

Supercritical Fluid Extraction

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Introduction to SCF

- Properties of SCF

Supercritical fluid Extraction (SFE)

- Components of SFE
- Critical properties of selected substances

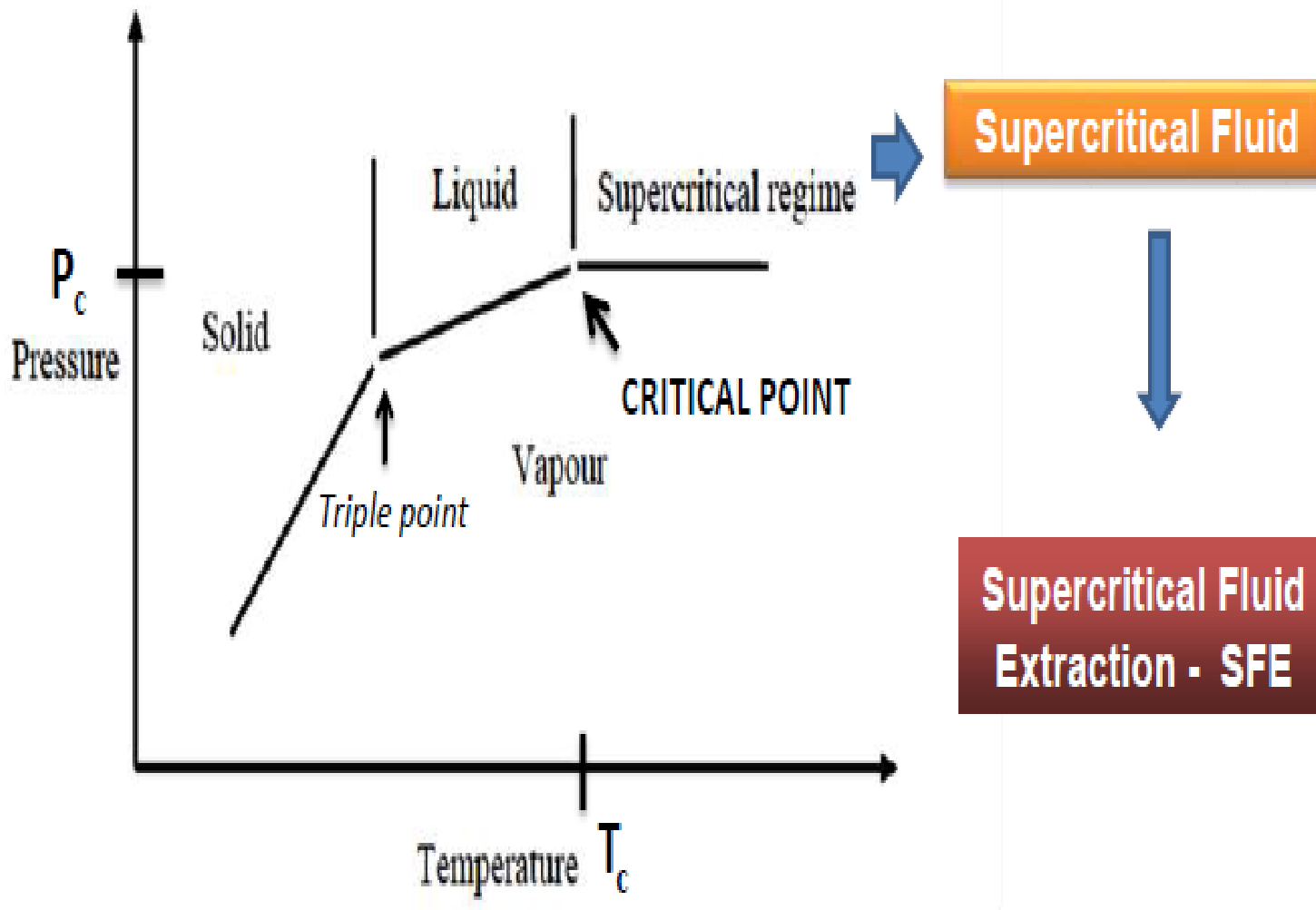
Modes of SFE

- Methods of developments of SFE

Objectives for commercialization

- Advantages
- Limitations
- Precautions
- Applications





At a certain temperature and pressure condition, liquid and vapour phases of a substance become indistinguishable. Known as **CRITICAL CONDITION**

