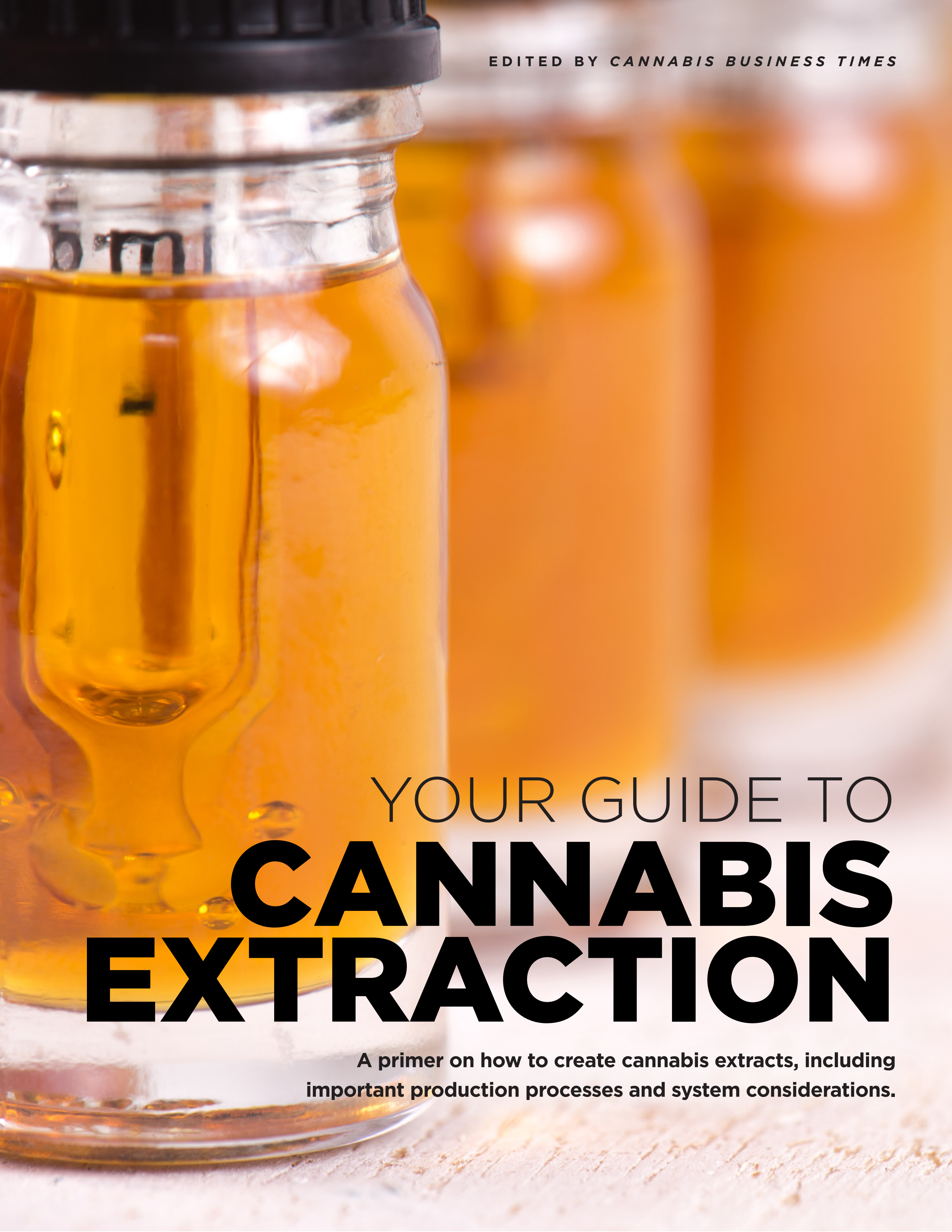

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YOUR GUIDE TO CANNABIS EXTRACTION

A primer on how to create cannabis extracts, including important production processes and system considerations.

CANNABIS
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Introduction

In 2017, flower comprised 50 percent of sales in the consumable cannabis market in the U.S. Half of the market was everything else, including concentrates and edibles, according to a report by Arcview Market Research and BDS Analytics. By 2022, the research firms predict that just 36% of the market will be flower as people become more familiar with the myriad options for consuming cannabis. Offering alternatives to flower is increasingly important. Extraction equipment can cost anywhere from thousands to millions of dollars, so it's important to determine what your company wants to produce, whether it be shatter, tincture, wax, resin, oils for vape pens or other products, before investing in expensive systems. The solvents and extraction methods available are just as varied as the end products that can be created with them.

Cannabis Business Times publishes extraction tips written by industry experts to help business owners determine which extraction method may be best for them and how to properly execute each. Now these articles can be found in one place. "Your Guide to Cannabis Extraction: A primer on how to create cannabis extracts, including important production processes and system considerations" delves into different extraction methods with step-by-step guides for some of the most common solvents, which types of end products they create, important post-extraction filtration processes and resources needed to execute each method. In addition, the guide provides information on potential bottlenecks and how to avoid them to make sure the extraction process is as efficient and cost-effective as possible.

A Crude Awakening

The lowdown on extraction-refinement processes of today & tomorrow.

BY KENNETH MORROW

THEMANWHOPHOTO | ADOBE STOCK

Crude extract is exactly that: crude. As an unprocessed material, it must be refined into a specific, desirable form—similar to how the petrochemical industry refines crude oil into petroleum jelly, kerosene, gasoline, diesel fuel, aviation fuel and hundreds of chemicals, among other forms.

Crude cannabis extracts are the result of basic extraction methodologies—be it ethanol or CO2 extraction. Further refinement is usually dictated by the desired end product. For example, a vape pen cartridge has slightly different extraction requirements than an edible. A cartridge may require an extract that has been completely refined to the point that all flavonoids (including pigments) have been filtered or distilled out, resulting in a colorless (clear) extract. An edible may not require use of a clear extract; therefore, some colorants (i.e., pigments and/or flavonoids) can remain, and the extract may require less filtering or refinement.

How you refine your extracts is dictated by your desired end products. Edibles in general require less refinement than vape cartridges.
DUG FOTO | ADOBE STOCK

Filtration

Filtration techniques have recently evolved, and some extraction companies offer remediation services for extracts and/or distillates that would otherwise not meet testing standards. An example is the removal of contaminants (e.g., fungicides). Filtration comes in many forms, but no matter the method, the first step is usually to winterize the extract, which involves dissolving the CO2 extract in ethanol or a similar solvent. This results in a cannabinoid-rich ethanol slurry. The cannabinoid-rich ethanol then is subjected to sub-zero temperatures to freeze fats, waxes, flavonoids and other undesirable compounds by solidifying and coagulating them, so that they can be filtered out via multiple methods. These filtration methods range from using a Buchner Funnel (a funnel with a perforated disc to filter out impurities) to the utilization of different-sized particulate filters (screen filtration).

Some advanced manufacturers mitigate this step by utilizing in-line, de-waxing apparatuses. Many hydrocarbon extractors utilize in-line de-waxers as well to avoid introducing ethanol that will eventually need to be removed to their extracts. Adding ethanol also changes the extract's composition and increases production costs.

Purification

After winterization and/or filtration (if necessary), any remaining ethanol must be removed from the extract. The most common ethanol removal method is to utilize a rotary evaporator, which heats the cannabinoids and ethanol in a vessel under vacuum from a vacuum pump. The heated ethanol evaporates and is collected by a condenser, leaving ethanol-free, concentrated extract in the vessel. At this point, the extract is ready to be used as-is for certain products that do not require further refinement, such as Rick Simpson Oil (RSO).

