Filtration Methods for Cannabis Extraction

2020
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Introduction to the Cannabis Industry

According to Business Insider, the legal cannabis market is “growing as fast as broadband Internet in the 2000s”\(^1\). This expansion shows no sign of stopping, and the market is developing into a vibrant, diverse landscape. According to Cowen analyst Vivien Azer, the industry will generate $85 billion in sales in 2030\(^2\).

As of November 2020, 35 states and the District of Columbia have legalized marijuana for medical use. Fifteen of these states also allow recreational cannabis use. Within the retail cannabis sector, extract and concentrate products are growing in popularity with an increasingly discerning customer base.

To ensure all consumers have access to safe and consistent cannabis concentrates, market leaders should strive to collaborate on industry standards and best practices. Whether it be BHO, dabs, distillates, kief, rosin, or sugar, processed products should be available to consumers with clear and accurate representation.

Sterlitech provides high-quality solutions for use in multiple cannabis filtration methods. This brief and regularly-updated guide is our contribution to the standardization of filtration methods and procedures across the industry.
Filtration in Cannabis Processing

Filtration is a necessary step in cannabis concentrate production. Depending on the specific process and end goal, filtration is used early or midway through the workflow. As you know, filtration removes unwanted particles from a fluid solution by passing the fluid through a membrane filter, with the help of applied vacuum or pressure. The choice of membrane dictates the size and characteristics of what is retained on its surface.

In conventional ethanol extraction, filtration usually occurs after winterization and before solvent recovery or further concentration (see the Cannabis Extraction Flow Diagram). Plant matter and waxes are the primary target for removal, but even smaller particles such as bacteria can be separated at this step. As new extraction methods develop, filtration technology is being explored to improve processes. The use of chemical-resistant nanofiltration membranes holds promise for simplifying and enhancing extraction using ethanol or hydrocarbon solvents.

In this guide, we outline standard steps for the ethanol extraction and purification process. We also provide an introduction to the use of nanofiltration membranes as an alternate method to accomplish these steps. The products and equipment required to run the methods described here are available at www.sterlitech.com.
Cannabis Extraction Flow Diagram

1. **Raw cannabis and ethanol**
   - Mixed with ethanol

2. **Crude ethanol extract**
   - Heated to 30-50°C

3. **Dissolved materials**
   - Potentially removed by mixing or blending

4. **Winterized extract**
   - Contains oil/wax solids
   - Multi-step filtration
     - Removes suspended particles, oil & wax solids, and bacteria

5. **Decolorized Extract**
   - Activated charcoal filtration
     - Removes chlorophyll

6. **Filtered extract**
   - Ethanol
   - Removes ethanol

7. **Concentrated Extract**
   - Rotary evaporation

**Filtration Methods for Cannabis Extraction**

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1. Preparing Extracts for Winterization

Description

Primary extraction using a solvent aims to produce oils that are free from particulates and contain the medically-relevant compounds (cannabinoids) in a higher concentration than raw cannabis. This can be accomplished using hydrocarbons (such as butane or propane), supercritical carbon dioxide, or ethanol to dissolve the components of raw starting materials while leaving behind most undesirable plant matter. Choice of solvent and process parameters varies depending on starting materials, desired end product, and other considerations. For example, carbon dioxide extraction eliminates chlorophyll early and leaves no residual solvent, but is more expensive and time intensive than other methods.\(^2\) This overview describes ethanol extraction, which is often used in the U.S. due to its availability, relative safety, and efficiency.

Purpose

To recover cannabis essential oils containing precious THC, CBD, terpene compounds, and other cannabinoids, solvent extraction, also referred to as primary extraction, is often the first step in refinement. Solvent extraction is usually followed by winterization and cold filtration to yield a highly concentrated final product.

Extraction with ethanol utilizes a defined ratio of solvent to extract, which changes depending on equipment and materials. A factor to consider here is the time required to filter solutions of higher and lower viscosity: solutions of lower viscosity (with a higher ratio of alcohol to extract) will take less time to filter and pose less threat of clogging. This set-up also provides an opportunity to change filters more frequently and improve filtration.

Materials

- Latex gloves
- Hot plate with stir bar or overhead stirrer
- Large glass beaker
- Thermometer
- Ethanol-resistant storage containers (ex: 1L LDPE/HDPE or borosilicate glass lab bottles)
- Food grade alcohol (ex: ethanol/Everclear, isopropyl alcohol, etc.)
- Cannabis extract
Method

1. Always wear gloves when handling chemicals. Fill a large glass beaker with ethanol. Make sure your beaker is sized to fit both solvent and cannabis extract without overflowing.

2. Place the beaker on a hot plate with stirrer function, and add a stir bar. Set the hot plate temperature between 30-50 degrees Celsius and stir on medium speed.
3. Slowly add cannabis extract to the heated ethanol while stirring.

