Lecture 3

Basic Principles of Unit Processes and Unit Operations in Organic Chemical Industries

LECTURE 3

BASIC PRINCIPLES CHEMICALPROCESSES, UNIT PROCESSES AND UNIT OPERATIONS IN ORGANIC CHEMICAL INDUSTRIES

Chemical processes usually have three interrelated elementary processes

- Transfer of reactants to the reaction zone
- Chemical reactions involving various unit processes
- Separation of the products from the reaction zone using various unit operations

Processes may involve homogeneous system or heterogeneous systems. In homogeneous system, reactants are in same phase-liquid, gases or solids while heterogeneous system include two or more phases; gas liquid, gas–solid, gas-gas, liquid–liquid, liquid solid etc. Various type reactions involve maybe reversible or irreversible, endothermic or exothermic, catalytic or non-catalytic. Various variables affecting chemical reactions are temperature pressure, composition, catalyst activity, catalyst selectivity, catalyst stability, catalyst life, the rate of heat and mass transfer. The reaction may be carried out in batch, semi batch or continuous. Reactors may be batch, plug flow, CSTR. It may be isothermal or adiabatic. Catalytic reactors may be packed bed, moving bed or fluidised bed

Along with knowledge of various unit processes and unit operation following information are very important for the development of a process and its commercialization [Austin,1984]

Basic Chemical data: Yield conversion, kinetics

- Material and energy balance, raw material and energy consumption per tone of product, energy changes
- Batch vs Continuous, process flow diagram
- Chemical process selection: design and operation, pilot plant data, Equipment required, material of construction
- Chemical Process Control and Instrumentation
- Chemical Process Economics: Competing processes, Material and, Energy cost, Labour, Overall Cost of production
- Market evaluation: Purity of product and uniformity of product for further processing

- Plant Location
- Environment, Health, Safety and Hazard
- Construction, Erection and Commissioning
- Management for Productivity and creativity: Training of plant personals and motivation at all levels
- Research, Development and patent
- Process Intensification

  Inorder to improve productivity and make the process cost effective and for improving overall economy, compact , safe, energy efficient and environmentally sustainable plant, process intensification has become very important and industry is looking beyond the traditional chemical engineering.

UNIT PROCESSES AND UNIT OPERATIONS IN CHEMICAL PROCESS INDUSTRIES

Chemical process is combination of unit processes and Unit operation. Unit process involves principle chemical conversions leading to synthesis of various useful product and provide basic information regarding the reaction temperature and pressure, extent of chemical conversions and yield of product of reaction nature of reaction whether endothermic or exothermic, type of catalyst used. Unit operations involve the physical separation of the products obtained during various unit processes. Various unit processes in chemical industries are given in Table M-I 3.1. Various chemical reactions and its application in process industries are given in Table M-I 3.2.

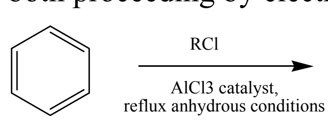
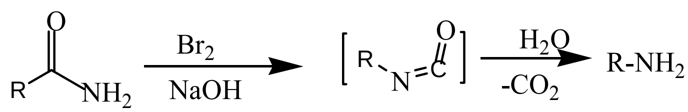
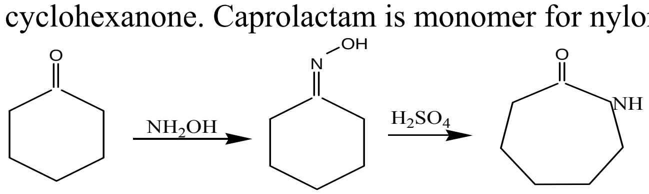
NITRATION

Nitration involves the introduction of one or more nitro groups into reacting molecules using various nitrating agents like fuming, concentrated, aqueous nitric acid mixture of nitric acid and sulphuric acid in batch or continuous process. Nitration products find wide application in chemical industry as solvent, dyestuff, pharmaceuticals, explosive, chemical intermediates. Typical products: TNT, Nitrobenzene, m-dinitrobenzene, nitroacetanilide, alpha nitronaphthalene, nitroparaffins