Short-path distillation is a commonly used method of separating ethanol and cannabinoids. SOONTHORN | ADOBE STOCK

Multiple options exist to further purify and refine the extract, should it be necessary. For example, some hydrocarbon extractors also utilize distillation when they are using trim, or lower-quality cannabis buds, to extract. In this case, even though the product may be cannabinoid-rich, the extract may have excessive fats/waxes and exhibit a darker color. This is a less desirable product. But it can be refined further via distillation to eliminate those undesirable qualities and produce a cleaner concentrate.

Distillation methods range from short-path distillation, wiped-film distillation, falling-film and thin-film distillation, as well as other, yet-to-be-adapted methods not commonly used by most current cannabis extractors, but that will soon become more relevant as production sizes increase.

The distillation process allows processors to further separate available cannabinoids, which yield purified compounds, such as CBD and THC exceeding 95-percent purity. The same principle would also separate other available cannabinoids such as Delta-8, THC-V and a host of others if present en masse. Distillation allows a processor to target specific boiling point temperatures of the available cannabinoids, resulting in purified cannabis compounds that can then be formulated into the final desired forms.

THE REFINEMENT PROCESS AT A GLANCE

STEP #1

WINTERIZATION

DISSOLVE THE CO_2 EXTRACT IN ETHANOL OR A SIMILAR SOLVENT.

FREEZE FATS, WAXES, FLAVONOIDS AND OTHER UNDESIRABLE COMPOUNDS.

STEP #2

FILTERING

FILTER OUT COMPOUNDS USING METHODS SUCH AS A BUCHNER FUNNEL OR SCREEN FILTRATION, OR BY USING IN-LINE DE-WAXING OFFERED ON SOME EQUIPMENT.

STEP #3

REMOVE EXTRACTION AGENT

REMOVE ANY REMAINING ETHANOL, OFTEN DONE WITH A ROTARY EVAPORATOR.

STEP 3 MAY BE THE FINAL STEP FOR PRODUCTS SUCH AS RICK SIMPSON OIL (RSO) THAT REQUIRE NO FURTHER REFINEMENT.

STEP #4

PURIFY

DISTILLATION METHODS RANGE FROM SHORT-PATH DISTILLATION, WIPED-FILM DISTILLATION, FALLING-FILM AND THIN-FILM DISTILLATION, AMONG OTHER LESS-COMMON METHODS.

USING TARGET CANNABINOIDS' BOILING POINTS, YOU CAN BEGIN DISTILLING SPECIFIC CANNABINOIDS TO MAKE ISOLATES OF THC, THC-A,CBD, ETC.

Distilled Innovation

As production increases, equipment manufacturers will evolve. Already, hydrocarbon, ethanol and CO_2 equipment manufacturers are rapidly scaling up their apparatuses (or completely re-engineering them to handle much higher capacities) and innovating large-scale automated systems that minimize production time and labor, thus lowering overall production costs.

Some companies utilize technologies common in industrial applications, such as spinning band distillation (SBD), which is used to refine fish oil on an industrial scale. Technologies such as these will ultimately migrate into cannabis extraction refinement companies. Petrochemical companies and many other chemical manufacturers, as well as flavor and fragrance industries, employ equipment and methodologies that will carry over into the cannabis space as well.

In the future, production costs will factor into what is considered crude extract. Crude will be considered cannabinoids and terpenes that have been separated from the biomass (the green vegetative plant material) through fresh-freezing. The reclaimed material is kief (aka resin gland heads) and it is the most desirable component of cannabis as it contains the active ingredients (cannabinoids and terpenes). The resin gland heads will then be dissolved into ethanol for winterization, filtration and distillation; made into rosin form via heat and pressure; or further refined by utilizing CO_2 or hydrocarbon extraction methods. Because extractors buy their extraction material by weight, removing the plant biomass before extractors purchase the product (i.e., separating the resin gland heads from the plant material) concentrates the amount of active ingredients available by weight, increases output capabilities and lowers production costs—enabling a concentrate company to provide final products without compromising quality. Superior quality at a superior price usually wins.

As we move into the future, manufacturers will continue to investigate other methodologies. For example, will it be possible to squeeze active ingredients out of large amounts of cannabis? While industrial-scale hydraulic presses that would be required for this process are expensive, and most do not have heating capabilities, I believe this form of extraction will be investigated thoroughly, especially when considering the possibilities of pressing 100 pounds of kief. If a manufacturer desires only cannabinoids and not to preserve terpenes, then pressing the cannabinoids

ELIMINATING THE BIOMASS AND PAYING TAXES ONLY ON WEIGHT SOLD CAN DICTATE SUCCESS OR FAILURE FOR SOME COMPANIES.

out of kief with heat would be perfectly acceptable and an economical extraction method—that is, if it can be accomplished en masse.

In California, separating active ingredients has other serious implications that must be considered by both growers and extraction companies. In the Golden State, cannabis is taxed by product weight. Eliminating the green vegetative material and concentrating the active ingredients minimizes the taxable material's weight, in that taxes paid on 1 gram of bud would be the same as 1 gram of kief, yet with kief, the available percentage of those desirable compounds is much higher.

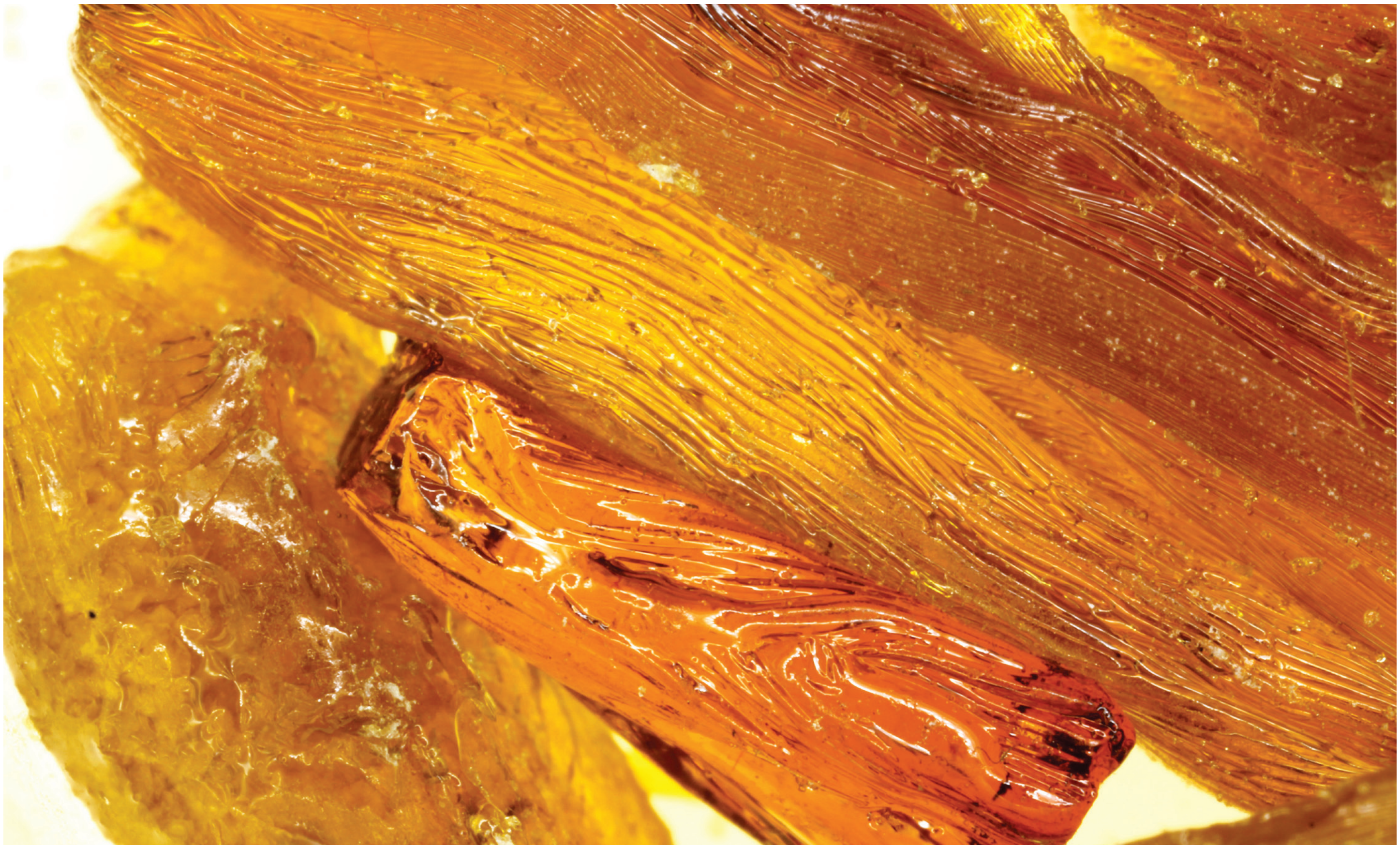
Separating biomass from active ingredients prior to selling product to an extraction company or conducting in-house extraction has numerous benefits. It is advantageous for extraction companies to simply purchase and refine raw resin, whether it is from fresh-frozen material, dry material, bud or trim. A friend once said to me, "But nobody wants to buy kief." That may be true—the consumer may not; but eliminating the biomass and paying taxes only on weight sold can dictate success or failure for some companies. The same friend then pointed out kief's low market price today, to which I replied, "Soon, the industry will adapt and price kief and other crude forms by percentage of available cannabinoids and terpenes, and producers will ultimately be paid according to the milligrams of cannabinoids and terpenes, because they are, in reality, all that is desired."

