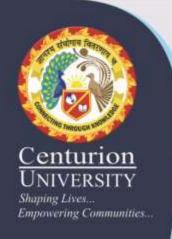


## **Super Critical Fluid Extraction**

**Skill Course** 



- 1. Principle of SCFE
- 2. Instrumentation



## Main functions performed in the SFE process

#### Extraction

Liquid CO 2 withdrawn from a gas cylinder is mixed with the appropriate modifier and is introduced at a specific pressure and temperature in the extraction chamber where the sample is extracted.

#### Collection

The solutes released from the supercritical fluid during expansion are collected. These compounds can be solids, liquids or even gases (very volatile compounds). The collection is done on a trapping material that can be an inert or adsorbing solid (silica, charcoal, Tenax) or a liquid.

#### Expansion

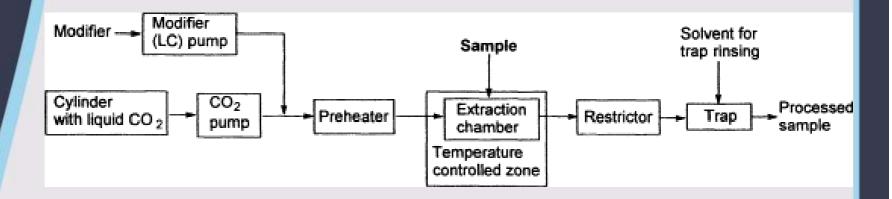
The fluid (in supercritical state) containing the extract is moved from the rest of the sample (matrix) through a restrictor. One side of the restrictor is at high extracting pressure while the other side is usually at ambient pressure, and the fluid expands. At the point of expansion, supercritical fluid becomes gas, and the solutes drop off from the solution.

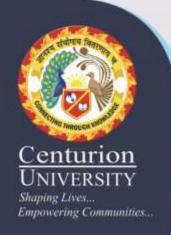
#### Reconstitution

The sample collected in the trap is transferred in a solution. A solvent must be chosen to rinse the trap, dissolve the solutes deposited there, and deliver a solution of the processed sample.



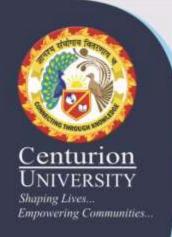
### Flow diagram of the SFE process





## **SCFE Systems**

- Supercritical Fluid Extraction (SCFE) Systems extract chemical compounds using supercritical carbon dioxide instead of an organic solvent.
- ❖ The supercritical fluid state occurs when a fluid is above its critical temperature (Tc) and critical pressure (Pc), when it is between the typical gas and liquid state.
- Manipulating the temperature and pressure of the fluid can solubilize the material of interest and selectively extract it.

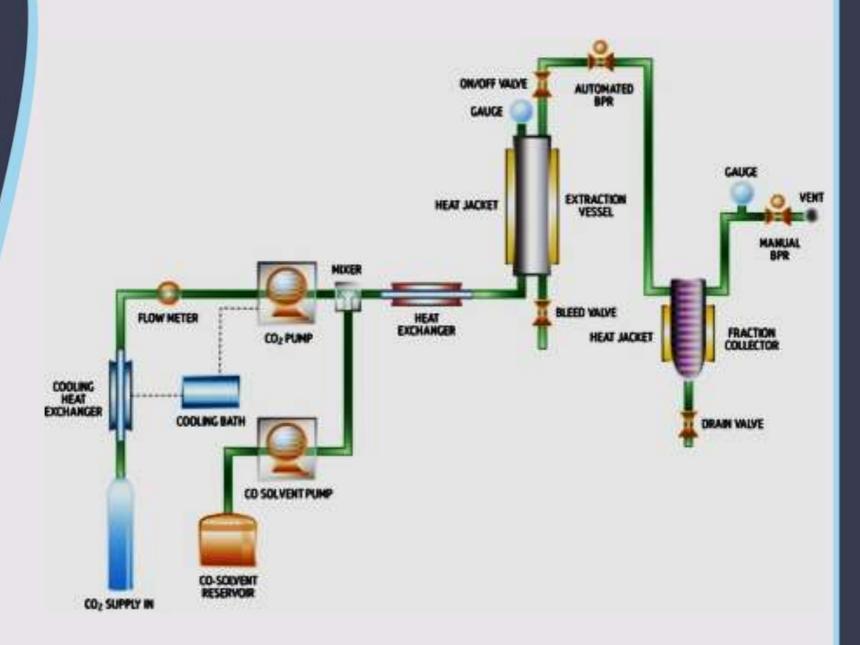


## **SCF Systems**

- ❖ The sample is placed in an extraction vessel and pressurized with CO₂ to dissolve the sample.
- ❖ Transferred to a fraction collector, the contents are depressurized and the CO₂ loses its solvating power causing the desired material to precipitate.
- ❖ The condensed CO₂ can be recycled.



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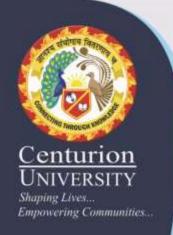




# Components of a commercial SCFE plant

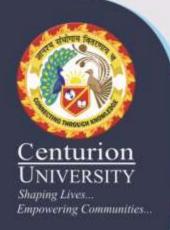
- Extractors
- Separators
- CO<sub>2</sub> hold-up tank
- Heat exchangers

The extraction process works in a closed loop with constant circulation of  $CO_2$  in the system. It is a semi-batch operation with typical batch time of 2 to 4 hours.



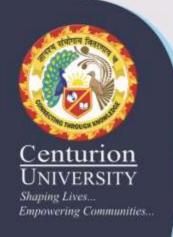
### Hold up tank and heat exchanger

- ➤ CO<sub>2</sub> is liquefied and fed to the hold-up tank through a heat exchanger (Condenser).
- ➤ Liquid CO₂ is then fed to the high-pressure metering pump, through a heat exchanger (subcooler) to ensure supply of CO₂ in liquid form.
- > Supercritical temperature is achieved by heating the high-pressure liquid from the pump discharge in a heat exchanger (pre-heater).
- ➤ A small quantity of other solvents such as water, ethanol etc. can be added in to the CO<sub>2</sub> stream through an entrainer pump.



#### **Extractor**

- This supercritical fluid then flows through the extractors containing raw material powder.
- ➤ The raw material powder, to be extracted is placed in these extractors with help of perforated baskets.
- > Extractors are equipped with specially designed quick acting closures for easy and fast opening.
- ➤ The supercritical CO<sub>2</sub> with dissolved extract then flows to separators.



#### **Separator**

- ➤ In the separators, lower temperature and pressure conditions are maintained for recovery of the extract from the CO<sub>2</sub> stream.
- ➤ Thus with proper selection of conditions, fractionation of extract can be achieved by using two separators operating in series.
- Separator-1 is maintained at supercritical conditions, where typically high molecular weight components of the extract precipitate.
- ➤ Whereas in separator-2 sub-critical conditions are maintained for complete recovery of any extract from CO₂ stream.
- ➤ The CO₂ vapor leaving separator-2 is condensed and fed back to CO₂ hold-up tank.