UNIT-2 : UNIT OPERATIONS

Notes by Mr. V. M. DESAI

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CONTENTS OF SYLLABUS :

1) Size Reduction: Principle, Jaw crusher, ball mill
2) Size Enlargement : Principle, Pellet mill, Tumbling Agglomerators
3) Separation : Magnetic Separation, Froth flotation,
4) Distillation Technique & its Types
   - Simple Distillation
   - Fractional Distillation
   - Distillation under reduced pressure (Vacuum Distillation)
   - Steam Distillation
   - Spinning band Distillation
5) Types of columns & packings
6) Types of condensers

B. Sc. Part-II (Sem-III/Paper-VI)

Industrial Chemistry

Academic Year: 2019-20

New Syllabus w.e.f. June-2019 (as per C.B.C.S. Pattern)

Notes-18/09/2019

"**S**uccess and **E**xcuses do not talk together.
If you want **E**xcuses, forget about **S**uccess.
If you want **S**uccess, do not give **E**xcuses..!"

TEACHING PLAN by V. M. DESAI (2Lectures/Week)

Unit No.	Lect. No.	Topic / Subtopic to be covered	Planned Date	Conducted Date	Teaching Aids Used
2	**UNIT OPERATIONS**				**(6Lectures)**
	1	Introduction: *Brief idea about COs and POs of this paper, Expected Learning Outcome, Syllabus Contents	26/09/19		
	2	**Distillation Technique & its Types** 1) Simple Distillation 2) Fractional Distillation	27/09/19		
	3 4	3) Distillation under reduced pressure(Vacuum Distillation) 4) Steam Distillation 5) Spinning band Distillation	03/10/19 04/10/19		
	5	Types of columns & packings Types of condensers	10/10/19		
	6	**Size Reduction:** Principle, Jaw crusher, ball mill **Size Enlargement :** Principle, Pellet mill, Tumbling Agglomerators **Separation :** Magnetic Separation, Froth flotation,	11/10/19 17/10/19		
	7	**Revision of completed points, Discussion on previous years questions and their answers**	18/10/19		

"Only I Can Change My Life. No One Can Do It for Me."

Size Reduction: Principle, Jaw crusher, ball mill

The process or method or technique used to cut or break the large particles into the smaller particles is known as Size reduction process.

Q. Give different purposes of size reduction. Explain Jaw crusher and Ball mill in detail. 10Marks

Marking Scheme: Purposes = 2M (Any 4)

Jaw crusher = 4M & Ball mill = 4M

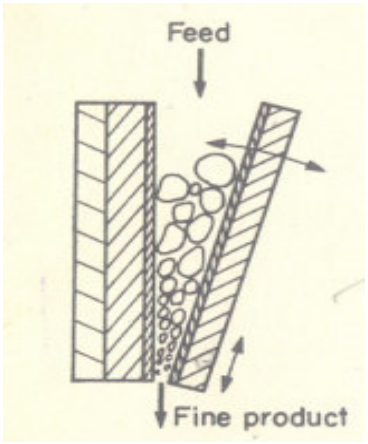
Purpose of Size Reduction: (Write any four purposes for 2Marks)

The following are some of the purposes of size reduction in industry:

1) To produce large solid particles into the smaller particles of workable size.
2) To crush chunks (i.e. thick solid) of raw materials to smaller workable size.
3) To increase surface area of materials by decreasing particle size
4) To increase reactivity of materials by providing large surface area because of its smaller size.
5) To achieve standard specification of size and shape of materials (products)
6) To reduce size of the bulk fibrous materials
7) To improve blending efficiency for formulations

1) **Jaw Crusher**: (Principle=1M, Fig.=2M, Construction=1M)

Principle: When solid is held between two planes and pressure is applied on one plane then solid is fractured and breaks into small fragments when pressure is removed.



