TN-141 - Cannabis Extraction by Supercritical Carbon Dioxide – Dewatering

INTRODUCTION

Carbon Dioxide (CO_2) extraction of Cannabis is becoming more prevalent as the extract market grows with the spread of medical and recreational legalization. However, due to the historically underground nature of working with the substance, little true scientific experimentation and process development has occurred; even less has been published. Those new to the field have limited guidance.

The following is a compilation of the process learnings regarding dewatering techniques that Supercritical Fluid Technologies (SFT) customers' companies currently working in the CO_2 Cannabis extraction field, have shared with us. Due to the proprietary nature of each company's specific process, the material here will be presented in a generic way that is applicable to the majority of processors.

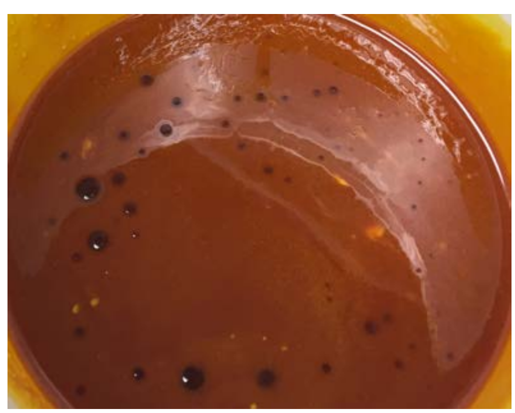
DEWATERING

All Cannabis feedstock contains water, even that which is properly dried and cured. Removing this water from the extract after processing raises the cannabinoid concentration of the extract, prevents it from spattering when consumed directly though vaping or in a rig, presents a cleaner taste to the consumer, and enables a more attractive appearance.

CO_2 processing creates extract that will be laden with dry ice. Depending on the equipment and processing conditions the extract will need to be de-gassed and/or heated prior to dewatering.

Gross Dewatering

Material from the extractor will usually contain some visible water, but unless heated, a large portion of the water extracted will remain entrained.



The easiest method that our customers have found to remove the majority of the water is to put the extracted material into a sealed glass container and heat it, with occasional gentle agitation (stirred rather than shaken) until the material separates cleanly into a water and extract layer. The time required to effect this phase separation depends on a number of factors including: the amount of material being separated, the heating method, the starting temperature of the extract, the temperature of the heat source, and innate properties of the extracted material.

Most of the effects of these parameters are common sense. More material will take longer to separate. A water bath will be a faster and more effective heat source than an oven. If the starting temperature of the material is higher it will take less time to warm to separation conditions. Increasing the temperature of the heat source will decrease the time needed to heat to temperature. The intrinsic properties of the feed stock such as the genetic makeup of the material, the feedstock format (bud, trim, shake, whole plant), material age, and storage/curing history also affect the separation time, but not in ways that are as obvious.

One consideration of this method is that THCA, the acidic, non-psychoactive form of cannabis, decarboxylates into THC, the psychoactive form of the cannabinoid, over time. The rate of decarboxylation increases with increasing temperature. Increasing temperature also increases the rate of terpenoid loss. With the increased interest in the role of terpenoids in both taste and effect produced during consumption, retaining terpenoids has become a goal of most processors. Most of our customers therefore balance speed of separation and decarboxylation against terpene retention by using a water bath set between 50 and 60 degrees C.

When there are two distinct phases with a clean demarcation in the glass container, the glassware is removed from the heat and the extract is allowed to cool at room temperature. It is essential that the material in the container be allowed to fully solidify so that extract does not inadvertently spill into the water phase during water removal. Once the extract is completely solid the container may be uncapped and inverted to pour off the water layer.

If further dewatering is desired, most of our customers use either a rotary evaporator or a vacuum oven for further processing.

Rotary Evaporation

Following gross dewatering the extract can be further dewatered via simple distillation or by using a rotary evaporator, which is generally preferred over simple distillation as both rotation and vacuum enable faster and more complete water removal. If using rotary evaporation for the secondary method of water removal, it is effective to use an evaporative flask for gross dewatering in order to limit both material loss during transfer and another heat cycle that would be required if transferring material between containers.

The most common rotary evaporation processing parameters are:

- water bath at 60 degrees C,
- rotation at 1/3 to ½ of equipment capacity, and
- vacuum gently decreasing to the vacuum pump limit.

Evaporation is complete when the vacuum has reached its limit and there have been no drips for at least 10 minutes.

At the start of evaporation the extract will have a matte look. By the end of the process the material will appear shiny.

Vacuum Oven

This secondary dewatering method has fewer set processing parameters as it is more dependent on what is in each separate sample being dewatered. The commonalities from our customers:

- 60 degree oven,
- gently reduce the vacuum down to the pump's limit,
- process a thin layer of material at a time, and
- start with cold or room temperature material.

The main issue with using this method is that there is spatter, which can be attributed to both water and terpenoids. Using a vessel with tall walls will help to contain the spatter. Starting the process with cold or room temperature extract allows the extract to release water as it "foams up" also reduces spatter.

In the photo on the left the material is just starting to heat up and is beginning to foam. The photo on the right shows two containers of extract that are fully foaming. They are reminiscent of a cake rising.

Vacuum in the oven must be reduced gently in the early stages of processing so that the foaming material does not overflow. Once the foaming has calmed down the material becomes clear and glassy as the vacuum is reduced.

The last remaining water bubbles will be around the edges of the container. Care must be taken at this point to prevent or reduce the amount of spatter (and this mess and material loss).

CONCLUSION

Our customers have found a convenient and easy method to remove the majority of the water from supercritical CO_2 Cannabis extract. Should additional dewatering be needed, heat and vacuum are required. Rotary evaporators or a vacuum oven are common pieces of equipment in even the most basic laboratory, and they can be used to effectively for second stage dewatering.

This article is the third in a series of articles that addressing the practical considerations of the extraction of Cannabis.   To learn more about our Cannabis extraction equipment, please contact Supercritical Fluid Technologies at 302-738-3420 or info@supercriticalfluids.com