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Oil Extraction from Orange Zest

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Objective Statement

Optimize orange oil yield using the extraction process and experimental apparatus developed by OilEx Tech.

OilEx Tech Research Data

Our research has been optimizing the extraction of oil from orange zest using microwaves. First the organic oranges were zested using Microplane zesters and used this zest as the biomaterial for experimentation. The orange zest was then distributed in a large glass cylinder with an open top. Inside the glass cylinder the orange zest was distributed around a glass beaker which was surrounded by a wire mesh through which microwaves cannot pass. A metal lid with a cone-shaped piece of ice (referred to as an “ice core”) frozen to it was placed on top of the large glass cylinder creating a closed system. The apparatus was then placed in a microwave with a cup of water to absorb excess energy and exposed to microwaves for a period of 5 ½ -8 minutes. The steam and oil from the zest vaporized and condensed when coming in contact with the ice core. The conical shape of the ice core allowed oil and water to drip down into the beaker surrounded by the wire mesh. The wire mesh kept the water and oil in the beaker from re-vaporizing. After microwave exposure was completed, the beaker, remaining solid biomaterial, and ice core were removed from the apparatus. The content of the beaker were emptied into a graduated cylinder where the volume of oil condensed could be calculated and, using a pipette, the oil was removed from the water in order to calculate oil mass. Volume and mass data for every trial was recorded.

When conducting this experiment, the objective was to maximize the amount of oil extracted in a single trial by manipulating multiple independent variables; duration of microwave exposure, mass of biomaterial sample, size of ice core, power of the microwave, biomaterial distribution, state of biomaterial (fresh or frozen), and cool down time. Among these variables a few remained constant, such as the power of the microwave, biomaterial distribution, and the size of the ice core. These variables were not changed during the duration of testing. The other variables, however, were varied and data was recorded to optimize duration of microwave exposure, mass of biomaterial sample, state of biomaterial, and cool down time.

For zesting the organic oranges, which were provided by “Nearly Normal’s”, Microplane zesters were used. In order to maintain the oil content of the orange peels and minimize evaporative losses outside of the experiment, the peels were frozen before zesting began. When zesting, the top 0.5mm-1.0mm of the orange peel was removed. This thin surface coating contains the majority of the oil. After the zest was successfully extracted, it was mixed in a Ziploc bag to ensure that a well-mixed sample was being used in experimentation.

The first trials varied the amount of time the biomaterial spent inside of the microwave. The mass and state of the biomaterial as well as the cool down time were held constant. The period of microwave exposure ranged from 5 ½ minutes to 8 minutes in 30 second increments using approximately 50 grams of biomaterial each trial. After 6 trials, it was found that the optimal exposure period is approximately 6 ½ minutes.

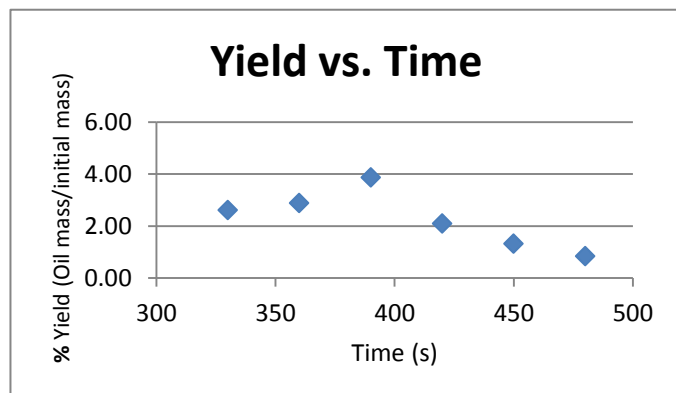


Figure 1 Oil yield vs. microwave exposure time

As the first ten trials were executed, it became apparent that there was a significant difference in the oil yield of fresh biomaterial grated the day of the trial, and the frozen biomaterial that had been previously prepared. Four different trials comparing frozen biomaterial and fresh biomaterial at 6 ½ minutes and approximately 50 grams of biomaterial were performed and conclusively showed that fresh biomaterial yielded much more oil than the frozen biomaterial. It was decided at this point to use only frozen biomaterial to ensure that all results were consistent.

