

## **Microwave Extraction of Essential Oil from Peppermint - Field Trial**

David Hackleman, Connor J. Smith and Jonathan Lebsack

Oregon State University, Corvallis, Oregon

### **Abstract**

An in-field test of the solvent free microwave extraction of peppermint oil was held in mid-August 2009 at Butler Valley Farms in Stayton, Oregon. Results of the trial were limited due to rapid coalescence of the mint oil prior to entry into the separator. This resulted in no mint oil actually received at the normal receiving site. A small quantity of mint oil was discovered during take-down and laboratory analysis indicates it to be pure. For the Solvent Free Microwave Extraction (SFME) system to work effectively in the large scale (farm operation) condition, it is believed that a significant change in the condensation apparatus will be necessary. Work demonstrating a solution to that issue will be presented. The 100KW 915MHz microwave unit (normally used for drying) performed well in the farm environment and after significant modifications, feed systems were developed to take chopped mint hay from existing mint tubs and move it into the extractor. The results of this field trial were adequate to enable design improvements for future development.

### **Introduction**

Peppermint has traditionally been a major crop in the northwest and in 2010 California, Idaho, Oregon, and Washington grew a combined total of 56,700 acres of peppermint (Jenkins 2012). Extracted mint oil is sold through distributors and specialty oil formulators such as A.M Todd and I.P. Callison to major brand companies such as Wrigley and Proctor and Gamble, to be used in cosmetics and food products.

In order to obtain the essential oil, it must be extracted; with steam distillation being the most commonly used method for extraction of mint oil from peppermint plants. It uses steam to break apart the oil glands and transport the peppermint oil out of the mint. The two main components of mint oil are menthone and menthol, which have boiling points of 207°C and 212°C, respectively (Sandborn 1929, C.R.C. 1971). Due to their large mole fractions the boiling point of peppermint will be somewhere within this region. The steam is able to remove the mint oil without reaching the boiling temperatures of menthone and menthol via Raoult's Law. Raoult's Law can only be considered valid when the system is ideal and all intermolecular forces are the same (Koretsky 2004). This case can be considered ideal because it is treated as a dilute system; the amount of mint oil in the mixture is considered to be approximately 1% mole fraction. At 1% we can assume the vapor pressure and boiling temperature will be very close to that of water.

The generation of steam is a very energy intensive process and the rising cost of natural gas and diesel has inspired research into possible ways to improve efficiency and yield, along with decreasing the carbon footprint. Additionally, due to a recent spike in diesel prices many farmers have been converting their boilers to natural gas. Natural gas is a much cleaner burning fuel and the conversion has only a 1-4 year payback. However, it is not clear if natural gas will remain at such a low price. The purpose of this

research was to explore more sustainable options for the extraction process. Rocky Lundy, Executive Director of the Mint Industry Research Council, reported that 44% of carbon emissions from the entire process come from steam distillation (Gregory 2012). A continuous microwave extraction process has been proposed, which promises a greater efficiency and oil yield utilizing the more stable energy price of electricity. However the open ends of the microwave allows air to enter the mixture. The air insulates the steam and traps it as humidity resulting in a loss of product. An improved condenser design is necessary if SFME is adopted and put to use for the extraction of mint oil.

In SFME mint is exposed to microwave radiation via a microwave applicator. The microwaves increase the vibrational energy in the water molecules, this continued increase in energy causes the water to boil and change phase. SFME been shown to be able to remove up to 65% of available oils and take anywhere from 1.5-45 minutes. SFME in comparison to steam distillation can yield up to three times the amount of oil and due to shorter processing times reduce the cost of energy (Velasco 2007, Lucchesi 2004). One main difference between SFME and steam distillation is the source of the steam for the extraction. In SFME the steam is generated from water remaining in the leaves of the plants. This causes greater disruption of the surface of the leaves and is hypothesized to be one of the reasons more oil is extracted.

Scanning electron microscopy (SEM) pictures were taken before and after mint leaves were processed by the solvent free microwave method. The before and after SEM images are shown in Figures 2.51 and 2.52 respectively (Velasco 2007). The images show the peltate glandules have broken apart after the microwave process has occurred. Similar SEM images have been reported for essential oil extraction of cardamom seed. Researchers observed differences in solvent free microwave extraction from hydro-distillation, showing that cells are broken and damaged much more noticeably during SFME (Lucchesi 2007).

