

Lecture 1: Orientation

1.1 Why a chemical engineer needs expertise in process technology?

A process engineer at operation in chemical plant shall have a deeper understanding of the technology on which the process plant is built to produce the profit making chemicals. A chemical engineer with sound knowledge in process technology has the following distinct advantages:

- a) Ability to clearly distinguish the functional role and importance of various processes and operations in the process plant
- b) Technical knowledge with respect to the selection of important parameters such as Temperature, Pressure and underlying physical principles of a process.
- c) Ability to distinguish various process streams and their conditions of operation (Temperature, pressure and phases)
- d) Basic knowledge for process troubleshooting and necessary safety precautions associated to a process/operation.

1.2 How to master fundamentals of process technology?

To master chemical process technology five crucial steps are involved namely:

- a) Raw-Materials and reactions: A chosen process route to manufacture desired chemicals with appropriate purities will eventually lead to preparing a list of raw-materials and utilities. Thereby, prominent reactions can be also known.
- b) Conceptual process flow-sheet: A conceptual process flow-sheet where a chemical engineer has an abstract representation of the actual process flow-sheet will enable quicker learning. A conceptual process flow-sheet typically constitute the following attributes:
 - Raw-material purification (Solid-fluid operations such as cyclone separators, bag filters etc.)
 - Raw-material processing (Heat exchange operations such as furnace heating, cooling etc.)
 - Raw-material to product transformation (Reaction operations using CSTR, PFR, PBR and Batch reactors)
 - Product purification (In separation processes such as flash, distillation, absorption and extraction)
 - Product processing (heat exchangers, phase change units)
 - Recycle of un-reacted raw-materials as recycle streams to the reaction operations.
- c) Process intensification in the form of heat-integration, stream utilization and waste reduction and multiple recycle streams: These options are in fact optional and they enrich the energy enhancement and waste reduction efficiency of a

process plant. Originally, chemical plants developed without such process intensification policies have been subjected to rigorous research and case study investigations to identify opportunities for cost reduction and better energy/waste management.

- d) Additional critical issues related to various unit operations/processes
 - Safety issues: What safety issues are most relevant and need frequent monitoring
- e) Alternate technologies: For a desired function of a process unit, can thereby alternate technologies that could reduce the cost and even then provide the same functional role and desired flow rates and compositions of the emanating streams.

1.3 How much to memorize for a chosen technology?

To a large extent, University education expects a chemical engineering undergraduate student to remember and draw at least a conceptual flow-sheet. However, when a systematic approach is not adopted in the learning process, it is rather difficult to remember all flow-sheets and relate to the logic behind their role in the process topology. Therefore, a well-trained student in process technology remembers process flow-sheets with logical sequence of unit processes/operations and not by strict memory.

1.4 Advantages of suggested learning approach for mastering process technology

- a) Trains student to be more analytical/concept-oriented rather than with memorized knowledge that is bereft of logical reasoning
- b) Systematic approach enables the growth of students' interest in the subject.
- c) Additional concepts further reveal to the student how to gradually complicate process technologies for maximum efficiency.
- d) Inculcate strong interest in the student towards technology research and innovation by enabling a learning environment that fundamentally targets the technological know-how.

1.5 Prominent unit-operations and unit-processes in chemical industry

A detailed summary of various prominent unit operations/processes and their functional role in the chemical plant are summarized in Table 0.1 along with suitable figures.

| Category | Unit operations/processes | Functional role |
|------------------------|---|---|
| fluid operations | a) Centrifugal pump b) Reciprocating pump c) Compressor d) Expander | a) To pressurize liquids and gases. b) To depressurize gases |
| solid operations | a) Crusher b) Grinder | a) To reduce the size of solids |
| Solid-fluid separators | a) Cyclone separator b) Centrifuge c) Electrostatic precipitator d) Classifier & Thickener e) Liquid-liquid separator | a) To separate solid particles from solid-liquid/gas mixtures |
| Heat exchangers | a) Shell & Tube heat exchangers b) Fired heaters and furnaces c) Coolers | a) To either remove or add heat to process streams so as to meet desired conditions in other units. b) Either utilities or other process streams are used to carry out heating/cooling requirements. |
| Mass transfer units | a) Phase separation b) Distillation c) Absorption d) Stripping e) Adsorption f) Extraction g) Leaching h) Crystallization i) Membrane | a) To separate a feed into products with different compositions. b) A third agent (heat or compound) is usually used to carry out separation. |
| Reactor units | a) Completely stirred tank reactor (CSTR) b) Plug flow reactor (PFR) c) Packed bed reactors | a) To carry out reactions in homogenous fluids (gases/liquids). b) To carry out catalytic and multi-phase reactions. |

| | | |
|--|--|--|
| | (PBR) d) Slurry & Trickle bed reactors | |
|--|--|--|

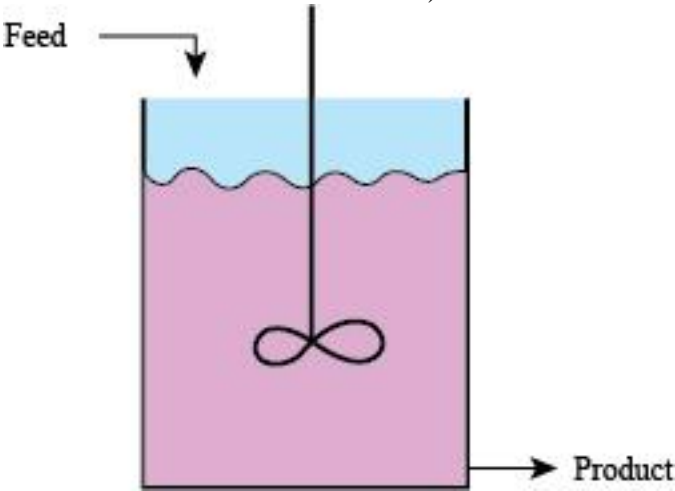
Table 0.1: Important unit operations/unit processes and their functional role in chemical process technology.

