UNIT: II **(DISTILLATION)**

CLASS:18

TOPIC: BASIC PRINCIPELS AND METHODOLOGY OF SIMPLE DISTILLATION, FLASH DISTILLATION

DISTILLATION

Distillation is defined as the separation of the components of a liquid mixture by a process involving vaporization and subsequent condensation at another place.

The distillation process involves two steps; (a) converting a liquid into vapour phase and (b) transferring the vapour to another place and recovering the liquid by condensation. The feed liquid is known as *distilland.* The condensed liquid is known as *distillate* or *condensate.*

If one component is volatile and others are non-volatile, it is possible to separate volatile components from nọn-volatile components by distillation. In such cases, distillation is considered as a separation or purification method. When heat is supplied to a mixture, a more volatile liquid evaporates readily than the less volatile liquid As a result, the condensed liquid consists of a high proportion of highly volatile liquid and less amount of less-volatile liquid." Therefore, distillation is said to be *partial separation method*. The extent of separation is governed by the properties of the components involved and the physical arrangements used for distillation.

In practice, it is difficult to distinguish three processes. namely evaporation, distillation and drying. Only working definitions help in differentiating them.

1. *Distillation* operation is used when condensed vapour is require as a product.

2. *Evaporation* operation is used when the concentrated liquid residue is needed as a product. The temperature of the liquid is maintained below its boiling point. Further vapour is not condensed, unless recovery is essential.

3. *Drying* operation is used when dried solid residue is required as a product.

Applications

Separation of volatile oils: Volatile oils are separated from cloves. anise seeds and eucalyptus leaves by the method of steam distillation.

Purification of organic solvents: Normally, simple distillation method is used for the purification of liquids having single component as a major fraction. Simple distillation method is also used for determining the boiling range of a liquid as per IP. 1996, as a method to decide the purity. Absolute alcohol (100% ethanol) can be obtained by azeotropic distillation.

Manufacture of official preparations: Spirit of nitrous ether and aromatic spirit of ammonia are prepared by simple distillation. Distilled water and water for injection are prepared as per the specifications of pharmacopoeia by simple and compression distillation methods.

Refining of petroleum products: In the petroleum industry, the crude oil is refined into different. fractions using flash distillation. Each Traction is a multicomponent system. Examples are petroleum ether 60, 80 etc.

Recovery of solvents: Solvents are used for extraction of drugs from plant parts and synthetic reaction mixtures. These solvents must be recovered, in order to prevent environmental contamination. The recovered solvent may be recycled for further use.

Quality control methods: Distillation method is used for determining alcohol content in liquid, dosage forms such as elixirs. as per IP. 1996. Azeotropic distillation 'method is used for the determination of water content in a substance using toluene according to IP, 1996.

Separation of drugs obtained from plant and animal sources: Drugs of natural origin (such as plants) are normally extracted using maceration or percolation methods. The menstruum (solvent) used for extraction is distilled off and the active constituents are separated. For example, vitamin A is separated from fish liver oil using the method of molecular distillation.

Purification of drugs obtained from chemical process: Many chemical processes involve the conversion of raw materials into products. The products are separated from the reaction mixture and purified using the methods of distillation.

In order to handle distillation effectively and economically, it is necessary to understand the theory behind this process. Several variables are involved in the process, which are often interrelated. Theory includes the understanding of different factors influencing the distillation process. The laws of conservation of matter and conservation of energy have to be applied

CLASSIFICATION OF DISTILLATION METHODS

- Simple distillation
- Flash distillation
- Fractional distillation
- Azeotropic and extractive distillation
- Distillation under reduced pressure
- Steam distillation
- Molecular distillation
- Destructive distillation
- Compression distillation

SIMPLE DISTILLATION

Simple distillation is a process of converting a single constituent from a liquid (or mixture) into its vapour, transferring the vapour to another place and recovering the liquid by condensing the vapour, usually by allowing it to come in contact with a cold surface.

This process is known *differential distillation*, as distillation is based on the differences in volatilities and vapour pressures of the components in the mixture. This method requires simple apparatus.

Principle: Liquid boils when its vapour pressure is equal to atmospheric pressure. Simple distillation is conducted at its boiling point. The higher the relative volatility of a liquid, the better is the separation by simple distillation. Heat is supplied to the liquid so that it boils. The resulting vapour is transferred to a different place and condensed. If the liquid of interest is volatile and remaining components are non-volatile then simple distillation is a useful means of purification and separation of liquids.

Applications:

(1) Simple distillation is used for the preparation of distilled water and water for injection.

(2) Volatile and aromatic waters are prepared.