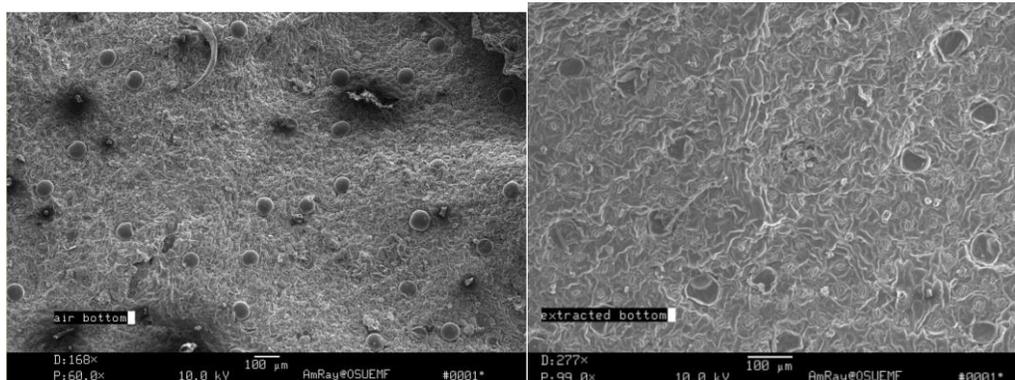


Figure 1: (left) SEM Peppermint leaf prior to microwave extraction. (right) SEM Peppermint leaf post microwave extraction (Velasco 2007).

## Experimental

Mint hay is received via a standard mint tub into an in-feed conveyor unit with counter rotating vanes above the belt to produce a constant four inch thickness layer of mint hay for deposition on the microwave applicator conveyor. A spray bar enables the

addition of water in the event the mint hay is deemed too dry for processing. It is then conveyed through a microwave choke unit and into the microwave applicator section. After entering the applicator, microwave energy (915 MHz) is applied to the mint hay and absorbed by the water in the hay. The microwave applicator system employed was a standard prototype testing unit from Industrial Microwave Systems, LLC (IMS) of Morrisville, NC. The unit selected included a 3-foot, 7-inch wide belt material application region. Roughly up to 150 Amperes are used during normal operation to produce up to 100 KW microwave power. A “circulator” is inserted between the generator and the transmission wave guide assembly. This unit directs reflected power (if any) that returns toward the generator into a termination device.

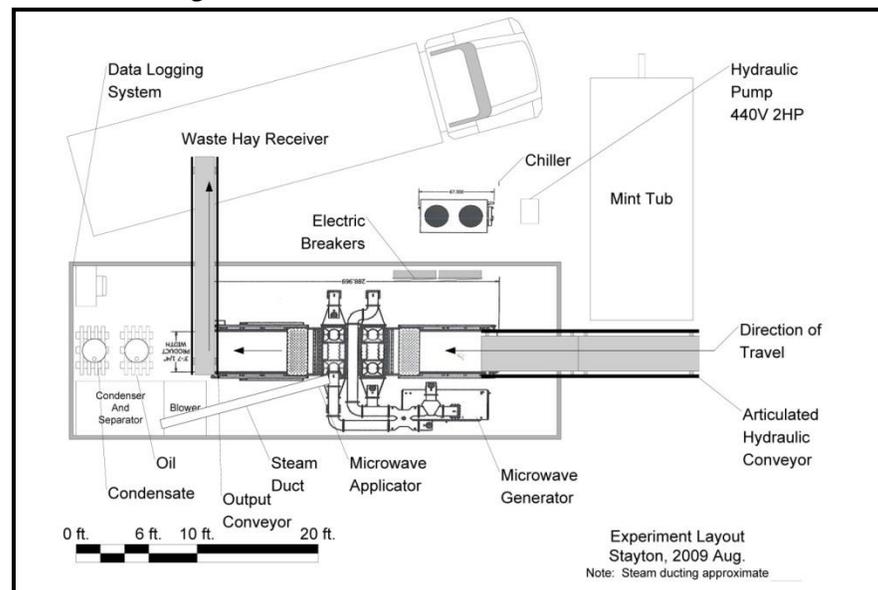


Figure 2: Experiment Layout (to scale)

The heated mint hay continues through a region within the applicator of slight negative pressure which is designed to withdraw the vapors to the condensing system. The hay then passes through another microwave choke unit to contain the microwave energy within the applicator region and then the hay is transported onto the open exit portion of the applicator belt. The microwave applicator belt then deposits the spent mint hay onto the out-feed conveyor belt which transports it to the waste receiving truck.