Table M-I 3.1: Unit Processes in Chemical Process Industries

Alkylation and Hydro delkylation	Decomposition
Acylation	Fermentation
Ammonoxidation	Halogenation
Amination by reduction	Hydsogenation
Amination	Hydrohenatlysis
Aromatisation	Hydroformylation
Amination by ammonalysis	Hydro lysis
Calcination	Hydration
Carbonation	Hydroammonalysis
Causticisation	Isomerisation
Chlorination and Oxy chlorination	Neutralistion
Condensation	Nitration
Biomethhanation	Methanation
Carbinisation	
Disproportination	Oxidation and partial oxidation
Cracking; Thermal, steam cracking, catalytic cracking	Pyrolysis
Dehydration	Polymeristion: Addition and condensation Chain growth and step growth,Bulk, Emulsion, suspension, solution, Radical and coordination polymeristion
Dehydrogenation	Reduction
Ditozitation and coupling	Reforming: Steam reforming Catalytic reforming
Gasification of coal and biomass	Sulphidation
Desulphurisation and hydro desulphurisation	Sulphonatiomn
Electrolysis	Sulphation
Etherification	Xanthation
Estertification and Trans Estrerificartion	

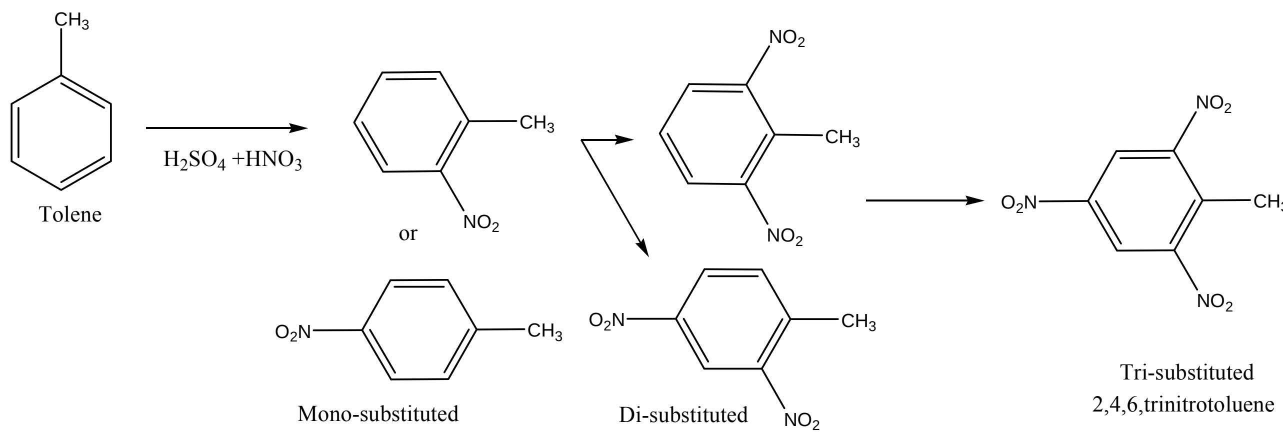
Table M-I 3.2: Important Chemical Reaction and their Application in Chemical Process Industries

Reaction	Description
Fisher-Tropsch (FT) Process	The Fisher-Tropsch process produce a variety of hydrocarbons (alkanes: $C_nH_{(2n+2)}$) by involves a series of chemical reaction. $(2n+1)\ H_2 + nCO \rightarrow C_nH_{(2n+2)} + nH_2O$ FT process is used for synthesis of alkanes.
Friedel-Crafts reactions	In this reaction attach substituent's to an aromatic ring. Two main types of Friedel-Crafts reaction are acylations reaction and alkylation reactions, both proceeding by electrophilic aromatic substitution.  Friedel-Crafts process used in alkelation reactions.
Oxosynthesis Reactions	In this process Isomeric mixture of normal- and iso-aldehydes get produces by utilizing syngas (CO and H_2) and olefinic hydrocarbons as reactants. It is exothermic process, this process thermodynamically favorable at ambient pressure and temperatures. This reaction also called as hydroformylation reaction. $RCH{=}CH_2 + CO + H_2 \rightarrow RCH_2CH_2CHO + R(CH_3)CHCHO$ Oxosynthesis used for production of alcohols.
Hofman Process	In this process, organic reaction of primary amide converts into a primary amine with one fewer carbon atom.  Hofman process typical examples are conversion of aliphatic amides to aliphatic amines and aromatic amides to aromatic amines.
Free-Radical Reaction	Any chemical reaction involving free radicals, generally radical generated from radical initiators such as peroxide or azo bis compounds. Radical reactions are chain reactions with chain initiation, propagation and termination steps. Free radical reactions are used many organic synthesis and polymerization reactions
Beckmann rearrangement	Beckmann rearrangement is acid catalyzed rearrangement of an oxime to an amide, which developed by German chemist Ernst Otto. A typical example of Beckmann rearrangement is synthesis of caprolactam from cyclohexanone. Caprolactam is monomer for nylon 6. 

