



EKC 316  
Separation Processes



Supercritical Fluid Extraction

# Course Learning Objectives

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- ▶ Apply the principles and theory of super critical fluid extraction.
- ▶ Develop design strategies.

## Principles and theory:

- ▶ Supercritical Fluid Extraction: Physical and transport properties of supercritical fluid,
- ▶ Phase diagrams of supercritical fluid and estimation of solid solubility at high pressure,
- ▶ Type of supercritical fluids, industrial applications of SFE: SFE of essential oils.

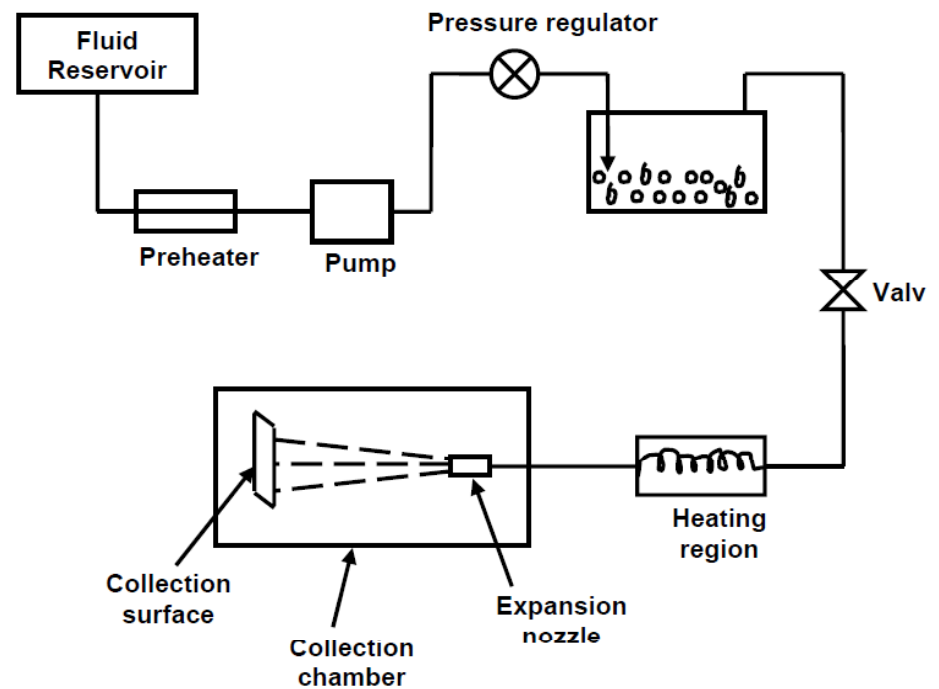
Course Strategies: Explain the supercritical fluid phase diagram and applications of supercritical fluid extraction in industry.

Course Activities: Lectures in class; Discussions; Tutorial; Test

# Case Study: Naphthalene Extraction

- ▶ A typical flow chart of a supercritical fluid extraction process is outlined as below.
- ▶ A supercritical solvent saturated with a solute is allowed to expand rapidly, leading to precipitation of solute. (Rapid expansion of supercritical solutions)

Rapid expansion is achieved by allowing it to pass through a nozzle at supersonic speed.



# Case Study: Naphthalene Extraction

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# Case Study: Naphthalene Extraction

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- ▶ Supercritical fluid (SF) is pumped through a pre heater into the vessel containing solid solute and the resultant solution is sent into a precipitation chamber by expansion through capillary or laser drilled nozzle.
- ▶ At precipitation chamber, pressure is much lower and solute solubility in SF is quite low and solute precipitates out of it.
- ▶ Size distribution and morphology of precipitated material is a function of pre-expansion concentration and its conditions.
- ▶ Pre-expansion concentration in term depends on SF, nature of solute, addition of co-solvent, operating pressure and temperature.
- ▶ Particle size is smaller and distribution is narrower if pre-expansion concentration is higher.

# Case Study: Naphthalene Extraction

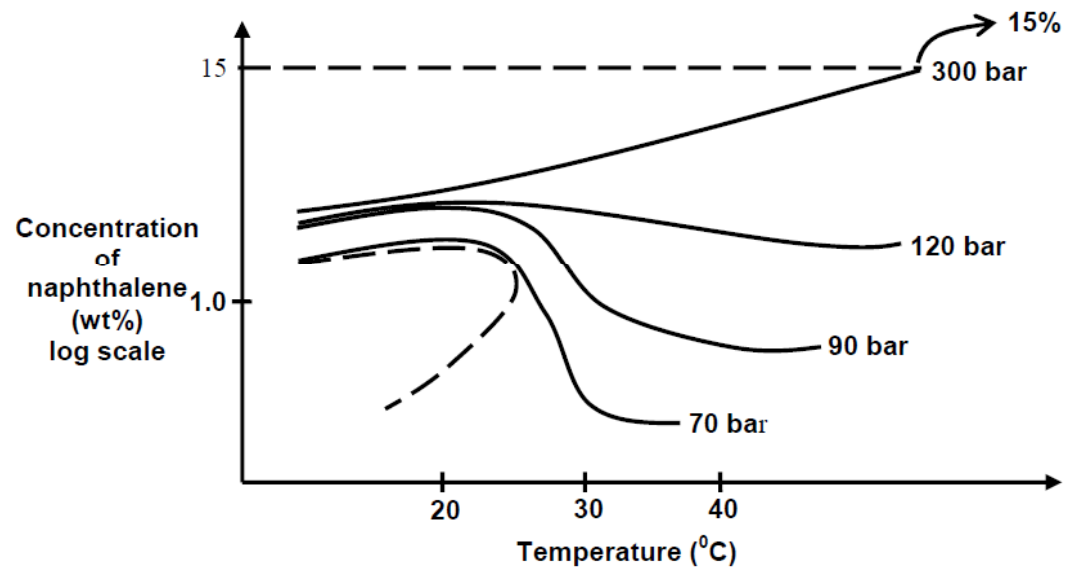
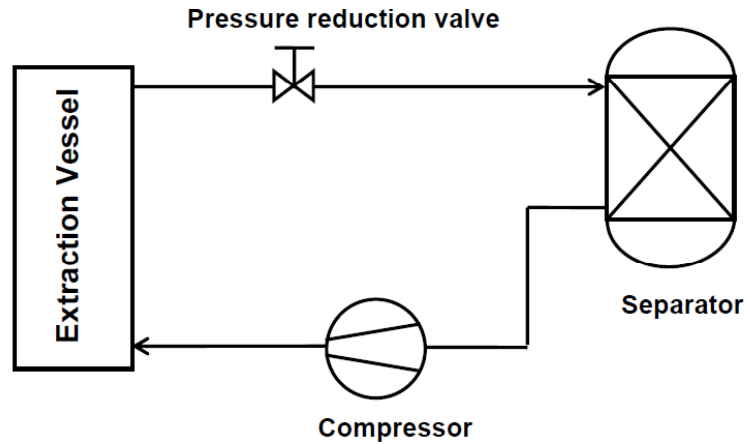