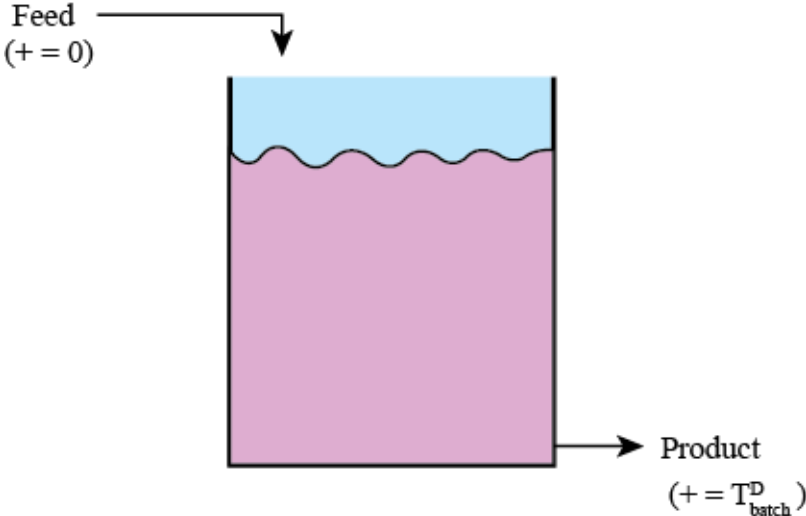
References:

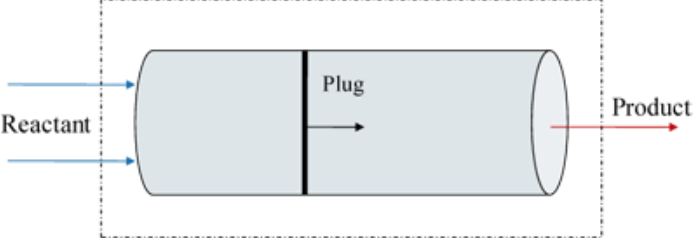
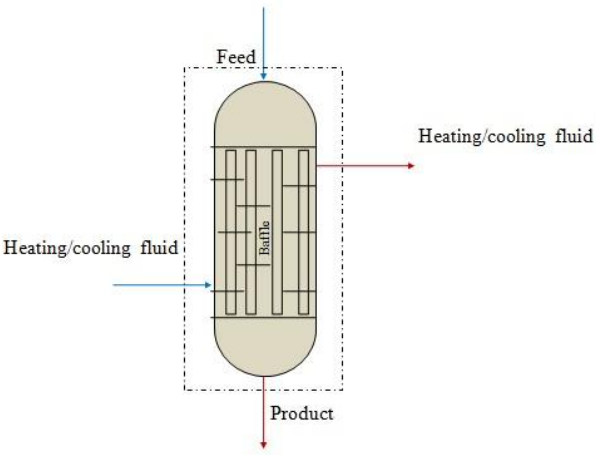
Dryden C. E., Outlines of Chemical Technology, East-West Press, 2008

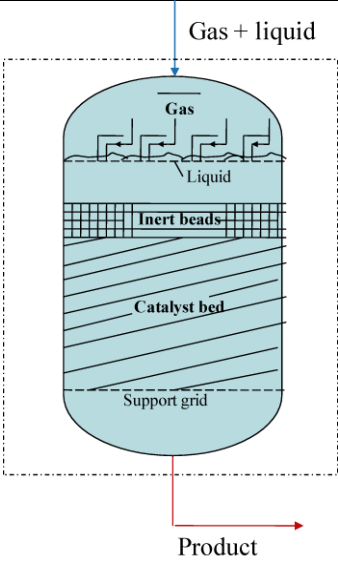
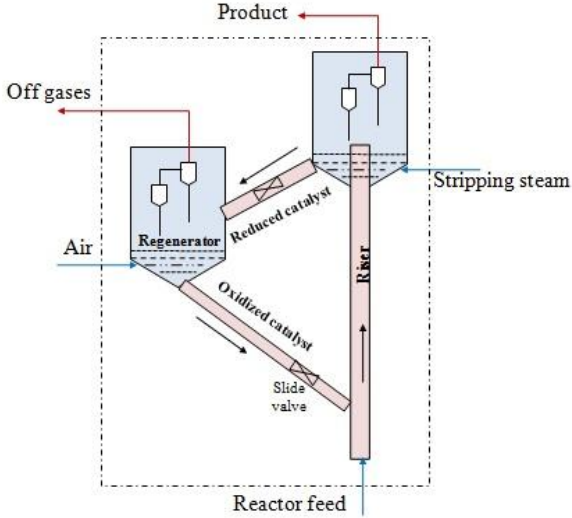
Lecture 2: Orientation (contd...)

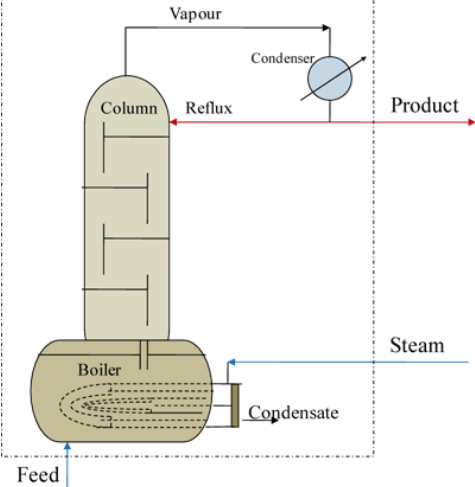
A pictorial representation of various unit processes and operations that are often encountered in chemical process flowsheets is presented in Table 0.2. Along with these figures, their function role in the process technology is also presented.

| Process Technology | Functional Role |
|---|--|
| Reactors a) CSTR b) Batch Reactor c) PFR d) Packed bed reactor e) Trickle bed reactor f) Fluidized bed reactor | <ul style="list-style-type: none"> - Central and most important process technology in process flow sheets - Carry out desired reactive transformations |
| <p style="text-align: center;">a) CSTR</p>  <p style="text-align: center;">CSTR</p> | <ul style="list-style-type: none"> - Well mixed reaction system set alignment - Homogeneous liquid/gas phase reaction - Most easy configuration - Temperature control through Jacket - Reactant instantaneously reaches lowest concentration - Most inexpensive to design and operate |

