North Carolina Agricultural and Technical State University

INDUSTRIAL HEMP RESEARCH
CANNABIS ANALYSIS, EXTRACTION AND PURIFICATION, OCTOBER 17, 2019

Matthew Todd, Mehrab Valizadehderakhshan

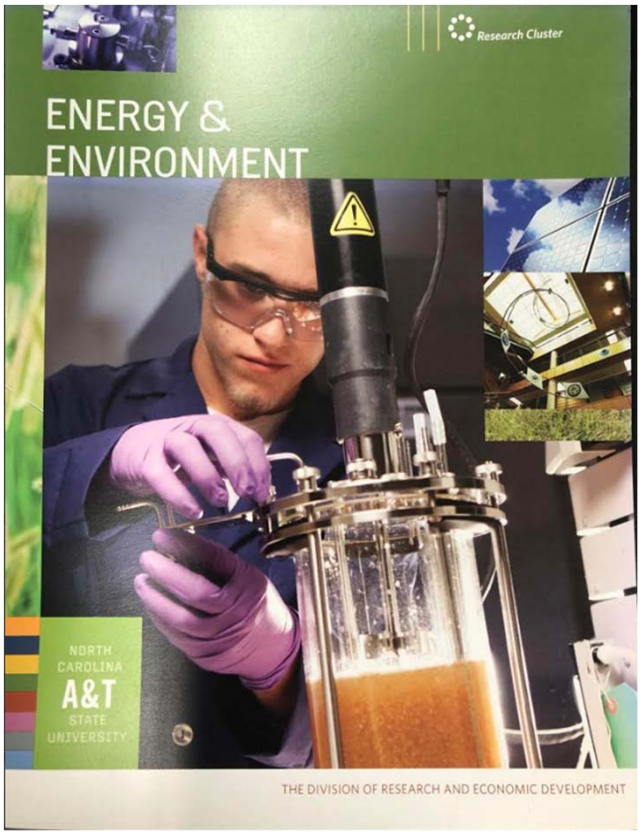
Ghasem Shahbazi

AGGIES DO

Mathew Todd, Research Engineer
NC A&T SU Biological Engineering

Responsible for Design, Construction, Operation, Optimization and Maintenance of Pilot Scale Bio-refineries at NC A&T SU for grant operated research for NSF, DOE, DOD, USDA and more.

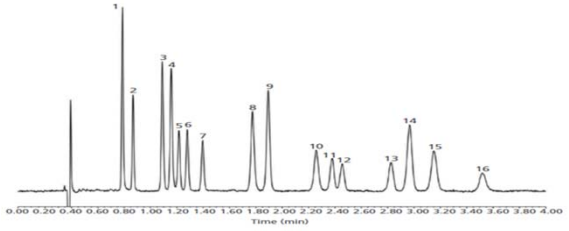
Currently Analytical Lead for Industrial Hemp Research



Instrumental methods are used for the identification and classification of Cannabis plants and products.

Because of the complex chemistry of Cannabis, separation techniques, such as GC or LC, often coupled with MS, are necessary for the acquisition of the typical chemical profiles.

However, especially for screening purposes and on-site field testing, non-instrumental techniques like thin-layer chromatography (TLC) and color reactions are being developed and will assist brokers and law enforcement.



All analysis conditions and Equipment listed in Hemp Analysis Protocol provided by NCDA have been replicated at NC A&T SU for all research related to Industrial Hemp.

Recent growth in federal regulations by the USDA and the testing at A&T for all samples reflects these changes.

- Cannabinoids
- Terpene Profiling: Flavors & Aromas
- Pesticide Residue Screening
- Extraction Efficiency
- Shelf-Life Stability
- Residual Solvents
- Basic Nutritional Panel
- Moisture Content / Moisture Balance
- Mycotoxins Analysis
- Food Borne Pathogens

https://www.avazyme.com/industrial-hemp-process-packet/

Cannabidiol (CBD)	$R_1 = H, R_2 = C_5H_{11}, R_3 = H$	Anxiolytic Antipsychotic Analgesic Anti-inflammatory Antioxydant Antispasmodic

CBD is a subject of research at NC A&T SU due to its **abundance** within certain strains of cannabis and high medical and **commercial value**.