Kenneth Morrow is an author, consultant and owner of Trichome Technologies™. Facebook: TrichomeTechnologies; Instagram: Trichome Technologies; k.trichometechnologies@gmail.com

The Economics of Extraction

Considerations business owners should keep in mind when building their extraction lab business plan.

BY KENNETH MORROW



Norwegian Wood shatter. Shatter is a form of "absolute" extract in cannabis. PHOTO BY MEL FRANK

Cannabis Business Times' May 2018 issue reported that vape cartridge sales in California reached $100 million for the combined months of November and December 2017. The next bestselling concentrate product during that time was wax, with $7.4 million in sales. The stated total of all concentrate revenue in California for that same period was $140.9 million.

Based on those figures, it's clear that vape cartridges dominate sales. But why? The simple answer: user convenience.

With this data, some companies will successfully focus strictly on cartridge sales, but the cartridge industry is destined to become competitive and saturated. Everyone has, or will soon have, their own branded cartridge without a strategic advantage like a rare cultivar, proprietary device, or efficient and/or superior extraction methodologies and practices. I believe many companies will produce a vape cartridge that is no better nor cheaper than the rest, and these companies will suffer due to little or no brand recognition and will struggle to compete with products that possess strategic advantages (such as the ones mentioned). Therefore, extract companies need to find means to efficiently produce a diversified range of concentrates—and not rely strictly on vape cartridge sales.

Consumers will ultimately demand a wide range of products and offerings. Future consumers will be educated on cannabis's nuances and characteristics. In turn, many will prefer to purchase superior- tasting, connoisseur-quality products over artificial or formulated flavors or poor-quality concentrates. If the superior offerings are affordable, that is all the better for the consumer.

In the first part of this special extracts series, we reviewed a wide range of concentrates concocted from different extraction processes. (See the sidebar to on p. 76 for the full list of products and methods discussed in Part I.) This is where production economics come into play. Extractors need to be mindful not only of a product's strategic market advantages over another, but also the costs associated with extraction equipment as well as the cost of required basic materials (such as solvents) that can accumulate rapidly and increase production overhead.

Each extraction apparatus, whether CO_2, ethanol, butane or a hand-press, requires labor of some sort. Some are more user-friendly than others. Typically, the more complex an extraction and refinement method is, the more training the technician will require. Therefore, a highly trained technician (or an employee with a Ph.D.) will require a higher wage than a person performing simple tasks, which ultimately impacts a business's cost of production.

The concentrate's final form is most often dictated by the primary extraction method or by the final super refinement. It all depends on what you want the final form to be and how much it costs to produce. CO_2 extraction equipment is costly depending on the unit's capabilities and requires a well-trained operator. Rosin, dry-sieve or water hash require more rudimentary equipment, and employees producing these products do not require extensive equipment training. Comparing the material and labor costs of CO_2 extraction to that of rosin presses reveals a stark dichotomy: CO_2 extraction equipment can cost hundreds of thousands of dollars, while commercial rosin presses might cost a few thousand. Odds are, the

EXTRACTION METHODS & PRODUCTS, AT A GLANCE

In Part I of this extract series, we identified the eight main extraction methods and the 12 most common forms of cannabis extracts.
Here they are at a glance:

EXTRACTION METHODS:

1. Dry-sieved then pressed hashish
2. Water extracted, dried, pressed hashish
3. Rosin, heat and pressure applied, squeezing out the resin
4. Liquid nitrogen extraction
5. CO_2 extraction
6. Ethanol alcohol extraction
7. Hydrocarbon extraction
8. Distillation, wiped film, thin film or short path

BASIC FORMS OF EXTRACTED CANNABIS:

1. Pressed resin glands (hashish)
2. Oil form sometimes referred to as RSO
3. Tincture
4. Shatter
5. Budder
6. Wax
7. HTFSE (high terpene full-spectrum extract)
8. HCFSE (high cannabinoid full spectrum extract)
9. Vape cartridges
10. CBD in crystalized form
11. THCA in crystalized form
12. Delta 8 in oil form

person operating the CO_2 equipment makes a higher wage than the rosin-manufacturing technician.

What follows is an abbreviated explanation of the idiosyncrasies and strategic advantages of each method and the associated requirements of each regarding ancillary costs. Again, the ability to produce a business plan around one method depends on the concentrate's final form, the overall volume of material you intend to process, the combined equipment cost, labor costs, ancillary cost of each, and the associated legal requirements regarding laboratory requirements and licensing considerations.

Hydrocarbon Extraction

- **Forms produced:** shatter, budder, wax, HTFSE, HCFSE, or crude extract for distillation, or terpenes to be added to distillate.
- **Apparatus cost:** $5,000 to $100,000 depending on size and capabilities.
- **Technician skill/cost:** educated in basic chemistry/moderate.
- **Ancillary requirements:** legally licensed, compliant laboratory; ability to store flammable fluids, liquid butane.
- **Compounds targeted:** all available cannabinoids, CBDA, THCA and all available terpenes. If required, material can be pre- or post-decarboxylated to produce a decarboxylated version of available cannabinoids at the expense of terpene content.
- **Note**: If extracting hemp CBD only, technicians may not be required to obtain all the same licenses or permits as they would if extracting THC. (This is also true regarding CO_2 extraction and other methods of extraction.)

CONSIDERATIONS FOR YOUR EXTRACTION OPERATION

Use this chart as a reference guide on the possibilities and limitations of the different methods used for cannabis extraction when deciding what extracted products and extraction methods on which you want to focus.

