text{Acid Value} = \frac{(V)(56.1)}{\text{mass of sample (g)}}$$

Where V is the volume (mL) of NaOH titrant used. The sample mass is referring to the mass of either the ginger or peppermint oil sample used.

Table 4. Acid value ranges for peppermint and ginger essential oil

Essential Oil	Acid Value Range
Peppermint	1-1.4
Ginger	6-8

Values from Table 4 were from the International Journal of Pharmaceutical and Phytopharmacological Research and the Asian Journal of Pharmaceutical and Clinical Research (Mohamed, 2004) (Alankar, 2009).

Relative Density

Relative density uses the a device called a pycnometer to compare the density of a sample fluid to the density of water in order to determine the sample fluid's unknown density. (UCSC, 2011)
Materials:
- Pycnometer
- Distilled Water
- Sample Fluid
- Mass Balance

Mehods:
1. Determine and write down the weight of empty dry pycnometer, m_0.
2. Add water such that pycnometer as well as capillary hole in the stopper is filled with water. Dry the spare water that leaks through the capillary hole with a filter paper and measure total weight m_1.
3. Empty the pycnometer. Rinse it once with the liquid whose density you are going to determine next. Fill pycnometer with the liquid as previously and measure the weight m_2.
4. Clean pycnometer carefully after finishing the experiment. Rinse it with distilled water and let dry.
5. Measure the laboratory temperature t, which determines the temperature of examined liquids and solid objects.
6. Calculate the weight of water $m_{H2O} = m_1 - m_0$
7. Calculate the pycnometer volume V following Equation 1
8. Calculate the weight of measured liquid $m_L = m_2 - m_0$ and determine density of liquid according to Equation 2.

$$V_T = V_{H2O} - V'_{H2O} = \frac{m_{H2O} - m'_{H2O}}{\rho_{H2O}} \text{ (eq 1)}$$

$$\rho_L = \frac{m_L}{m_{H2o}}\rho_{H2O} \text{ (eq 2)}$$

Table 5. Densities of peppermint and ginger oil

Essential Oil	Density Range (g/mL)
Peppermint	0.900-0.916
Ginger	0.870 - 0.882

The values from Table 5 were taken from the Food and Agriculture Organization of the United Nations (Plotto, 2002).

Refractive Index

The refractive index of essential oil measures how the oil bends light. It measures the change of the speed of light when passing through an oil. The index varies depending on the fluid being measured, however it is a simple method to ensure the quality of oil (Robinson, 2006).
Materials:
- Refractometer
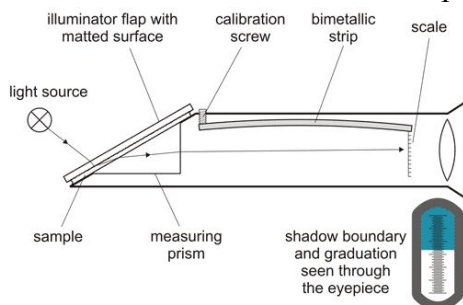- Pipette
- Daylight
- Sample Oil

Figure 6. Common handheld refractometer diagram (Robinson, 2006)

Methods:
1. Position the refractometer so that the measuring prism is open to the daylight
2. Pipette two to three drops of the oil sample onto where sample is indicated on Figure 6
3. Close the illuminator flap and press it so that the sample spreads across the entire surface of the prism without any air bubbles or dry spots
4. After thirty seconds look through the eyepiece to view the refractive index
5. Rinse the main prism with distilled water and pat dry

Table 6. Refractive index range for peppermint and ginger oil

Essential Oil	Refractive Index Range
Peppermint	1.457-1.467
Ginger	1.4880-1.4950

The values from Table 6 were from standards listed by journals from The 4th Annual Seminar of National Science Fellowship and the Asian Journal of Pharmaceutical and Clinical Research (Mohamed, 2004) (Alankar, 2009).

APPENDIX G: Safety Protocols/ Job Safety Analysis

Oil Distillation

Job Function	Potential Hazards	Safe Practice/Equipment
Handling and moving heavy items and equipment	• Ergonomic hazards including muscle strain, awkward motions, heavy items, crushing or pinching injuries	• Get help lifting all items that cannot be safely moved by one person. • Use mechanical means to move heavy objects at all possible times. (e.g cart, dolly) • Use proper lifting techniques. For example use your legs to pick up items off the ground.
Steam/Boiling Water	• Physical hazards including skin burns and respiratory stress.	• Wear proper personal protective equipment such as gloves, long sleeves, long pants, respirator mask and eye protection.
High Pressure	• Safety hazards include potential explosions and flying objects.	• Properly attach safety clamps to the lid of the boiling chamber. • Pay attention to the pressure levels based on the gauge values. • Do not leave distiller unattended.

Testing

Job Function	Potential Hazards	Safe Practices/Equipment
Chemical Storage (AV & PV)	• Chemical hazards include fire, combustion, corrosion, toxins, and extreme temperatures	• Read the labels and the MSDS protocols for each chemical to ensure proper storage. • Store all chemicals in their designated container, shelf, and cabinet.
Chemical Handling (AV & PV)	• Chemical hazards include illness, burns, respiratory stress, skin irritation, and toxins.	• Read the labels and the MSDS protocols for each chemical to ensure proper handling. • Wear proper personal protective equipment such as gloves, long sleeves, long pants, respirator mask and eye protection
Metrohm 848 Titrino plus (AV & PV)	• Chemical hazards include illness, burns, respiratory stress, skin irritation, fire, combustion, corrosion, toxins, and extreme temperatures.	• Read relevant SOP • Handle all chemicals carefully and use clean storage containers. • Check chemistry of the reaction to ensure stable reaction.
Refractometer	• None	• Read relevant SOP and user manual
Pycnometer	• Physical Hazards include broken glass and flammable fluids.	• Be careful when handing pycnometers as they are made of glass and are extremely fragile. • If fluids spill, clean up immediately