Fig. 10.5: Solubility diagram of naphthalene under supercritical condition

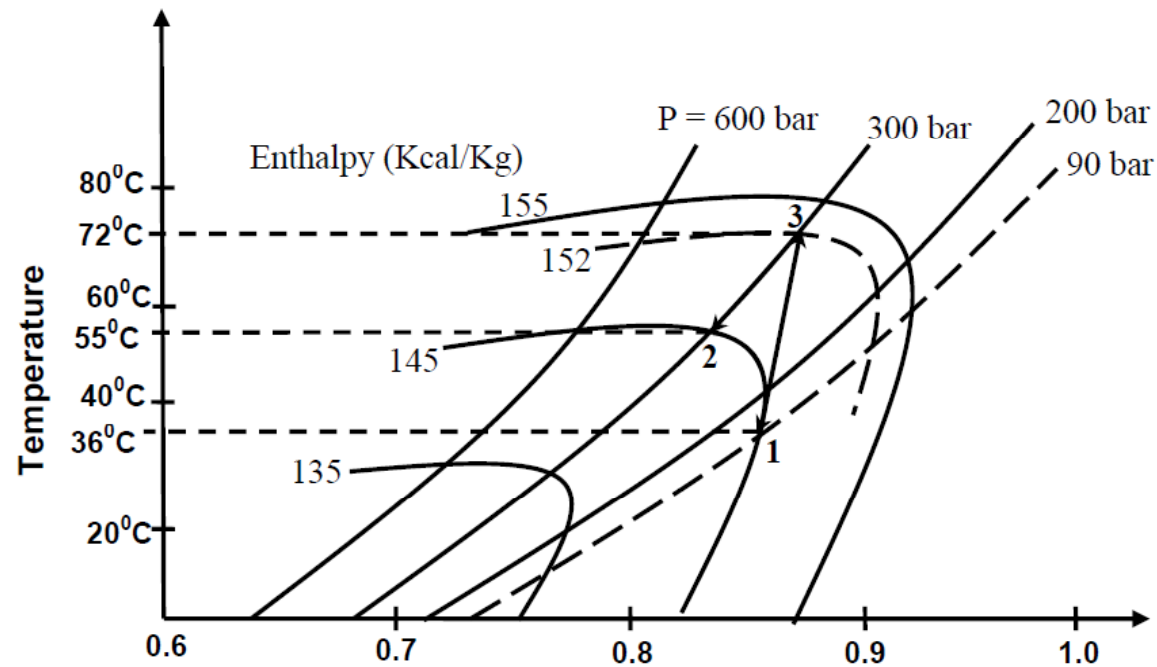
# Case Study: Naphthalene Extraction

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- ▶ A naphthalene-chalk dust mixture is fed to the extraction vessel.
- ▶ Assume, extraction condition is 300 bar and 550C. At this condition CO<sub>2</sub> SCF contains naphthalene dissolved at 15 wt%.
- ▶ Then it is expanded to 90 bar through pressure reduction valve (assuming isenthalpic expansion). After expansion its temperature is 360C at 90 bar.
- ▶ Solubility of naphthalene is 2.5%. So, it falls out of solution.
- ▶ Precipitated naphthalene is collected and CO<sub>2</sub> is compressed to 550C and 300 bar and recycled to extractor.

# Case Study: Naphthalene Extraction

## ► Mollier Diagram





# Case Study: Naphthalene Extraction

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- (1) 55°C, 300 bar
- (2) 36°C, 90 bar (after expansion)
- (3) 72°C, 300 bar (after compression)

Energy required to compress from 90 to 300 bar is, (152-145)=7 Kcal/kg (12.6 BTU/lb)

Then it is cooled to 55°C

- (4) 72°C, 300 bar to 55°C, 300 bar

Decrease in solubility is 15 to 2.5 wt%

*1 lb CO<sub>2</sub> contains 0.15 lb Naphthalene*

After extraction

*1 lb CO<sub>2</sub> contains 0.025 lb Naphthalene*

Extracted naphthalene 0.150-0.025 = 0.125 lb

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*0.125 lb naphthalene extracted for 1 lb CO<sub>2</sub>*

*So, 0.125 lb naphthalene extracted for  $1000/125 = 8$  lb CO<sub>2</sub>*

*So, we require 8 lb CO<sub>2</sub> to recycle for extraction of 1 lb naphthalene.*

*Energy required for compression from 90 to 300 bar is  $= 12.6 \times 8$  BTU*

*= 100.8 BTU for extraction of 1 lb naphthalene*

# Type of Supercritical Fluid

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- ▶ Principle of selection of the SFE solvent:
  - ✓ Good solvating property
  - ✓ Inert to the product
  - ✓ Easy separation from the product
  - ✓ Cheap
  - ✓ Low PC because of economic reasons

Solvent	Critical Temperature, K	Critical Pressure, MPa	Critical Density, kg/m <sup>3</sup>
Methane	192	4.60	162
Ethylene	283	5.03	218
Carbon dioxide	304	7.38	468
Ethane	305	4.88	203
Propylene	365	4.62	233
Propane	370	4.24	217
Ammonia	406	11.3	235
Water	647	22.0	322

# Type of Supercritical Fluid

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Fluid	$T_c$ (K)	$P_c$ (bar)
Carbon dioxide	>304.1	> 73.8
Trifluoromethane (Fluoroform)	>299.3	>48.6
Chlorotrifluoromethane	>302.0	>38.7
Trichlorofluoromethane	>471.2	>44.1
Cyclohexane	>553.5	>40.7
n-Pentane	>469.7	>33.7
Toluene	>591.8	>41.0

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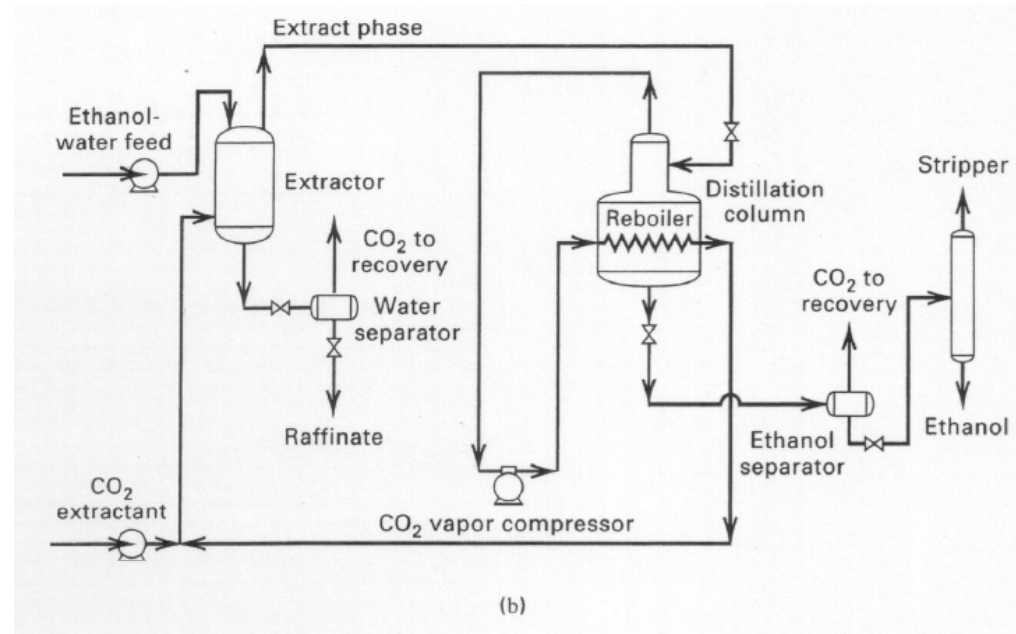
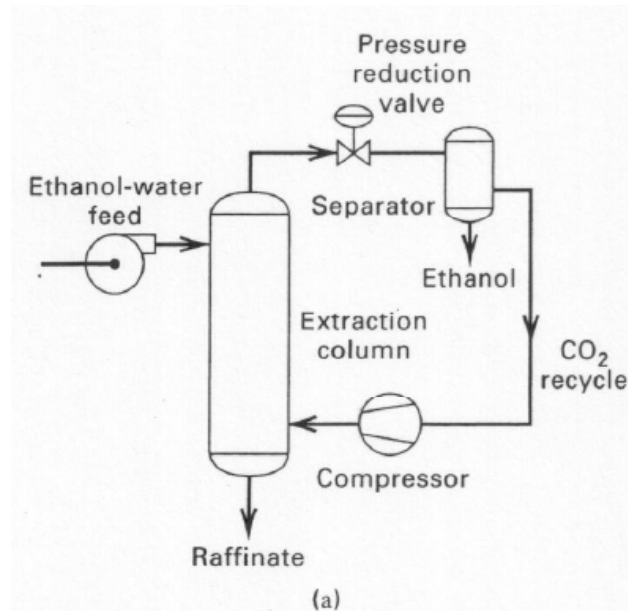
# Type of Supercritical Fluid

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- ▶ Carbon dioxide is the most commonly used SCF, due primarily to its low critical parameters (31.1°C, 73.8 bar), low cost and non-toxicity.
- ▶ Following properties of CO<sub>2</sub> make it qualified to be an SCF.
  - ▶ Low critical pressure (74 atm) and low critical temperature (320C)
  - ▶ Relatively non-toxic
  - ▶ Non-flammable
  - ▶ Available as high purity
  - ▶ Low cost
  - ▶ Easily removable from extract
  - ▶ It has polarity like liquid pentane at supercritical conditions and thus, best suited for liophilic compounds.
- ▶ Drawback: It cannot extract polar solutes.