Cannabidiol (CBD) type: CBD was isolated in 1940, but its correct structure was first elucidated in 1963 by Mechoulam and Shvo . Seven CBD-type cannabinoids with C1 to C5 side chains have been described. CBD and its corresponding acid CBDA are the most abundant cannabinoids in fiber-type Cannabis (industrial hemp).

Adams, R., Hunt, M., and Clark, J. (1940) Structure of cannabidiol, a product isolated from the marihuana extract of Minnesota wild hemp. I. J. Am. Chem. Soc. 62, 196–199. 14.

Mechoulam, R. and Shvo, Y. (1963) Hashish—I. The structure of cannabidiol. Tetrahedron 19, 2073–2078.

The need for Processing Industrial Hemp **arises at the moment of harvest**. Hemp is a perishable product that deteriorates within days without proper drying but can have the shelf life of up to TWO years with proper Processing and Packaging.

Processing is the act of first preserving and possibly concentrating the components of value from the plant material while meeting the preconceived burden of purity required by the market.

Drying/ Curing the plant material far below the moisture content of smoking flower is required for efficient extraction.

Grinding the plant material is required to increase extraction vessel capacity and efficiency.

Extraction removes the oil from the plant material and concentrates the essential components by 5x. This is achieved by passing a Class II (food safe) solvent across the bud material, then evaporating that solvent, leaving cannabinoids and contaminants with similar chemical properties.

Winterization remove some of the natural waxes and slightly improves purity and improves efficiency during Distillation.

Extraction

Distillation separates light (terpenes) and heavy (chlorophyll, heavy metals, plant residue) components to further increase purity of CBD and remove shelf unstable byproducts. During this step the CBD is de-carboxilated to make the cannabinoids biologically available.

Isolation can be achieved thru physical chemistry (direct crystallization) and chromatography. By repeating these steps any purity deemed optimal for any end use market can be achieved.

Product Mixing is required to achieve a specific dosing and deliver products trans-dermally or by ingestion. Specific chemistry is required in most cases.

Packaging is required to distribute products before shipping.

Isolation

Grinding is essential to increasing the surface area of the material for extraction.

The finer the grind, the greater access to waxes, and chlorophyl, the main contaminants.

Hydrocarbon Extraction: lower wax and chlorophyll(some conditions), fastest extraction, very dangerous due to high flammability, excellent affinity to terpenolic compounds

Because of their nonpolar nature, almost all hydrocarbon solvents are useful for extracting cannabinoids and terpenes. Butane and Propane are the most common. Closed Loop Systems must be run by highly trained technicians and due to the high flammability of these hydrocarbons, a “C1D1” facility, a class one, division one safety-rated room to house the equipment, is required.

CO2 Extraction: lowest wax and chlorophyll content, energy intensive and slower compared to other methods

For cannabinoid extractions, the supercritical phase is used, the CO_2 appears a clear liquid and can be “pumped” . The supercritical phase occurs at higher temperature and pressure combinations, which changes the polarity of the molecules to be more specific for dissolving cannabinoids.

Subcritical CO_2 (liquid phase CO_2) can also be used for terpene extraction, but that requires somewhat more technical skills for the operator and an extraction vessel with tunable parameters

Ethanol extraction: higher wax and chlorophyll content, higher throughputs normally achieved due to ease in scaling up

The rapid extraction times and high throughput for large capacities as well as the relative safety of residual levels in finished products lends to the popularity.
The molecular properties are such that even at very cold temperatures, it is still a good solvent for cannabinoids, while it is not a good solvent for fats and lipids, so a winterization step is not necessary.
Ethanol is also flammable, but like the other hydrocarbons, it can be performed quite safely in closed systems that aren't complicated to operate.

IOSITY Labs donated their services to extract the the A&T 2019 crop, and contribute knowledge to the Doctorate thesis of a student at A&T.

Extraction Methodology

Solvent Pre-Temp: -30 ° C

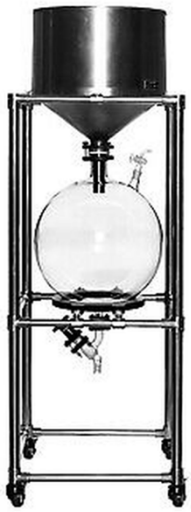
Material Column: 2 kilos per column

Solvent Post-Temp: -60 ° C

Winterization: 500 micron filter

The Precision X10 unit was used to extract the industrial hemp.