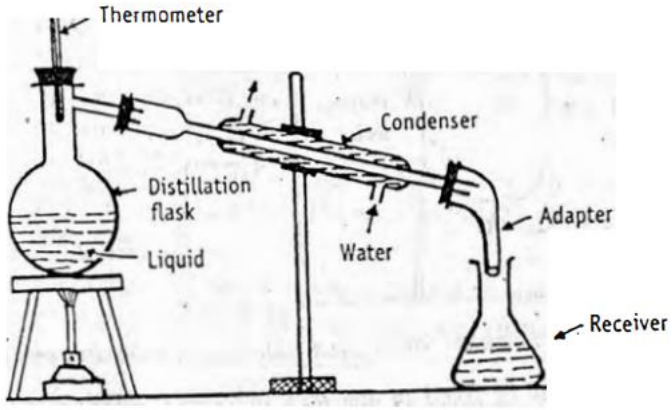
(3) Organic solvents are purified.

(4) A few official compounds are prepared by distillation. Examples are spirit of nitrous ether and aromatic spirit of ammonia.

(5) Non-volatile solids are separated from volatile liquids.

Laboratory Scale Apparatus for Distillation

Assembling of apparatus : The construction of a simple distillation apparatus is shown in Figure. It consists of a distillation flask with a side arm sloping downwards. Condenser is fitted into the side arm by means of a cork. The condenser is usually water condenser. i.e., jacketed for circulation of water. The condenser is connected to a receiver flask using an adapter. On a laboratory scale. the whole apparatus is made of glass.



Apparatus for simple distillation (on laboratory scale).

Procedure : In the laboratory scale, the liquid to be distilled is filled into the flask to one-half to two-third of its volume. Bumping is avoided by adding small pieces of porcelain or porous pot before distillation. A thermometer is inserted into the cork and fixed to the flask. The thermometer bulb must be just below the level of the side arm. Water is circulated through the jacket of the condenser as shown in Figure.

The contents are heated gradually. The liquid begins to boil after some time. The vapour begins to rise up and passes down the side arm into the condenser. The temperature rises rapidly and reaches a constant value. The temperature of the distillate is noted down, which is equal to the boiling point of the liquid.

The vapour is condensed and collected into the receiver. The flame is adjusted so hat the distillate is collected at the rate of one to two drops per second. Distillation should be continued until a small volume of liquid remains in the flask.

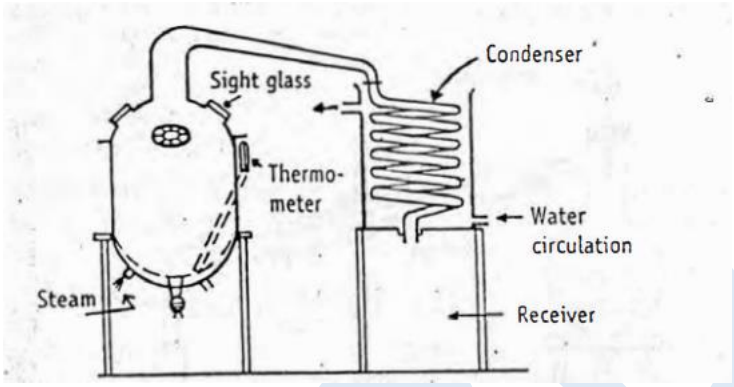
Large Scale Equipment for Simple Distillation

Construction:

A simple still as shown in Figure is used for large-scale distillation. It is made up of stainless steel, copper or any other suitable material. A still of this kind has a limited heating surface and functions

perfectly, with volatile solvents, but is not useful for concentrating dilute solutions. Specially designed stills suited to one product or a group of products are used for frequent and continuous use.

A thermometer is fixed to the still to note the temperature of the boiling liquid. An observation window in the hood is helpful to the operator to see the progress of the distillation and the level of the contents to be distilled. The still is connected to a condenser and then to a receiver. The still is heated using steam. Therefore, a steam inlet at the bottom of the still and an outlet for removing the condensed steam are provided.



Equipment for simple distillation on large scale operation.

Working: A liquid to be distilled is filled into the still to one-half to two-third of its volume. Bumping is avoided by adding small pieces of porcelain or porous pot before distillation. A thermometer is inserted into the still. Water is circulated through the jacket of the condenser as shown in Figure

Steam is passed through the inlet. The contents are heated gradually. The liquid begins to boil after some time. The vapour begins to rise up and passes into the condenser. The temperature rises rapidly and reaches a constant value. The temperature is noted down, which is equal to the boiling point of the liquid.

The vapour is condensed and collected into the receiver. The pas- sage of steam is regulated so that the distillate is collected at a slower rate (a few drops per second). Distillation should be continued until a small volume of liquid remains in the still.

Water stills are used for producing distilled water including water for injection on a continuous basis.