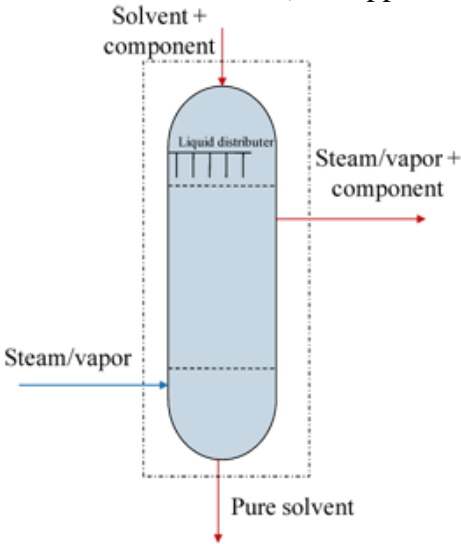
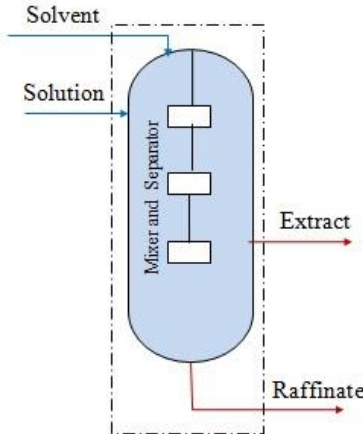
| | |
|--|---|
| <p style="text-align: center;">b) Batch Reactor</p>  <p>The diagram shows a rectangular vessel representing a batch reactor. The vessel is partially filled with a pink liquid, with a light blue layer on top. An arrow labeled "Feed (+ = 0)" points to the top of the vessel. An arrow labeled "Product (+ = T_{batch}^D)" points to the right side of the vessel.</p> | <ul style="list-style-type: none">- Has a simple design, with the requirement of very little supporting equipments- Ideal for small scale experimental studies on reactor kinetics- Can be used industrially for treatment of very small quantities of materials. |
|--|---|

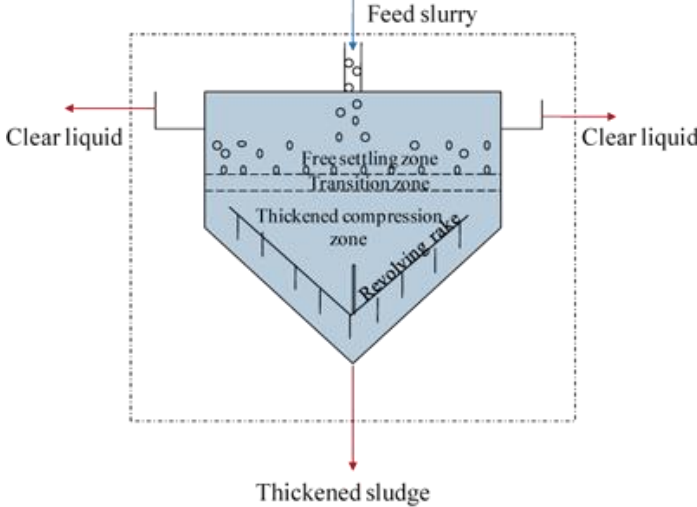
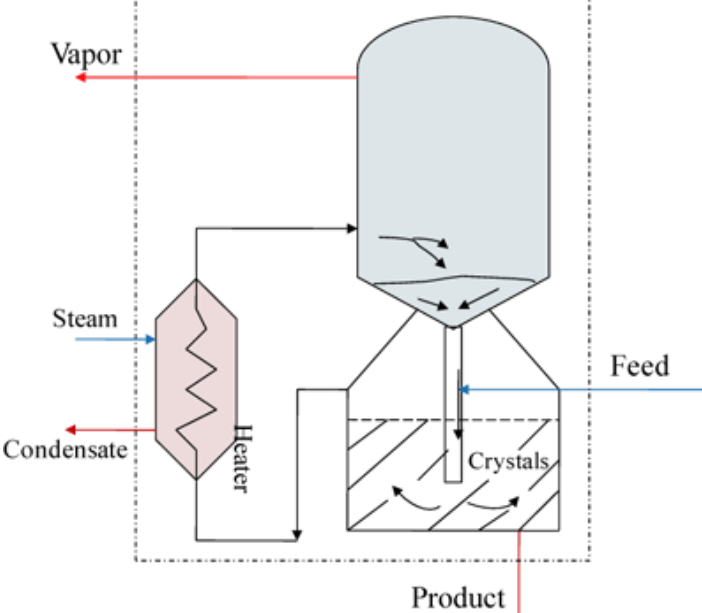
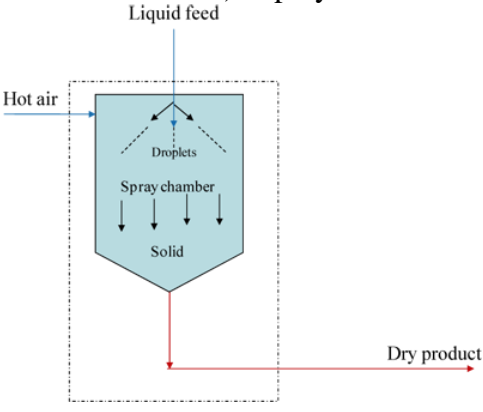
| | |
|--|---|
| <p style="text-align: center;">c)PFR</p>  | <ul style="list-style-type: none"> - Homogeneous liquid/gas phase reaction - Reactant gradually reaches low concentrations - Good control over temperature - Temperature control through jacket (not shown) |
| <p style="text-align: center;">d)Packed Bed Reactor (PBR)</p>  | <ul style="list-style-type: none"> - Heterogeneous reaction - Packing to act as catalyst - Packing packed in tubes - Shell fed with heating/cooling fluid (optional) set alignment - continuous sentence |
| <p style="text-align: center;">e)Trickle Bed Reactor</p> | <ul style="list-style-type: none"> - Multi-phase reaction - If the reaction is not catalytic, packing serves to enhance interfacial area |