The last independent variable that was optimized was mass that was used during the trial. Time was consistently held at 6 ½ minutes for all trials while mass was varied from 10 grams to 90 grams in 10 grams increments. The final results of these trials showed that 30 grams of biomaterial yields the optimal oil yield by mass of biomaterial. Trials using samples greater than 30 grams did not have higher percent yields by mass of biomaterial.

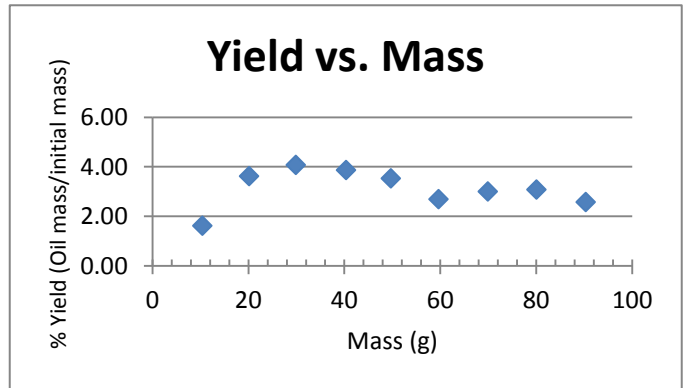


Figure 2 Oil yield vs. mass of biomaterial sample

Hypothetic Maximum Amount of Steam Condensable

$$M_{\text{H}_2\text{O}(\text{Ice})} * \Delta H^{\text{s} \rightarrow \text{l}} = M_{\text{H}_2\text{O}(\text{steam})} * \Delta H^{\text{v} \rightarrow \text{l}}$$

Average Ice Core Mass: .19397 kg

$\Delta H^{\text{s} \rightarrow \text{l}}$: 334 kJ/kg

$\Delta H^{\text{v} \rightarrow \text{l}}$: 2257 kJ/kg

$$.19397 \text{ g} * 334 \text{ kJ/kg} = M_{\text{H}_2\text{O}(\text{steam})} * 2257 \text{ kJ/kg}$$

$$M_{\text{H}_2\text{O}(\text{steam})} = .02870 \text{ kg} = 28.70 \text{ g}$$

Moisture Content of Orange Zest

Moisture Content (Kg H₂O and Oil/Kg dry biomaterial) = $(M_{\text{Initial}} - M_{\text{Dry biomaterial}}) / M_{\text{Dry biomaterial}}$

Appendix A shows M_{Initial} values, $M_{\text{Dry biomaterial}}$ values, and individual moisture content values

Average moisture content of orange zest = 0.995 Kg H₂O and Oil/Kg dry biomaterial

Orange Oil Optimum Yield Recipe

To extract a maximum amount of oil from orange zest using an 1100 watt microwave and an OilEx Tech apparatus, one must first zest approximately 30 grams of orange zest and mix it thoroughly. Place the mesh microwave-resistant wire into the large glass cylinder and place a 200mL beaker inside the mesh cylinder. Evenly distribute 30 grams of zest around the wire mesh. Next, place the metal lid with an attached ice core on top of the glass cylinder. Place the glass cylinder in the microwave and include a cup of water so excess energy is absorbed by this water rather than any other component of the system. Expose the apparatus to microwaves for 6 ½ minutes with approximately 2 minutes of cool down time inside or outside of the microwave after exposure is complete. Finally, remove the beaker and empty it into a graduated cylinder. From there, the oil can be pipetted into a container for storage or mass calculation.

