# IMPROVED LIVE TERPENE EXTRACTION BY MICROWAVE FOR ENHANCED FLAVOR

Competition within the cannabis industry is rapidly growing and producers now find themselves competing for consumers who demand increasingly more personalized experiences. Producing just a strong "high" effect is no longer sufficient to meet consumer demand, and producers are now “wheeled” towards creating products that both meet consumer expectations for both palate preference and the targeted effect. Terpenes are now a cornerstone ingredient for producing highly differentiated, tailor-made cannabis products. Microwave terpene extraction offers cannabis processors the unique ability to capture all pivotal live terpenes from fresh flowers, enabling them to develop strain specific, targeted, and functional high-quality consumer products.

## INTRODUCTION

As the market expands, demand is growing for unique, targeted, and highly differentiated cannabinoid products for targeted “effects,” including sleep enhancement, pain management, improved focus, and increased energy.

Terpenes play an important role in creating differentiation and brand loyalty for the products that compete in different “effect” categories.. Aroma and flavor is characterized by the unique terpene profile contained within every cannabis strain.. These senses are closely linked to memory and experience and, as a result, different terpenes can produce different emotional and even physical responses amongst consumers. The majority of cannabis consumers now seek targeted functional solutions, which requires producers to rebuild the ‘Entourage Effect’ and introduce specific, targeted terpenes.

Consumer products with “live terpene” experiences are considered more appealing to consumers, which is why many manufacturers are looking for different technologies that can enable them to maximize terpene recovery to develop superior products.

The three extraction techniques most commonly used for cannabis today are super-critical carbon dioxide ($sCO_2$), pressurized hydrocarbon, and ethanol extraction (EtOH).

These techniques are effective for producing highly potent consumer products however they offer only limited capabilities for terpene processing.

Milestone's patented microwave terpene extraction solutions, comprised of the ETHOS platforms, XL and X 2.0, offer a unique and specific terpene-driven solution for the production of high quality live terpenes, prior to cannabinoids extraction.

## I EXPERIMENTAL

### INSTRUMENT

- ETHOS XL, equipped with 7.0 kW chiller
- ETHOS X 2.0, equipped with 2.1 kW chiller

### SAMPLE AND REAGENT

- Cannabis samples: fresh frozen buds of Blueberry strain, Slurricane strain, Kosher Kush strain, ACDC strain, Dosi Cake strain, OGKB 2.0.
- Distilled water

### PROCEDURE

*ETHOS XL:* Up to 10 kg of fresh-frozen cannabis material was loaded into material socks (approximately 2.5 kg of material per material sock). The material socks were sealed and placed inside the ETHOS XL cavity. After the pre-loaded method "full load" was selected, approximately 9L of water filled the cavity and the extraction process began.

Figure 1. ETHOS XL

| Time | MW Power [W] | $T_{max}$ [°C] |
|---|---|---|
| 00:30:00 | 3,000 | 102 |
| 01:30:00 | 2,200 | 102 |

Table 1. Typical ETHOS XL extraction method

*ETHOS X 2.0:* Up to 2.5kg of fresh-frozen cannabis material was placed in a mixing container. The cannabis buds were mixed with 1.25L of distilled water for 10 minutes (1:0.5, material:water ratio). The mixture was then transferred into the 15L reactor glass vessel and placed inside the X 2.0 cavity. After the desired pre-uploaded method was selected, the extraction process started.

Figure 2. ETHOS X 2.0

| Time | MW Power [W] | $T_{max}$ [°C] |
|---|---|---|
| 00:05:00 | 1,800 | 102 |
| 00:05:00 | 1,400 | 102 |
| 01:15:00 | 1,600 | 102 |

Table 2. Illustration of typical ETHOS X 2.0 extraction methods utilized

## I RESULTS AND DISCUSSION

One of the biggest drawbacks with conventional extraction methodologies like hydrocarbon and subcritical $CO_2$ is the required use of solvents.

Hydrocarbon extraction: With hydrocarbon extraction, the use of butane and/or propane solvents are essential to the process. This technique enables the process of fresh or fresh-frozen cannabis flowers to capture live terpenes, however recovery requires additional clean-up steps that causes terpenes to be partially purged and lost.

Subcritical $CO_2$: This technique, while considered efficient overall, has several major limitations, including its inability to process wet or fresh-frozen material. This limitation is caused by the carbonic acid that is formed by water and carbon dioxide during the extraction process. The carbonic acid

causes an acidic pH change in the final product, which negatively impacts flavor in wet or fresh-frozen materials. Additional drawbacks with this technique include the significant up-font capital investment required, and the need to hire specially-trained users to run the system.

Microwave-assisted extraction by comparison offers a far more affordable and efficient approach that requires minimal operator expertise to run the system.

Microwave: ETHOS XL and ETHOS X 2.0 utilize microwave technology. Both systems do not require use of solvents, and unlike hydrocarbon extraction, does not necessitate the construction of a specially designed extraction area to house the equipment, in accordance with C1D1 NFPA environmental building code.