Preparation of Purified Water (BP) and Water for Injection (BP) By Distillation

The principle involved in the preparation of water for injection by distillation is same as that of the simple distillation. However, it is a special case for the following reasons.

(1) Gases dissolved in the raw. water must be removed. These should not be allowed to contaminate the distillate. Such gases include carbon dioxide. Ammonia is the most important gas to be avoided.

(2) The carryover of soluble materials in the droplets must be avoided, particularly if the product is required for use as water for injection.

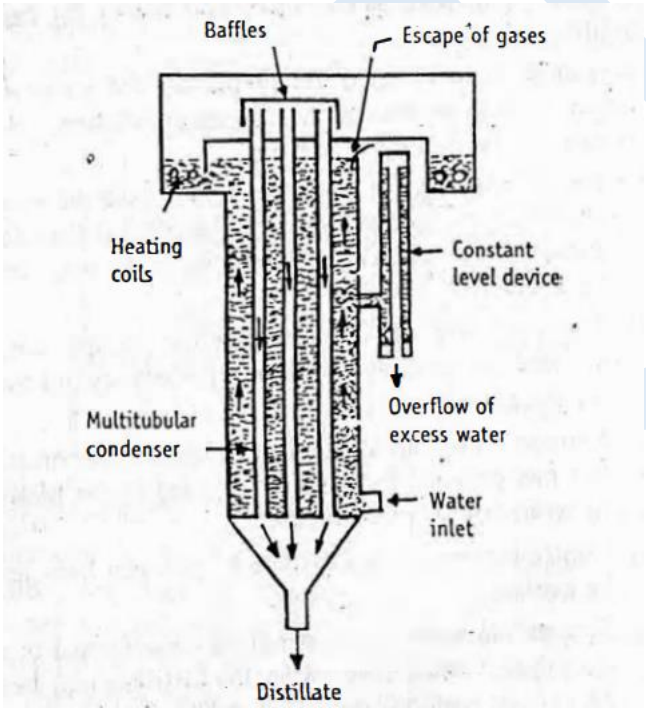
(3) Entrapment of liquid droplets by the vapour must be prevented. For this purpose, baffles are included in the path of the vapour between boiler and condenser.

(4) Contamination of the distillate by pyrogen from feed water must be avoided.

(5) The residue of solids must not be concentrated to a point where hydrolysis occurs. Otherwise, the distillate may be contaminated by volatile material produced during hydrolysis. For example, hydrolysis of chlorides produces hydrochloric acid.

Construction: The arrangement of an apparatus for the continuous production of distilled water or water for injection is shown in Figure. The distillation apparatus consists of a boiler, which may be made of cast iron. Baffles and condenser tubes are made up of stainless steel or monel metal. The top of the condenser jacket is open, so that gases from water can escape into atmosphere. The condenser tubes are vertical and open at both ends, as shown in Figure.

Working: Water (feed) enters at the base of the still and rises in the. jacket, which contains a number of tubes. In the condenser tubes, the condensed liquid descends. The rising feed water gets heated on account of condensate in the tubes. The rate of flow is adjusted in such a way that the water gets heated to 90-95 °C, before it enters the boiler. The dissolved gases in water escape to the atmosphere. The heated water then enters the boiler, in which steam is circulated under pressure through a copper coil. The steam that is obtained by feed water cannot escape except through the condenser tubes, whose upper ends are protruded into the boiler head. The descending steam is condensed into distilled water, which flows from the lower ends of the tubes.



Construction of a distillation unit for the preparation of water for injection.

Advantages: This process is economical as the amount of steam used in coils is reduced on account of preheating of feed water by counter-current flow of the condensate. This also facilitates the escape of dissolved gases without any additional effort.

FLASH DISTILLATION

Flash distillation is defined as a process in which the entire liquid. mixture is suddenly, vaporized (flash) by passing the feed from a high-pressure zone to a low-pressure zone.

Flash distillation is also known as *equilibrium distillation*, i.e., separation is attempted when the liquid and vapour phases are in equilibrium. This method is frequently carried out as a continuous process and does not involve rectification.

Principle: When a hot liquid mixture is allowed to enter from a high-pressure zone into a low-pressure zone, the entire liquid mixture is suddenly vaporised. This process is known as flash vaporisation. During this process the chamber gets cooled. The individual vapour phase molecules of high boiling fraction get condensed while low boiling fraction remains as vapour. This. process requires certain amount of time. Therefore, the liquid and vapour is kept in intimate contact until equilibrium is achieved. The liquid fraction is collected separately. The vapour is separated from the liquid and further allowed to condense.