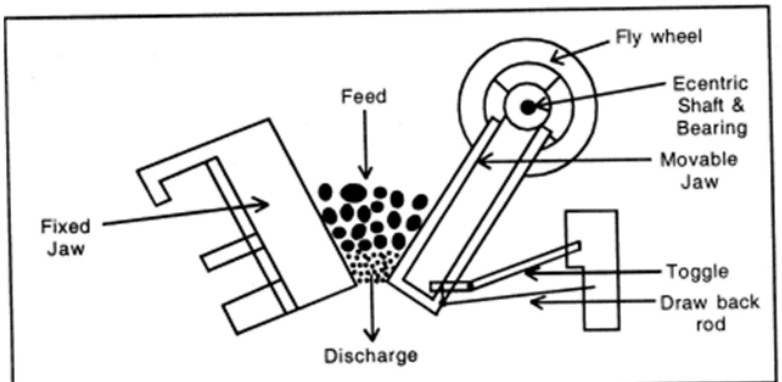


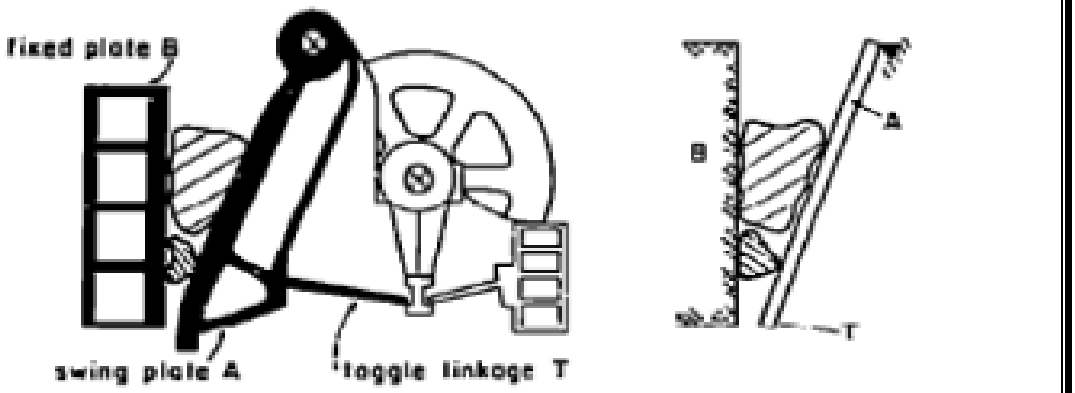
Fig. 2.1 : Schematic diagram of Jaw crusher.

Construction: **(Draw diagram for 2Marks)**

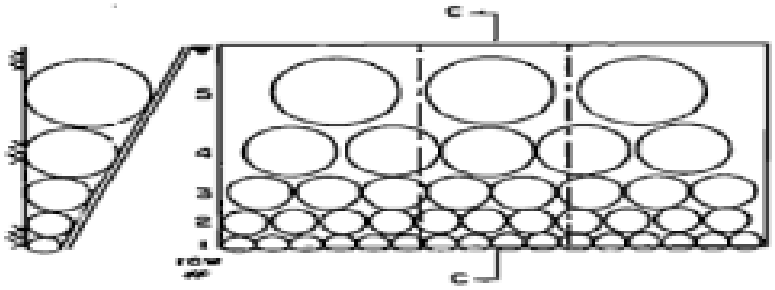
1) Feed is admitted between two jaws, which are open at the top like V as shown in fig.
2) One of the jaws is fixed and vertical, while the other is the swinging jaw (Movable jaw)
3) This fixed jaw reciprocates in a horizontal plane and makes the angle of 20-30° with the fixed jaw.
4) Movable jaw is operated by an **eccentric unit** so as to impart great compressive force on feed (solid materials)
5) Solids which has to be broken is caught between the two jaws

6) Large lumps of solid materials are caught between the upper parts of the jaws
7) Subsequently broken and dropped into the narrower space below
8) Broken pieces are further reduced next time when jaws come closer.
9) No. of strokes given to the movable jaw ranges between 250 to 400 times per minute

Following diagrams are **not expected in exam,** it is only for understanding of concept.