Wackers Process	Wacker process is similar to hydroformylation and used for aldehyde compounds. A typical example of wacker process is oxidation of ethylene to acetaldehyde in the presence of Pd catalyst. $[PdCl_4]^{--}+C_2H_4+H_2O \rightarrow CH_3CHO+Pd+2HCl+2Cl^-$ Pd^{++} $CuCl_2+2Cl^- \rightarrow [PdCl4]^{--}+2CuCl$ $2CuCl+ 0.5\ O_2+2HCl \rightarrow 2CuCl_2+H_2O$

Example Preparation of TNT (trinitrotoluene)

TNT is produce in a three-step process: First, toluene is nitrated with a mixture of sulfuric acid and nitric acid to produce mono-nitrotoluene or MNT. The MNT is separated and then renitrated to dinitrotoluene or DNT. In the final step, the DNT is nitrated to trinitrotoluene or TNT using an anhydrous mixture of nitric acid and oleum.



HALOGENATION

Halogens involve introduction of one or more halogen groups into a organic compound for making various chlorine, bromine, iodine, fluorine organic derivatives. All though chlorine derivatives find larger application, however some of the bromine and fluorine derivatives are also important. Various chlorinating agents are chlorine, HCl, phosgene sulfuric chloride, hypochlorite, bromination, bromine, hydrobromic acid, bromide, bromated, alkaline hypobromites. In iodination iodine, hydroiodic acid and alkali hypoiodites

Example

Typical important chemicals are chlorinated products: Ethylene dichloride, chlorinated methanes Chloroform, Carbon tetra chloride etc) Chlorinate ethane, Chloro propane, chloro butanes, chloroparaffins, chlorination of acetaldehyde (Chloral), alkyl halhides, Chlorobenzene, Ethylene diiodide, Chloroflurocarbon (CFCs).

Preparation of chloroform and chloroflurocarbon (CFCs)

1. $CH_4 + Cl_2 \xrightarrow[- HCl]{\text{photochemically}} CH_3Cl \xrightarrow[- HCl]{Cl_2} CH_2Cl_2 \xrightarrow[- HCl]{Cl_2} CHCl_3$

Methane — Choloroform

2. $CHCl_3 + 2HF \longrightarrow HCF_2Cl + HCl$

Choloroform — chlorodifluoromethane (CFCs)

SULPHONATION AND SULPHATION

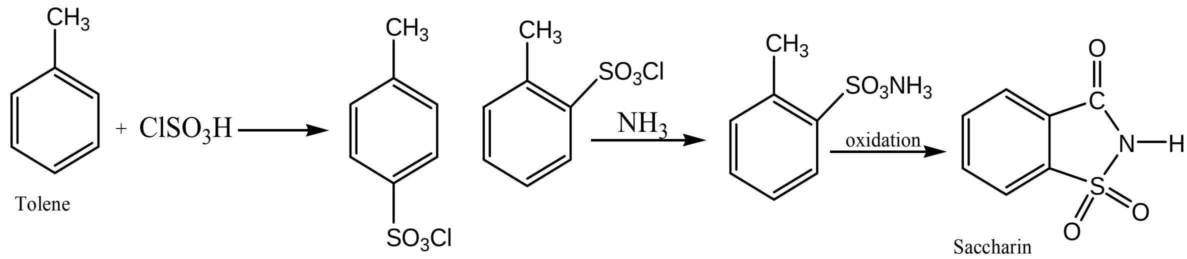
Sulphonation involves the introduction of sulphonic acid group or corresponding salt like sulphonyl halide into a organic compound while sulphationinvolves introduction of $-OSO_2OH$ or $-SO_4$-. Various sulphonating agents are sulphur trioxide and compounds, sulphurdixide, sulphoalkylating agents. Some of the sulphaming agents are sulphamic acid. Apart from sulfonation and sulphamate sulpho chlorinated, sulfoxidation is also used.