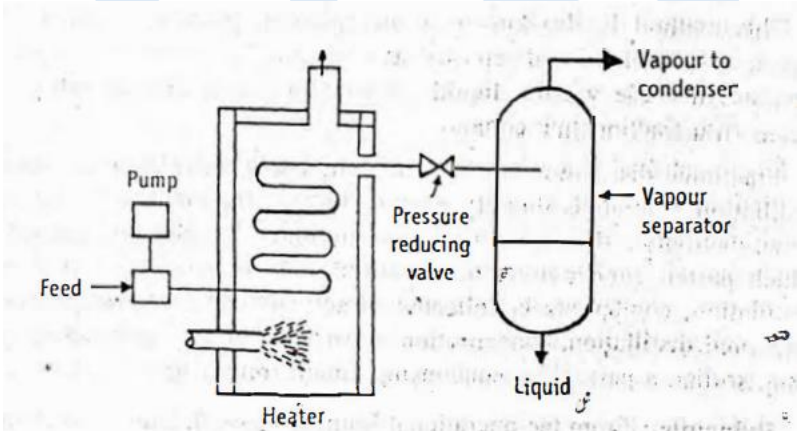
Uses: Flash distillation is used for separating components, which boil at widely different temperatures. It is, widely used in petroleum industry for refining crude oil.

Advantages: Flash distillation is a continuous process. It is' used for obtaining a multi-component systems of narrow boiling range, especially in oil refinery. Examples are petroleum ether 60, 80 etc.tt

Disadvantages: Flash distillation is not effective in separating com ponents of comparable volatility. It is not suitable for two component systems. It is not an efficient distillation when nearly pure components are required, because the condensed vapour and residual liquid are far from pure.

Equipment

Construction: The construction of a flash distillation apparatus is shown in Figure. It consists of a pump, which is connected to a feed reservoir. Pump helps in pumping the feed into the heating chamber which contains a suitable heating mechanism. The other end of the pipe is directly introduced into the vapour-liquid separator through a reducing valve. The vapour outlet is provided at the top of the separator and liquid outlet is provided at the bottom.



Apparatus for flash distillation.

Working: The feed is pumped through a heater at a certain pressure. The liquid gets heated, which enters the vapour-liquid separator through a pressure-reducing valve. Due to the drop in pressure, the hot liquid flashes, which further enhances the vaporisation process. The sudden vaporisation induces cooling. The individual vapour phase molecules of high boiling fraction get condensed, while low boiling fraction remains as vapour. The mixture is allowed for a sufficient time, so that vapour and liquid portions separate and achieve equilibrium. The vapour is separated through a pipe from above and liquid is collected from the bottom of the separator.

By continuously feeding into the still, it is possible to obtain continuous flash distillation. The operating conditions can be adjusted in such a way that the amount of feed exactly equals the amount of material removed. Therefore, vapour and liquid concentrations at any point remain constant in the unit.

MCQ Each carry 1 mark

1. Distillation operation involves one of the following steps
    a. Vaporization
    b. Vaporisation and condensation
    c. Vaporisation, condensation and crystallization
    d. Vaporisation, condensation, crystallization and drying
2. The separation of liquid by distillation is based on one of the following principles
    a. Boiling point
    b. Miscibility
    c. Vapor pressure
    d. Viscosity
3. Distillation does not involve in one of the following processes
    a. Evaporation
    b. Extraction
    c. Purification
    d. Separation
4. Which part in the distillation apparatus represents the heat exchanger?
    a. Adapter
    b. Condenser
    c. Receiver
    d. Still
5. Which one of the methods is also known as differential distillation?
    a. Azeotropic distillation
    b. Molecular distillation
    c. Simple distillation
    d. Steam distillation
6. 2. Porcelain pieces are put into the distillation flask to avoid _____
    a) Overheating
    b) Uniform boiling
    c) Bumping of the solution
    d) None of the mentioned options
7. The process of heating a liquid mixture to form vapours and then cooling the vapours to get pure component is called _____
    a) Crystallisation
    b) Distillation
    c) Chromatography
    d) Sublimation
8. The process of distillation is used for the liquids having _____
    a) Sufficient difference in their boiling point
    b) Sufficient difference in their melting point
    c) Sufficient difference in their solubility
    d) None of the mentioned
9. The residue in the round bottom flask is _____

a) Volatile
b) Non volatile
c) None of the mentioned
d) Volatile & Non volatile

Each carry 2 marks

1. What are constant boiling mixtures? How are they separated?
2. What is meant by constant boiling mixtures? Give two examples
3. Define distillation. Mention two applications of it as per 1P.
4. Define flash distillation. List applications

Each carry 5 marks

1. Describe the construction and working of a distillation apparatus for the preparation of distilled water.
2. Describe the construction and working of a distillation apparatus for the preparation of water for injection.
3. Describe flash distillation

Each carry 10 marks

1. Describe laboratory and large scale process of simple distillation