Substances above critical point- **“SUPERCRITICAL FLUIDS” (SCF)**



Properties of SCF

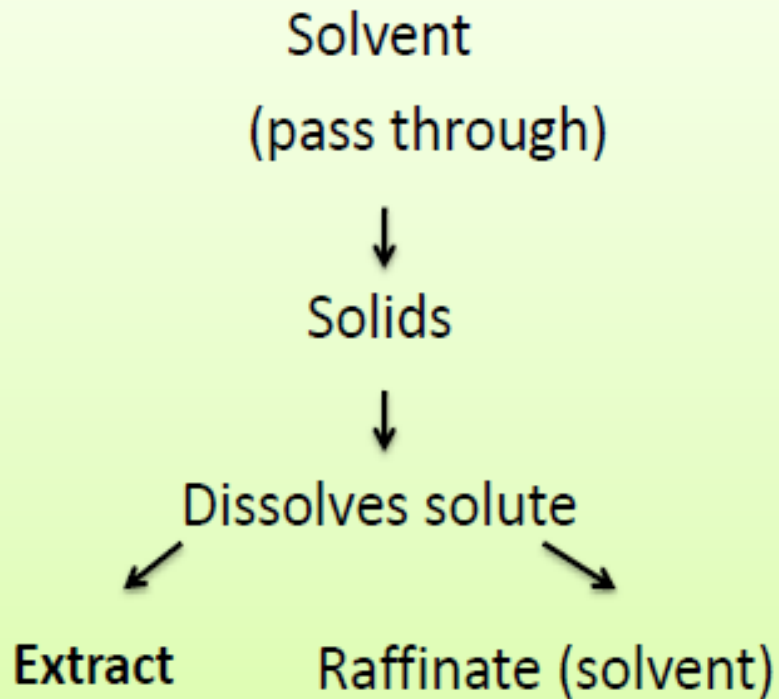
Physical and thermal properties of SCFs are in between pure liquid and gas, hence can also be known as '**Compressible liquids**' or '**dense gases**'

- Changes in properties are for a SCF are as follows:
 - Liquid like densities (100-1000 times greater than gases)
 - Diffusivities higher than liquids (10^{-3} and 10^{-4} cm²/s)
 - Good solvating power
 - Reduction in surface tension
 - Low viscosity (10-100 times less than liquid)
 - Gas like compressibility properties

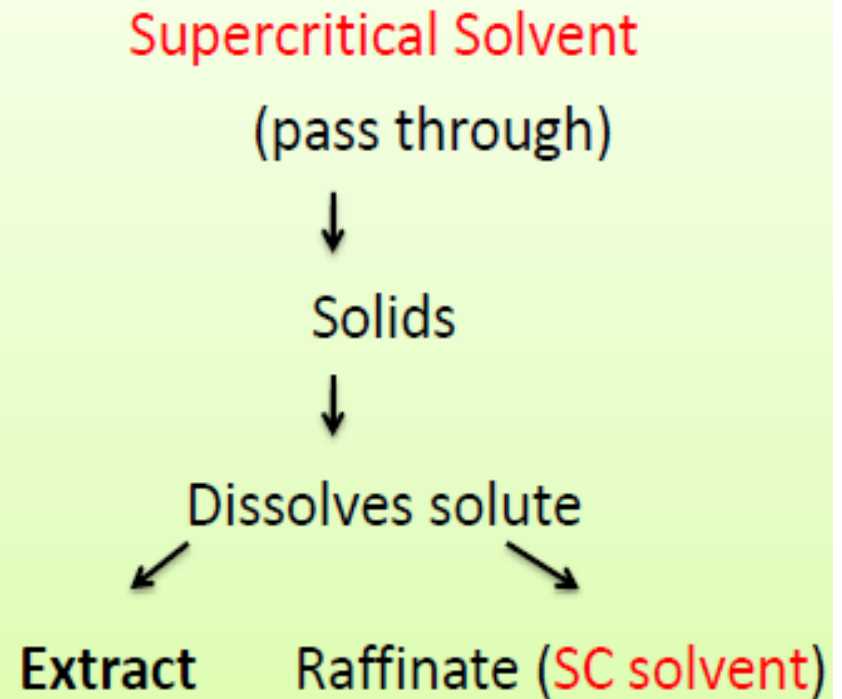
Therefore they possess high penetrating power

