Mass Transfer Operation II

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Lecture 17 - Supercritical fluid extraction, equipment for leaching

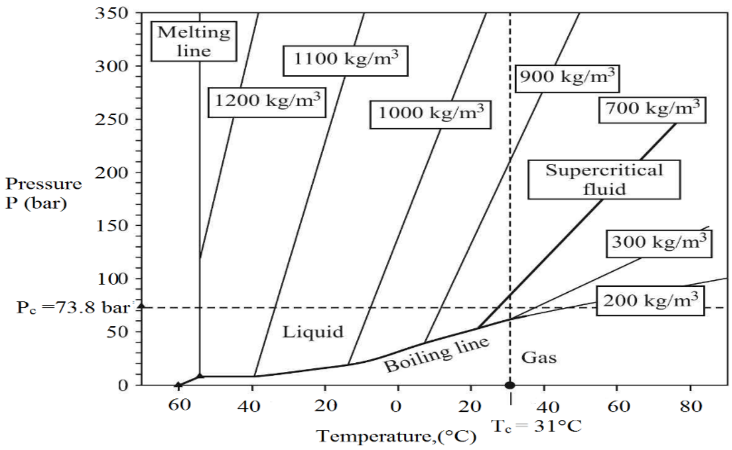
Welcome back to Mass Transfer Operations II course and we were discussing on leaching process that is solid liquid extraction. Now we will be discussing on the last topic that is supercritical fluid extraction and equipment for leaching.

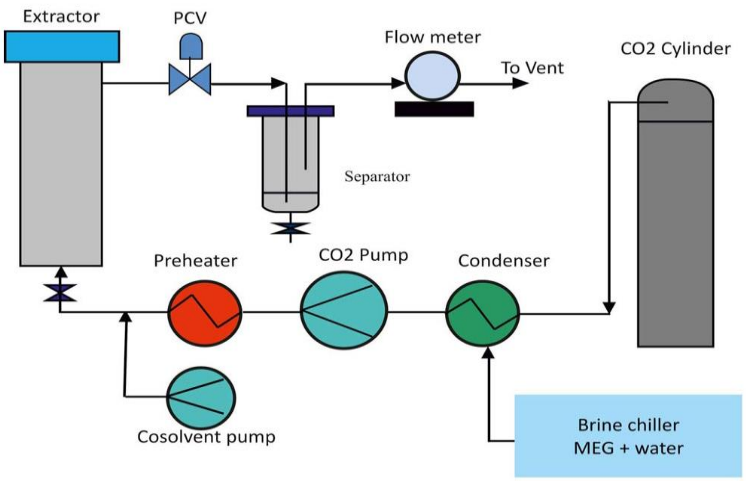
1. Supercritical Fluid Extraction:

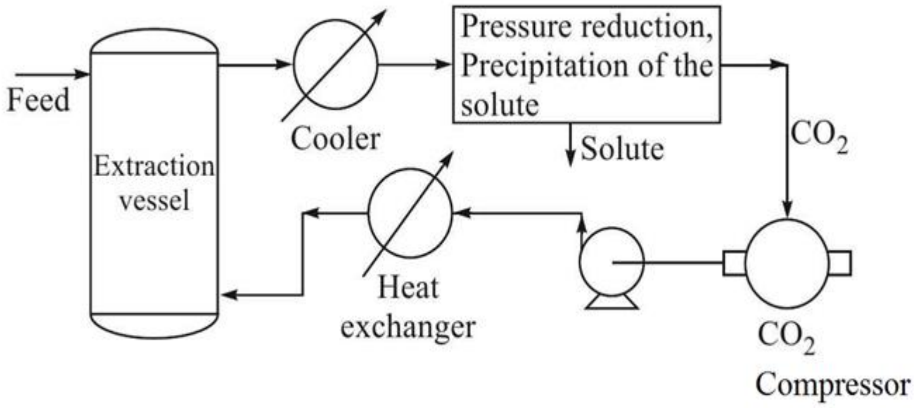
Extraction of certain solutes from solids or liquids using supercritical fluid is a recent addition to separation technology. A fluid is called supercritical if both T and P are above critical values. Its density and viscosity are low and diffusivities of solutes in it are ~100 times greater than those in ordinary liquids. Only disadvantages of supercritical fluid extraction is that it is high pressure operation.