a) Elevation view b) Idealized view



c) Cross Section CC d) Plan View of Plate A

Figure 3 Idealizations of particles within jaw crusher

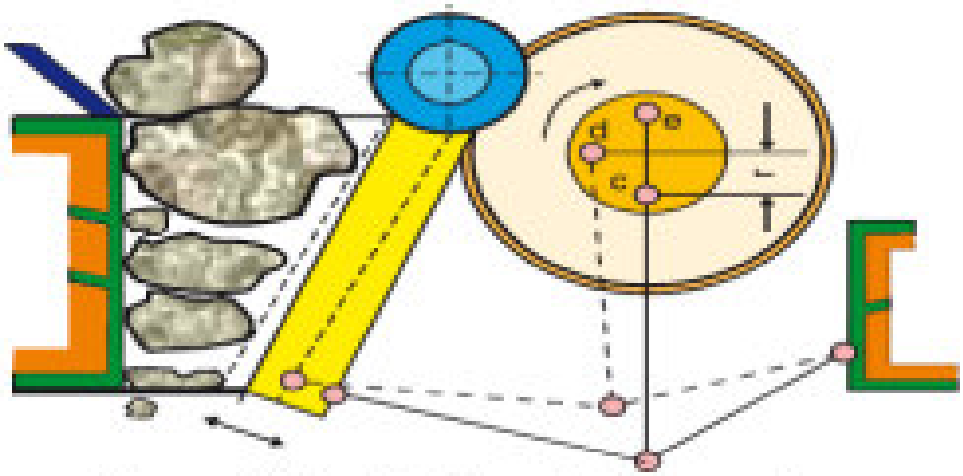


Figure 2 Double Toggle Jaw crusher

2) Ball Mill : (Principle=1M, Fig.=2M, Construction=1M)

Principle: Size reduction is achieved by impact of the balls when they drop from near the top of the shell and energy consumed in lifting the balls is utilized for grinding solid particles

Construction:

1) A ball mill consists of hallow cylindrical or conical shell which is slowly rotating about its horizontal axis.
2) Half of its volume is filled with solid grinding balls (made up of steel) as shown in fig.
3) Size reduction is achieved by impact of the balls when they drop from near the top of the shell and energy consumed in lifting the balls is utilized for grinding solid particles
4) Solid material (feed) is introduced from left and small solid particles (product) is discharged at the right as shown in fig.