Winterization: a phase separation or precipitation in which the mixture of raw extract dissolved in ethanol is chilled and the fats and lipids solidify and can be physically separated from the liquid cannabinoid–ethanol solution



Once some of the waxes have been removed through winterization, now the solvent must be removed.

A Roto-Vap will remove the Ethanol but some residual solvent will be left and must be removed.

If the extract is the final product the residual solvent could be removed by vacuum oven. Then the Cannabinoids can be de-carboxylated by heating.

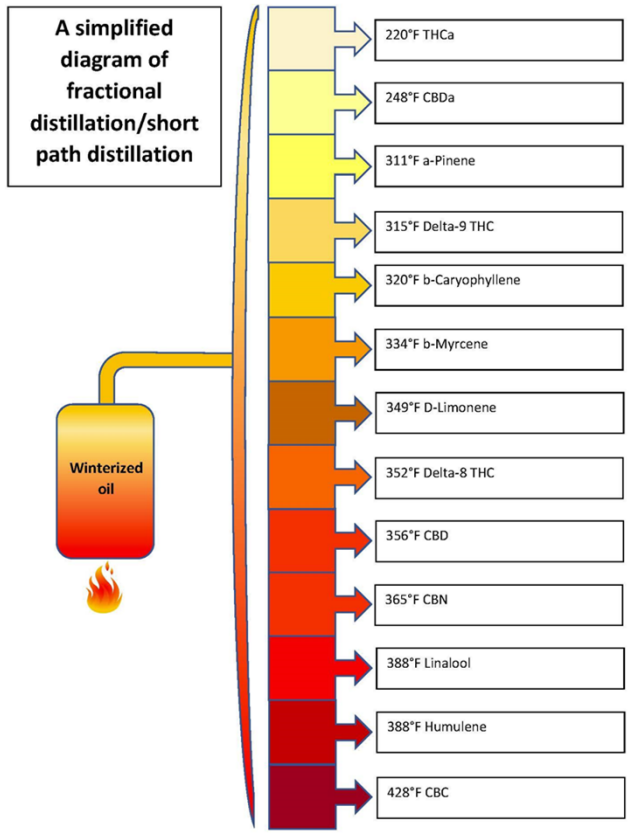
The extract is a combination of cannabinoids and other components.

Depending on the conditions under which the hemp was run, the hemp and the solvent used; more or less purity of any and all of the constituents can be achieved.

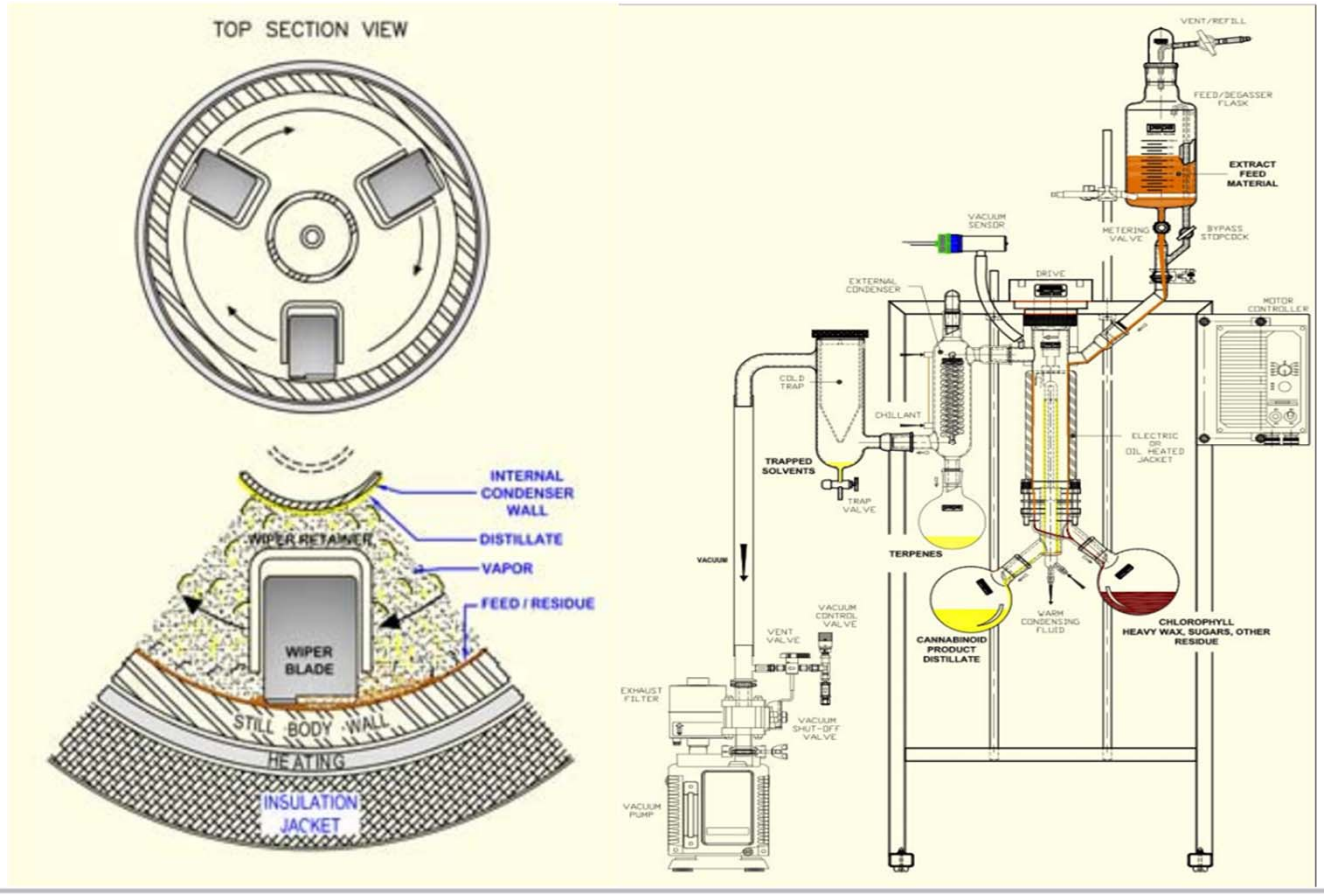
There are components within the extract that can sour or biodegrade and removal of these will improve quality and shelf life.