Solubility and selectivity in SCE are strong functions T and P.

CO_2 is the useful solvent for supercritical fluid extraction, especially in food processing.







Advantages:

1. CO_2 is inexpensive
2. It is non toxic
3. Non-flammable
4. Available in high purity

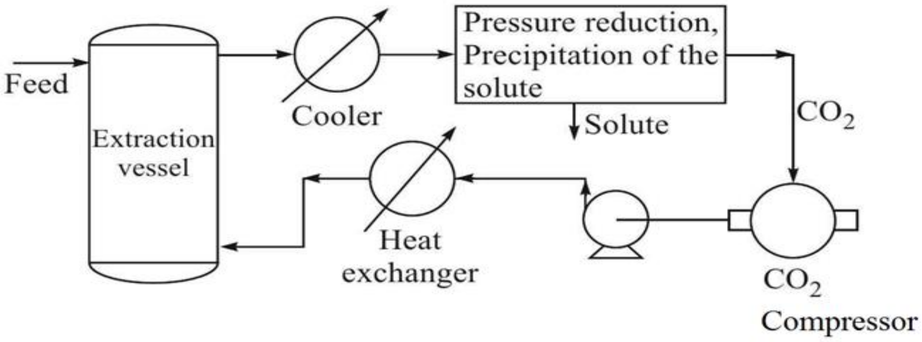
CO_2: normal boiling point: -78.5°C

P_c (bar): 73.8 bar

T_c (°C): 31.1°C

Density (kg/m^3): 468kg/m^3

It is also called cleanest solvent



Example:

1. Decaffeination of coffee → Coffee beans are soaked in water

↓

Loaded into a extraction vessel (Super critical CO_2 is circulated) ← Scrubbing vessel (Caffeine is transferred from CO_2 to water)

→ Water is depressurized and distilled away

2. Extraction and recovery of active ingredients in natural products and in food industries.
3. Extraction of flavours, fragrance and aroma from flowers (jasmine and rose).
4. Food colour from plants and fruits.
5. Extraction of herbal medicine and antibiotics like β - carotene.
6. Extraction of oils from flaked seeds and grains.
7. Spice extraction.

Application of supercritical fluid extraction:

Supercritical fluid extraction is the most effective and efficient way to extract valuable constituent botanicals. Supercritical Fluid Extraction (SFE) is the process of separating one component (the extractant) from another (the matrix) using supercritical fluids that is CO_2 as the extracting solvent. CO_2 is the king of extraction solvents for botanicals. Extraction conditions for supercritical CO_2 are above the critical temperature of 31°C and critical pressure of 74 bar. Supercritical fluids are highly compressed gases, which have combined properties of gases and liquids in an intriguing manner. Supercritical fluids can lead to reactions, which are difficult or even impossible to achieve in conventional solvents. It is a fast process completed in 10 to 60 minutes. A supercritical fluid can be separated from analyte by simply releasing pressure, leaving almost no trace and yields pure residue.

Supercritical Fluid Extraction (SFE), which has received much interest in its use and further development for industrial applications, is a method that offers some advantages over conventional methods, especially for the palm oil industry. SC-CO_2 refers to supercritical fluid extraction (SFE) that uses carbon dioxide (CO_2) as a solvent which is nontoxic, inexpensive, non-flammable, and non-polluting supercritical fluid solvent for the extraction of natural products. Almost 100% oil can be extracted and it is regarded as safe, with organic solvent-free extracts having superior organoleptic profiles.

Over the past three decades, supercritical CO_2 has been used for the extraction and isolation of valuable compounds from natural products.

Supercritical fluid technology is now recognized as an effective analytical technique with favourable and comparable efficiencies to existing chemical analysis methods and when applied for the qualitative and quantitative identification of constituents of naturally occurring products and heat – labile compound.

Alkaloids, organic compounds with bitter taste and toxic effects on animals and humans, but present therapeutic effects when applied in moderate doses, are found in many natural plants. Alkaloids such as caffeine, morphine, emetine, pilocarpine, among others, are the active components in a variety of stimulates and medicinal products and their recovery from natural plants is of great interest to the food, pharmaceutical, and cosmetic industries. Supercritical carbon dioxide proved to be highly selective for caffeine prompting its use as the selected solvent in the commercial decaffeination of coffee and black tea. Recent investigations have demonstrated the potential exploration of solvent and anti-solvent properties of carbon dioxide in the recovery of alkaloids such as theophylline, theobromine and pilocarpine, among others.