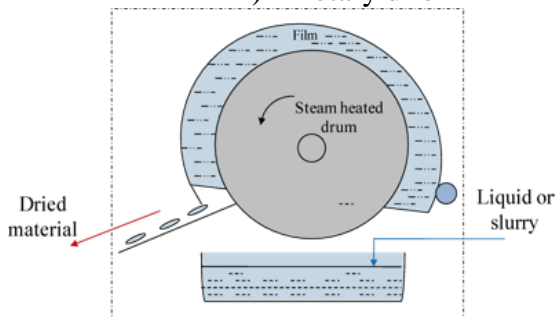
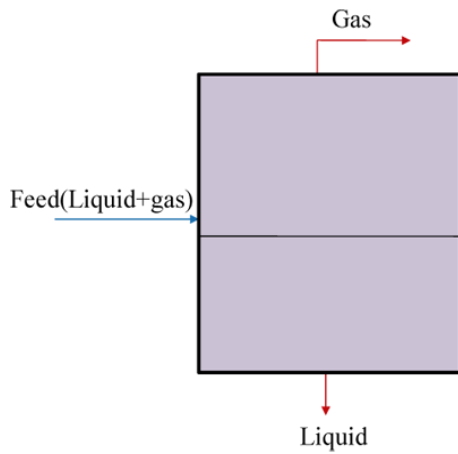
| | |
|---|---|
|  <p style="text-align: center;">Gas + liquid</p> <p style="text-align: center;">Product</p> | <ul style="list-style-type: none"> - If the reaction is catalytic, packing acts as a catalyst as well - Complicated design |
| <p>f) Fluidized bed reactor</p>  <p style="text-align: center;">Product</p> <p style="text-align: center;">Reactor feed</p> | <ul style="list-style-type: none"> - Provides highest mass, heat and hence reaction rates for solid-fluid reactions - Very commonly deployed in petroleum refineries (catalytic cracking) - Complicated accessories (shown) and control system required - The accessories are for catalyst re-generation and transport. |
| | |

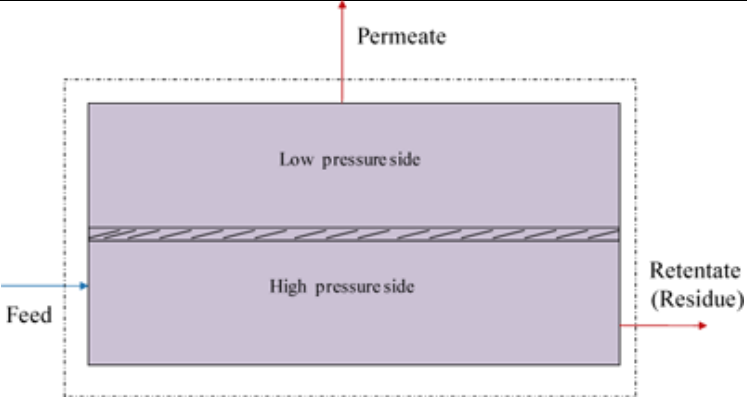
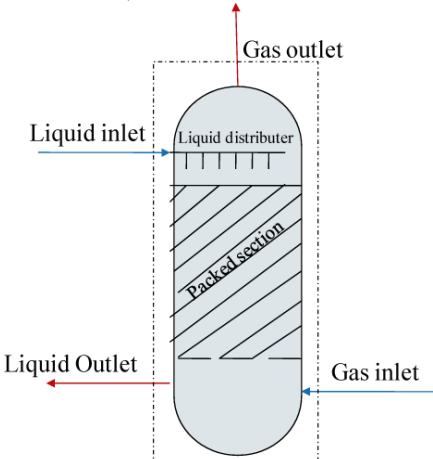
| | |
|---|--|
| <p>Separators:</p> <ol style="list-style-type: none"> Batch distillation Continuous distillation Absorption Stripping Liquid-liquid extraction Leaching Crystallization Drying Flash separator Membrane separator Packed bed contactor | <ul style="list-style-type: none"> - Most important process technology - Provides desired separation between phases and streams - Located next to the reactor as 100 % conversions are very rare in industrial practice |
| <p>a) Batch distillation column</p>  | <ul style="list-style-type: none"> - Used to separate a liquid mixture based on relative volatility (differences in boiling points) - Operated in batch mode |
| <p>b) Continuous distillation (Fractionator) column missed</p> | <ul style="list-style-type: none"> - The most important separation technology in process flow sheets - Provides very pure products - Differences in boiling points is the working principle |

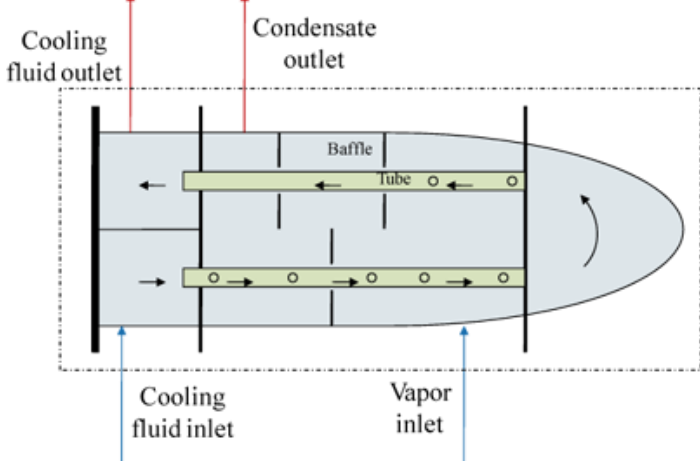
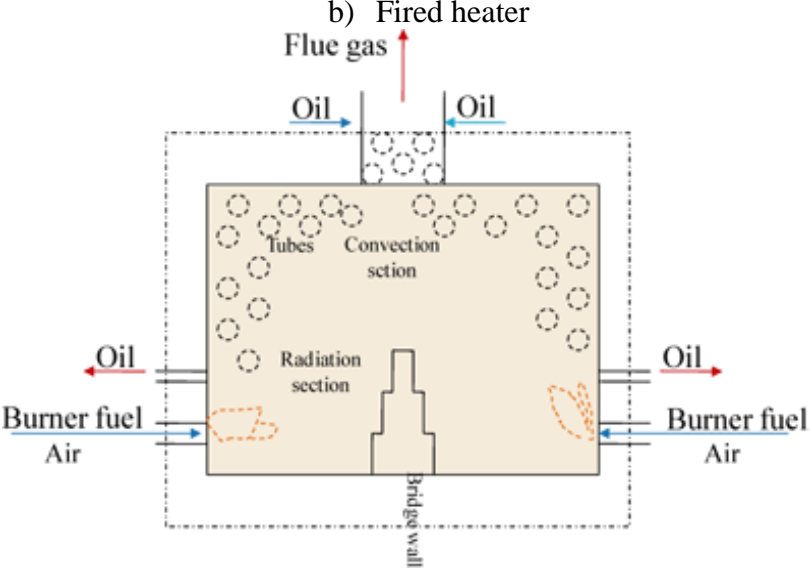
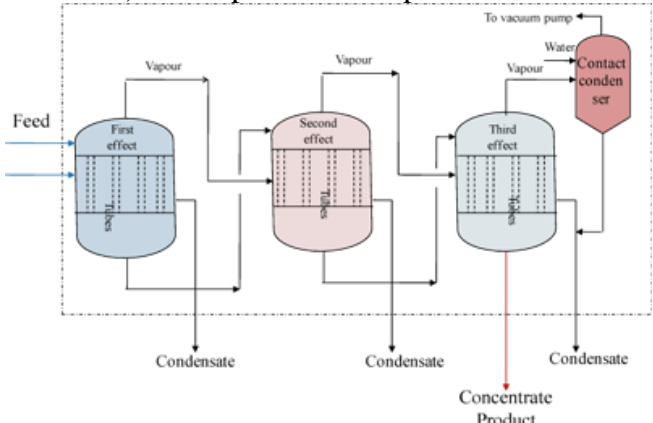
| | |
|---|--|
| | <ul style="list-style-type: none"> - Energy intensive operation - |
| <p style="text-align: center;">c) Absorption column</p> <p style="text-align: center;">Absorber used for taking up a soluble gas in a solvent liquid.</p> | <ul style="list-style-type: none"> - Used to absorb components from gaseous stream - Solvent is used - Usually followed with stripper to regenerate the fresh solvent - Operated at low temperature and moderate/high pressure |
| | |