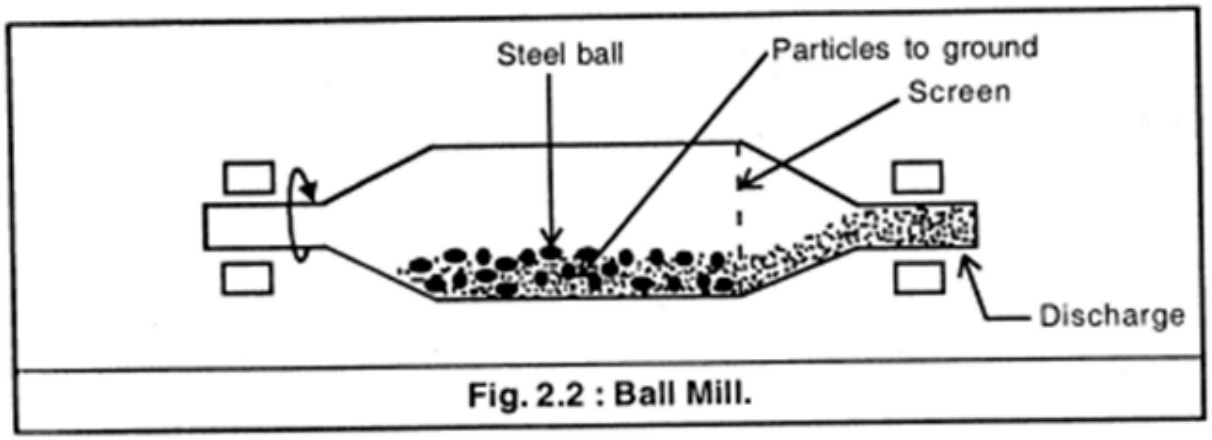
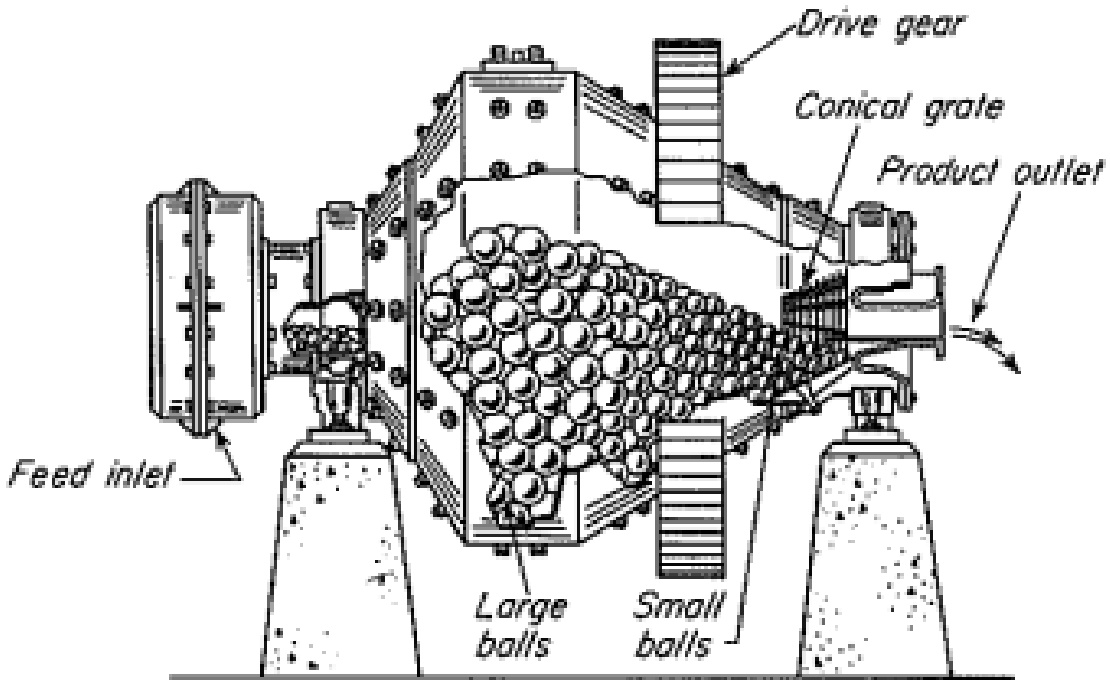


Fig. 2.2 : Ball Mill.

(Draw above diagram in exam, following diagram is given for understanding the concept.)



Size Enlargement / Agglomeration:

The process of combining smaller powder particles to produce particles of desirable (required) size and shape, is known as Size Enlargement process. It is also known as Agglomeration.

Q. Give different purposes of size enlargement. Explain Pellet mill or Tumbling Agglomerators. 10Marks

Marking Scheme: Purposes = 2M (Any 4)

Pellet mill = 4M & Tumbling Agglomerators =4M

Purposes of Size Enlargement / Agglomeration:

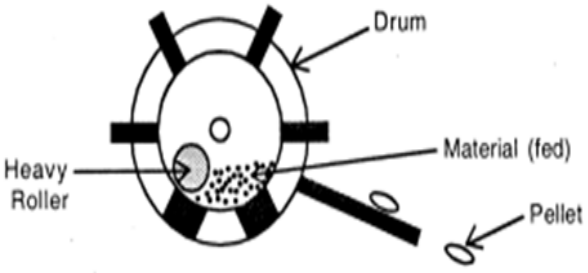
(Write any four purposes for 2Marks)

1) To reduce handling hazards particularly with respect to irritating obnoxious powders.
2) To render particles free flowing.
3) To densify materials.
4) To reduce dusting losses
5) To prevent caking and lump formation
6) To provide definite quantity of units.
7) To produce useful structural forms
8) To create uniform blends of solids which do not segregate
9) To improve appearance of products
10) To remove particles from liquids
11) To permit control over properties of finely divided solids e.g. solubility, porosity, surface volume ratio, heat transfer
12) To separate multicomponent particle size mixtures by selective wetting and agglomeration

Remark: In size enlargement, small particles are gathered into larger, relatively permanent masses in which the original particles can still be identified. The products of size enlargement are either regular shapes e.g. bricks, tiles, tablets, or irregular shapes such as sintered ore.