The mint oil, steam and air vapor mixture is drawn from the microwave applicator through an 8” round duct to a standard “DU-100” condenser and separator system manufactured by Newhouse, Inc. of Redmond, OR. The duct route included an eight foot vertical rise above the microwave applicator and a gradual incline down to the receiving end of the vertical tube and shell condenser, similar to that experienced from a standard mint tub to a condenser in a conventional system. To enable the vapor transport to the condenser, a variable speed blower was attached to the exhaust portal of the condenser to produce a modest negative pressure at the inlet end of the condenser. This was deemed

necessary as the vapor transported from the microwave applicator is a mixture of steam, mint oil vapors and air drawn in from the ends of the open conveyor belt containing mint hay. The variable nature of the exhaust blower was to enable total flow rate to be controlled based on the rate of steam condensation and hence negative pressure applied during the process when vapors arrived at the condenser. The exhaust air line then travelled from the building. The condensate line from the tube and shell condenser was attached directly to a standard “teepee” condenser of Newhouse design, identical to the other such units at the Butler Farm operation. From the separator, an oil transport line was sent to a barrel on a platform balance and the water separated was sent to another barrel on a separate platform balance.

In order to measure the specific conditions during the operation, a set of sensors, associated data logging equipment and software were assembled. Pressure transducers, thermistors and a vortex flow meter were ordered from Labjack® and Omega Engineering Inc.<sup>1</sup> Current sensors for measuring the energy demands of the blower, hydraulic conveyor belts, and microwave generator were obtained from Enercorp Instruments Ltd<sup>2</sup>. A barrel scale for measuring condensate flow rate was purchased through Rice Lake Weighing Systems<sup>3</sup>. Analog signals of temperature, pressure, weight, flow, and current were wired into a Labjack® U3 PLC unit ordered from Labjack Corporation<sup>4</sup> and interpreted with the software package, “DAQFactory<sup>5</sup>.” The flow meter was attached to the condenser cooling water input line and output both temperature and flow rate. Thermistors were installed on cooling water output, vapor input, and condensate output lines. Pressure sensors were placed at the vapor input and output streams to ensure that exit pressure was high enough to prevent condenser shell collapse. The Newhouse Tube and Shell condenser comes equipped with a thermistor sensor on the condensate line as well as a flow controller that increases coolant flow on demand from a set point. The microwave generator and applicator system had incorporated within it three IR sensors above the belt track in the applicator to allow monitoring of the hay surface temperature to take place. Power sensors on the microwave system enabled the measurement of forward, reflected and “pass through” power. Pass through power is based on energy that, for example, is not absorbed by the mint hay and continues along the applicator wave structure to a termination load. Above the exit site for spent hay from the applicator, an Infra-red detecting camera was imaging and observing the surface

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<sup>1</sup> Pressure transducers: Solid State Pressure Transducer, 0 to 5 Vdc output. Actual units used were the model PX-209-030A5V. [http://www.omega.com/pptst/PX209\\_PX219.html](http://www.omega.com/pptst/PX209_PX219.html) ; Temperature probes: <http://labjack.com/catalog/ei1034-temperature-probe> ; Flow meter: FV105-T with 24V DC supply: <http://www.omega.com/pptst/FV100.html>

<sup>2</sup> Current sensors: <http://mcmaster.com/#6583k26=2fyr91> (200 ampere, clip-on 5V output model 6583K26).

<sup>3</sup> Rice Lake RoughDeck® BDP with IQ-355 indicator and Analog output: <http://www.ricelake.com/product.aspx?CatID=1852>

<sup>4</sup> LabJack U3 PLC unit: [http://www.labjack.com/labjack\\_u3.php?prodId=52](http://www.labjack.com/labjack_u3.php?prodId=52) ; with C B-15 terminal board: <http://www.labjack.com/details.php?prodId=28&category=4>

<sup>5</sup> DAQFactory is available from Azeotech: <http://www.azeotech.com/index.php> .

temperature of the exiting hay. It was believed that a hay exit temperature on the order of 105°C would be satisfactory in that the hay temperature would have exceeded the boiling point of water and yet not initiated combustion. Such was set and reproducibly accomplished with an input power of 45 KW and a belt speed of 3 fpm. Upon establishment of these operating conditions, large quantities of steam and vapor were emitted within seconds from the mint hay samples while in the applicator and modest quantities continued to be emitted from the hay upon exit from the applicator region.