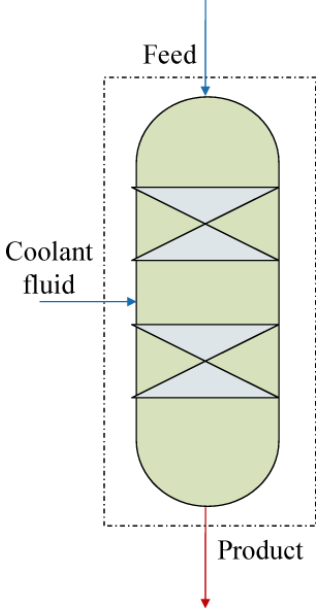
| | |
|--|---|
| <p style="text-align: center;">d) Stripper</p>  <p style="text-align: center;">Stripper used for removing a soluble gas from solution by counter current contact with an inert gas.</p> | <ul style="list-style-type: none"> - Steam/Hot gas is used to strip the gas - Regenerated solvent used for absorption |
| <p style="text-align: center;">e) Liquid Liquid extraction</p>  | <ul style="list-style-type: none"> - Used to separate components from a liquid with a liquid solvent - Consists of a series of mixers and separators - Produces extract (rich with solvent and components extracted) and raffinate (product with lean extractants) |
| <p style="text-align: center;">f) Leaching</p> | <ul style="list-style-type: none"> - A liquid solvent extracts components from a solid - High interfacial area |

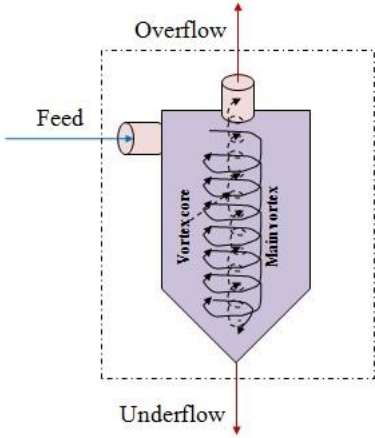
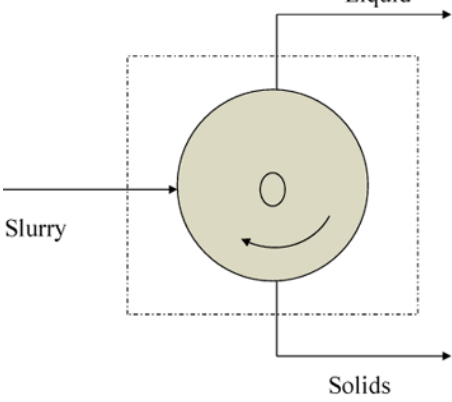
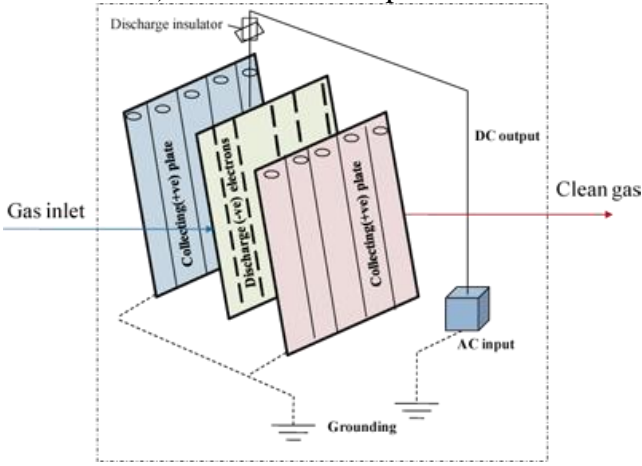
| | |
|--|---|
|  | <p>between solid/liquid is required to enhance extraction capability</p> |
| <p style="text-align: center;">g) Crystallization</p>  | <ul style="list-style-type: none"> - Used to crystallize solids from a slurry/super-saturated solution - Fine crystals added to serve as nucleating agent |
| <p style="text-align: center;">h) Spray drier</p>  | <ul style="list-style-type: none"> - Liquid slurry is sprayed in the form of droplets - Hot gas (air) dries the solid - Enables very good control over the product particle size |

