

SUPERCRITICAL FLUID EXTRACTION UNIT

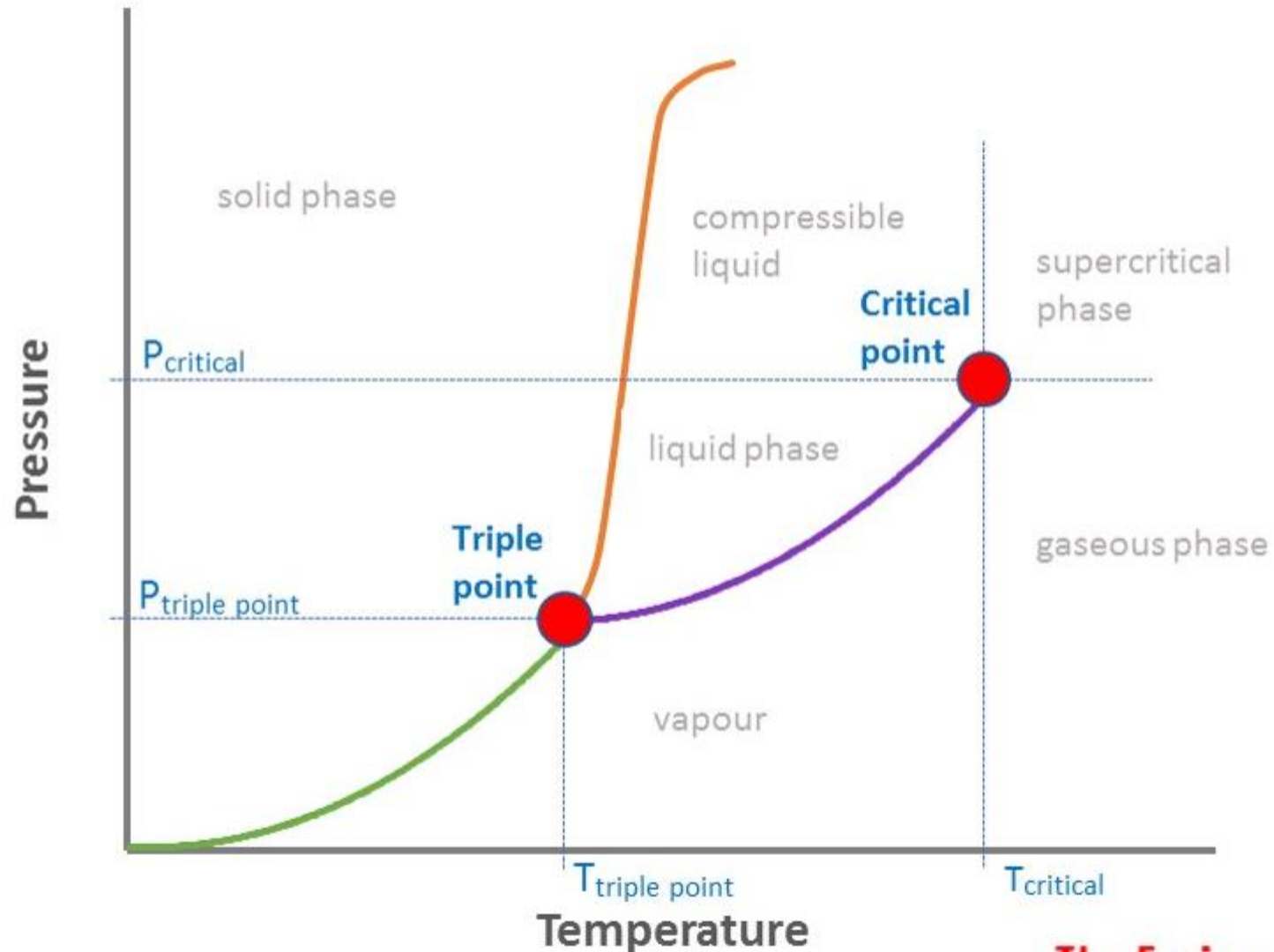
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INTRODUCTION

- Supercritical fluid is a state of matter that is intermediate between a gas and liquid in its properties.
- This state formed when a gas or liquid solvent is subjected to temperature and pressure condition exceeding a particular **critical point**.
- The temperature and pressure at which this point occurs are known as the Critical Temperature and Critical Pressure and are characteristic of the solvent.