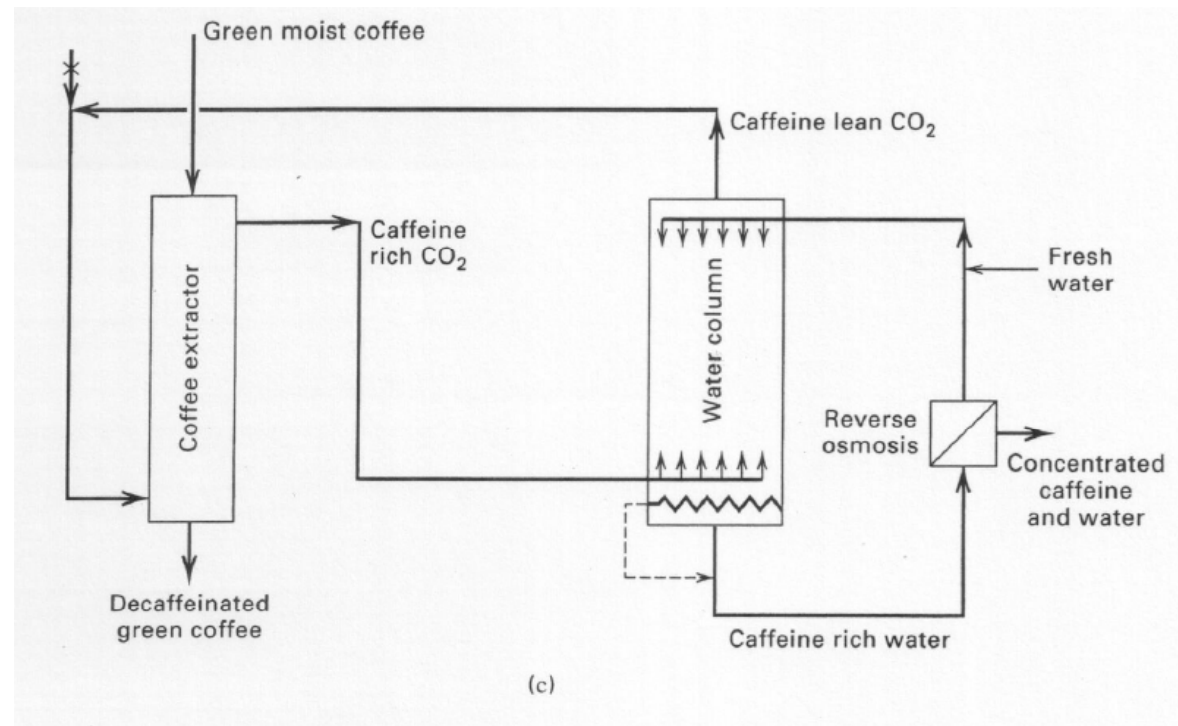
# Type of Supercritical Fluid

Methods to recover CO<sub>2</sub> in supercritical extraction processes (a) P reduction, (b) high P distillation



# Type of Supercritical Fluid

Methods to recover CO<sub>2</sub> in supercritical extraction processes (c) high pressure absorption with water



# Type of Supercritical Fluid

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## Nitrous Oxide:

- ▶ It is good for removal or solubilize polar solutes, as it has a permanent dipole moment. N<sub>2</sub>O is better than CO<sub>2</sub> for extraction of polychlorinated dibenzodioxines from fly ash.
- ▶ Disadvantage: It is highly explosive

## H<sub>2</sub>O:

- ▶ Disadvantage:
  - (i) High P<sub>c</sub> and T<sub>c</sub> (T<sub>c</sub> > 374<sup>0</sup>C, P<sub>c</sub> > 221 bar)
  - (ii) At these conditions, H<sub>2</sub>O is corrosive.

## Co-solvents/ modifiers

- ▶ Co-solvents are added to modify the polarity of the SCF, so that the power of SCF to solvate polar solutes increases.
- ▶ Ex: CO<sub>2</sub> should be mixed with 1-10% of methanol to solubilize more polar solutes.



# Industrial Applications

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- ▶ In 1980s, the application of SCF in the extraction of byproducts from pharmaceuticals, purification of medical polymers, and separation of complex synthesis mixtures were explored.
- ▶ The formation of ultra fine particles using SFC is of current interest. foremost among them. The recrystallization of materials is under intense study at many European and US pharma companies.
- ▶ The general impression that supercritical fluids involve high processing costs is not true as supercritical fluid decaffeinated coffee available at competitive market prices (with organic solvent decaffeinated coffee) certainly would contradict this impression. Besides food and flavoring industry, SFC is applied in petrochemical and pharmaceutical industry.

## Food and Beverage Industry

- ▶ SFE is applied as the residual solvent could be easily removed. The widest application is the decaffeination of tea and coffee. Other application is the extraction of essential oils and aroma materials from spices. Brewery industry uses SFE for the extraction of hop. The method is used in extracting some edible oils and producing cholesterol-free egg powder.

# Industrial Applications

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## Petrochemical Industry.

- ▶ The distillation residue of the crude oil with SFE has been developed into large-scale procedure (ROSE Residum Oil Supercritical Extraction). The method is applied in the regeneration of used oils and lubricants.

## Pharmaceutical industry

- ▶ Producing of active ingredients from herbal plants to avoid thermo or chemical degradation and eliminate residual solvents from the products.

# Industrial Applications

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- ▶ Advantages and disadvantages of SCFs compared to conventional liquid solvents for separations:

## Advantages

- ▶ Dissolving ability of the SCF is controlled by P and/or T
- ▶ SCF can be easily recovered from the extract due to its volatility
- ▶ Non-toxic solvents leave no harmful residue
- ▶ High boiling components are extracted at relatively low T
- ▶ Thermally labile compounds can be extracted without degradation

## Disadvantages

- ▶ Elevated pressure required
- ▶ Compression of solvent requires elaborate recycling measures to reduce energy costs
- ▶ High capital investment for equipment

# Industrial Applications

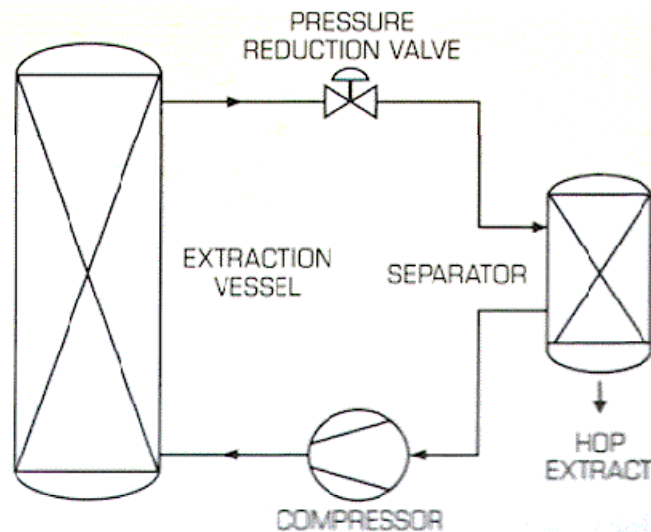
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## Food and Beverage Industry

- ▶ Since 1960s, many researchers primarily in Europe and then in U.S., studied SCF for developing extraction processes. European researchers focused on botanical substrates such as spices, herbs, coffee, tea, and etc. using supercritical CO<sub>2</sub>.
- ▶ By 1980s, several industrial scale of SCF extraction processes were operated in Germany, UK and US to decaffeinate coffee and tea, and extracting flavors and essential oils from hops, spices, and herbs. A coffee decaffeination plant in Bremen processes more than 60,000,000 kg/year.
- ▶ The major motivation of the mentioned development was the elimination of residual solvents in the products, especially methylene chloride used to decaffeinate coffee.
- ▶ Solvent residues in food and pharmaceutical products were then becoming the focus of regulatory attention in the 1970s. Besides the elimination of solvent residues, enhanced flavor and aroma characteristics that cannot be obtained by the traditional organic solvent extraction processes.

# Industrial Applications

- ▶ A schematic diagram of a generic supercritical fluid extraction process is shown in below figure.