	HYDROCARBON	CO_2	ETHANOL	DISTILLATION	LIQUID NITROGEN	ROSIN	DRY SIEVE	H2O
FORMS PRODUCED	shatter, budder, wax, HTFSE, HCFSE, or crude extract for distillation	crude extract; shatter, budder and wax (top-of-line equipment)	crude extract; top-of-line equipment can produce extract that needs less refinement	super-refined oil rich in cannabinoids; oil can be refined into crystals	sieved resin glands separated by size (needs further refinement)	raw resin	raw resin	raw resin
EQUIPMENT COST	starting at $5,000	starting at $100,000	starting at $5,000	starting at $5,000	starting at $1,000	starting at $100	starting at $100	starting at $100
ANCILLARY REQUIREMENTS	liquid butane; ability to store flammable fluids	liquid CO_2; ability to store liquid CO_2 tanks	ethanol; ability to store ethanol alcohol	–	liquid nitrogen; ability to store liquid nitrogen	–	–	–
COMPOUNDS TARGETED	all available cannabinoids and terpenes	all available cannabinoids; very low amounts of monoterpenes	all available cannabinoids; very low amounts of monoterpenes	all available cannabinoids; very low amounts of monoterpenes	all available cannabinoids and terpenes	all available cannabinoids and terpenes	all available cannabinoids and terpenes	all available cannabinoids and terpenes (minus water-soluble terpenes)

CO_2 Extraction

- **Forms produced:** typically a crude extract that requires further refinement of one form or another, be it winterization or filtration. That said, equipment manufacturers have begun producing superior equipment that produces a more refined product. Some processors are producing shatter, budder and wax.
- **Apparatus cost:** between $100,000 and millions of dollars, depending on size, capabilities or automation abilities.
- **Technician skill/cost:** educated in basic chemistry/moderate.
- **Ancillary requirements:** legally licensed, compliant laboratory; ability to store liquid CO_2 tanks.
- **Compounds targeted:** all available cannabinoids; resulting extract has very low amounts of monoterpenes.

Ethanol Alcohol

- **Forms produced:** typically a crude extract that requires further refinement of one form or another, be it winterization or filtration. Similar to modern CO_2 extraction equipment, manufacturers are now producing superior equipment that produces a more refined product that requires less labor to super-refine. But most extraction facilities further refine ethanol extracts through distillation after winterization and/or filtration.
- **Apparatus cost:** between $5,000 and millions depending on size, capabilities or automation abilities.
- **Technical skill/cost:** educated in basic chemistry/moderate.
- **Ancillary requirements:** legally licensed, compliant laboratory; ability to store ethanol alcohol.
- **Compounds targeted:** all available cannabinoids, resulting extract has low amounts of monoterpenes.

Distillation: Thin film, short film, wiped film

- **Forms produced:** a super-refined oil rich in desired cannabinoids, be it THC, CBD or a multitude of others in composition. The resulting product can be left in oil form, meaning the THC (or other cannabinoids) can be crystallized.
- **Apparatus cost:** between $5,000 and millions depending on size, capabilities or automation abilities.
- **Technician skill/cost:** highly educated in chemistry/high.
- **Ancillary requirements:** legally licensed, compliant laboratory.
- **Compounds targeted:** all available cannabinoids; resulting extract has low amounts of monoterpenes.

LNZ (liquid Nitrogen)

- **Forms produced:** sieved resin glands separated by size (crude form of extract that can be further processed or refined into almost any form).
- **Apparatus cost:** $1,000 to millions depending on size and capabilities. As of now, there is no version of this apparatus for sale. Extraction facilities that utilize this method are currently manufacturing their own equipment to suit their requirements.
- **Technical skill/cost:** basic understanding of chemistry and gravity/moderate.
- **Ancillary requirements:** legally licensed, compliant laboratory or ability to extract directly in growth environment (i.e., directly in greenhouse or in the field), lots of liquid nitrogen.
- **Compounds targeted:** all available cannabinoids and terpenes.

Rosin (heat and pressure press)

- **Forms produced:** raw resin, typically stable and slightly hard when cool, ranging from a stable mass to viscous fluid depending on starting material and method, temperature, pressure and duration time of pressing.

- **Apparatus cost:** $100 to $10,000+, depending on size and capacity capabilities.
- **Technician skill/cost:** basic understanding of chemistry/moderate.
- **Ancillary requirements:** legally licensed, compliant facility.
- **Compounds targeted:** all available cannabinoids and terpenes.

Dry Sieve (specific sized resin glands)

- **Forms produced:** raw resin, preferably of targeted size mature glands.
- **Apparatus cost:** $100 to $10,000+.
- **Technician skill/cost:** basic understanding of chemistry/moderate.
- **Ancillary requirements:** legal, licensed and compliant facility.
- **Compounds targeted:** all available cannabinoids and terpenes.
- **Note:** This form of extract can be refined and manufactured into a multitude of extract forms.

H_2O Extraction (water extraction) (specific-sized resin glands)

- **Forms produced:** raw resin, preferably of targeted-size, mature glands.
- **Apparatus cost:** hundreds to thousands of dollars.
- **Technician skill/cost:** basic understanding of chemistry/moderate.
- **Ancillary requirements:** legally licensed, compliant facility.
- **Compounds targeted:** all available cannabinoids and terpenes.
- **Note:** This form of extract can be refined and manufactured into a multitude of extract forms. Water-extracted resin glands contain lower amounts of water-soluble terpenes because they are absorbed and lost in the waste water.

Most of these concentrate forms can be refined and manufactured into a multitude of consumer goods and products. The key to efficiency is to determine what final extract form you desire, then decide which method or apparatus suits your requirements, and how that equipment and the resulting ancillary requirements fit within your business plan.

I have seen many interesting things in my years of concentrate consulting. For example, one individual who insisted he could produce a better rosin press went to China, had some sort of press fabricated and shipped it to Oregon where it has sat unused for two years, never to press a speck of cannabis.

Another group called me rand stated that their intent was to take hydrocarbon extract and increase its value via further distillation, which I explained was unlikely unless they were extracting very poor-quality hydrocarbon extracts.

The point from these two misadventures is this: Don’t over-complicate your extraction process. Figure out exactly what concentrate form fits your business plan, then figure out exactly which method suits your needs and stick to it.

Kenneth Morrow is an author, consultant and owner of Trichome Technologies™. Facebook: TrichomeTechnologies; Instagram: Trichome Technologies; k.trichometechnologies@gmail.com

Your Guide to Supercritical Extraction

Understanding the principles of carbon dioxide extraction and its output capabilities can help you evaluate equipment and anticipate potential production bottlenecks.

BY MARK JUNE-WELLS

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Cannabis extracts are an important part of the rapidly growing marijuana industry, especially in medical markets. The essential oil of cannabis, which is a concentrate of all the active pharmaceutical ingredients in the marijuana plant, is a dynamic substance that can be transformed into numerous forms for user consumption. As a starting point, extracts can be converted (with a bit of basic chemistry know-how) into products such as tinctures, transdermal patches, effervescent tablets, drink powders, suppositories and oral tablets, not to mention the standard vaporizing and dabbing oils.

A variety of suitable solvents is available for extracting the active ingredients from cannabis—each of which has strengths, weaknesses, laboratory infrastructure requirements and production-scaling considerations.

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This column explores supercritical carbon dioxide extraction ($SCCO_2$), including its functionality, laboratory requirements and what features should be considered during extractor selection.