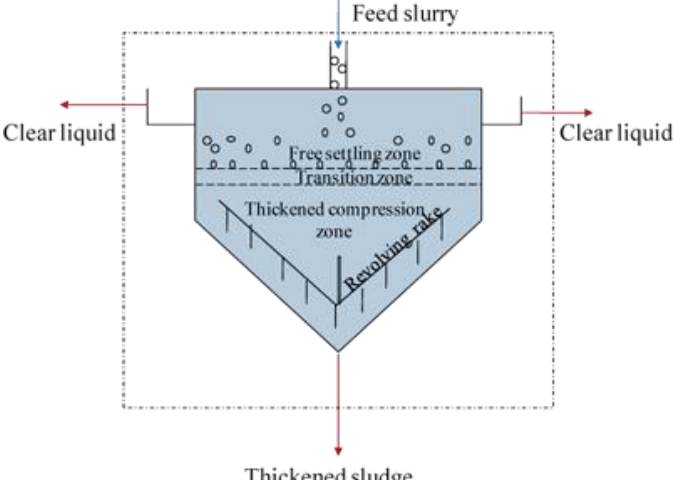
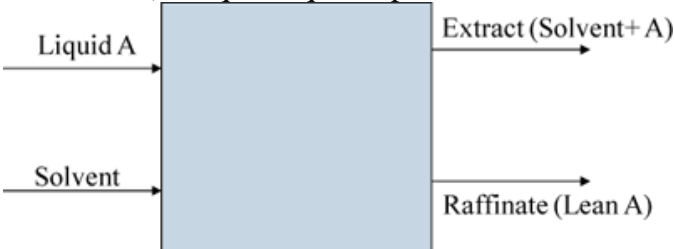
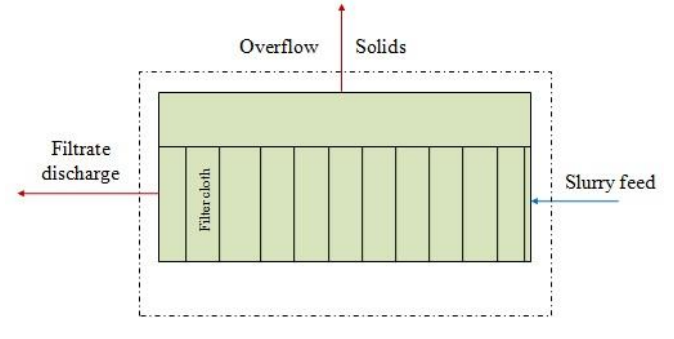
| | |
|--|--|
| <p>i) Rotary drier</p>  | <ul style="list-style-type: none"> - Through rotation, an agitated liquid film is dried to obtain the dried solid. |
| <p>j) Flash separator</p>  | <ul style="list-style-type: none"> - Very common technology to separate liquid streams at high pressure and lower temperatures - Upon pressure reduction/heating, low boiling components separate as vapor phase and yield a liquid phase. - Complete separation only possible for fewer components |
| <p>k) Membrane separation</p> | <ul style="list-style-type: none"> - A semi-permeable barrier (membrane) is used to separate feed streams based on concentration set |

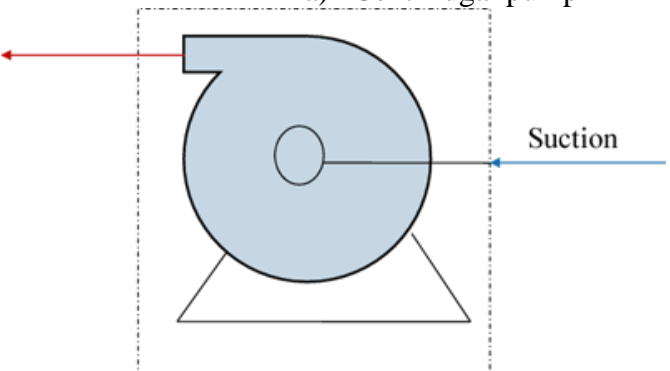
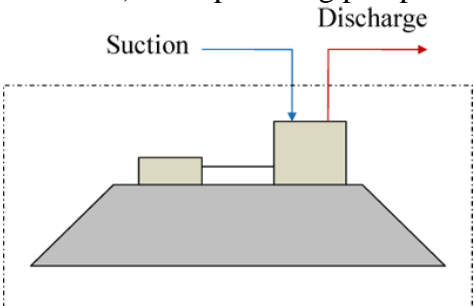
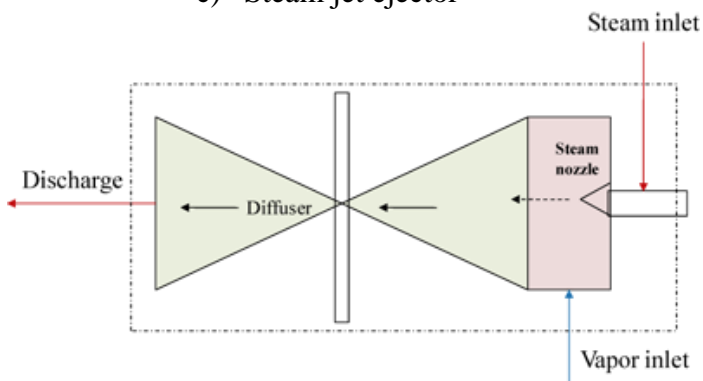
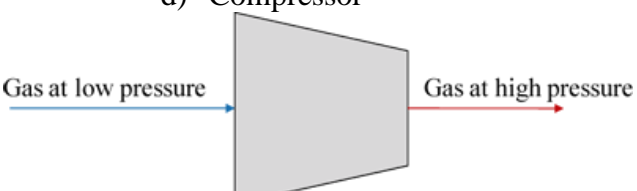
| | |
|---|--|
|  | <p>alignment difference/pr essure difference.</p> <ul style="list-style-type: none"> - Various types of processes available - New technology in process industries. |
| <p>1) Packed bed contactor</p>  | <ul style="list-style-type: none"> - Used for absorption/stripping operations - Packing serves to enhance gas/liquid interfacial area |
| <p>Heat exchange equipment</p> <ol style="list-style-type: none"> Shell & Tube heat exchanger Fired heater Multiple effect evaporator Quenching | <ul style="list-style-type: none"> - Very prominent equipment to heat/cool process fluids - Include steam/power generation as well! |
| <p>a) Shell & Tube heat exchanger</p> | <ul style="list-style-type: none"> - Most common equipment in process industries - Tube fed with a fluid and shell is fed with another fluid - Process heat |