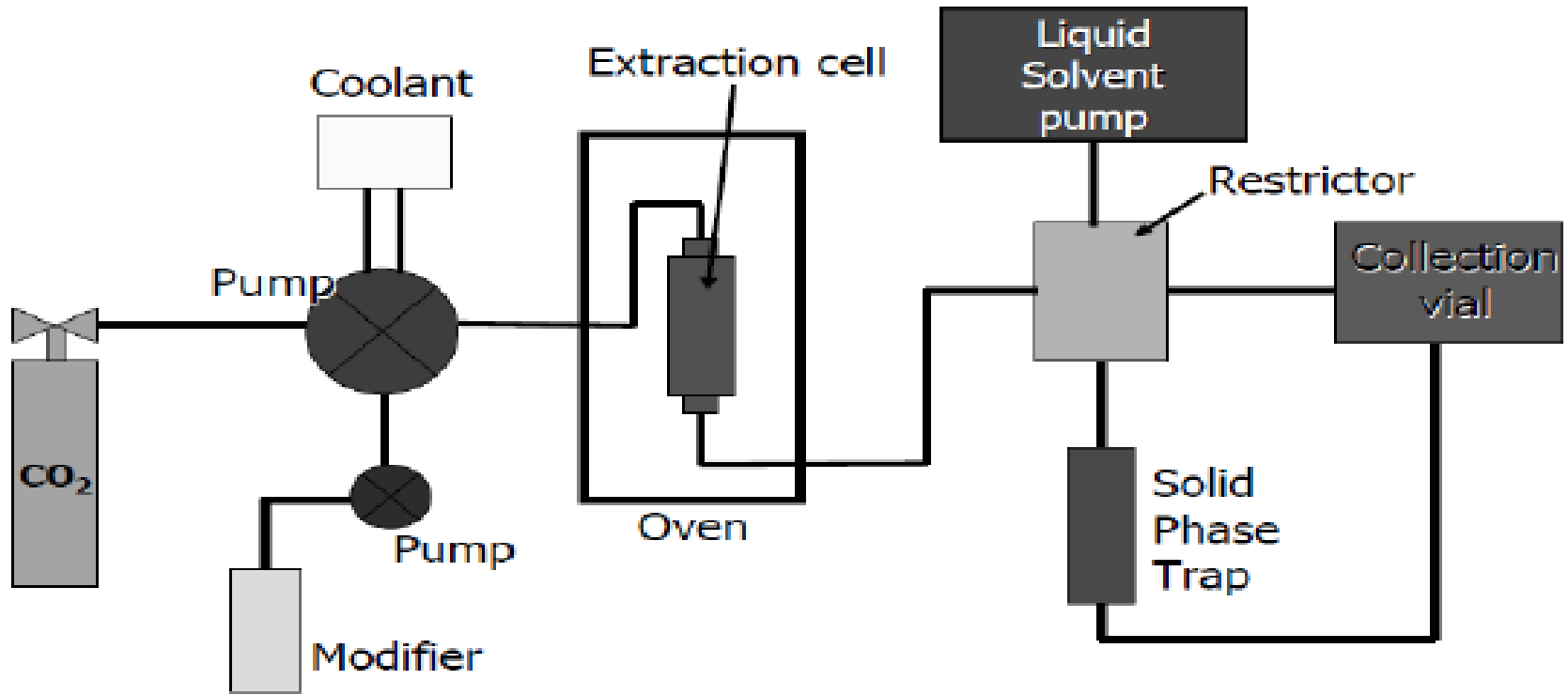


Solvent extraction method



Supercritical fluid extraction





Supercritical fluid extraction is the process of separating one component from another (the matrix) using supercritical fluids as the extracting solvent



OPERATION FLOW CHART

Introduction of feed into extractor (solid feed) or extractor in modified column either co-currently or counter currently



Formation of mobile phase: mixing of solutes with supercritical fluid.



Exposure of mobile phase to pressures (50-500 atm) and temperatures (ambient to 300°C) near or above the critical point for enhancing the mobile phase solvating power.