Medicinal Value

A logical place to begin a conversation about carbon dioxide extraction (or any type of extraction, for that matter) is a quick overview of the medicinally valuable solutes to be extracted from the cannabis plant.

Two classes of cannabis substances receive the most attention in this growing industry: cannabinoids and terpenes.

At least 113 cannabinoids have been isolated, and these molecules range in weight from 250 to 350amu (atomic mass units). Their physical form can be liquid or solid (depending on identity), contain a variety of functional groups and are non-volatile.

Terpenes are a large and diverse group of compounds produced by plants and some animals. This group of molecules is classified by the number of base isoprene units. (Isoprenes are common organic compounds produced by plants.) Furthermore, terpenes and their associated mixtures are responsible for the pleasant—or unpleasant—aromas given off by plants. Terpenes vary widely in mass based on the number of carbon atoms (or isoprene units), can include a variety of functional groups and are physically liquid or oil.

Flavonoids and carotenoids are also present in cannabis. While they are not frequently recognized as valuable in the cannabis industry, they are well-known bio-botanical compounds in the nutritional and medical industries.

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Flavonoids are polyphenolic compounds that give plant extracts their golden and brown colors. There are more than 5,000 known flavonoids that vary in molecular weight and numbers of functional groups. They are usually solid in their pure form.

Carotenoids are a group of pharmaceutically important molecules with more than 600 known constituents. They tend to be of very high molecular weight, contain a variety of functional groups and are orange to red in color.

Finally, numerous fatty acids and chlorophylls can be extracted from plant material. Though they are not generally thought of as medicinally valuable in the cannabis industry, some evidence exists for bioactivity in the nutraceutical industry. Fatty acids are commonly 16 to 20 carbons long, but can be much larger; they tend to solidify at room temperature, and the level of saturation (i.e., number of hydrogen-carbon bonds) can vary.

Chlorophylls are the large molecules responsible for a plant's ability to produce sugars from sunlight and water. Chlorophylls range between 800 and 900amu and give plant extracts their green to black coloration. (Black coloring occurs when chlorophyll is oxidized.)

The CO_2 Process

Now that we have covered the majority of the extractable solutes in cannabis, let's explore how carbon dioxide functions as a solvent.

Before diving in, a quick review of some relevant physical properties of carbon dioxide can be helpful. Carbon dioxide is a gas at standard temperatures and pressure. It forms a liquid at pressures above 5bar (i.e., 73psi), and its critical point (the vapor-liquid boundary) is 73bar (1060psi) at 33.1 degrees Celsius.

Here, we'll describe the solvent properties of carbon dioxide in its supercritical state—because the gas state cannot act as a solvent, and the liquid state is not an efficient solvent in cannabinoid extraction.

So, what features of supercritical carbon dioxide ($SCCO_2$) make it an efficacious solvent in the extraction of cannabis? Supercritical carbon dioxide—and all supercritical fluids—have the density of a liquid, diffusivity of a gas and a low viscosity (thickness). In simpler terms, this means that $SCCO_2$ has: a high-solute carrying capacity (i.e., it can hold a lot of material), the ability to penetrate into the smallest spaces (like a gas) and very little flow resistance. Additionally, its polarity and density can be manipulated. Polar manipulation can be achieved with the addition of co-solvents, such as ethanol. Density manipulation is the true power of supercritical carbon dioxide as a solvent. While other solvents such as hydrocarbons and ethanol are more efficient at stripping plant material of their cannabinoids and terpenes, $SCCO_2$ has the unique ability to target specific fractions in the parent (plant) material or separate solutes. Those processes are possible because $SCCO_2$ density is dependent on pressure and temperature parameters.

Solute-carbon dioxide interactions are solute-specific. Each solute in a mixture (i.e., the parent plant material) has a unique solubility profile that is related to the density of the $SCCO_2$; a density exists where specific solutes become highly soluble in $SCCO_2$. This is called the crossover phenomenon. It is characterized by an exponential increase in a solute's solubility in $SCCO_2$. Because the crossover point is solute-specific—if the critical density is known for target solutes, they can be individually removed by using temperature and pressure gradients.

We can also view this crossover phenomenon from a different perspective: Imagine using temperature and pressure settings that result in the extraction of all solutes from your feed material, then reducing density downstream of the extraction location. This process is called retrograde solubility and can be taken advantage of to separate the components of the $SCCO_2$/solute mixture.

Essentially, this process starts with $SCCO_2$ at a very high density, followed by sequential depressurizations that result in consistent reductions in $SCCO_2$ density throughout that process. As this process is occurring, certain solutes are no longer soluble and are collected at specific locations (i.e., separation vessels).

That ability to target or separate solutes from a mixture is the most valuable feature of $SCCO_2$ extraction. Other beneficial features of carbon dioxide extractions include the fact that it is generally regarded as safe (i.e., high exposure limits), it is relatively cheap, and it is available in high purity from numerous sources.

THAT ABILITY TO TARGET OR SEPARATE SOLUTES FROM A MIXTURE IS THE MOST VALUABLE FEATURE OF $SCCO_2$ EXTRACTION.

CO_2 System Considerations

So, what are the important features of a supercritical carbon dioxide extraction system? As previously mentioned, density, which is determined by pressure and heat, is one physical property of $SCCO_2$ that dictates extraction efficiency and separation. Therefore, three variables are of the utmost importance:

1. maximum pressure rating
2. the ability to measure the temperature of the carbon dioxide (not the surface of the vessels) and
3. high wattage heaters.

Those features are important because it is necessary to obtain high pressures, deliver the heat in an efficient manner and have a real-time knowledge of carbon dioxide temperatures to tailor the density appropriately.

An extractor also should have a pump/flow monitoring system that evaluates the mass of carbon dioxide being delivered to the extraction vessel. Additionally, that pump should have the ability to deliver high flow rates to the parent material in the extraction vessel. This is because an important calculated variable to optimizing a supercritical carbon dioxide extractor is the ratio of carbon dioxide mass used during extraction to the mass of the parent material—a ratio of 50 or more is usually necessary to reach 90-percent to 95-percent extraction completeness.

Finally, separation vessels with high maximum-pressure ratings are extremely important because they give the technician the ability to use a variety of pressures in the development of separation (i.e., product development) protocols.

A shortcoming of $SCCO_2$ extraction is that numerous waxes and fatty acids are also soluble in supercritical carbon dioxide. From a manufacturing perspective, this is an important point because those materials need to be removed during the refinement process before product development. This is achieved through a process called winterization, which takes advantage of the differing solubility of waxes and cannabinoids in a solvent at low temperatures (i.e., -30 degrees Celsius or lower).

The winterization process frequently represents the slowest part of the refinement process if the infrastructure does not match the extractor production rate. The standard protocol uses a funnel and filter paper in concert with a vacuum. Depending on volume, this process can take between four and eight hours. Furthermore, it must be repeated multiple times to account for the dissolution of waxes back into ethanol during the protracted filtering process. However, the redundancy and length of this process can be circumvented through the use of low-pressure cartridge filter techniques that can process large volumes of winterized material in an expeditious manner while controlling temperature.

The next consideration is the solvent recovery that follows the winterization process. It is important to size your solvent recovery units to match your winterizing/filtering production rates. Most commonly, rotary evaporation systems are used to recover your winterizing solvent. It is also important to scale this system to match your pre-solvent recovery step production and extraction rates.

To put these notes in perspective, here is an example of a production system followed by an identification of the bottleneck.

First, it is necessary to lay out the assumptions:
1. extractor input of 2,000 grams
2. return ratio 0.18
3. two extractions per day
4. five days a week run time.