Evaporative distillation is used to fractionate crude CBD oil to its many chemical constituents.

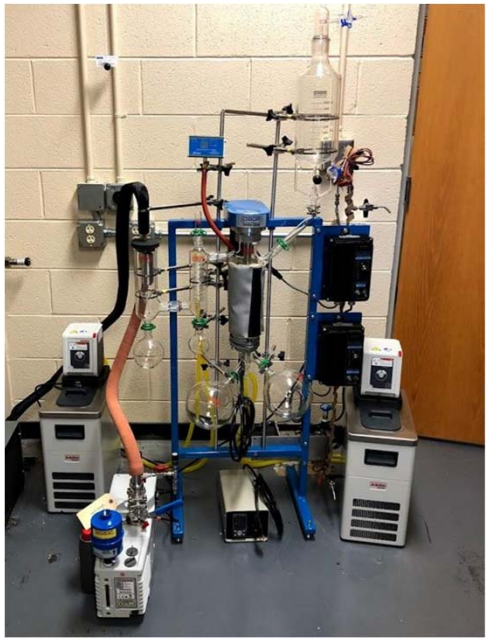
Distillation can yield high purity and high value products for the farmers and processors



Distillation: Wiped Film Molecular Sieve



Distillation has been reported to remove any residual particals, fat, metals and many other naturally occurring products that reduce value and shelf life of CBD. At the same time distillation can be used to further purify internal substances to again improve their value.



A wiped film molecular sieve reported to produce the highest purities and allows for the preservation and purification of the terpenolic compounds, will be used to conduct research at A&T.

First Pass: Remove Lights, in this case Terpenes & residual solvents

Conditions:

1. Head Pressure: 10,000 microns Hg
2. Outer Head Temperature: 160 ^{o}C
3. Internal Head Temperature: 20 ^{o}C
4. Cold Trap 1: -15 ^{o}C
5. Cold Trap 2: -45 ^{o}C

Second Pass Distillation: Cannabinoids Removed from Color Bodies and Waxes

Second Pass: Remove Heavies, in this case Waxes Meatals and color bodies

Conditions:

1. Head Pressure: <50 microns Hg
2. Outer Head Temperature: 117 ^{o}C
3. Internal Head Temperature: 80 ^{o}C
4. Cold Trap 1: -15 ^{o}C
5. Cold Trap 2: -45 ^{o}C

Questions?

Matthew Todd
336-327-7977
mstodd@ncat.edu