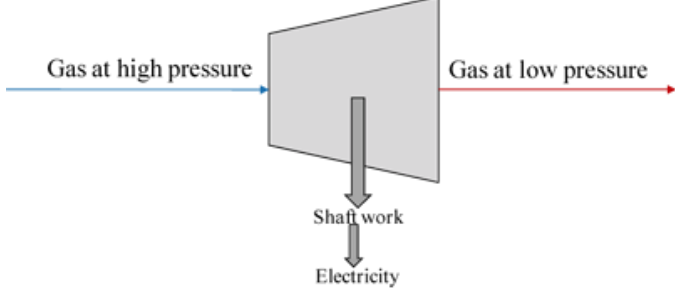
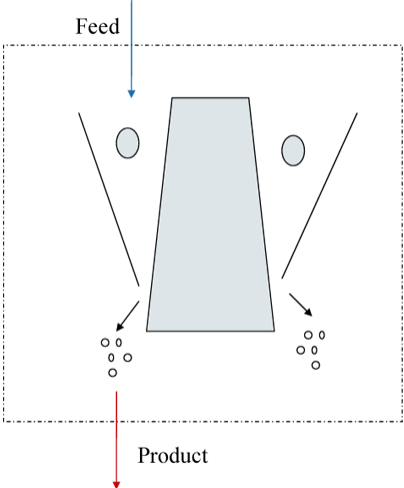
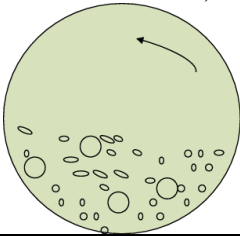
| | |
|---|--|
|  | <p>is transferred across the tube</p> <ul style="list-style-type: none"> - No mixing of tube fluid and shell fluid allowed |
| <p>b) Fired heater</p>  | <ul style="list-style-type: none"> - Used to heat streams to extremely high temperatures - High temperatures generated by burning fuel oil/fuel gas - Complicated design for maximum heat transfer efficiency - Shell & tube type/radiation type designs usually adopted |
| <p>c) Multiple effect evaporator</p>  | <ul style="list-style-type: none"> - Common equipment to concentrate a solid-liquid stream from low concentration to high concentrations. |

| | |
|--|--|
| | <p>alignment</p> <ul style="list-style-type: none"> - Steam utility is optimized by adopting process intensification method. |
| <p>d) Quenching</p>  | <ul style="list-style-type: none"> - Direct heat transfer equipment - Involves cooling/heating a fluid with direct contact with a stream - Commonly used for streams emanating with very high temperatures from reactions/furnaces. |
| <p>Solid-fluid process technology</p> <ul style="list-style-type: none"> a) Cyclone separator b) Centrifuge c) Electrostatic separator d) Thickener e) Liquid- liquid separator <p>Filter press</p> | <ul style="list-style-type: none"> - Used for separating solids from solid-liquid or solid-gas mixtures |
| <p>a) Cyclone separator</p> | <ul style="list-style-type: none"> - Separates fine solid particles from a gas-solid mixture - Uses centrifugation as working principle. - Very good separation of solid and |

| | |
|--|---|
|  | <p>liquid possible set alignment</p> |
| <p>b) Centrifuge</p>  <p>Ex: Sugar is extracted from sugar slurry.</p> | <ul style="list-style-type: none"> - Separates solids from solid-liquid mixture - Uses the principle of centrifugation for separation - Very good separation of solid and liquid possible <p>Put the fig no. b</p> |
| <p>c) Electrostatic separator</p>  | <ul style="list-style-type: none"> - Separates solids from solid-liquid mixture - Uses the principle of charged surfaces to separate the solids - Very common in process technologies |
| <p>d) Thickener</p> | <ul style="list-style-type: none"> - Separates a slurry |

| | |
|--|--|
|  | <p>(solid-liquid) into a sludge and clarified liquid</p> <ul style="list-style-type: none"> - Settling is adopted as working principle. |
| <p>e) Liquid-liquid separator</p>  | <ul style="list-style-type: none"> - Uses decantation as working principle based on density difference. |
| <p>f) Filter press</p>  | <ul style="list-style-type: none"> - Separates a solid from solid-fluid mixture - Uses a fine mesh/cloth to separate under pressure. Adjust the text |
| <p>Fluid transport</p> <ol style="list-style-type: none"> Centrifugal pump Reciprocating pump Steam jet ejector Compressor Expander | <ul style="list-style-type: none"> - Very important to achieve process conditions desired in other important processes such as reactors and separators. |

| | |
|---|---|
| <p>a) Centrifugal pump</p>  | <ul style="list-style-type: none"> - Energizes liquids to moderately high pressure. |
| <p>b) Reciprocating pump</p>  | <ul style="list-style-type: none"> - Energizes liquids to very high pressures. |
| <p>c) Steam jet ejector</p>  | <ul style="list-style-type: none"> - Used for providing vacuum (low pressure) in various units - Common in process flow sheets. |
| <p>d) Compressor</p>  | <ul style="list-style-type: none"> - Enhances pressure of gases to high values. |
| <p>e) Expander</p> | <ul style="list-style-type: none"> - Reduces pressure of gas to lower values - Recovered energy used for shaft work or |

| | |
|--|--|
|  <p>Gas at high pressure</p> <p>Gas at low pressure</p> <p>Shaft work</p> <p>Electricity</p> | <p>power generation (electricity).</p> |
| <p>Size Reducer:</p> <p>a) Crusher</p> <p>b) Grinder</p> | <ul style="list-style-type: none"> - Used for reducing size of solids and enhance their surface area to facilitate higher mass transfer and reaction rates. |
| <p>a) Crusher</p>  <p>Feed</p> <p>Product</p> | <ul style="list-style-type: none"> - Continuous operation - Size control is very easy |
| <p>b) Grinder</p>  | <ul style="list-style-type: none"> - Batch operation - Achieving size control is difficult. |
| <p>Storage:</p> <p>a) Storage tank</p> <p>b) Pressurized spheres</p> | <ul style="list-style-type: none"> - Used to store fluids and gases. |
| <p>a) Storage tank</p> | <ul style="list-style-type: none"> - Used |

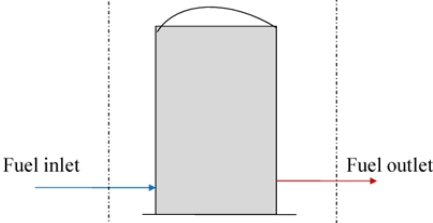
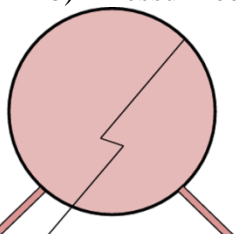
| | |
|---|---------------------------------------|
|  | <p>especially for liquid fuels</p> |
| <p>b) Pressurized spheres</p>  | <p>- Used to store gaseous fuels.</p> |

Table 0.2: Summary of various prominent process technologies and their functional role in process flow sheets.