With those assumptions, the output per run is 360 grams per day and 3,600 grams a week. Therefore, the total volume of material to be filtered would be 36 liters with a 10:1 ratio of winterization solvent to extract.

IT IS IMPORTANT TO SIZE YOUR SOLVENT RECOVERY UNITS TO MATCH YOUR WINTERIZING/FILTERING PRODUCTION RATES.

That material can be filtered in 34 minutes with a positive-pressure filtration system capable of an aqueous flow of 125 liters per hour and an equal volume of solvent to wash the waxes. The final volume for solvent recovery is 72 liters, which can be recovered in four and a half hours with a rotary evaporator capable of processing 16 liters per hour. Evaluating these numbers suggests that your post-processing equipment is capable of refining a week's worth of extract in roughly five hours. Therefore, your extraction parameters or the extractor are the bottleneck in the described production system.

While this analysis oversimplifies the process in some ways, it does exemplify the importance of planning your total production system to meet the output at each stage because capital might be better leveraged to obtain a system with a higher overall output. Imbalanced systems can cause manufacturing equipment to lay idle for periods of time, which is not an optimal use of capital, labor or equipment.

Mark June-Wells, Ph.D. is principal owner of Sativum Consulting Group and a Ph.D. in botany/plant ecology (Rutgers University).

Your Guide to Hydrocarbon Extraction

A primer on how to create cannabis extracts using butane gas, including important production processes and system considerations, as well as potential bottlenecks.

BY MARK JUNE-WELLS & MITCHELL LINDBACK

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Demand for extracted cannabis products is quickly gaining traction. For example, edibles companies saw $1.5 billion in sales in major markets in 2017—and cartridge sales last year cleared $610 million in those same markets, according to Brightfield Group. When executed properly, cannabis extracts can be also used to produce other high-quality products such as topicals, transdermal patches, capsules and other concentrates. Yet, cannabis extraction can be a complex process—one that needs careful attention and a skilled workforce to be completed safely and efficiently.

The first part of this special extraction series explored the products that can be derived from cannabis' essential oils—such as tinctures, transdermal patches, oral tablets, vaporizing and dabbing oils, and many more—and how those extract formulations can be achieved through supercritical carbon dioxide extraction ($SCCO_2$).

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The second installment of this series explores hydrocarbon extraction, butane in particular, including its functionality, laboratory requirements and what features should be considered during extractor selection.

Introduction to Butane

Before diving in, a quick review of relevant physical properties of n-butane can be helpful. Butane is a non-polar, Class 2 flammable liquefied gas that the Food and Drug Administration (FDA) has Generally Recognized as Safe (GRAS). It has a low boiling point of 31.1°F (-0.5°C), which is helpful when cold-boiling the residual solvent from the concentrate solution. This process leaves behind the temperature-sensitive terpenes. Many extraction technicians prefer to blend their butane with propane to create a gas mixture that will strip additional terpenes and purge more efficiently than butane alone. (The boiling point of propane is -43.6°F (-42°C).)

So, what features of n-butane (C_4H_{10}) make it an effective solvent in cannabis extraction? Hydrocarbons are arguably the most efficient solvent for cannabis extraction. Of the two standard hydrocarbons used for extraction (butane and propane), butane is a low-pressure system where extractions occur between 0 psi and 30 psi (pounds per square inch). One advantage of hydrocarbon extraction is the sheer number of products you can create from a single standard extraction without further refinement. Fresh, frozen extractions are the "fresh-squeezed juice" of the concentrate world. Currently, the preferred method is to separate the crystalline high-cannabinoid extract (HCE) from the aqueous, high-terpene extract (HTE). These fractions can be sold as separate SKUs or recombined at a ratio of the processor's choosing to create a full-spectrum extract (FSE). Full-spectrum extracts are very popular.

A few shortcomings of butane exist, the primary one being its flammability and the regulatory compliance costs surrounding any hydrocarbon extraction. Hydrocarbons as extraction solvents are currently outlawed in Canada and some U.S. counties have enacted or are considering bans on hydrocarbon extraction. The second shortcoming is the lack of automated options. Unlike CO_2 and the new ethanol systems, hydrocarbon extraction is still a very hands-on process, which can make extraction-outcome predictability difficult. (The hands-on nature of hydrocarbon systems puts added reliance on operator skill to achieve product consistency.) Only a few automated extraction machines are currently available to the cannabis industry; however, the price tag and throughput can be a tough pill to swallow for some. Automated hydrocarbon extraction systems will improve in quality and decrease in price as their safety features and internal monitoring systems become more reliable.

The Butane Process

Parent Material Quality

The starting material's quality has a direct effect on the finished concentrate's quality, regardless of extraction methodology. Always store plant material in a cool, dark place, and in vacuum-sealed or nitrogen-filled bags. These precautions limit exposure to ultraviolet rays, heat and oxygen, which are the three primary factors contributing to cannabinoid degradation.

The Wash

Cold butane is released from the solvent tank into the material column, where it slowly washes over the plant material, dissolving the cannabinoids and terpenes from the cannabis. Once the plant material has been washed, the solution can be collected directly, or it can be processed through an in-line de-waxing column.

Winterization/Filtration

Butane extractions are not typically winterized and filtered because the low extraction temperatures dissolve almost no chlorophyll and because the low temperature limits the amount of dissolved lipids/waxes. Additionally, many closed-loop hydrocarbon extraction machines come equipped with in-line de-waxing systems. Like winterization, in-line de-waxing requires a minimum -22°F (-30°C) environment, but it is a single-solvent system, where winterization uses a secondary solvent. In-line de-waxing employs the extraction solvent and takes advantage of the surface area created by baffles, stainless beads or other media to retain the undesirable materials as concentrate/extraction-solvent solution pass through.

For winterization, a polar solvent like ethanol is used at a 10:1 ratio, which is chilled until separation occurs. It is then poured over various-sized micron filters to sep-

CHECK YOUR CERTIFICATIONS

Not all extraction equipment is created equal, and some is downright unsafe. For more on this, see "Buyers Beware" in the April 2018 issue of *Cannabis Business Times*, or read it here: bit.ly/CBT-buyers-beware.

ONE ADVANTAGE OF HYDROCARBON EXTRACTION IS THE SHEER NUMBER OF PRODUCTS YOU CAN CREATE FROM A SINGLE STANDARD EXTRACTION WITHOUT FURTHER REFINEMENT.

arate out the lipids and waxes from the concentrate solution. While winterization is a more thorough process than in-line de-waxing, the polar ethanol might degrade some terpenes, which could result in a less flavorful product. Note that if the extraction system has in-line de-waxing built into the process, the technician should account for an additional 30 to 90 minutes of processing time.

Collecting Concentrate

Once the concentrate solution enters the collection pot, the residual butane is purged off passively by heating the vessel, which pushes the butane out of the concentrate solution back to the colder solvent tank. This process completes the "closed-loop." Once the majority of the butane has been removed from the solution within the collection pot, the extraction technician collects the concentrate solution and places it on a parchment sheet or into a glass media bottle for separation.

Removing Residual Solvent

Purge methods and durations are dependent on the desired finished product. If the desired end product is wax, the concentrate solution can be whipped for a couple of hours to remove all residual butane. If shatter is the desired product, the concentrate solution is spread thin across Teflon sheets and purged inside a vacuum oven for a minimum of 48 hours.