Cholesterol was shown to be soluble in supercritical carbon dioxide and even more soluble in supercritical ethane. Extraction with supercritical fluids requires higher investment but can be highly selective and more suitable for food products.

As ethane is much more expensive than CO_2, the use of CO_2/ethane and CO_2/propane mixtures can be an attractive alternative for the removal of cholesterol from foods due to the compromise between higher ethane cost and better cholesterol removal efficiency. Cholesterol removal was also improved through the coupling of carbon dioxide extraction with an adsorption process operating at the same extraction conditions. Literature data also point to potential fractionation of fat simultaneously with the removal of cholesterol from dairy products. The extraction/fractionation operation was also coupled with an adsorption step that uses alumina as the adsorbent. The 0combined extraction/adsorption operation resulted in the removal of more than 97% of the cholesterol in the original butter oil.

The carbon dioxide extraction has also proved efficiency for the production of high quality cocoa butter from cocoa beans. Recent investigation point to the potential use of supercritical CO_2 for microbial inactivation of foods and the implementation of an innovative technique for the sterilization of thermally and pressure sensitive materials.

Another variation is the solution-enhanced dispersion by supercritical fluids. In this process, the supercritical fluid is first mixed with the solution and it is a mixture that is subsequently sprayed into a vessel controlled at the operating temperature and pressure and where particle formation takes place.

Finally it is important to mention that supercritical fluids are known to provide good reaction media due to their capacity to homogenize a reaction mixture, high diffusivity and controlled phase separations and distribution of products.

Problem 3:

In a supercritical fluid extraction vessel, a naphthalene and chalk dust mixture is fed at 300 bar and 55^0C. 15 wt.% naphthalene is dissolved at this condition. The pressure is decreased to 90 bar through pressure reduction valve by isenthalpic expansion. At 90 bar pressure, its temperature becomes 36^0C and the solubility of naphthalene is 2.5

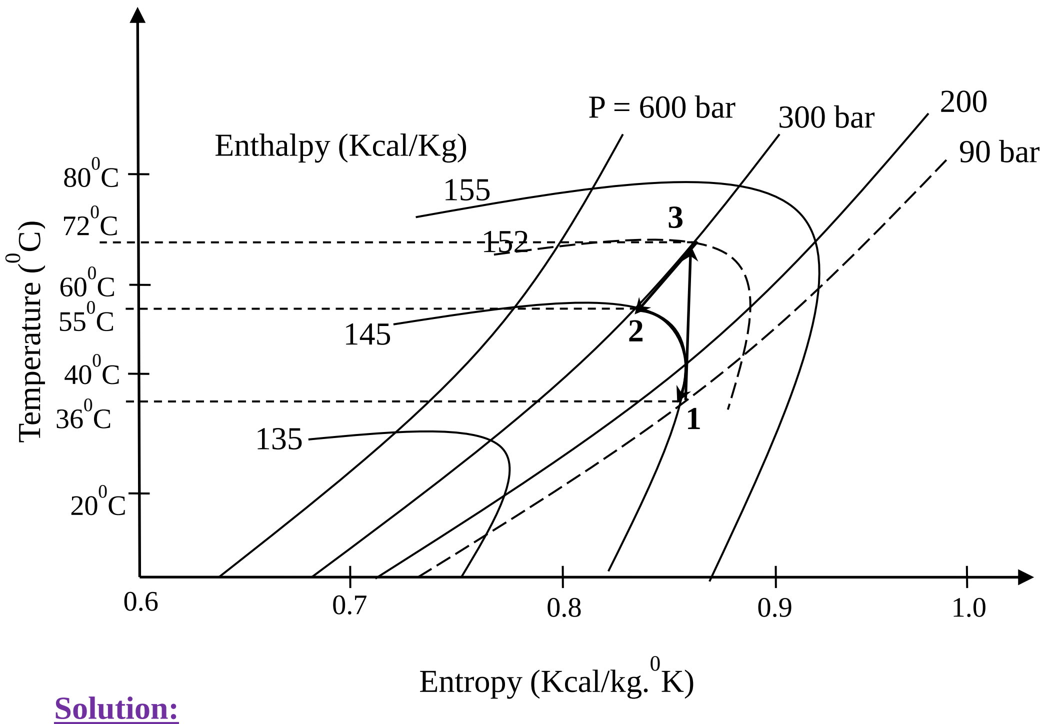
wt.%. Hence, it is separated from the solution and is collected. CO_2 is compressed to 300 bar at 55^0C and recycled to extractor.