Typical application of sulphonation and sulphation are production of lingo sulphonates, linear alkyl benzene sulphonate, Toluene sulphonates, phenolic sulphonates, chlorosulphonicacd, sulphamates for production of herbicide, sweetening agent (sidiumcyclohexysulphamate). Oil soluble sulphonate, saccharin

Preparation of Saccharin

The industrial synthesis entails the reaction of hydrogen chloride with a solution of sulfur trioxide in sulfuric acid. Sulfonation by chlorosulfonic acid gives the ortho and para substituted chlorosulfones. The ortho isomer is separated and converted to the sulfonamide with ammonia. Oxidation of the methyl substituent gives the carboxylic acid, which cyclicizes to give saccharin.

$$HCl + SO_3 \rightarrow ClSO_3H$$



OXIDATION

Oxidation used extensively in the organic chemical industry for the manufacture of a large number of chemicals. Oxidation using oxygen, are combinations of various reactions like oxidation via dehydrogenation using oxygen, dehydrogenation and the introduction of oxygen and destruction of carbon, partial oxidation, peroxidation, oxidation in presence of strong oxidizing agent like $KMnO_4$, chlorate, dichromate, peroxides H_2O_2, PbO_2, MnO_2; nitric acid and nitrogen tertra oxide, oleum, ozone. Some of the important product of oxidation are aldehyde, ketone, benzyl alcohol, phthalic anhydride, ethylene oxide, vanillin, bezaldehyde, acetic acid, cumene, synthesis gas from hydrocarbon,, propylene oxide, benzoic acid, maleic acid, benzaldehyde, phtathalic anhydride. Oxidation maybe carried out either in liquid phase or vapour phase.

Preparation of synthesis gas from hydrocarbon

By using the Fischer–Tropsch process, or Fischer–Tropsch synthesis, is a collection of chemical reactions that converts a mixture of carbon monoxide and hydrogen into liquid hydrocarbons.

$$H_2O + CH_4 \longrightarrow CO + 3\,H_2$$

$$CO + 3\,H_2 \longrightarrow \underbrace{H_2O + CO}_{\text{synthetic gas}}$$

HYDROGENATION

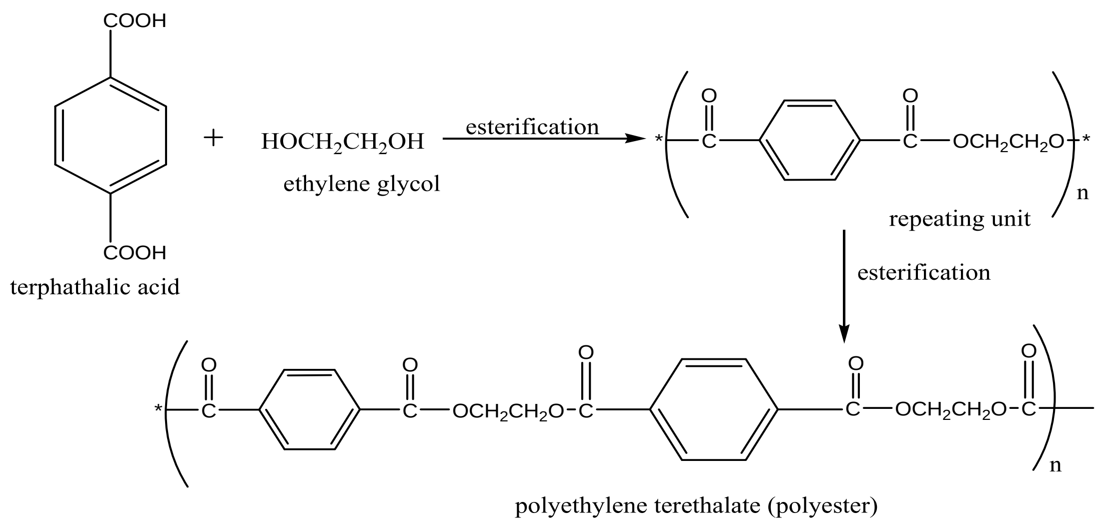
Hydrogenation involves the reaction of a substance with hydrogen in the presence of a catalyst. Some of the other reaction involving hydrogen are, hydrodesulphurisation, hydrcracking, hydro formylation, oxosynthesis, hydroammonylsis, synthesis of ammonia.