## Results and discussion

During the entire process, no mint oil was received in the separator unit. However, when the condenser line was disconnected from the separator, a stream of fluid was observed that had the distinct odor of pure mint oil. A small portion of this fluid was captured and analyzed via gas chromatography and the results are pictured in Figure 3. In essence, it appears that more Menthol and less Menthone were found in the oil extracted than in that from the normal process. A tube and shell heat exchanger was used to condense the steam and oil stream. However, the tube and shell was unable to successfully condense much of the mixture. It was determined that air was getting drawn into the stream with the steam and mint oil through the open ends of the microwave. It is

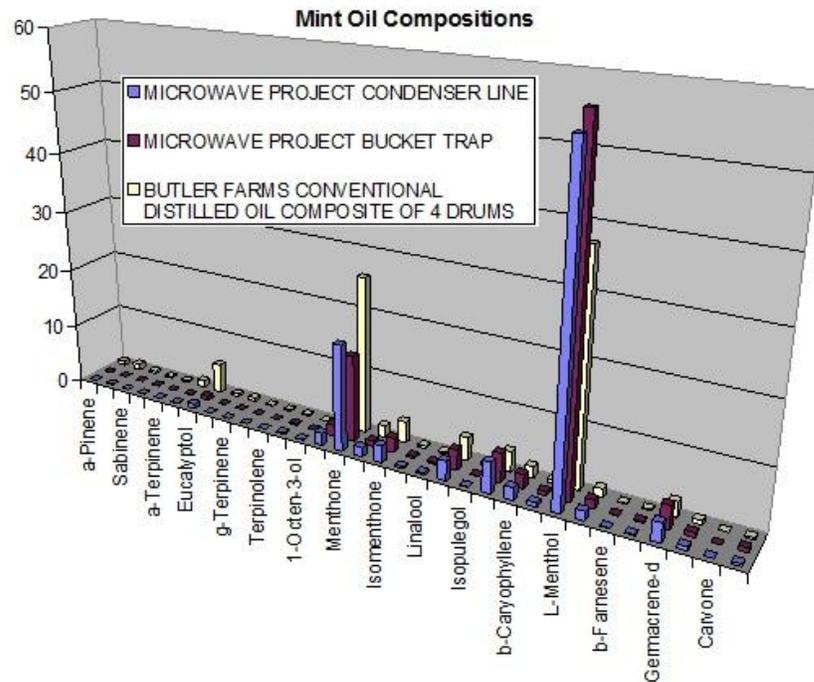


Figure 3: Recovered mint oil composition

known that in the presence of a noncondensable vapor, tube and shell condenser performance will be greatly reduced (Collier 1981). This is due to a gas boundary layer developing as condensation is formed along the condenser tube and it is this boundary

layer which resists the steam from condensing (Seunguim 2006). It is more difficult to condense because the air acts as an insulator against heat transfer.

Microwave extraction could also change the way that farmers harvest their crop. Currently after being cut the plants must sit in the field to dry for several days to enable a chopping process to take place on the plant material. Research suggests that during this waiting time period no significant mass of oil is lost (Gershenzon 2000). However there is a significant risk of weather conditions affecting the oil in the plants. In addition, the composition of the oil is changed during this drying period. After drying the hay is then chopped and placed into large mobile trailers called mint tubs for processing. The microwave system requires higher moisture content in the mint hay than steam distillation during processing or ignition and combustion can take place. Table 1 shows some of the power ratings and belt speeds used for the microwave experiments.

Table 1: Data for experimental conditions on 8/19/09

Run	Belt Speed [FPM]	Power [KW]	Comments
1	3	40	Airflow 10% Using 4-port
2	3	50	44gpm-condenser
3	3	60	Water spray on
4	3	70	Airflow 15%
5	3	70	15%, higher spray dispersion
6	1.5	50	Slow belt down for more oil
7	1.5	60	Steam emitting from front section
8	1.5	70	Hay burning took place after 20
9	3	30	“Y” adapter in place (2-port
10	3	30	Operating normally
11	3	30	Restarted (1-port collection)
12	3	35	Continued – power increase
13	3	35	Water condensing – very slow
14	3	40	Operating, condensation rate
15	3	45	Same
16	3	50	Operation halted due to arc
17	3	45	No arc, operation restarted
18	3	45	Hay output temp 105°C +/- 1C

Slow belt speeds and higher power rating resulted in higher outgoing temperatures and in some cases combustion. The mint collected for these experiments was taken from a farm after it had been chopped and dried for steam distillation. To avoid combustion, additional water had to be sprayed onto the mint before passing it through the microwave. More testing is needed but this is a strong indication that mint could be cut and immediately processed via SFME.