- ▶ To extract hops, flavors used in the brewing of beer, compressed and pelleted hop flowers (lupelones) are charged to the extraction vessel, which is subsequently secured for pressure operation.
- ▶  $\text{CO}_2$  from a reservoir is pumped into the vessel, and after T and P are adjusted to supercritical conditions of about  $60^\circ\text{C}$  and 300 atm, continuous flow of carbon dioxide is initiated.
- ▶ Hop flavors and lipids are extracted as the carbon dioxide flows through the charge of hops, and the solution (of carbon dioxide and flavors) is decreased in P to about 60 atm across the P reduction valve shown.
- ▶ Because the loss of dissolving ability, the flavors and lipids precipitate from the gas phase and collect in the separator; the  $\text{CO}_2$  is recompressed and recycled to the extractor, and the process  $\text{CO}_2$  flow continues until all the flavors are extracted.

# Industrial Applications

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- ▶ The extraction vessel is then vented, the spent hops are removed, fresh pellets are charged to the vessel, and the process started again.
- ▶ Besides the enhanced flavor and higher yield, SCF offer technical and economic advantages. Organic solvents such as methylene chloride or hexane were used to extract hops. To obtain concentrated flavors, organic solvents were distilled, off but some top note aromas are lost. Supercritical CO<sub>2</sub> produces a superior product without distillation step.

# Industrial Applications

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## Propane Deasphalting

- ▶ A de-asphalter is a unit in a crude oil refinery or bitumen upgrader that separates asphalt from crude oil or bitumen. Petroleum-refinery solvent process using propane to remove and precipitate asphalt from petroleum stocks, such as for lubricating oils.
- ▶ Asphalt also known as bitumen, is the sticky, black and highly viscous liquid or semi-solid present in most crude petroleums and in some natural deposits. The primary use of asphalt is in road construction, where it is used as the glue or binder mixed with aggregate particles to create asphalt concrete. Its other main uses are for bituminous waterproofing products, including production of roofing felt and for sealing flat roofs
- ▶ Propane Deasphalting. This entire process is not, strictly speaking, a SFE process because the initial extraction step is carried out at near-critical condition. But propane deasphalting does make use of the substantial change in the solvent power of a liquid in the vicinity of its critical point.

# Industrial Applications

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- ▶ Propane deasphalting is also an example of a process using near-critical property changes to improve energy and phase separation efficiency. Since the solvent characteristics of propane can be changed dramatically in various regimes of P-T phase space, this single solvent can be used selectively to separate a lube oil feedstock into paraffin wax, asphalt, heavy ends, naphthenes, color bodies, and the desired product, a purified light oil.
- ▶ Compressed liquid propane is used to dissolve all the constituents of a lube oil feedstock except for the asphalt. Because the propane-lube oil mixture possesses a low viscosity, the asphalt fraction is readily separated from the mixture, hence it is easily recovered.



# Industrial Applications

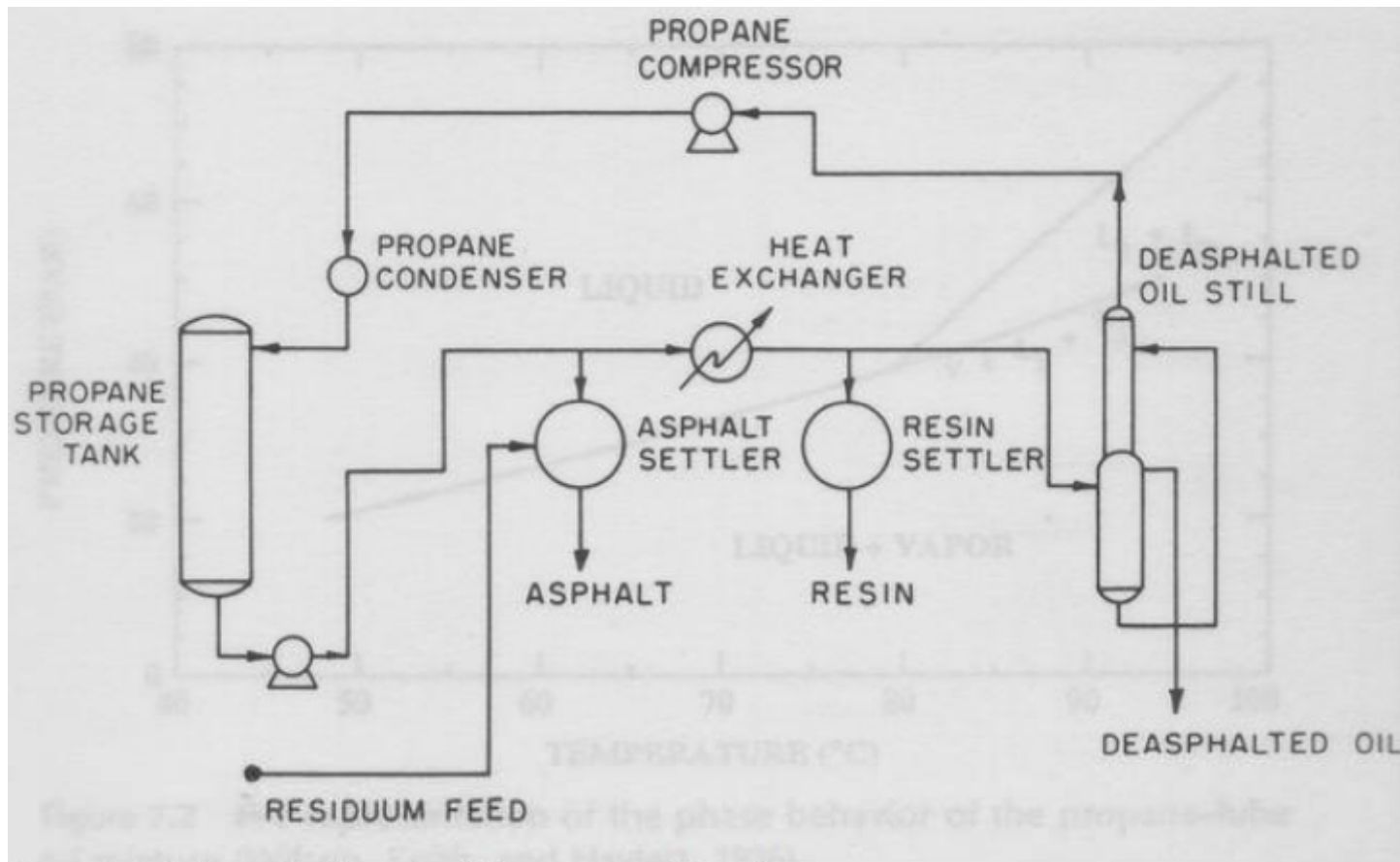


Figure 1.2 Schematic diagram of the propane-lube oil process

# Industrial Applications

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## Solexol Process

- ▶ This process is similar to propane deasphalting. It was developed a few years later for the purification and separation of vegetable and fish oils.
- ▶ It too uses propane as a selective solvent and its purpose is to concentrate the polyunsaturated triglycerides (the so-called drying oils) in vegetable oils and to extract the vitamin A values from fish oil.
- ▶ Fish oil triglycerides are complex compounds consisting of various saturated and unsaturated fatty acids on a glycerol backbone. Staging the temperature within an extraction column and supplying a recycle stream allows the separation of the fish oil triglycerides by virtue of the variable dissolving power of the near-critical propane and by virtue of the lower propane solubility of unsaturated triglycerides relative to the propane solubility of saturated triglycerides. This process is yet another example of the use of the unique solvent characteristics of near-critical and supercritical fluids