Preparation of aldehyde (Hydro-formylation):

$$H_2 + CO + CH_3CH{=}CH_2 \xrightarrow{HCo(CO)PBu_3} CH_3CH_2CH_2CHO$$

ESTERIFICATION

Esterification is an important unit process in the manufacture of polyethylene terephathalate, methyl metha acrylate, cellulose ester in viscose rayon manufacture (xanthation of alkali cellulose with carbon disulphide), nitroglycerine.



HYDROLYSIS

Hydrolysis is used both in inorganic and organic chemical industry. Typical application is in oil and fats industry during soap manufacture where hydrolysis of fats are carried out to obtain fatty acid and glycerol followed by addition of sodium hydroxide to form soap. Other application is in the manufacture of amyl alcohols. Some of the major product using hydrogen is ethylene from acetylene, methanol, propanol, butanol, production of alcohol from olefins (eg. Ethanol from ethylene).

Various types of hydrolysis reaction may be pure hydrolysis, hydrolysis with aqueous acid or alkali, dilute or concentrated, alkali fusion, hydrolysis with enzyme and catalyst.

Preparation of ethanol from ethylene:

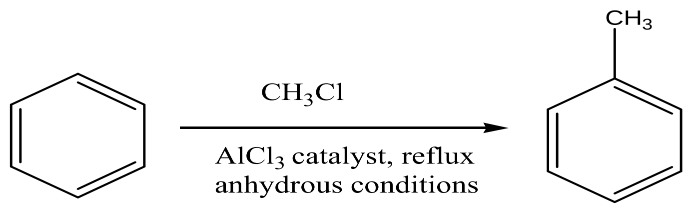
$$CH_2 = CH_{2(g)} + H_2O_{(g)} \xrightleftharpoons{\text{catalyst}} CH_3CH_2OH_{(g)} \qquad H = -45 \text{ kJ mol}^{-1}$$

ALKYLATION

Alkylation involves the introduction of an alkyl radical into an organic compound by substitution or reduction. Products from alkylation find application in detergent, lubricants, high octane gasoline, photographic chemicals, plasticizers, synthetic rubber, chemicals etc. Some of the alkylating agents are olefins, alcohols, alkyl halides. Although sulphuric acid and phosphoric acid were commonly used as catalyst in alkylation process, however due to the corrosive nature of these acid now solid acid catalyst is finding wide application in new alkylation processes.

Preparation of toluene

Alkylation is the transfer of an alkyl group from one molecule to another.