Calculate:

(a) The amount of CO_2 required to extract 1 kg naphthalene,

(b) Energy required for extraction of 1 kg naphthalene

Mollier diagram is supplied for this system.



Solution:

(1) 36^0C, 90 bar (after expansion)

(2) 55^0C, 300 bar (extraction)

(3) 72^0C, 300 bar (after compression)

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

Then it is cooled to 55^0C

(4) 72^0C, 300 bar to 55^0C, 300 bar for recycle

Decrease in solubility is 15 to 2.5 wt%

1 Kg CO_2 contains = 0.15 Kg Naphthalene

After extraction,

1 Kg CO_2 contains = 0.025 Kg Naphthalene

Extracted naphthalene = 0.150 – 0.025 = 0.125 Kg

Now, 0.125 Kg naphthalene is extracted for 1Kg CO_2

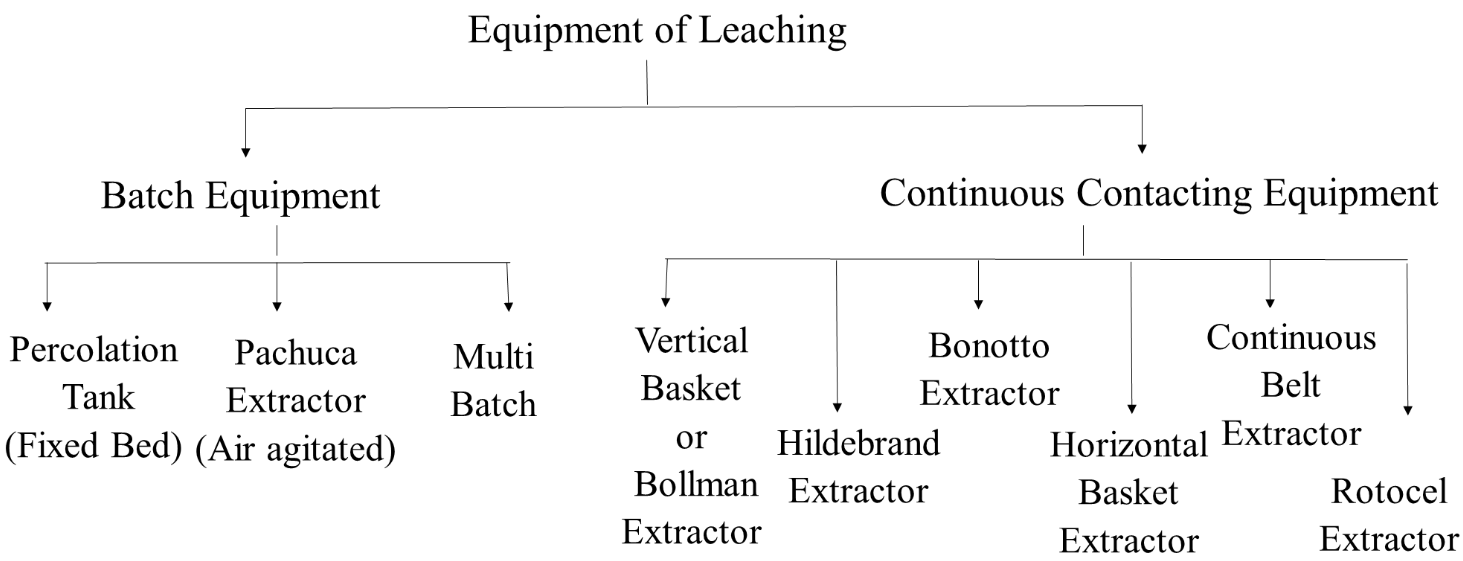
So, 1 Kg naphthalene is extracted for = $\frac{1000}{0.125}$ = 8 Kg CO_2

So,

(a) 8 Kg CO_2 is required to recycle for extraction of 1 Kg naphthalene.