# Industrial Applications

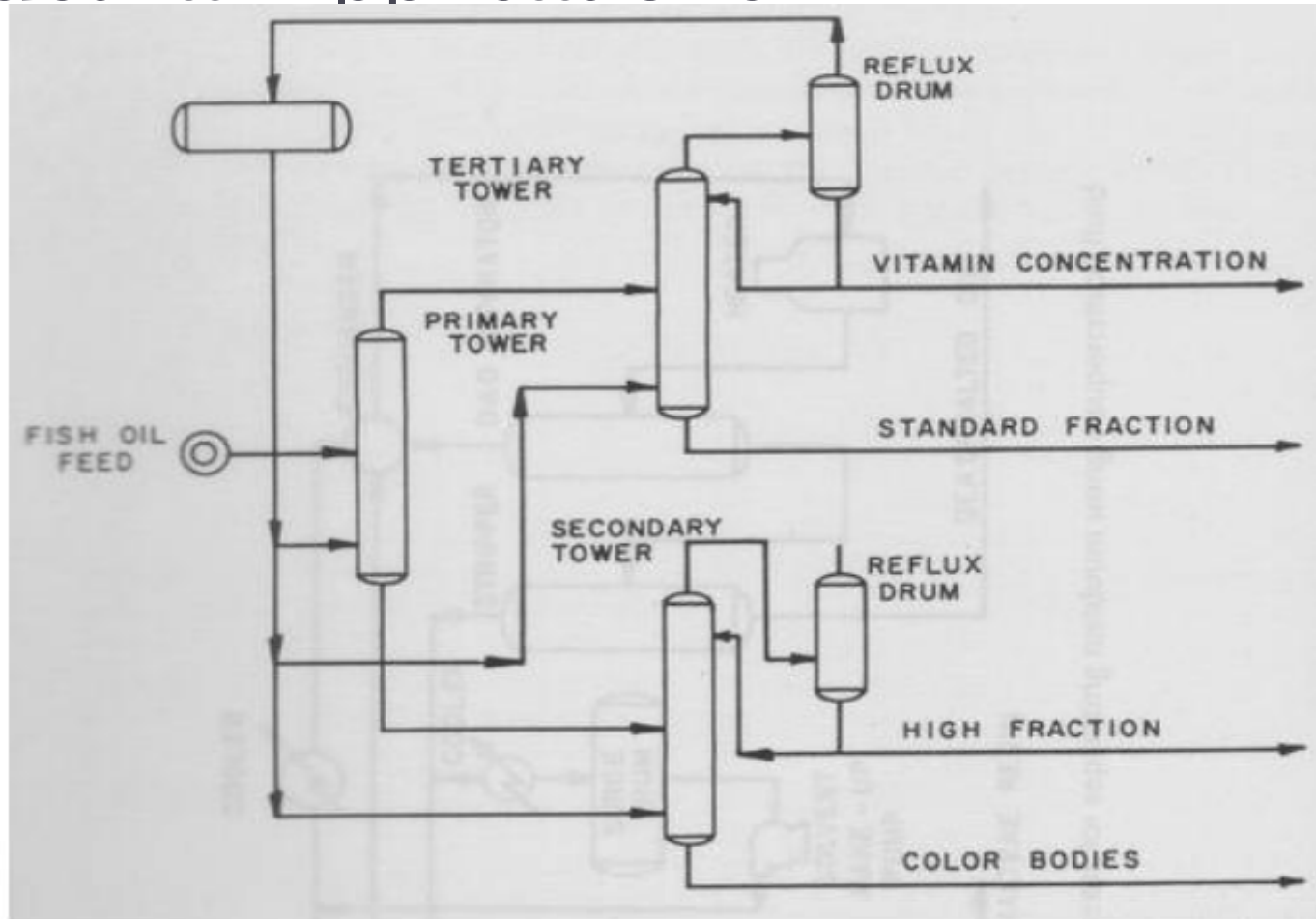


Figure 1.3 Schematic diagram of the Solexol process for separating fish oils

# Industrial Applications

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## Rose process

- ▶ This process is not entirely a supercritical process. Its primary extraction step is carried at compressed liquid conditions, not at supercritical condition. Nevertheless, the process has advantage of the variable solvent power of a near-critical liquid at various stages of the extraction/separation process.
- ▶ In order to separate residuum, the raw material is first mixed with compressed liquid butane or pentane and the undesired asphaltene fraction is precipitated.
- ▶ Butane is used because it has a higher solvent power for the heavy hydrocarbons. For the higher molecular weight residual, butane is an effective solvent in dissolving the desired high molecular weight components and precipitating the asphaltene.
- ▶ It is too effective in the lube oil refining process because the molecular weights of all the components, desired and undesired, are much lower, so that the components cannot be differentiated finely enough by butane.
- ▶ If an intermediate resin fraction is desired, another separator and stripper system would be installed right after the asphaltene separator in Figure 1.3.



# Industrial Applications

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## Activated carbon regeneration

- ▶ The potential advantages of SCF regeneration are reduced energy requirements and lower carbon loss compared with the thermal regeneration process currently in operation.
- ▶ It is suggested that, because many organic compounds can be dissolved by supercritical fluid solvents, the organic materials adsorbed onto activated carbon during the cleanup of a wastewater stream can be desorbed by supercritical carbon dioxide at condition less severe than the 1,000°C temperature level of an industrial regeneration furnace.
- ▶ For small-scale processes, which use activated carbon at a rate of a few thousand pounds per day, the desorbers can also serve as fixed-bed adsorption vessels. Hence, in situ carbon regeneration can be carried out when the carbon bed is spent, that is, fully loaded with adsorbed organic stem.

# Industrial Applications

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- ▶ Processes that use much more activated carbon and system that require many large adsorption vessels, such as those at municipal waste treatment facilities, the spent carbon can be removed from the adsorbers and transferred to the desorbers for regeneration. The transfer of the spent activated carbon from adsorbers to the thermal regeneration furnace is currently standard industrial practice. The spent carbon is transferred by water slurry pumps, sequenced valves, and motor-activated closures.
- ▶ Although the cost of CO<sub>2</sub> is rather low, its consumption should be considered in evaluating the viability of a process, especially if the cost of the material being extracted is low. The CO<sub>2</sub> that is lost represents an economic penalty for this process, but part or all of it can be recovered with the installation of an additional CO<sub>2</sub> surge tank system.

# Industrial Applications

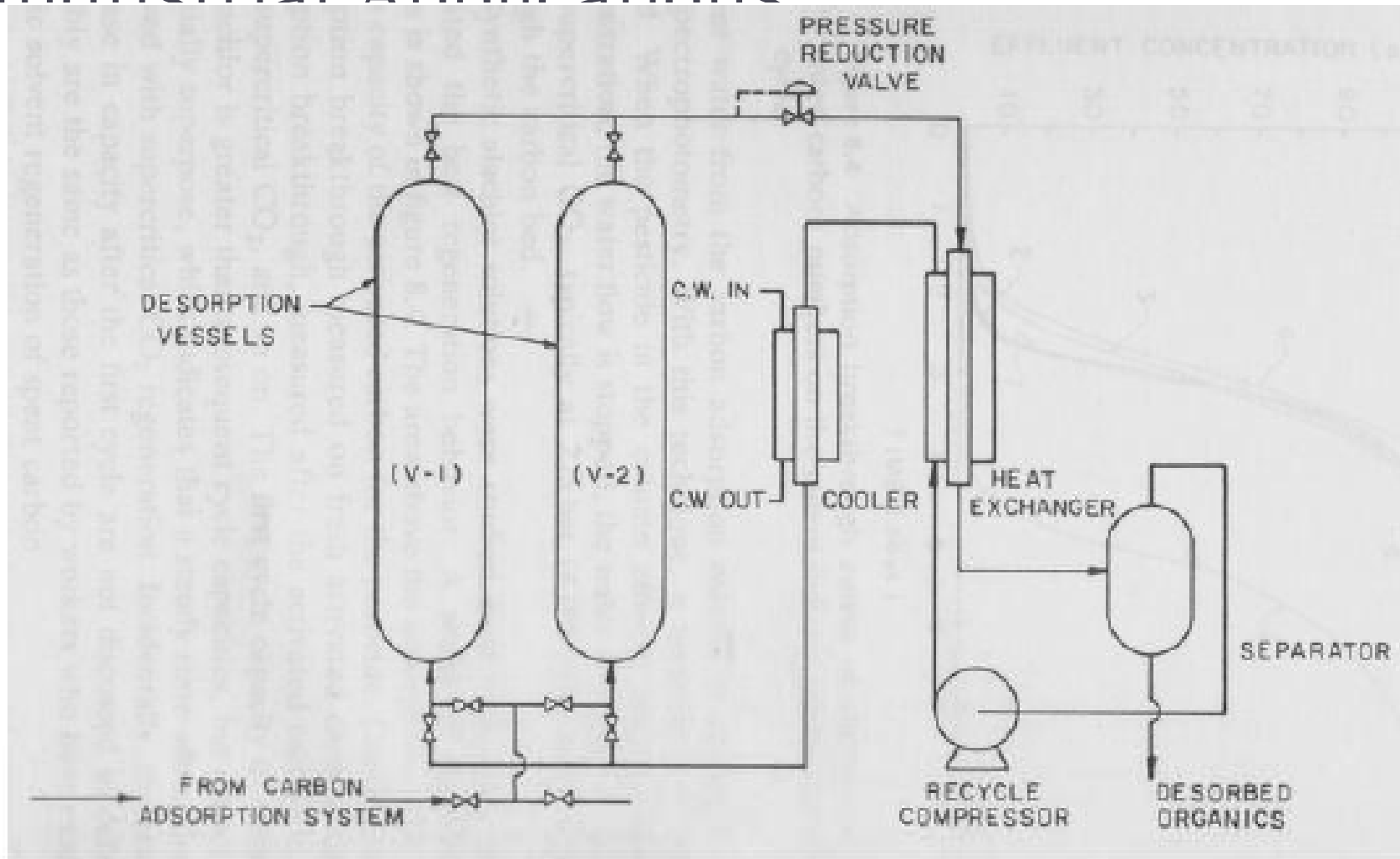


Figure 1.5 Schematic diagram of regeneration activated carbon with SCF solvent



# Industrial Applications

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## Separation of organic-water solutions