Pellet Mills: (4Marks)

1) A schematic diagram of a pellet mill is shown in Fig



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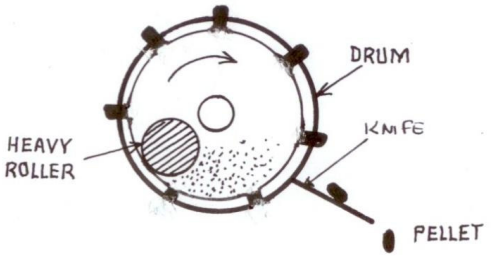


2) Moist Material (fed) is poured through a die containing holes.
3) The die is supplied with power to rotate around a freely rotating roller.
4) The friction (घर्षण) of material in the die holes supplies resistance necessary for compaction.
5) A knife cuts the exudates into pellets. Bonding agents such as glue or starch may be mixed with the feed.

Pellet quality and capacity depends on:

1) Feed (Material) properties e.g. moisture
2) Lubricating characteristics
3) Die characteristics and speed
4) Particle size
5) Abrasiveness (घर्षण)

Applications:

1) To produce KBr pellets used in IR spectroscopy characterization /application
2) To produce animal feed pellets
3) To produce desired pelletized product for characterization

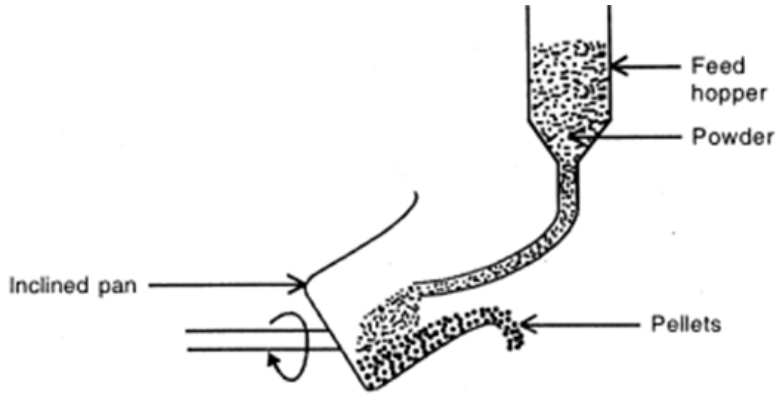
Tumbling Agglomerators (4Marks)

Principle: When powder mass along with suitable binder is mixed and tumbled (i.e. fall suddenly) the binding surface, destructive (tension) forces or cohesive forces are developed which result in agglomeration (एकत्रीकरण).

Two types of tumbling agglomerators are used;

a) Inclined pan agglomerator and
b) Drum agglomerator

Inclined Pan Agglomerator: (2Marks)



1) It consists of inclined pan rotating about its axis.
2) It is inclined to about 45^0 to its axis.
3) It is fed with the powdery raw material from the hopper as shown in fig.

4) Due to rolling action, material layers form over a nucleus particle to form balls. Enlarged balls roll off the pan.
5) Fine powdered materials silts down through the large balls and remain in the pan.

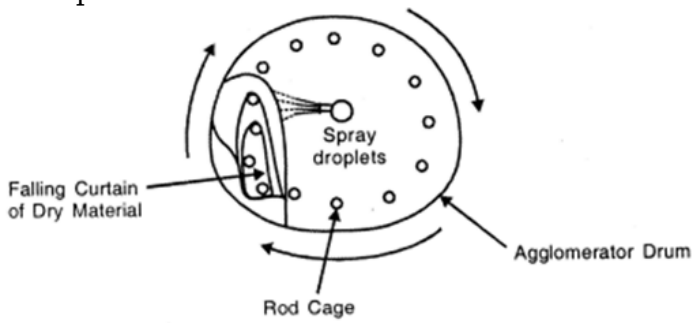
Advantages of an Inclined Pan:

1) Uniform product without need for a screen
2) Low equipment cost which is simple to control
3) Easy observation of the balling action

Disadvantage: It produces dust.

Drum Agglomerator: : (2Marks)

1) A drum agglomerator consists of a drum which itself rotates in clockwise direction.
2) When liquid binder is sprayed onto curtain as shown in fig. and small particles are lifted up by rod cage, it undergoes agglomeration to form larger particles.