Recommendations for Further Testing

Because so much variance must be accounted for when using orange zest as a biomaterial, executing more identical trials that can be averaged for a single data point will yield better and more accurate results on the relationship between microwaving period and oil yield and the relationship between biomaterial mass and oil yield. Other tests that could have interesting implications are investigation of the relationship between ice core mass and oil yield and the relationship between biomaterial distribution and oil yield. Orientation of the botanical, whether it is spread out evenly or in denser less distributed clumps, may affect the percent oil yield by mass. Furthermore, there may be a linear relationship between the initial mass of biomaterial and the duration it is exposed to microwaves. This will again be related to the ice core mass, as longer duration will melt more of the ice core and require more initial ice mass in order for condensation to continue.

Acknowledgements

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References

Bejar, Asma et. Al. "Effect of Microwave Treatment On Physical and Functional Properties of Orange (*Citrus Sinensis*) Peel and Leaves." *Omicsonline*. Food Processing and Technology, 2011 2.2. Web. 1/15/13. <<http://www.omicsonline.org/2157-7110/2157-7110-2-109.pdf>>.

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Appendix A: Moisture Content Data

| Trial (day.run) | M_Initial (g) | Mass Lost (g) | Dry Mass Md (g) | Moisture Content (M - Md)/Md) (Kg H2O and oil/Kg Dry Biomaterial) |
|-----------------|---------------|---------------|-----------------|--|
| 1.1 | 86.73 | 32.31 | 54.42 | 0.593715546 |
| 1.2 | 85.2 | 27.42 | 57.78 | 0.474558671 |
| 2.1 | 49.96 | 21.26 | 28.7 | 0.740766551 |
| 2.2 | 49.74 | 22 | 27.74 | 0.793078587 |
| 2.3 | 49.54 | 21.53 | 28.01 | 0.768654052 |
| 2.4 | 50.35 | 23.42 | 26.93 | 0.869662087 |
| 3.1 | 50.75 | 26.01 | 24.74 | 1.051333872 |
| 3.2 | 50.1 | 21.75 | 28.35 | 0.767195767 |

Appendix A (continued)

| Trial (day.run) | M_Initial (g) | Mass Lost (g) | Dry Mass Md (g) | Moisture Content (M - Md)/Md) (Kg H2O and oil/Kg Dry Biomaterial) |
|-----------------|---------------|---------------|-----------------|--|
| 3.3 | 50.1 | 21.08 | 29.02 | 0.726395589 |
| 3.4 | 49.81 | 14.32 | 35.49 | 0.403493942 |
| 4.1 | 50.23 | 26.04 | 24.19 | 1.076477883 |
| 4.2 | 49.77 | 22.91 | 26.86 | 0.852941176 |
| 4.3 | 49.9 | 23.88 | 26.02 | 0.917755573 |
| 4.4 | 50.44 | 22.92 | 27.52 | 0.832848837 |
| 5.1 | 50.7 | 19.7 | 31 | 0.635483871 |
| 5.2 | 35.5 | 21.03 | 14.47 | 1.453351762 |
| 5.3 | 34.87 | 21.6 | 13.27 | 1.627731726 |
| 5.4 | 35.06 | 16.91 | 18.15 | 0.931680441 |
| 6.1 | 30.58 | 18 | 12.58 | 1.430842607 |
| 6.2 | 30.12 | 17.73 | 12.39 | 1.430992736 |
| 7.1 | 15.23 | 9.63 | 5.6 | 1.719642857 |
| 7.2 | 30.86 | 14.28 | 16.58 | 0.861278649 |
| 8.1 | 10.48 | 6.62 | 3.86 | 1.715025907 |
| 8.2 | 20.19 | 11.23 | 8.96 | 1.253348214 |
| 8.3 | 29.94 | 17.63 | 12.31 | 1.432168968 |
| 8.4 | 40.39 | 20.69 | 19.7 | 1.050253807 |
| 9.1 | 59.71 | 22.78 | 36.93 | 0.616842675 |
| 9.2 | 69.95 | 27.66 | 42.29 | 0.654055332 |
| 9.3 | 80.06 | 27.06 | 53 | 0.510566038 |
| 9.4 | 90.34 | 28.82 | 61.52 | 0.46846554 |
| Averages | 47.88666667 | 20.94066667 | 26.946 | 0.955353642 |