4. Mix until the solution appears homogeneous.

5. Increase heat if necessary to improve mixing. Use a thermometer to monitor the temperature of the solution, ensuring that the temperature does not approach or exceed ethanol's boiling point (78 degrees Celsius. Heating beyond this temperature poses a safety hazard and results in the loss of certain volatile cannabinoids.

6. Let the solution cool to room temperature, then pour into an ethanol-compatible container for cold storage before further purification or use.
2. Winterization

Description

After the initial extraction, most products require further processing before they are ready for consumption. Winterization is often the next step to purify cannabis oils, where the main goal is to precipitate plant waxes/lipids so that they can be filtered out. When ethanol is used as a solvent, the plant lipids and cannabis extracts are both soluble at room temperature and cannot be separated. However, when the temperature is decreased, the waxes/lipids are no longer soluble and can be removed.

Purpose

Not all products undergo winterization, especially if the producer is interested in retaining terpenes or other properties plant waxes may carry. However, when cannabis is intended to be ingested by smoking, removing fats is often desirable for improved potency and quality. A household freezer is theoretically cold enough to solidify plant fats from an extract solution, but the process can take days. Cryofreezers and dry ice are often employed to speed up the process. The precipitated solids can be rewashed after rapid freezing to ensure no desired products are co-precipitated out.

Containers for winterization should not be excessively large, as this will slow cooling. Borosilicate glass lab bottles are recommended, and some even come with convenient GL45 threading to fit many bottle caps. The time required for full freezing and precipitation will vary depending on the method of cooling, though erring on the longer side will maintain higher yield.
Filtration Methods for Cannabis Extraction

Materials

- Freezer or dry ice
- GL45 1L lab containers

Method

1. Pour ethanol/cannabis solution into solvent and temperature-compatible containers.

2. Allow the solution to freeze completely, usually 12-24 hours.
3. Filtration

Description

After fats and waxes solidify, they can be separated from the cannabis solution. Filtration accomplishes this separation, as well as eliminating any particulates that reduce overall product quality.

Purpose

The goal of filtration is to improve clarity, increase potency, enhance stability, and create a repeatable customer experience. Therefore, selection of appropriate filter membranes is essential. To learn more about how membranes filters are rated, please see our blog post. As experts in membrane separation processes, we have a wide selection of filters and a wealth of application knowledge. We recommend using a multi-step process with a combination of sequential filtrations to achieve products with high quality and consistency. The suggested filtration steps are explained below.

1. Coarse cellulose filter (19-26 micron pore size range) to remove waxes/lipids.

2. Medium cellulose filter paper (6 micron pore size range) to remove any other particulates before very fine filtration.

3. Fine PES filter (0.2 or 0.45 micron pore size) to remove bacteria and micro-particulates to result in a highly refined extract.

Setting up the filtration apparatus is just as important as filter selection. Buchner funnels are great filter holders for coarse filtration, like the first two steps designated above. However, the final filtration on the sub-micron (0.2 or 0.45 micron pores size) level requires a filter holder with a clamping mechanism to facilitate an impenetrable seal and solid membrane placement. Sterlitech microanalysis filter holders designed exactly for this, with the added convenience of easy cleaning. The receiving flask should obviously be chosen to fit the volume, tube fittings, and mouth size.

For vacuum pump selection, it is important to consider the max vacuum and compatibility with the chemical solvent - i.e. ethanol. The suction should be strong enough to pull fluid through the filter, but not so powerful as to pull through unwanted material. Sterlitech recommends oil-free, compact, and quiet pumps like the PILOT and Rocker models in our catalog. If the pump is not resistant to chemical exposure, we recommend adding a cold trap to your apparatus to prevent ethanol from entering the pump.
Materials

- Filters
- Buchner funnel with rubber stopper
- Microanalysis filter holder
- Vacuum pump
- Vacuum tubing
- Vacuum manifold
- Funnel
- Receiving flask

Method

1. Prepare filtration equipment by setting up the receiving flask, vacuum pump, hoses, filter funnel and/or manifolds.
2. Insert the appropriate membrane into the filter holder assembly.

3. For filtration using a Buchner funnel, wet the membrane before adding solution. This secures the filter for optimal performance.

4. Turn on the vacuum pump. For manifolds, also turn on the vacuum port.

5. Decant/pour the winterized alcohol/cannabis solution slowly into the filter funnel. Attempt to minimize the amount of contact between heavy precipitate and the filter to reduce clogging and extend filter function.
6. When the solution has been emptied onto the membrane, allow the vacuum to run for an additional 30 seconds to ensure completion.

*Pro-tip: Spritz the surface of the cake layer that accumulates on the membrane with cold ethanol to re-dissolve the extract.