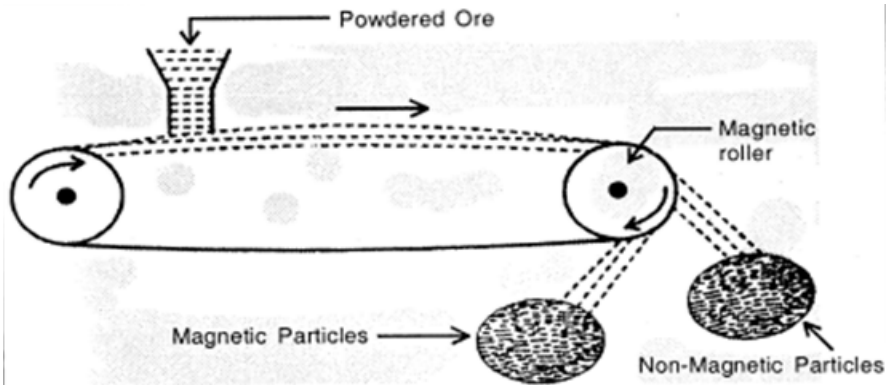
Advantages over a pan agglomerator:

1) It has large capacity
2) It has large retention time if required
3) It has less sensitivity

Disadvantage: By using Drum Agglomerators, particles of various sizes are produced and hence a screening is required to separate enlarged particles from the smaller particles.

Magnetic Separation of Ore: (Not imp in exam)

Principle: This method is based on the differences in magnetic properties of ore and gangue.



i) Either ore or gangue must have magnetic properties, which is attracted by a magnet and get separated.
ii) In this method, an electromagnetic separator consisting of leather or brass belt moving over two rollers one of which of fitted with magnets is used.
iii) When the finely powdered ore is dropped over the moving belt at one the magnetic portion of the ore is attracted and forms one heap near to the roller while non-magnetic gangue falls away and forms another heap. Hence ore can be separated from the impurities.
iv) E.g. It is used for separating iron particles from granules and powders

Froth Floatation Method: (5Marks)

Principle: This method is used for removal of gangue (i.e. unwanted impurities) from sulphide ores. This is based on the principle that the metallic sulphide particles of ore preferentially wetted by oil and the gangue particles by water.

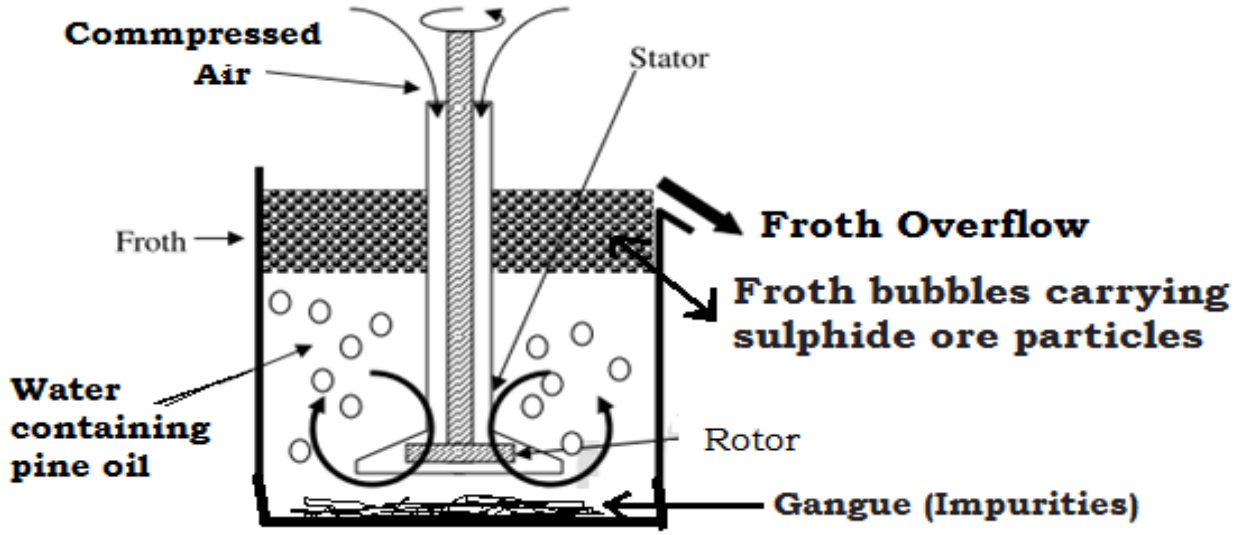
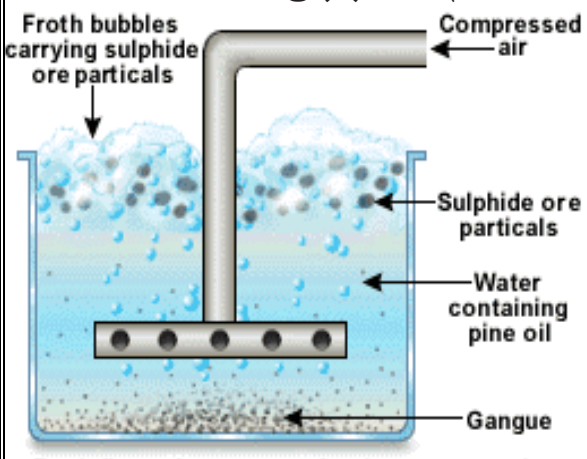