Hydrocarbon System Considerations

When employing a flammable solvent like butane, it is important to understand that the processing laboratory—not only the extractor—must be in compliance with Occupational Safety and Health Administration (OSHA) and National Fire Protection Association (NFPA) regulations as they apply to flammable solvents and explosive atmospheres. These include implementing a Class 1, Division 1 (C1D1) extraction/manufacturing space that has gas monitoring, zero ignition points, adequate ventilation and a fire-suppression system that can negate catastrophic equipment failure. A well-ventilated space dedicated to temperature regulation where the chillers and heaters are plumbed into the C1D1 extraction space is recommended. There are also important oven, refrigeration and vacuum requirements (see OSHA 29CFR, NFPA 45, 99, 70, etc.).

Production Considerations

When it comes to the extraction system, these three variables are of the utmost importance:

1. Ensure the machine is approved for use at the processing location. If the closed-loop system has not been peer-reviewed in that jurisdiction, then it will not pass inspection.
2. Ensure the extraction system can handle the current throughput as well as the ability to scale.
3. Control temperature. The equipment must be able to keep 25 kilograms of butane at -40°F (-40°C), while maintaining sub-cooled injection coils and cooling of the de-wax column. These ancillary chiller and heater expenses, including HVAC to vent the heat generated by these units, can run upwards of $50,000 on top of the closed-loop extraction system (CLS) price.

Production Bottlenecks

Below is an example of a production system followed by an identification of the bottleneck(s). First, it is necessary to lay out the following assumptions:

1. Starting material is trim
2. Extractor input of 4,500 grams
3. Return ratio of 0.15 (meaning the weight of extracts will be 15 percent of the weight of the plant material used)
4. Five extractions per shift
5. One shift per day/five work days per week

With hydrocarbon extraction, there are two bottlenecks within the process. First is the *vacuum oven space required* to purge the residual solvent out of the concentrate solution, and the second is *packaging the finished product*.

Extracting 4,500 grams of biomass every 90 minutes results in 22.5 kilograms (22,500 grams) of biomass per shift being extracted. Based on the average yield from properly grown trim (15 percent), 3,375 grams of concentrate is produced per shift. A 5-cubic-foot oven can hold about 1,200 grams of concentrate. Therefore, three 5-cubic-foot ovens would be required for each production day. Since each purging event requires 48 hours, a manufacturing laboratory would require a minimum of six ovens of that size to meet the weekly production of the extractor.

The packaging and fulfillment bottleneck is the most significant because none of the processes outside of labeling and sealing are automated. Currently, every process except labeling requires staff labor. Packaging will continue to be the most intrusive bottleneck in all successful processing facilities until the industry can figure out a way to automate handling various forms of concentrate and weighing it out to exact amounts.

Mark June-Wells, Ph.D. is principal owner of Sativum Consulting Group and a Ph.D. in botany/plant ecology (Rutgers University).

Mitchell Lindback is an extraction specialist for MJardin Group, a highly specialized professional operating company.

Your Guide to Ethanol Extraction

Overall, ethanol extraction is an effective process most suited to high-throughput, bulk-processing laboratories that focus on a few products.

BY MARK JUNE-WELLS

RACHAN | ADOBE STOCK

For years, flower was king. As long as there was an established pipeline for flower distribution, it was almost impossible for investors to lose money in the early stages of this industry's growth. While this might remain true in some markets, flower profits are becoming leaner. There has been a significant decrease in flower price in the most mature markets due to the expansion of outdoor growing techniques that produce high-quality flower in large quantities, as well as market saturation driving prices down.

Let's create a fiscal example as a starting point. First, let's assume, for this example, that the price of high-quality outdoor flower is wholesaling at $700 per pound. The second assumption is that two potential outcomes of yield exist from the extraction and refinement process (i.e., 10 percent and 15 percent of dried flower weight). The final assumption is the potential wholesale prices for high-quality refined oils, which we will estimate at two potential levels of $20 and $25 per gram.

At 15-percent extraction yield, an extracted pound would wholesale for $1,360.80 (at $20/g) and $1,701 (at $25/g). At 10-percent extraction yield, the wholesale price for a pound would be $907.20 (at $20/g) and $1,134 (at $25/g).

This example framework is somewhat simplified, but it characterizes the potential for a basic, value-added approach to dealing with the cannabis flower market's rapidly expanding supply side. (A gram of marijuana flower can retail between $1 to $15, according to data from Leafbuyer.) More revenue can be gained through scale and the manufacturing of extracted products than through flower sales.

In the first two parts of this special three-part extraction series in Cannabis Business Times, we explored supercritical CO_2 extraction (March 2018) and hydrocarbon extraction (May 2018]; in this column, we will delve into ethanol's properties, the different types of extraction strategies, safety considerations for ethanol systems and laboratory infrastructure considerations.

Ethanol and Its Guidelines

The Food and Drug Administration (FDA) classifies ethanol as a Class 3 solvent with low risk for acute or chronic toxicity in pharmaceutical manufacturing processes where the residual is less than 5,000 ppm or 0.5 percent. The FDA also implies that residual solvents in this category should be limited to 0.5 percent through rigorous quality assurance and quality control programs.

Despite those FDA guidelines, some states have adopted more conservative safety limits suggested by the Occupational Safety and Health Administration (OSHA) and the National Institute for Occupational Safety and Health (NIOSH). OSHA and NIOSH set the worker environmental exposure limit for ethanol at 1,000 ppm of Total Weighted Average (TWA) over an eight-hour work shift, which means that some states are allowing only 0.1 percent residual ethanol in extracted products.

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WHAT IS ETHANOL?

Have you ever drunk grain alcohol? If so, you were drinking ethanol. Ethanol is "a colorless volatile flammable liquid C2H5OH that is the intoxicating agent in liquors and is also used as a solvent and in fuel—called also ethyl alcohol, grain alcohol," according to Merriam-Webster.
It "can be fermented from many sources of starch, including corn, wheat, grain sorghum, barley, and potatoes, and from sugar crops such as sugar cane and sweet sorghum. Because there has been an abundant supply of corn, most of the ethanol made in the United States is from corn," the University of Illinois Extension explains.

- CBT

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The National Fire Protection Association (NFPA), for its part, classifies ethanol as a Class 1 Division 2 Group D flammable liquid. As such, OSHA requires that ethanol vapors be held at 25 percent of the 3.3-percent by volume Lower Explosive Limit (LEL) through adequate ventilation in storage areas. Therefore, areas containing ethanol in production facilities must maintain no more than a 0.83 percent by volume ethanol vapor in the ambient air. (Adequate ventilation—as defined by OSHA and NFPA—is a system capable of cycling a room's total air volume six times per hour.)

Numerous other considerations apply to the storage and use of ethanol in manufacturing laboratories that could fill a column of their own, so I will simply outline that, when it comes to storage, the maximum flammable cabinet storage is 60 gallons, and the maximum storage permissible outside of a flammable cabinet or storage room is 25 gallons.

The Process

Ethanol extraction is a single-stream process that can be conducted under warm or cold conditions. An example of a warm ethanol extraction processes is the Soxhlet technique. This technique essentially boils ethanol in a flask or pot, then condenses the alcohol on a cooled-coil, which then drips through the packed flower material, stripping the cannabinoids and terpenes during the process. The advantage to this approach is that the extraction is time efficient and of relatively low solvent-to-feed ratio. However, the warm-ethanol technique is generally a small-batch approach that extracts chlorophyll/

waxes and decarboxylates the cannabinoids due to the heat involved. (Decarboxylation is the conversion of THCA, for example, to THC through heating and agitation that yields carbon dioxide during the process.) Therefore, heated ethanol extractions might require additional dewaxing and clarification steps.