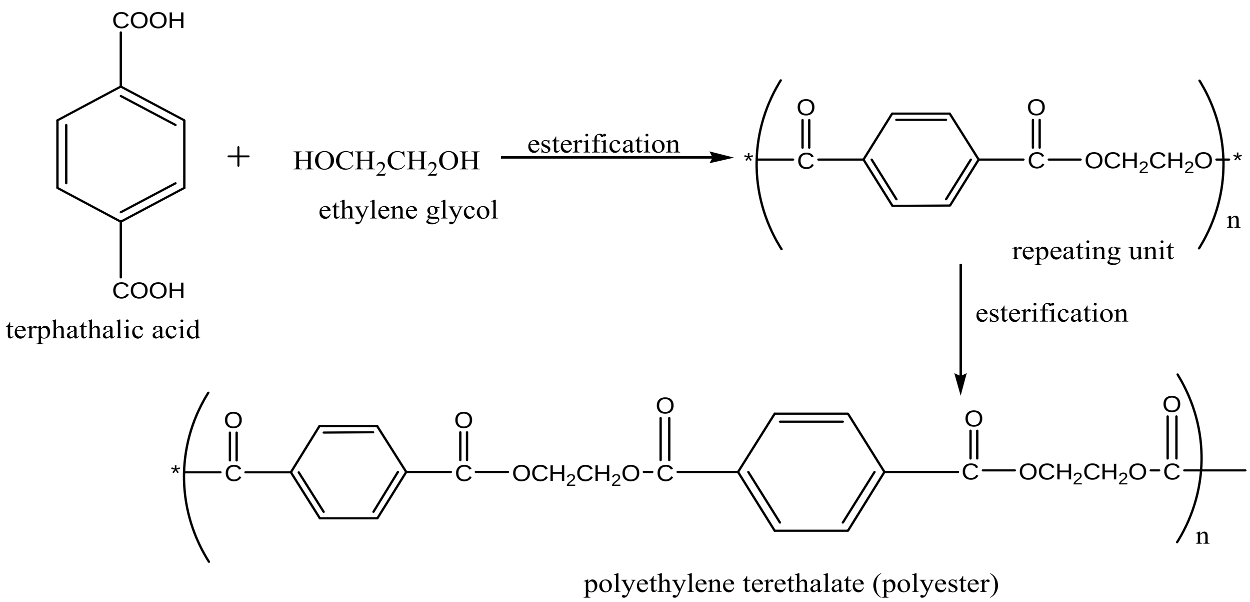
POLYMERIZATION

Polymerization is one of the very important unit processes which find application in manufacture of polymer, synthetic fibre, synthetic rubber, polyurethane, paint and petroleum industry for high octane gasoline. Polymerisation maybe carried out either with single monomer or with comonomer. Polymerisation reaction can be addition or condensation reaction. Various Polymerisation methods may be bulk, emulsion, solution, suspension. Typical important product from polymerization are, Polyethyelene, PVC, poly styrene, nylon, polyester, acryicfibre, poly butadiene, poly styrene, phenylic, urea, melamine and alkyd resins epoxy resin, silicon polymers, poly vinyl alcohol etc.

Preparation of Polyethylene or polythene

It is the most common plastic.

$$n\ CH_2 = CH_2 \longrightarrow \left(CH_2\text{-}CH_2 \right)_n$$



UNIT OPERATIONS IN CHEMICAL INDUSTRIES

Unit operations are very important in chemical industries for separation of various products formed during the reaction. Table M-I 3.3 give the details of unit operation in chemical process industries.

Table M-I 3.3: Unit Operations in Chemical Process Industries

Absorption and stripping	Membrane Process: Reverse osmosis, Ultrafiltration, Dialysis, Electrodialysis, Perevaporation
Adsorption and desorption Pressure Swing adsorption Chromatography	Crushing Grinding, Pulverizing and Screening
Distillation: Batch distillation Flash distillation, Azeotropic distillation, Extractive distillation Reactive distillation	Solid liquid extraction
Evaporation	Striping
Fluidisation	Sublimation
Crystallisation	Solvent extraction
Liquid- Liquid extraction	

DISTILLATION

Distillation has been the king of all the separation processes and most widely used separation technology and will continue as an important process for the foreseeable future [Olujie et al., 2003]. Distillation is used in petroleum refining and petrochemical manufacture Distillation is the heart of petroleum refining and all processes require distillation at various stages of operations.

MEMBRANE PROCESSES

Membrane processes have emerged one of the major separation processes during the recent years and finding increasing application in desalination, wastewater treatment and gas separation and product purification. Membrane technology is vital to the process intensification strategy and has continued to advance rapidly with the development of membrane reactors, catalytic membrane reactor, membrane distillation, membrane bioreactors for wide and varied application [Sridhar, 2009]

Membrane process classified based on driving force. Various type of membrane process and driving force are given in Table M-I 3.4.

Table M-I 3.4: Membrane Processes

Membrane process	Driving force
Reverse osmosis	Pressure difference
Ultrafiltration	Pressure difference
Microfiltration	Pressure difference
Nanofiltration	Pressure difference
Dialysis	Concentration difference
Pervaporation	Concentration difference
Liquid membrane	Concentration difference
Electrodialysis	Electrical potential
Gas Permeation	Concentration difference
Thermo-osmosis	Temperature difference

Based on lower operating costs, comparable capital cost and only slightly product loss (including fuel), membranes have demonstrated a flexible, cost, effective alternative to amine treating for some natural gas processing applications [Cook & Losin, 1995]. Gas membrane and its application areas are mention in Table M-I 3.5.