### Ethanol-Water.

- ▶ The energy shortages of the 1970s were an impetus for evaluating the use of SCFs for separating organic compounds from water. A process that received substantial governmental and industrial funding is supercritical CO<sub>2</sub> separation of ethanol from water.
- ▶ The primary goal of the work is to break the ethanol-water azeotrope of 95.5wt% ethanol. But if the azeotrope cannot be broken, a parallel goal is the demonstration of a less energy-intensive process using SFE in tandem with normal azeotropic distillation.
- ▶ The goal was that it might be possible to combine supercritical fluid extraction and distillation to reduce processing costs in much the same manner that combines reverse osmosis and evaporation, for example. A simplified schematic diagram of the continuous and countercurrent process of ethanol-water separation is shown in Figure 1.6

# Industrial Applications

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- ▶ The ethanol-water feed stream and the supercritical CO<sub>2</sub> recycle stream can be pumped continuously to a column, the produce stream can be drawn off from a separator, and the raffinate (depleted in ethanol) can be discharged from the column through a valve shown in the figure.
- ▶ The internals of the extractor may be sieve trays or various packing arrangements. The feed flows down the column from plate to plate through down-comers against the upward flow of CO<sub>2</sub>.
- ▶ There is an upper limit on the operating pressure if the process is to be operated as described since at high pressures the density of the CO<sub>2</sub>-rich phase can approach or exceed that of the water rich phase. This results in no phase separation and an inability to operate the column, although if CO<sub>2</sub> were the heavy phase, it could be introduced at the top of the column.
- ▶ During the plate-to-plate contact of the supercritical CO<sub>2</sub>-rich phase with the ethanol-water solution, the ethanol is preferentially extracted.

# Industrial Applications

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- ▶ The extract consists of mostly CO<sub>2</sub>, some ethanol, and a little water; it leaves at the top of the column.
- ▶ Figure 1.6 Shows the CO<sub>2</sub>-rich solution is expanded across a pressure reduction valve; this reduces the solvent power of the CO<sub>2</sub> and causes the ethanol to precipitate in the separator.
- ▶ The CO<sub>2</sub> is then recompressed and recycled to the extractor.

# Industrial Applications

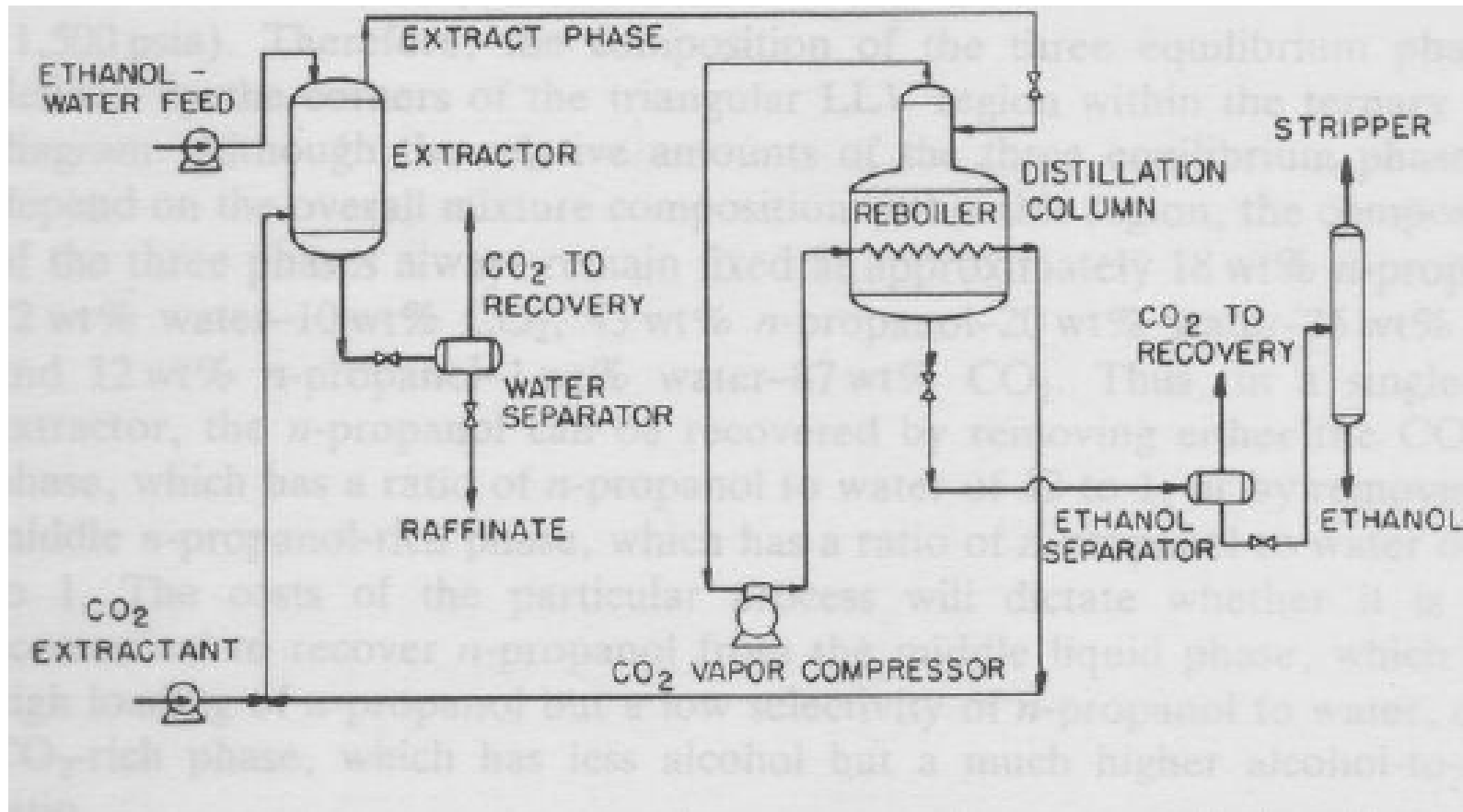


Figure 1.6 Schematic diagram of a process using SC CO<sub>2</sub> for extracting ethanol from water combined with distillation for CO<sub>2</sub> cleanup.

# Industrial Applications

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## Acetic acid-Water

- ▶ The potential for achieving a lower-energy process also motivated a substantial effort to examine supercritical fluid extraction for separating acetic acid from water.
- ▶ The work falls into two subcategories: (a) separation of fermentation derived acetic acid and (b) separation of acetic acid-water solutions that are formed during production of cellulose acetate.
- ▶ The recovery of acetic acid from fermentation broths is similar to the recovery of ethanol from fermentation broths. In the cellulose acetate production process, cellulose reacts with glacial acetic acid and acetic anhydride to form cellulose triacetate.
- ▶ Water is then added to the cellulose triacetate-glacial acetic acid solution, which results in the removal of a fraction of the acetate moieties from the cellulose backbone. Water also causes the precipitation of cellulose acetate and, of course, dilutes the acetic acid.