7. Repeat this process as much as needed to improve yield.
4. Solvent Recovery

Description

If previous steps are conducted successfully, there should be low to no contaminants present in solution after filtration: only ethanol and cannabis essential oils should remain. At this point, ethanol can be recovered to yield the final cannabis concentrate product. Any remaining water will also be removed here. The most common practice to accomplish solvent recovery is the use of a rotary evaporator. This apparatus uses heat, vacuum pressure, and centrifugal force to separate solutions.

Purpose

Evaporation of ethanol, especially using a rotary evaporator, is an extremely efficient mechanism of recovery for further use. The machine boils the solution to vaporize the ethanol, then traps the gas and sends it through a cooling condenser. The ethanol then forms a liquid again and drips into a new container, leaving behind the un-boiled extracts in the original glass bulb.

Rotary evaporator setups are customizable, and the options can be confusing. Dry ice condensers and coiled condensers both work, although chilled coiled condensers are often favorable as they require less monitoring. Other considerations are bath and evaporator flask sizing/capacities, coating, and heating elements. Size will determine production capabilities, while coating and heating element selection will influence the longevity of the equipment. Typically, powder-coated stainless steel and electropolished heating baths will be more expensive but suffer less oxidation or corrosion.

When programming the rotary evaporator, producers have the opportunity to develop unique methods for their process and potentially run a hands-free recovery. Most rotary evaporators, including those offered by Sterlitech, can continuously feed solution into the boiling flask to maximize run-time and minimize time spent in disassembly and clean-up. A low temperature/slow rotation program will optimize volatile terpene preservation, while a high temp/fast rotation program will increase throughput.
Filtration Methods for Cannabis Extraction

Materials

- Rotary Evaporator (chilling condenser or dry ice)
- Round-bottom flask
- Collection flask
- Chiller with tubing
- Vacuum pump

Method

1. Turn the system on and ensure the chiller is at the desire temperature.

2. Lower the feed tube into your filtered cannabis solution and turn on the vacuum to pull solution into the round-bottom flask. Fill only 25-50% of the volume capacity for this flask.

3. Turn on the rotation function and allow the bath to heat to the set temperature.

4. Monitor the progress of the separation and stop when the ethanol ceases to drip into the collection flask. This process can be repeated multiple times to remove residual alcohol. To perform continuous evaporation, continue feeding filtered solution up to 50% of the flask volume as needed.
Organic Solvent Nanofiltration Method

Legalization and the increasing demand for cannabis products have transformed technology used for extracts from simple to sophisticated in recent years.

Organic Solvent Nanofiltration (OSN) using Duramem™ Membranes has the potential to simplify the cannabis extraction process by reducing the multistep processes of Winterization, Filtration, Evaporation, and Color Removal to a single filtration step. They also offer a solution for removing color (i.e. chlorophyll) and impurities while preserving up to 90% concentration of THC and CBDs.

Duramem™ membranes are stable in organic solvents, such as in ethanol and other alcohols, and their tight pore structures make them an ideal solution for simplifying the extraction process and removing color.

Image from Evonik Corporation, 2018
References


Glossary

Kief: usually refers to trichomes that are physically separated from the rest of the plant material using a sieve. THC content can be anywhere between 20-60%.

Bubble Hash: hash produced by ice water extraction to isolate trichomes from other plant matter.

Butane hash oil (BHO): oil extracted using butane, which can be processed to create a variety of end products (shatter, holy water, honeycomb, crumble, sap, etc) with THC content between 80-90%.

Supercritical CO₂ oil: oil extracted using high pressure supercritical liquid CO₂.

Rick Simpson Oil (RSO): whole plant cannabis oil that can be applied to skin, and is also known as hemp oil, cannabis oil, or Phoenix Tears. This product is named after Rick Simpson, who is attributed to creating this method of whole plant oil and using it to treat his own skin cancer.

Rosin: solid form of cannabis resin created by applying high temperature and pressure to melt raw material through a screen.

Nominal pore size: refers to a filter capable of preventing passage of a minimum percentage (usually between 60% and 90%) of solid particles of greater than the stated pore size, which is normally expressed in micrometers or microns.

Absolute pore size: specifies the pore size at which a challenge organism or particle of a particular size will be blocked by the filter with nearly complete efficiency (greater than 99.8%) under strictly defined test conditions.
Founded in 2001 in Kent, WA, Sterlitech Corporation manufactures and markets filtration focused laboratory products to a broad spectrum of scientific and industrial sectors. Its line of flat sheet membranes and tangential flow cells deliver industry-leading performance and reliable results. Configured for reverse osmosis, nanofiltration, ultrafiltration, and microfiltration applications, Sterlitech’s bench scale test equipment provides the versatility required to innovate.

Sterlitech’s comprehensive line of products is supported by the expertise of its technical specialists who can assist with application-specific product selection, and provide customized solutions where necessary. Unique problem-solving approaches, flexibility, and consistent quality have made Sterlitech Corporation a renowned global provider of filtration products and equipment.

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