Membrane distillation is a membrane separation process, which can overcome the limitation of more traditional membrane process. Membrane distillation has significant advantage over other processes, including low sensitivity to feed concentration and the ability to operate at low temperature [Patli and Patil, 2012]. Various type of membrane processes are mention in Table M-I 3.6.

Table M-I 3.5: Gas Membrane Application Areas

Common Gas Separation	Application
O_2/N_2	Generation oxygen enrichment, inert gas
H_2 /hydrocarbons	refinery hydrogen recovery
H_2/CO	Syn. gas adjustment
H_2/N_2	Ammonia purge gas
co_2/hydrocarbons	Acid gas removal from natural gas
H_2O/hydrocarbons	Natural gas dehydration
H_2S/hydrocarbons	Sour gas treating
He/hydrocarbons	Helium separation
He/N_2	HELIUM RECOVERY
Hydrocarbon/ air	Hydrocarbon recovery
H2O/AIR	Air dehumidification

Source: Spillman, 1989

Table M-I 3.6: Various Types of Membrane Processes

Separation Process	Separation Mechanisms	Feed Stream
Microfiltration	Sieving	Liquid or Gas
Ultra-filtration	Sieving	Liquid
Dialysis	Sieving And Sorption Diffusion	Liquid
Reverse Osmosis	Sorption- Diffusion	Liquid
Evaporation	Sorption- Diffusion	Liquid
Gas And Vapour Permeation	Sorption- Diffusion	Liquid or Vapour

ABSORPTION

Absorption is the one of the most commonly used separation techniques for the gas cleaning purpose for removal of various gases like H_2S, CO_2, SO_2 and ammonia. Cleaning of solute gases is achieved by transferring into a liquid solvent by contacting the gas stream with liquids that offers specific or selectivity for the gases to be recovered. Unit operation and is mass transfer phenomena where the solute of a gas is removed from being placed in contact with a nonvolatile liquid solvent that removes the components from the gas.

Solvent: Liquid applied to remove the solute from a gas stream.

Solute: Components to be removed from entering streams.

Some of the commonly used solvents are:

Chemical Absorption

Amine Processes: Mono-ethanol amine (MEA), di-ethanol amine (DEA), tri-ethanol amine (TEA), diglycol amine (DGA), methyl diethanol amine (MDEA)

Carbonate Process: K_2CO_3, K_2CO_3+MEA, K_2CO_3 +DEA, K_2CO_3+arsenic trioxide

Physical Absorption

Polyethylene Glycol Dimethyl Ether (Selexol), N-methyl pyrrolidine,NMP (Purisol), Methanol (Rectisol), Sulphonane mixed with an alkanolamine and water (sulfinol).

ADSORPTION

Adsorption technology is now used very effectively in the separation and purification of many gas and liquid mixtures in chemical, petrochemical, biochemical and environmental industries and is often a much cheaper and easier option than distillation, absorption or extraction. Some of the major applications of adsorption are gas bulk separation, gas purifications, liquid bulk separation, liquid purifications [Keller II, 1995]. One of the most effective method for recovering and controlling emissions of volatile organic compounds is adsorption Some of the commercial adsorbent s are silica gel, activated carbon, carbon molecular sieve, charcoal, zeolites molecular sieves, polymer and resins, clays, biosorbents. some of the key properties of adsorbents are capacity, selectivity, regenerability, kinetics, compatibility and cost [Knaebel, 1995]. Some of the methods used for regeneration of adsorbent are thermal swing, pressure swing, vacuum (special case of pressure swing), purge and gas stripping, steam stripping [Crittenden, 1988]. Commercial adsorption processes is given in Table M-I 3.7. Some of the important criteria of good adsorbent are [Keller II, 1995].