(b) Energy required for compression from 90 to 300 bar is = 8 × 7 Kcals = 56 Kcals for extraction of 1 Kg naphthalene

Equipment for Leaching:



Now we will be discussing about the equipment for leaching, so there are two different types of leaching equipment like we can say extraction units also like batch equipment and we can say this continuous contacting equipment. In batch equipment say we can say percolation tank is there that is fixed bed one then Pachuca extractor unit that is air agitated one then Multi batch system also is there.

In case of the continuous contacting equipment we can say vertical basket or Bollman extraction unit is there then Hildebrand extractor is there then Bonotto extractors also are there and some horizontal basket extractors are there. Some continuous belt extraction units also are there and then Rotocel extractors also are there. So these are we can say these continuous contacting equipments, few these ones pictorial views of the equipments are as follows:

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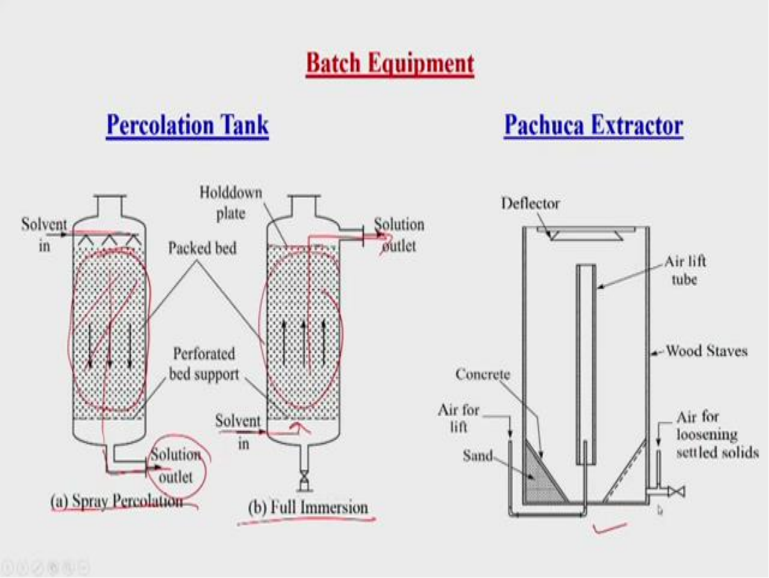

Like percolation tank suppose the solvent will be in from here and then say packed bed is here packing material is filled here and then we can say the solution will be out with we can say the solute like this is a spray percolation system. Then full immersion system is like this where we can say this one hold down plate is there and then packing material is here but solvent will be in from the bottom and then solvent will be out from the top like this and then Pachuca extractor is there that type of thing using this air lifting.

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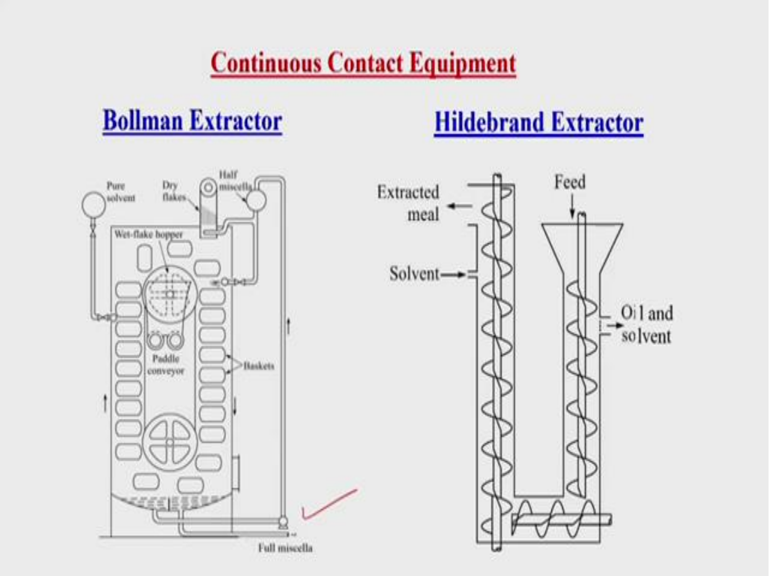

Then we have this Bollman extractor like this, it is also we can say one type of extraction unit that is leaching unit, then Hildebrand extractor is also like this.