Isolation of dissolved solute by precipitation

Components of SCF extraction

1. **Fluid reservoir** (gas cylinder in case of CO₂)
2. **Pump**
 - Reciprocating pump
 - Syringe pump (pulse-free flow at large range of flow rates)
3. **Extraction cell/column** (stationary phase)
 - Usually stainless steel Chamber or vessel in compartment
 - Capable of withstanding high pressure (300-600 atm) [for solids]
 - Open tubular capillary columns or packed columns [liquids]



4. **Restrictor**

– Maintaining pressure change inside the extraction vessel

Two types-

– fixed (linear restrictor, tapered restrictor, integral restrictor, ceramic frit restrictor, metal restrictor)

– Variable (variable nozzle, back pressure regulator)

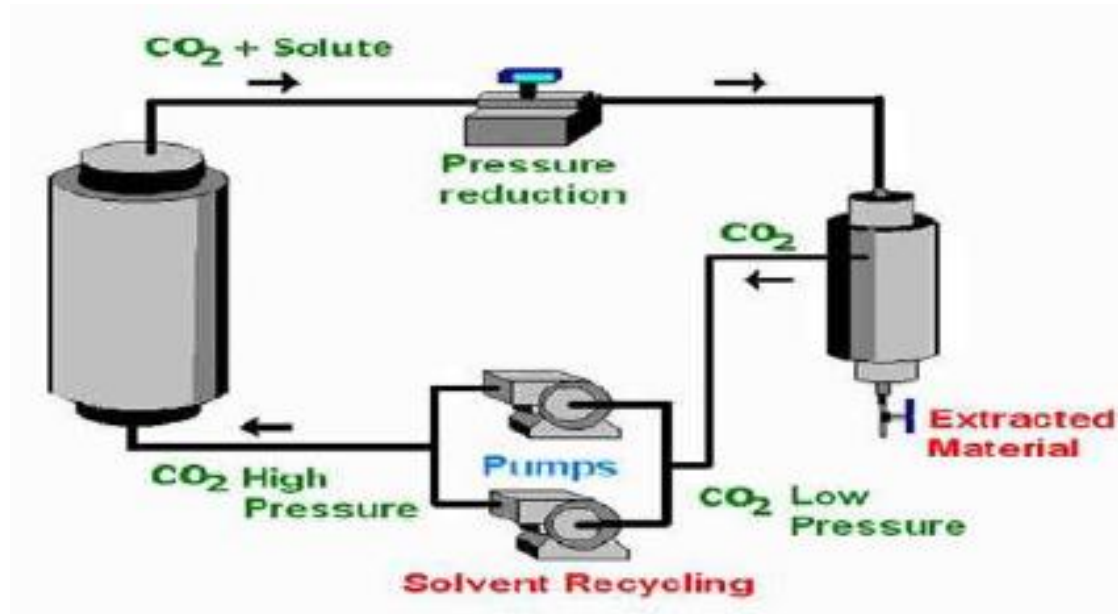
5. **Collector** (trapping system)

6. **Detectors** (flame ionization detector of gas chromatography)



Recycling of SFE can be done-

1. *Reduction of pressure*
2. *Reduction of temperature*
3. *Pumping SCF to expansion tank*



1. Reduction of pressure- SCF unable to dissolve the solute, separation of solid under gravity and the gas at low pressure is compressed back to the supercritical conditions.

2. Reduction of temperature - solute drops and recovery of solvent without recompression

3. Pumping SCF to expansion tank - where it becomes gas resulting in very less solubility i.e. separation of solute. Spent gases are then recompressed and recycled. Heat exchangers are used to maintain temperature and prevent excessive cooling at throttling valve called as Joule-Kelvin effect.

Sample Matrix Parameters that influence SFE:

- Particle size and shape
- Surface area and porosity
- Moisture content
- Changes in morphology
- Sample size
- Extractable level
- **The parameters effect on solubility:**
 - The vapour pressure of the component
 - Interaction with the supercritical fluid
 - Temperature, pressure, density and additives



ADVANTAGES

Elimination of organic solvents i.e. reduces the risk of storage.

- Rapid (due to fast back-diffusion of analytes in the SCF reduces the extraction time since the complete extraction step is performed in about 20 min)
- Suitable for extraction and purification of compounds having low volatility present in solid or liquid
- Susceptible to thermal degradation (low operating conditions)
- Complete separation of solvent from extract and raffinate
- Continuous process
- Low handling cost
- Solvent recovery is easy
- Versatile and efficient (use of co-solvents and co-solutes)



APPLICATIONS

Food science

- Fat and oil samples in meat, egg, meals, chocolate, dairy products, seeds and food snacks,

Natural products

- Flavors & spices of ginger, eucalyptus, soyabean, coffee, soybean, basil, lime peels, potato chips, popcorn

By-products recovery

- Fruit and vegetable waste