- Beyond this point , the solvent will be neither a gas nor a liquid , but will possess the properties of both phases.
- Whether this supercritical fluid acts more like a gas or liquid will depend on the pressure as well as temperature.
- **Critical point** of pure substances is defined as the highest temperature and pressure at which the substances can exist in vapour- liquid equilibrium.

Triple point, critical point and supercritical phase



- At temperature and pressure above this point, a single homogenous fluid is formed, which is known as **SCF**.
- **SCF** is heavy like liquid but has penetration power like gas.
- **SCF**'s are produced by heating a gas above its critical temperature or compressing a liquid above its critical pressure but in this molar volume remains same irrespective of original form(liquid or gas).

1. Supercritical Fluid Extraction (SFE) is a eco-friendly alternative of extraction replacing organic solvents.
2. In SFE use of **Supercritical fluids (SCF's) like CO₂** is used as solvent .
3. SCF are increasingly replacing organic solvents because of regulatory and environmental pressures on hydrocarbon and ozone depleting emissions.
4. SFE can be used to extract active ingredients or analytes from various plants and microbial samples and useful in extraction of unknown natural products.
5. Manipulating temperature and pressure of the fluid can solubilize the material of interest and selectively extract it.

Why Carbon dioxide??

The density of the supercritical CO₂ at around 200 bar pressure is close to that of hexane, and the solvation characteristics are also similar to hexane; thus, it acts as a non-polar solvent. Around the supercritical region, CO₂ can dissolve triglycerides at concentrations up to 1% mass. The major advantage is that a small reduction in temperature, or a slightly larger reduction in pressure, will result in almost the entire solute precipitating out as the supercritical conditions are changed or made sub critical.

- Non-toxic,
- Non-flammable,
- Odorless,
- Tasteless,
- Inert, and
- Inexpensive

CO₂ has critical temperature of 31°C and critical pressure of 74 bar. Due to its low critical temperature 31°C, carbon dioxide is known to be perfectly adapted in food, aromas, essential oils and nutraceutical industries.

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: –
 1. Liquid like densities (100-1000 times greater than gases)
 2. Diffusivities higher than liquids (10^{-3} and 10^{-4} cm²/s)
 3. Good solvating power
 4. Reduction in surface tension
 5. Low viscosity (10-100 times less than liquid)
 6. Gas like compressibility properties
 7. Therefore they possess high penetrating power

FLOWCHART FOR SFE PROCESS

INTRODUCTION OF FEED TO EXTRACTOR

MIXING OF SOLUTE AND
SUPERCRITICAL FLUID

{ MOBILE PHASE }

EXPOSURE OF MOBILE PHASE TO 50-500atm AND
TEMPERATURE-300°C NEAR CRITICAL POINT

ISOLATION OF DISSOLVED SOLUTES BY
PRECIPITATION

CO₂ IN VAPOUR FORM IS
COMPRESSED INTO A LIQUID
BEFORE BECOMING
SUPERCRITICAL AND THEN
EXTRACTION TAKES PLACE

COMPONENTS OF SFE

1. Reservoir
2. Pump
3. Extraction cell
4. Restrictor
5. Collector
6. Detector

- Fluid reservoir (gas cylinder in case of CO₂)
- Pump – Reciprocating pump – Syringe pump (pulse-free flow at large range of flow rates)
- 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]

- Restrictor – Maintaining pressure change inside the extraction vessel
It is of two types:
 - a. Fixed (linear restrictor, tapered design)
 - b. Variable (variable nozzle, back pressure regulator)
- Collector (trapping system)
- Detectors (flame ionization detector of gas chromatography)

ADVANTAGES

- Elimination of organic solvents i.e. reduces the risk of storage.
- Rapid (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)

LIMITATIONS

1. Prolonged time (penetration of SCF into the interior of a solid is rapid, but solute diffusion from the solid into the SCF).
2. Modeling is inaccurate.
3. Scale is not possible (due to absence of fundamental, molecular-based model of solutes in SCF)
4. Expensive
5. Consistency & reproducibility may vary in continuous production