(1) it must selectivity concentrate one or more components called adsorbate to from their fluid phase levels

(2) the ability to release adsorbate so that adsorbent can be reused,

(3) as high as possible delta loading the change of weight of adsorbate per unit weight of adsorbent between adsorbing and desorbing steps over a reasonable range of pressure and temperature

Table M-I 3.7: Commercial Adsorption Processes

Sorbex process	Application
Parex	Separation of paraxylene from mixed C_8 aromatics isomers
MX sorbex	Meta xylene from mixed C_8 aromatics isomers
Molex	Linear paraffins from branched and cyclic hydrocarbons
Olex	Olefins from paraffins
Crsex	Para cresol or meta cresol isomers
Cymex	Para cymene or meta cymene from cymene isomers
Sarex	Fructose from mixed sugar
UOP ISOSIV processor	separation of normal paraffins from hydrocarbon mixture
Kerosene Isoiv process	For separation of straight chain normal paraffins from the kerosene range(C10-C18) used for detergent industry

Pressure swing adsorption (PSA) is based on the principle of relative adsorption strength, is a milestone in the science of gas separation [Shiv Kiran and Chakravarty, 2002]. Some of the commercial application of PSA are air drying, hydrogen purification, bulk separation of paraffins, air separation for oxygen and nitrogen production,

Chromatography is a sorptive separation process. in choromatography feed is introduce in column containing a selective adororbent(stationary phase) and separated over the length of the column by the action of a carrier fluid (mobile phase)that is continually supplied to the column following the introduction of the feed. The separation occurs as a result of the different partitioning of the feed solutes between the stationary phase. The separated solutes are recovered at different time in the effluent from the column [Rangrajan,2010].

CRYSTALLIZATION PROCESS

Crystallization processes are used in the petroleum industry for separation of wax. The process involves nucleation, growth, and agglomeration and gelling. Some of the applications of crystallization is in the separation of wax, separation of p-xylene from xylenes stream. Typical process of separation of p-xylene involves cooling the mixed xylene feed stock to a slightly higher than that of eutectic followed by separation of crystal by centrifugation or filtration.

LIQUID –LIQUID EXTRACTION

Liquid –liquid extraction has been commonly used in petroleum and petrochemical industry for separation of close boiling hydrocarbons. Some of the major applications are:

- Removal of sulphur compound from liquid hydrocarbons
- Recovery of aromatics from liquid hydrocarbon
- Separation of butadiene from C_4 hydrocarbons
- Extraction of caprolactam
- Separation of homogenous aqueous azeotropes
- Extraction of acetic acid
- Removal of phenolic compounds from waste water
- Manufacture of rare earths
- Separation of asphaltic compounds from oil
- Recovery of copper from leach liquor
- Extraction of glycerides from vegetable oil

Some of the important property of a good solvent

- High solvent power/capacity
- High selectivity for desired component
- Sufficient difference in boiling points of the solvent and the feed for effective separation
- Low latent heat of evaporation an specific heat to reduce utility requirement
- high thermal an chemical stability
- Low melting point
- Relatively inexpensive
- Non toxic and non –corrosive

- Low viscosity low interfacial tension

Technological development in unit operations-

- Distillation, Azeotropic, extractive distillation, reactive distillation, membrane distillation
- Random packing to Structured Packing
- Single and two pass to Multiple down comer
- Rasching rings and berl saddles to Intalox sadles, pall rings, nutter rings, half rings, super rings,Fleximax
- Pan park to Wire gauge packing, Goodloe, Mellpark, Flexipack, Gempack, Intalox
- Fixed bed to Fluidised bed reactor
- Conventional reactor to Micro reactor
- Ball mill grinding to Vertical roller mill and press roll Mill
- Open circuit grinding to Closed circuit grinding
- Batch digester to continuous digester
- Low speed and low capacity cipper to High speed chipper and high capacity chipper
- Low speed Paper machine to high speed machine
- Drum displacer, Pressure diffuser, Displacement presses, Combined deknotting and Fine screening,
- High temperature screening before washing, Reverse cleaners
- Adsorption(Olex, Parex and Molex), Crystallisation and Membrane separation processes
- Solvent extraction processes and New solvents
- Conventional distillation Short path distillation, divided wall column
- Conventional bubble cap, sieve plate to valve tray
- Random packing to structured packing
- Axial flow reactor to radial flow reactor
- Conventional instrumentation to smart (intelligent) instrumentation

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