Milestone’s ETHOS X products offer a “greener” approach to extraction that does not require the use of chemical or cancerogenic solvents. The technology also utilizes enhanced heating efficiency that reduces energy consumption without compromising terpene taste or quality.

As microwaves pass through the sample matrix, every change in the electric field of the wavelength causes a dipole rotation of the water molecules. This rotation generates friction, which creates a tremendous amount of heat, rapidly. The focalized phenomena distends botanical cells and leads to rupturing of the glands and oleiferous receptacles.

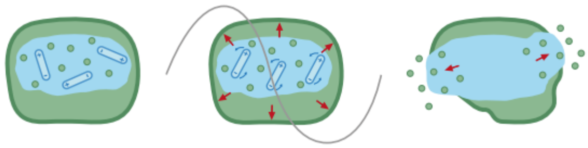

Figure 3. The microwave selective heating process

The heat generated during the process eventually causes water to change from liquid to steam. The increased heat and pressure caused from this steam, releases the terpenes from the plant material. As the steam exits the microwave cavity and enters the distillation head, it carries these newly released terpenes with it. The water and terpenes in the steam is then distilled and separated in two phases according to density.

In this application report, we report the productivity and terpene relative concentration results for many cannabis strains that were microwave processed using ETHOS XL and X 2.0. The material processed and analyzed includes fresh-frozen buds for the following strains: Blueberry, Slurricane, Kosher Kush, ACDC, Dosi Cake, and OGBK 2.0.

Please see table 3 to see recovery concentration percentages for each strain we analyzed. Several other monoterpenes and sesquiterpens were microwave distilled, but only the most predominant and strain specific terpenes were analyzed *via* GC-MS.

Both ETHOS XL and X 2.0 were used in this study for processing 10 kg and 2.5 kg (respectively) per run, resulting in comparable yields (table 4).

| BLUBERRY | | SLURRICANE | | KOSHER KUSH | | ACDC | | DOSI CAKE | | OGKB 2.0 | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| TERP. | RELATIVE CONC. | TERP. | RELATIVE CONC. | TERP. | RELATIVE CONC. | TERP. | RELATIVE CONC. | TERP. | RELATIVE CONC. | TERP | RELATIVE CONC. |
| α-pinene | 6.77% | α-pinene | 1.71% | α-pinene | 2.40% | α-pinene | 11.20% | α-pinene | 2.44% | δ-Limonene | 26.96% |
| camphene | 0.23% | camphene | 0.46% | camphene | 0.60% | camphene | 0.23% | camphene | 0.60% | β -Myrcene | 12.83% |
| β-pinene | 2.81% | β-pinene | 2.54% | β-pinene | 3.79% | β-pinene | 3.97% | β-pinene | 3.77% | Linalool | 6.03% |
| β-myrcene | 36.40% | β-myrcene | 9.51% | β-myrcene | 32.30% | β-myrcene | 72.20% | β-myrcene | 33.10% | α-Humulene | 4.93% |
| α-phellandrene | 0.01% | α-phellandrene | 0.01% | D-limonene | 31.10% | D-limonene | 7.76% | D-limonene | 33.40% | α-Pinene | 2.58% |
| α-terpenine | 0.02% | δ-3-carene | 0.00% | trans-β-ocimene | 0.00% | terpinolene | 0.16% | γ-terpinene | 0.00% | Tans-Nerolidol | 2.26% |

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| p-cymene | 0.00% | α-terpenine | 0.02% | γ-terpinene | 0.03% | L-fenchone | 0.13% | terpinolene | 0.46% | β-Pinene | 1.61% |
| D-limonene | 20.80% | D-limonene | 12.20% | terpinolene | 0.44% | linalool | 1.39% | L-fenchone | 0.19% | Cis-Nerolidol | 1.18% |
| trans-β-ocimene | 19.80% | trans-β-ocimene | 0.04% | L-fenchone | 0.12% | α-fenchol | 0.56% | linalool | 7.40% | Camphene | 0.96% |
| γ-terpinene | 0.03% | γ - terpinene | 0.03% | linalool | 9.43% | α-ocimene | 0.74% | α-fenchol | 2.04% | | |
| trans-4-thujanol | 0.00% | terpinolene | 0.24% | α-fenchol | 2.08% | borneol | 0.13% | α-ocimene | 1.06% | | |
| terpinolene | 0.15% | L-fenchone | 0.27% | α-ocimene | 0.00% | α-terpineol | 0.40% | borneol | 0.30% | | |
| L-fenchone | 0.08% | linalool | 8.31% | borneol | 0.31% | β-caryophyllene | 2.12% | α-terpineol | 1.53% | | |
| linalool | 0.76% | α-fenchol | 1.41% | α-terpineol | 1.57% | α-humulene | 0.61% | geraniol | 0.13% | | |
| α-fenchol | 0.51% | α-ocimene | 0.78% | geraniol | 0.18% | guaiol | 0.11% | β-caryophyllene | 5.94% | | |
| α-ocimene | 1.03% | camphor | 0.01% | β-caryophyllene | 4.88% | α-bisabolol | 0.12% | α-humulene | 1.71% | | |
| camphor | 0.00% | borneol | 0.25% | α-humulene | 1.35% | | | | | | |
| borneol | 0.10% | α-terpineol | 0.89% | α-bisabolol | 0.06% | | | | | | |
| α-terpineol | 0.35% | Nerol | 0.01% | | | | | | | | |
| geranyl acetate | 0.00% | geraniol | 0.04% | | | | | | | | |
| β-caryophyllene | 5.87% | β -caryophyllene | 22.80% | | | | | | | | |
| α-humulene | 1.71% | α-humulene | 5.78% | | | | | | | | |
| trans-nerolidol | 0.03% | caryophyllene oxide | 0.23% | | | | | | | | |
| caryophyllene oxide | 0.07% | α-bisabolol | 0.41% | | | | | | | | |
| α-bisabolol | 0.03% | | | | | | | | | | |
| *Total Mono* | 89.85% | | 38.71% | | 84.35% | | 98.87% | | 86.40% | | 50.97 |
| *Total Sesqui* | 7.70% | | 29.22% | | 6.29% | | 2.96% | | 7.65% | | 8.37 |