Not n2 i.e h2, condenser ,

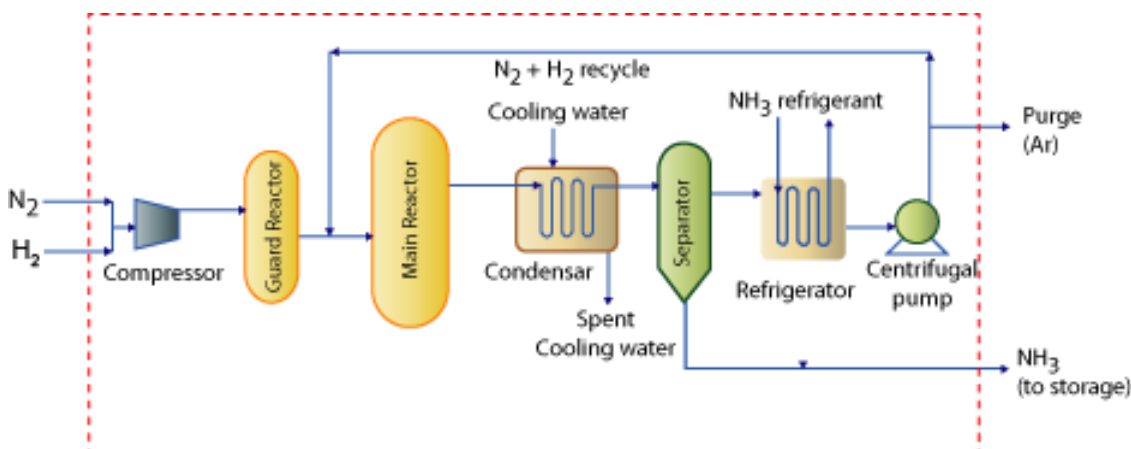


Figure 1.1: Process flow sheet for ammonia manufacture using Haber's process.

Illustration of quickly learning process technology: Ammonia manufacture using Haber's process (Figure 1.1)

- a) **Reaction:** The Haber process combines nitrogen from the air with hydrogen derived mainly from natural gas (methane) into ammonia. The reaction is reversible and the production of ammonia is exothermic. Nitrogen and hydrogen will not react under normal circumstances. Special conditions are required for them to react together at a decent rate forming a decent yield of ammonia. These conditions are $T = 400^\circ\text{C}$; $P = 200\text{ atm}$; Iron catalyst with KOH promoter.
- b) **Raw materials:** H_2 from synthesis gas, N_2 from synthesis gas/air liquefaction process
- c) **Process technology:** Illustrated in Figure 1.1
- d) **Unit processes:** Feed Guard Converter; Main Reactor

- e) **Unit operations in the technology:** Condensation/Gas Liquefaction; Separation; Refrigeration; Centrifugal Re-circulator
- f) **Striking feature:** Conversions are low (8 - 30 % per pass) and hence large recycle flow rates exist.
- g) **Functional role of various processes**
- a. **Feed guard converter:**
 - CO and CO₂ conversion to CH₄ and removal of traces of H₂O, H₂S, P and Arsenic.
 - These compounds could interfere with the main haber's reaction as well as poison the catalyst.
 - b. **Main reactor:**
 - Cold reactants enter reactor from reactor bottom and outer periphery to absorb heat generated in the reversible reaction.
 - Carbon steel used for thick wall pressure vessel and internal tubes
 - Gas phase reaction at 500 – 550 °C and 100 – 200 atms.
 - Pre-heated gas flows through the tube inside with porous iron catalyst at reaction conditions.
 - Catalyst removed from the converters is re-fused in an electric furnace.
 - c. **Condenser:**
 - Complete liquefaction not possible due to vapor liquid equilibrium between NH₃ in vapor and liquid phases.
 - Cooling fluid: Chilled water
 - Incoming stream has: Gaseous NH₃, un reacted N₂ and H₂, impurity gases like CO, CO₂, CH₄
 - Product stream has: Liquefied NH₃, Vapor phase NH₃ sub3in equilibrium with liquid, non-condensable gases such as N₂ and H₂
 - System pressure is high therefore NH₃ bound to be present in higher concentrations in the vapor.
 - d. **Separator:**
 - Working principle: Density difference between vapor and liquid
 - Liquefied NH₃ separated from the un-reacted gases (NH₃ still present in the vapors).
 - e. **Refrigeration:**
 - Why?: NH₃ available in vapors needs to be condensed. Therefore, refrigeration is required for gaseous product emanating from the separator.
 - The condensed NH₃ emanates at -15 °C.
 - After refrigeration, the un-reacted N₂ and H₂ are recycled to the reactor.

f. Centrifugal pump:

- A centrifugal pump adjusts the pressure of the stream from the separator to the pressure of the feed entering the reactor
 - A purge stream exists to facilitate the removal of constituents such as Argon.

g. Striking feature: All units such as Condenser, Separator, Refrigerator operate at high pressure. This is because losing pressure is not at all beneficial as the un-reacted reactants (corresponding to large quantity in this case due to low conversion in the reversible reaction) need to be supplied back to the reactor at the reactor inlet pressure conditions.

1.7 Technical Questions

1.7.1

- i) Only reactants and not product (Ammonia) are fed to the reactor. According to Le-Chatelier principle, higher yields are obtained at high pressures. Exothermic reaction implies that the temperature cannot be too high.
- ii) Ammonia removal from vapor phase emanating from the condenser is according to Le-Chatelier principle, which indicates that the presence of ammonia in the feed can reduce conversions and hence yield.

1.7.2

- i) The purge stream consisting of Argon impurity would go to pressure swing adsorption process for Argon recovery. Therefore, it will be advantageous to have as much high pressure as possible for the purge stream and hence it is desired to take the purge stream after the centrifugal pump.

1.7.3

- i) Ammonia's boiling point is -33°C at 1 atm pressure. At higher pressures, the boiling point of ammonia will increase. Therefore, at the system pressure (which is close to 200 atm.), the boiling point of ammonia would be much higher and hence liquefaction is possible at -15°C .
- ii) The ammonia refrigerant is at 1 atm pressure and hence can be a liquid upto a temperature of -33°C . The stream undergoing refrigeration is at 200 atm. And therefore liquefies at about -15°C . degree

References:

Dryden C. E., Outlines of Chemical Technology, East-West Press, 2008