# Industrial Applications

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- ▶ The ethanol-water feed stream and the supercritical CO<sub>2</sub> recycle stream can be pumped continuously to a column, the produce stream can be drawn off from a separator, and the raffinate (depleted in ethanol) can be discharged from the column through a valve shown in the figure.
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# Industrial Applications

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# Industrial Applications

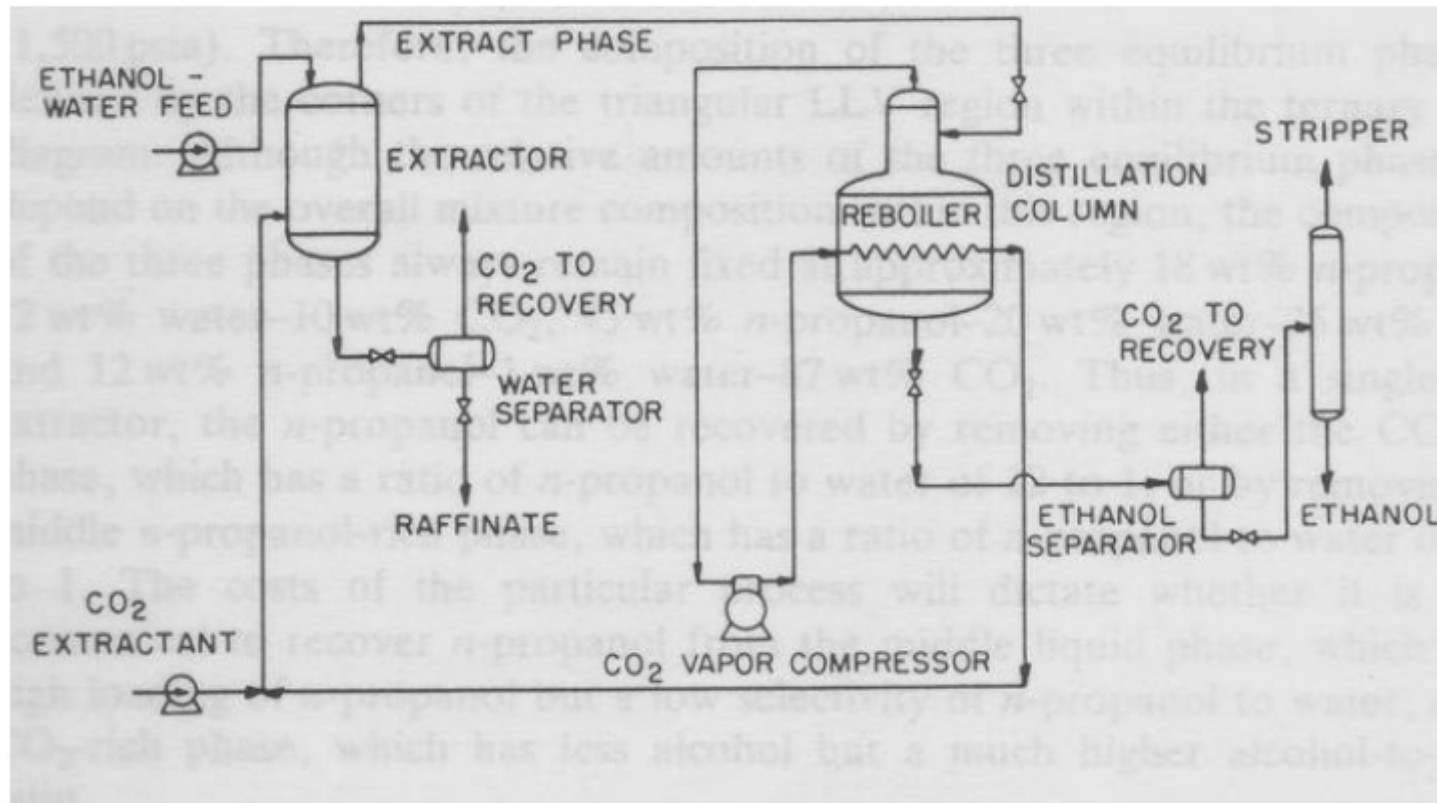


Figure 1.6 Schematic diagram of a process using SC CO<sub>2</sub> for extracting ethanol from water combined with distillation for CO<sub>2</sub> cleanup.



# Industrial Applications

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## Polymers and monomer processing

- ▶ High-pressure polyethylene polymerization. Either a well-stirred autoclave or a tubular reactor can be used to carry out the polymerization.
- ▶ Ethylene is compressed to 2,700 bar and a free-radical initiator, e.g., trace amounts of oxygen or a peroxide, is injected into the feed stream to promote the free-radical polymerization.
- ▶ The polyethylene polymer that is formed remains dissolved in the supercritical ethylene phase at the operating temperature which ranges from  $\sim 140$  to  $250^{\circ}\text{C}$ . The heat of reaction is removed by through-wall heat transfer when the tubular reactor is used and by regulating the rate of addition of initiator when the autoclave reactor is used.
- ▶ Downstream of reactor section the polyethylene-ethylene solution is expanded to a lower pressure, typically to about 350bar. Because the solvent power of supercritical ethylene is lowered during the pressure reduction, the polyethylene formed in the reactor nucleates, precipitates from solution, and is collected in the separator.

# Industrial Applications

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- ▶ The low-pressure ethylene from the polyethylene separator is recompressed and recycled to the reactor. Interestingly, high-pressure ethylene is both the reactant and the solvent for the product in this process. Many polymer fractionation process fractionation patents of the 1940s were derived directly from this process.

# Industrial Applications

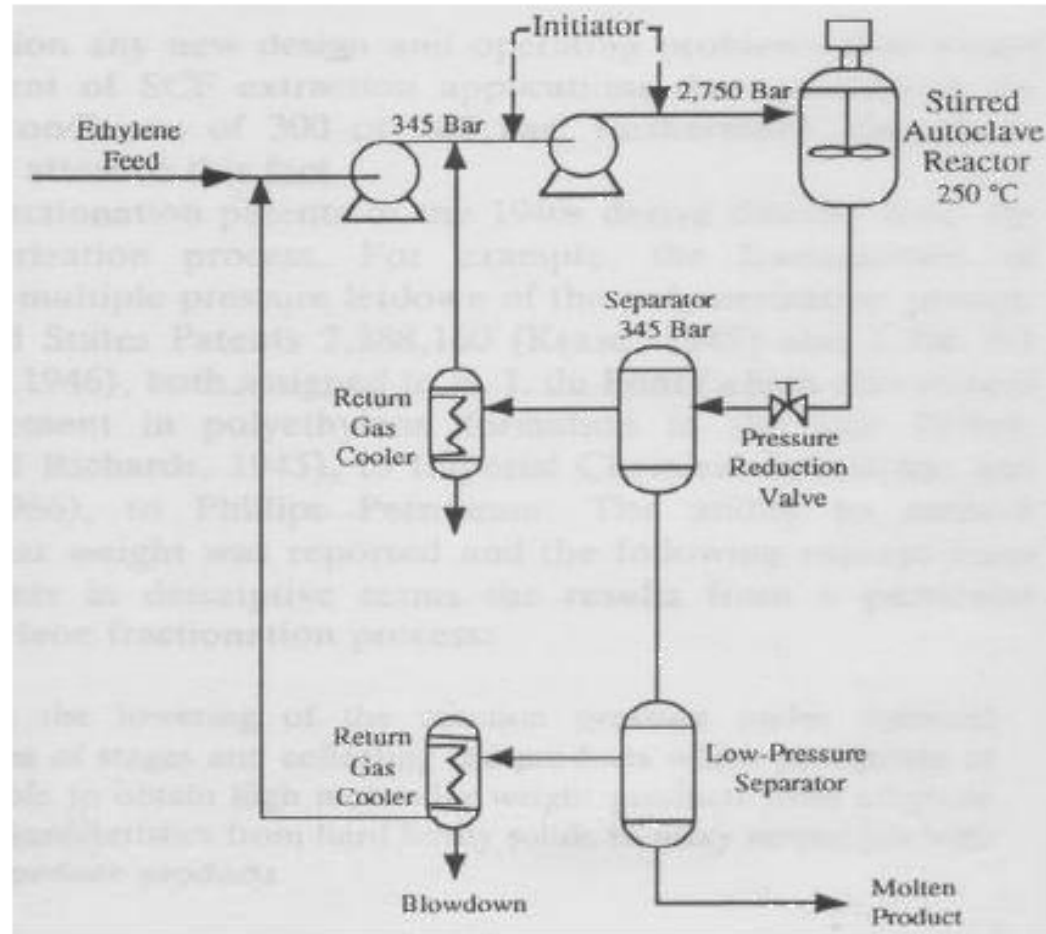


Figure 1.7 Schematic diagram of high-pressure polyethylene process (Stirred Autoclave)