Table 3. ETHOS XL and X 2.0 extractions – Terpenes-relative concentration.

| CANNABIS STRAIN | TERPENES (g) | | EXTRACTION YIELD [%]*** |
|---|---|---|---|
| | ETHOS X 2.0* | ETHOS XL** | |
| Blueberry | 16 | 63 | 0.625 |
| Slurricane | 5.5 | 22 | 0.219 |
| Kosher Kush | 16.5 | 66 | 0.661 |
| ACDC | 22 | 87.4 | 0.874 |
| Dosi Cake | 13 | 51.4 | 0.514 |

Table 4. *Extraction Yields using ETHOS XL and X 2.0*

** Up to 2.5 kg material per run*
*** Up to 10 kg material per run*
**** (w/w)*

## CANNABINOIDS

Thus far, we've examined the use of microwave extraction to produce high-quality terpenes. But what happens to critically important cannabinoids during this process?
To evaluate if cannabinoids are impacted by microwave extraction processing, we studied the results of materials that were previously run through microwave extraction to see if there was any residual impact on cannabinoids with subsequent hydrocarbon butane extraction. After running OGKB 2.0 material through our microwave extraction system, these same materials were then dried in a ventilated oven and than run through a hydrocarbon butane extraction system. Table 5 shows the total potential tetrahydrocannabinol (THC) in the post extraction fresh frozen plant material closely matches the pre-extraction values. One considerable difference is that tetrahydrocannabinolic acid (THCA) is almost fully decarboxylated, following microwave assisted extraction.
This proves cannabinoids remain intact in spent plant material, following microwave terpene extraction.

| Compound | *Pre* Microwave treatment | *Post* Microwave treatment |
|---|---|---|
| THCa | 6.043 | 0.954 |
| $\Delta^9$-THC | 0.164 | 3.905 |
| $\Delta^8$-THC | 0.144 | <LOQ |
| THCV | <LOQ | <LOQ |
| CBDa | <LOQ | <LOQ |
| CBD | <LOQ | <LOQ |
| CBDVa | <LOQ | <LOQ |
| CBDV | <LOQ | <LOQ |
| CBGa | 0.22 | 0.157 |
| CBG | <LOQ | 0.195 |
| CBN | 0.087 | <LOQ |
| CBC | <LOQ | <LOQ |
| Total Potential THC | 5.464 | 4.742 |
| Total Cannabinoids | 6.658 | 5.211 |

Table 5. Assessing cannabinoid quality after microwave processing

## CONCLUSIONS

Microwave extraction performed with ETHOS platforms, X 2.0 and XL, successfully enabled the capture and isolation of live terpenes on numerous fresh-frozen cannabis strains prior to cannabinoid extraction. Microwave extraction represents a "greener" approach for capturing valuable terpenes without the use of chemical solvents or other purification steps. Materials processed through microwave extraction can then be successfully dried and run through any cannabinoid extraction protocol without any negative residual impact on the cannabinoid molecules themselves (Figure 4).

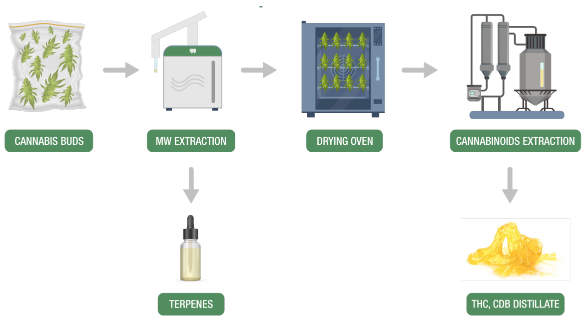


Figure 4. Illustration of the production plant's workflow with microwave processing integrated

Use of microwave extraction enables cannabis processors to organize their production facilities according to cannabinoid and terpene extraction, resulting in a smoother, more efficient, and more profitable facility workflow.

## REFERENCES


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