Appendix B: Raw Experimental Data

| Trial (day.run) | Sample mass (g) | Output oil mass (g) | % yield (Oil mass/initial mass) | Duration (s) | Cool down duration (s) | Fresh/Frozen biomass |
|-----------------|-----------------|---------------------|---------------------------------|--------------|------------------------|----------------------|
| 1.1 | 86.73 | 1.17 | 1.35 | 420 | 240 | Fresh |
| 1.2 | 85.2 | 1.22 | 1.43 | 360 | 240 | Fresh |
| 2.1 | 49.96 | 1.16 | 2.32 | 390 | 240 | Fresh |
| 2.2 | 49.74 | 1.04 | 2.09 | 420 | 240 | Fresh |
| 2.3 | 49.54 | 0.65 | 1.31 | 450 | 240 | Fresh |
| 2.4 | 50.35 | 0.42 | 0.83 | 480 | 240 | Fresh |
| 3.1 | 50.75 | 2.23 | 4.39 | 390 | 240 | Fresh |
| 3.2 | 50.1 | 1.14 | 2.28 | 390 | 240 | Frozen |
| 3.3 | 50.1 | 1.44 | 2.87 | 360 | 240 | Fresh |
| 3.4 | 49.81 | 1.3 | 2.61 | 330 | 240 | Fresh |
| 4.1 | 50.23 | 1.94 | 3.86 | 390 | 240 | Frozen |

Appendix B (Continued)

| Trial (day.run) | Sample mass (g) | Output oil mass (g) | % yield (Oil mass/initial mass) | Duration (s) | Cool down duration (s) | Fresh/Frozen biomass |
|-----------------|-----------------|---------------------|---------------------------------|--------------|------------------------|----------------------|
| 4.2 | 49.77 | 1.76 | 3.54 | 390 | 240 | Frozen |
| 4.3 | 49.9 | 2.94 | 5.89 | 390 | 240 | Fresh |
| 4.4 | 50.44 | 2.49 | 4.94 | 390 | 240 | Fresh |
| 5.1 | 50.7 | 2.39 | 4.71 | 390 | 60 | Fresh |
| 5.2 | 35.5 | 2.34 | 6.59 | 390 | 120 | Fresh |
| 5.3 | 34.87 | 2.09 | 5.99 | 390 | 180 | Fresh |
| 5.4 | 35.06 | 1.5 | 4.28 | 390 | 240 | Frozen |
| 6.1 | 30.58 | 0.93 | 3.04 | 390 | 60 | Frozen |
| 6.2 | 30.12 | 1.31 | 4.35 | 390 | 120 | Frozen |
| 7.1 | 15.23 | 0.46 | 3.02 | 390 | 120 | Frozen |
| 7.2 | 30.86 | 0.78 | 2.53 | 390 | 120 | Frozen |
| 8.1 | 10.48 | 0.17 | 1.62 | 390 | 120 | Frozen |
| 8.2 | 20.19 | 0.73 | 3.62 | 390 | 120 | Frozen |
| 8.3 | 29.94 | 1.22 | 4.07 | 390 | 120 | Frozen |
| 8.4 | 40.39 | 1.56 | 3.86 | 390 | 120 | Frozen |
| 9.1 | 59.71 | 1.61 | 2.70 | 390 | 120 | Frozen |
| 9.2 | 69.95 | 2.1 | 3.00 | 390 | 120 | Frozen |
| 9.3 | 80.06 | 2.47 | 3.09 | 390 | 120 | Frozen |
| 9.4 | 90.34 | 2.33 | 2.58 | 390 | 120 | Frozen |