This type of technique is also limited in the number of products it can produce because all the acid-form cannabinoids are decarboxylated during the extraction. While heating ethanol can increase the extraction process's efficiency, ethanol is a good solvent for extracting terpenes and cannabinoids. Therefore, it can be used as an extraction solvent at room temperature or under supercooled conditions. Using ethanol at room temperature or under cooled conditions are the most common practices because these conditions allow for the retention of cannabinoid acid forms that can be leveraged to manufacture shatters, THCA crystals or THCA-rich oral formulations.

Know the Difference

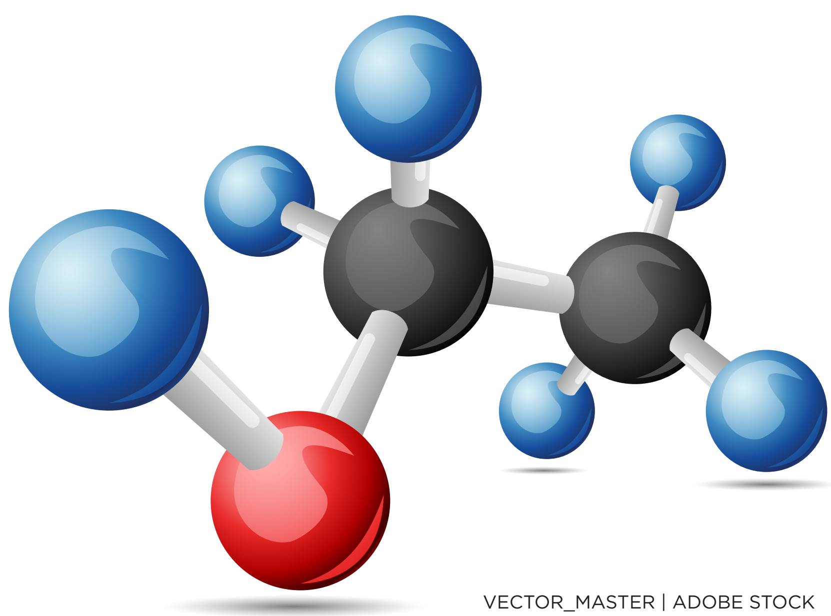
A few differences exist between the outcomes of room temperature and supercooled ethanol extractions. First, room-temperature extractions generally extract more waxes and pigments than supercooled techniques, which results in additional dewaxing and clarification steps. However, room-temperature extraction techniques are more efficient. In short, ethanol is a very good solvent as it applies to the extraction of cannabinoids and terpenes. In the literature that describes the solubility of cannabinoids in ethanol, there is no definitive carry capacity, but many sources suggest that cannabinoids are soluble in ethanol at a 1:1 ratio (meaning that 1g of THC is soluble in 1mL of ethanol).

Finally, ethanol extraction can be conducted as expensively or inexpensively as the manufacturer desires. It can be conducted in simple vessels where the ethanol/plant material mixture is agitated by hand or in automated extractors that control temperature, inject a specific ethanol volume and undertake inline dewaxing/clarification.

Solvent Handling

Ultimately, there is always one major problem to address with ethanol extraction beyond the safety requirements: downstream solvent handling.

All types of ethanol extraction require that between 0.6 and 1 gallon of ethanol be used during the extraction process. The reason for this requirement is not due to the solubility of the cannabinoids or terpenes; it is due to the absorbent nature of the plant material. To extract



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the solutes from the feed material, ethanol must fully saturate the flower or trim. For that reason, a significant volume of ethanol is needed to execute the process with an efficiency rate of more than 90 percent. While some automated machines have built-in processes to minimize the required ethanol volume, the best-case scenario is that the amount of ethanol required ranges from 0.5 gallons to 0.6 gallons per pound.

Machines with automation features have also compensated for the problem of squeezing the ethanol from the plant material by adding spin-of-compression cycles to their processes. This is helpful because recovering all the ethanol from saturated plant material—which holds cannabinoids and terpenes—can be a difficult and messy affair with basic approaches. So, how does one deal with the large amount of ethanol required to extract the cannabinoids from a highly productive cannabis grow? The answer: planning and large solvent-recovery systems.

Scaling for Ethanol Extraction

To help elucidate the laboratory requirements associated with properly scaling ethanol extraction equipment to a grow operation, let's look at another hypothetical example.

First, let's assume that the grow is producing 720 pounds of extractable material (i.e., flower/trim) per month. With that information, the extraction system will need to be able to extract a total of 36 pounds of feed material every workday, each month (720 pounds ÷ 20 days). To meet that requirement, the ethanol extraction system will need to process 4.5 pounds per hour of each eight-hour workday. (There are solutions that claim to meet this specification; they often carry a heftier price tag.)

For this example, let's also assume that we own a supercooled system capable of extracting at the aforementioned rate. That type of machine uses the least amount of ethanol (0.6 gallons/pound) during the process compared to other ethanol extraction technologies. So, the total volume of alcohol to be recovered and processed per day would be 21.6 gallons (36 pounds x 0.6 gallons/pound) or 81.8 liters.

To recover that volume of alcohol, the laboratory would require a large rotary or falling film evaporator (equipment made to gently remove solvents from samples by evaporation). For this example, a rotary evaporator is probably the most cost-effective choice. A large rotary evaporator capable of handling 16 liters/hour would recover the ethanol in about five and a half hours, which means that there would still be room for additional throughput on the extraction side; roughly, 17 pounds of additional feed material throughput per day is feasible under this example.

Ethanol is a solvent capable of extracting cannabinoids and terpenes efficiently. It also has a relatively low boiling point, which makes it easy to remove from final product, and a favorable toxicological profile including FDA limits in the range of 0.5 percent. However, there is a high solvent-to-feed requirement, which can create ethanol storage compliance issues and a need for an expensive, high-throughput rotary evaporator. Finally, ethanol cannot be tailored to separate cannabinoids or terpenes during the extraction phase to work into a predetermined product pipeline. Overall, ethanol extraction is an effective process most suited to high-throughput, bulk-processing laboratories that focus on a few products.

Mark June-Wells, Ph.D. is principal owner of Sativum Consulting Group and a Ph.D. in botany/plant ecology (Rutgers University).

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CANNABIS BUSINESS TIMES

PUBLISHER
Jim Gilbride
jgilbride@gie.net

EDITORIAL
Noelle Skodzinski, Editorial Director
nskodzinski@gie.net
Michelle Simakis, Editor
msimakis@gie.net
Jonathan Katz, Managing Editor
jkatz@gie.net
Brian MacIver, Senior Editor
bmaciver@gie.net
Andrea Sparr-Jaswa, Science Editor
asparr-jaswa@gie.net

DIGITAL
Eric Sandy, Digital Editor
esandy@gie.net
Melissa Schiller, Assistant Digital Editor
mschiller@gie.net

ADVERTISING SALES
Scott Anthony, National Sales Manager
santhony@gie.net
Craig Thorne, Account Manager
cthorne@gie.net
Sarah Birckhead, Account Manager
sbirckhead@gie.net
Steve Webb, Classified Account Manager
swebb@gie.net
Michaela Dodrill, Market Coordinator
mdodrill@gie.net

CREATIVE
Jim Blayney, Creative Director
jblayney@gie.net
Katelyn Boyden, Graphic Designer
kboyden@gie.net

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gie media incorporated
5811 Canal Rd., Valley View, OH, 44125
Phone: 800-456-0707
Editorial Fax: 216-525-0515
Internet: www.cannabisbusinesstimes.com
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