Later in 2009, after the tests in Oregon, a SFME pilot plant was assembled in North Carolina to test the efficiency of the microwave process on a larger than lab scale. Results from the experiments showed that the tube and shell condenser was unable to effectively condense the mint oil. The problem was determined to be the addition of air

to the mixture due to the open ends of the microwave. However it was discovered that a spray scrubber after the condenser was able to collect a visible amount of oil. This inspired the design of a direct contact condenser (Pommerenck 2012).

The direct contact condenser they designed, built, and tested showed vast improvements in steam capturing efficiency when compared to a tube and shell condenser. However due to the materials used for its construction it could not sustain operating temperatures seen in the microwave pilot plant. Using their design a new direct contact condenser was built using materials that would be able to withstand increased temperatures. The condenser was constructed out of aluminum and contained stainless steel spray nozzles, both for their non-corrosive properties. Tests were conducted using 8 and 16 nozzles and tested over a range of 20-100% steam by mass. Additional tests were completed using the full 24 nozzles but due to the location of some of the nozzles coolant was lost as an aerosol with no way to quantify the loss. Comparing the data to research completed by Pommerenck et al. on efficiency of a tube and shell condenser used for the mint distillation process found that with increasing amounts of air there is a greater loss of heat transfer. This is believed to be the effects of a boundary layer of the noncondensable fluid, air, which forms along the tube and resists condensation from forming (Seunguim 2006). Pommerenck's tube and shell condenser used a coolant flow rate of 24 L/min while the flow rates tested in this research were 18 L/min and 36 L/min. The direct contact condenser showed a considerable increase in performance even with the smaller flow rate compared to the tube and shell unit, indicating removal of the boundary layer.

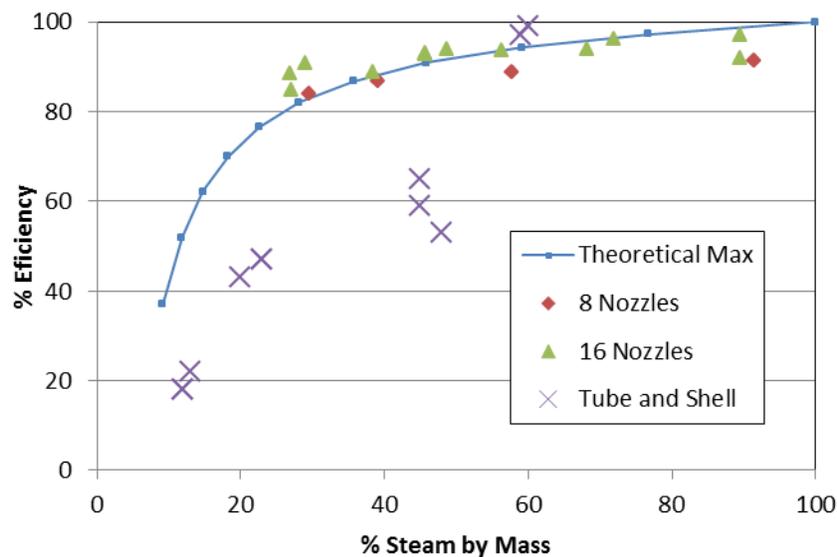


Figure 4: The measured efficiencies plotted against the theoretical maximum for 8 and 16 nozzles. The tube and shell data points reported come from a previously published paper (Pommerenck et al 2012).

The efficiency tends to follow the maximum theoretical efficiency while the tube and shell condenser lowers in efficiency. The overall goal of this project is to determine the feasibility of the use of a direct contact condenser for implementation in the solvent free microwave extraction of peppermint oil when air is present.

## Conclusion

For this experiment, an attempt was made to utilize as much of the traditional process equipment as possible to enable a comparison to be made for just the microwave applicator as a replacement for the essential oil extraction device. From the results, it is clear that a more radical approach is necessary to utilize the microwave system effectively in essential oil extraction. Much of the needed information to enable such a system to be constructed has been obtained from this experiment. The next steps for such a development involve an equipment design and development phase followed by a full scale prototype. The logistics necessary to accomplish this project were daunting as it is a brand new method of extracting essential oils from plants. The author wishes to thank the team and sponsors for the support of this opportunity to explore technology. An additional study was performed at IMS in North Carolina in December, 2009 utilizing 400 pounds of mint hay collected and frozen during this operation.

## Acknowledgements

The authors would like to acknowledge the contributions of Rocky Lundy, Gregory Biza, Kevin Allen, Ted Carter, Wes Miller, Jim Guynn, and Dr. Ganti Murthy. We would also like to thank the Mint Industry Research Council and the OSU foundation for generously funding this research.

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