# Industrial Applications

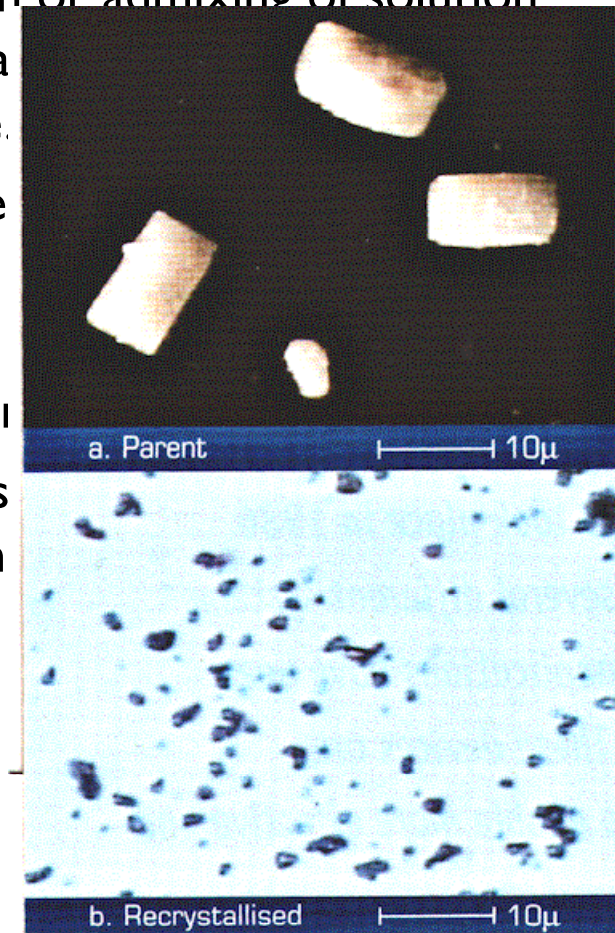
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## Pharmaceutical industry: Recrystallization

- ▶ Some pharma compounds are difficult to be micronized by grinding or jet milling. Materials with low melting point ( $< 60^{\circ}\text{C}$ ) or waxy material, cannot be ground or milled to fine size ( $< 1$  or  $2$  microns) as the material will smear or form amorphous and size unstable particles.
- ▶ 2 supercritical fluid processes have been developed for reducing such their particle size.
  - (a) Supercritical fluid is employed as a solvent to dissolve a pharma compound, then, by pressure decrease, cause precipitation;
  - (b) Gas acts as an anti solvent, causing recrystallization from a liquid solution because of a solubility decrease when the gas and liquid solvent contract.
- ▶ With a gas anti solvent, the rapidity and intimacy of mixing far exceeds what liquid anti solvents can achieve, and thus GAS recrystallization can achieve narrow particle size distribution in the 100s of nanometer range.

# Industrial Applications

- ▶ Both processes require rapid pressure reduction or admixing of solution and gas in millisecond timescale, which creates a resulting ultrafine particles of narrow size range.
- ▶ Photomicrographs shows  $\beta$ zlig-pregnanolone, re supercritical  $\text{CO}_2$ .
- ▶ The parent material ranges in size from about 2 supercritical fluid processed (by RESS) material
- ▶ Many European and US pharma companies are s nano-sized pharma compounds in the developm formulations using supercritical fluids.



# Industrial Applications

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## Pharmaceutical industry: Purification of Surfactants

- ▶ Liquid and solid surfactants are used in the formulation of many pharma products, including food products, cleaning solutions and floor wax.
- ▶ Most surfactants have color, odor, byproducts which are incompatible for pharma applications. Supercritical fluid extraction can be used to purify several types of surfactants of importance for pharma formulations.
- ▶ Below figure shows an example of surfactant with different color species, the color of the parent materials is a result of polymerizing reactions that occur during synthesis.
- ▶ As the vapor pressure of the surfactant is very low, they cannot be distilled to improve the color. Supercritical CO<sub>2</sub> separates materials on the basis of solubility, not vapor pressure, and thus it can purify heat sensitive materials that cannot be processed by distillation or very low vapor pressure.



# Industrial Applications

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## Pharmaceutical industry: Extraction of Byproducts from polymer

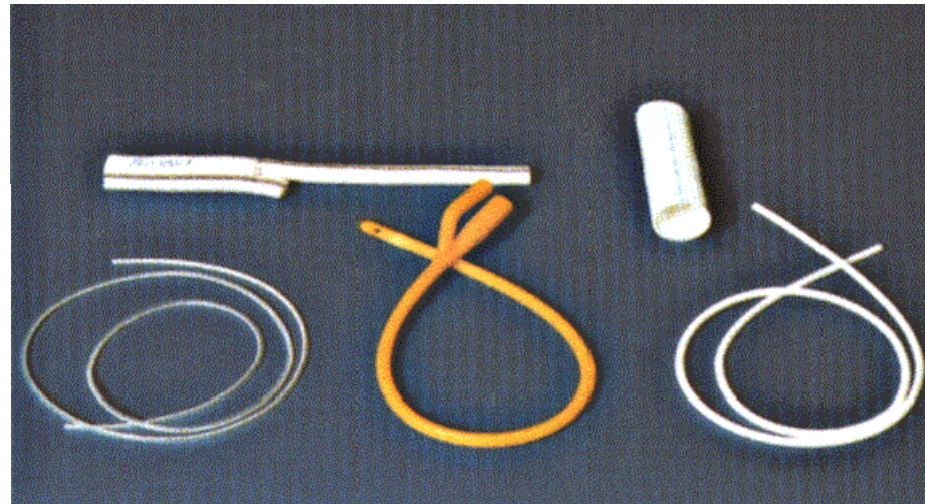
- ▶ There has been increased study of potentially migratable species from polymeric devices that implanted in the body such as urinary catheters, brain shunts and aorta grafts..
- ▶ The silicone or polyester based polymers normally contain several percent residual byproducts which can be removed before it is manufactured into the mentioned device.
- ▶ Silicone raw materials, however, react further during the manufacture of the device, producing the byproducts directly in the finished part.
- ▶ Organic solvents such as hexane and methylene chloride can dissolve and extract objectionable components, but these solvents are difficult to be removed without degrading or altering the characteristics of the polymer or device.



# Industrial Applications

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- ▶ Supercritical fluids especially  $\text{CO}_2$ , wets polymers sufficiently to allow the interior volumes to be reached in order to dissolve and carry away the undesired materials and, when the pressure on the part is reduced, all the carbon dioxide is removed.
- ▶ Below figure shows selected medical components that are currently being processed with supercritical fluids to remove undesired species.





# Industrial Applications

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- ▶ Organic solvents are usually explosive so a SFE unit working with them should be explosion proof and this fact makes the investment more expensive. The organic solvents are mainly used in petrolchemistry.
- ▶ CFC-s are very good solvents in SFE due to their high density, but the industrial use of chloro-fluoro hydrocarbons are restricted because of their effect on the ozonosphere.
- ▶ CO<sub>2</sub> is the most widely used fluid in SFE.
- ▶ Beside CO<sub>2</sub>, water is the other increasingly applied solvent. One of the unique properties of water is that, above its critical point (374°C, 218 atm), it becomes an excellent solvent for organic compounds and a very poor solvent for inorganic salts. This property gives the chance for using the same solvent to extract the inorganic and the organic component respectively.

# Industrial Applications

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## Other plant extractions

- ▶ Denicotin tobacco.
- ▶ Removal of residual solvents from wastes recovery of contaminated soil.
- ▶ Removal of fat from foods
- ▶ Extraction of vitamin E from natural resources
- ▶ Removal of alcohol from wine and beer
- ▶ Extraction of pesticides
- ▶ Extraction of polyaromatic hydrocarbon, polychloro benzene

# References

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- ▶ Hannay, J.B. and Hogarth, J., *On the Solubility of Solids in Gases*, Proc. Roy. Soc., (London). 29:324. 1879
- ▶ Buchner, E.G., *Die beschränkte Mischbarkeit von Flüssigkeiten das System Diphenylamin und Kohlensäure*, Z. Phys. Chem., 56:257. 1906
- ▶ Krukonis, V., Brunner, G., and Perrut, M., *Industrial Operations with Supercritical Fluids: Current Processes and Perspectives on the Future*, Proceedings 3rd International Symposium on Supercritical Fluids, Tome I. p I. Strasbourg, 17th October 1994
- ▶ Andrews, T., The Bakerian Lecture - *On the Gaseous State of Matter*, Proc. Roy. Soc., (London). 24:455. 1875