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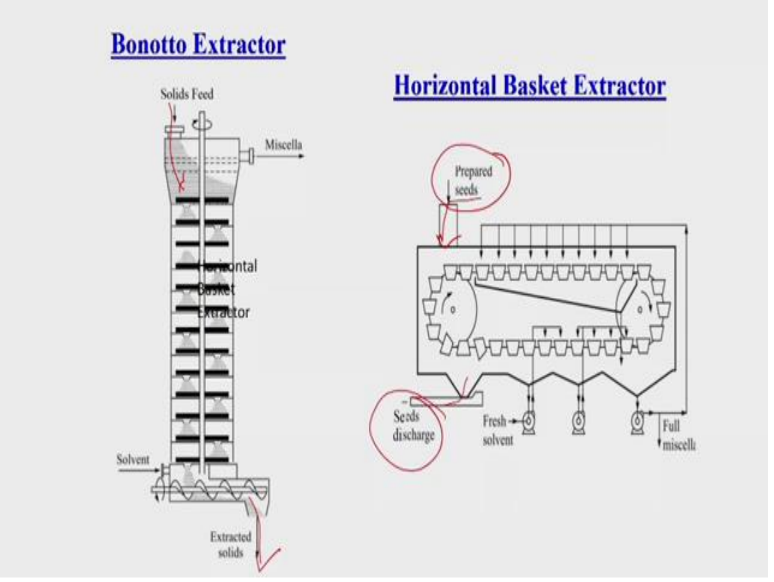

And then we have this Bonotto type extractor where we can say this one solids will be entering from here and then we can say this one extracted solid will be separated from this one bottom of this one through this conveyor belt like horizontal basket extractor also say suppose seeds will be or we can say this one feed will be entering from here and then we can say this one and or inert material will be discharged from the bottom of this we can say basket extractor.

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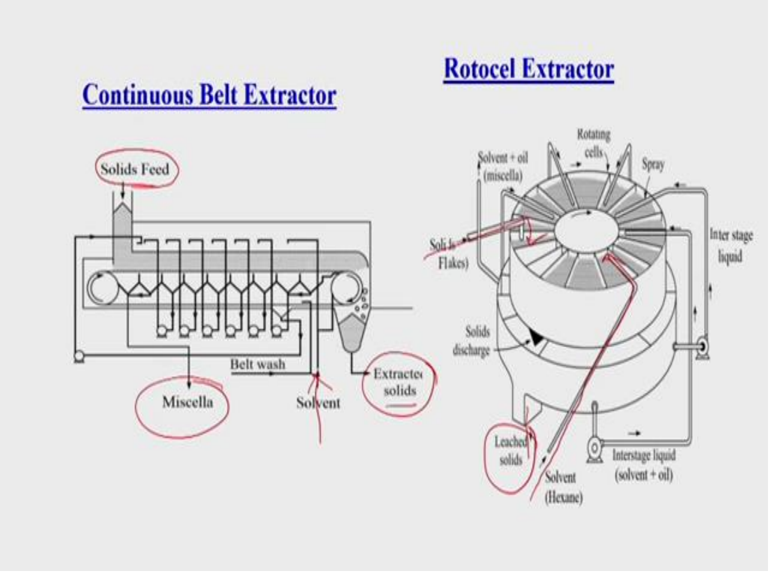

And like continuous extractor we can say this one belt type extractor, suppose this using this belt actually suppose solid feeds will be entering from here and then we can say this solvent will be supplied from this bottom of this we can say this one belt extractor and then extracted solid or we can say inert material will be separated from here.

And we can say this Miscella also will be coming out from the other side of this we can say this extractor. Like Rotocel extractor also like here also so many we can say these rotating cells are available here and solid will be supplied from this top and we can say this one this what is called inert solid or leached solid or we can say this one extracted solids will be coming out from here and this we can say this one extracting solvent is supplied from the top of this extraction unit.

Thank you very much. In the next class, we shall discuss Membrane Separation Technology.