Fig.(a) *(In exam, draw **any one** diagram for 2Marks)*



Froth floatation process for the concentration of sulphide ores.

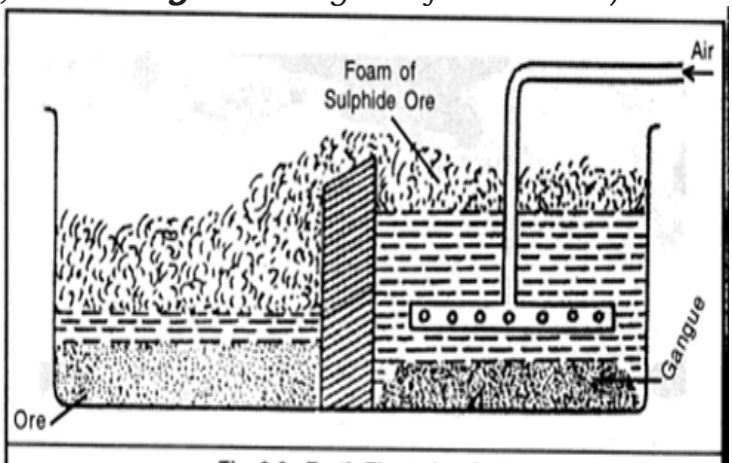


Fig. 2.8 : Froth Floatation Cell.

Fig.(b) **Fig.(c)**

Explanation:

1) In this process, powdered ore is mixed with water. Oil is added. (oil added are pine oil, eucalyptus oil, xanthates or fatty acids).
2) A compressed air is passed as shown in fig.
3) The ore particles become wet by oils while the gangue particles by water.
4) As a result, froth is formed which carries the ore particles.
5) The froth is light and is skimmed off. It is then dried for recovery of the ore particles.

Applications:

1) This method is used for removal of gangue (i.e. unwanted impurities) from sulphide ores.

2) It is widely used for industrial waste-water treatment

3) It is used in mineral processing

4) It is also used in paper recycling process where it remove the hydrophobic contaminants like printing ink.

4.1) Simple Distillation:

a) Definition: The process in which liquid is converted into its vapour phase at its boiling point (B.P.) and the vapour is then cooled back to liquid **on cooling** is known as **'Distillation'**

b) Principle: Distillation involves both **evaporation of a liquid** and **condensation of its vapour**.

c) Use of simple distillation:

Simple distillation is **effective for** separating;

i) two liquids which **differs largely** in their boiling point (B.P.)
ii) liquid from non-volatile solids
iii) volatile liquid from non-volatile oils /liquids

d) Example:

1) Purification of low boiling point (volatile) like CCl_4 (B.P.350K)
2) Purification of high boiling point (non-volatile) like Aniline (B.P.458K)

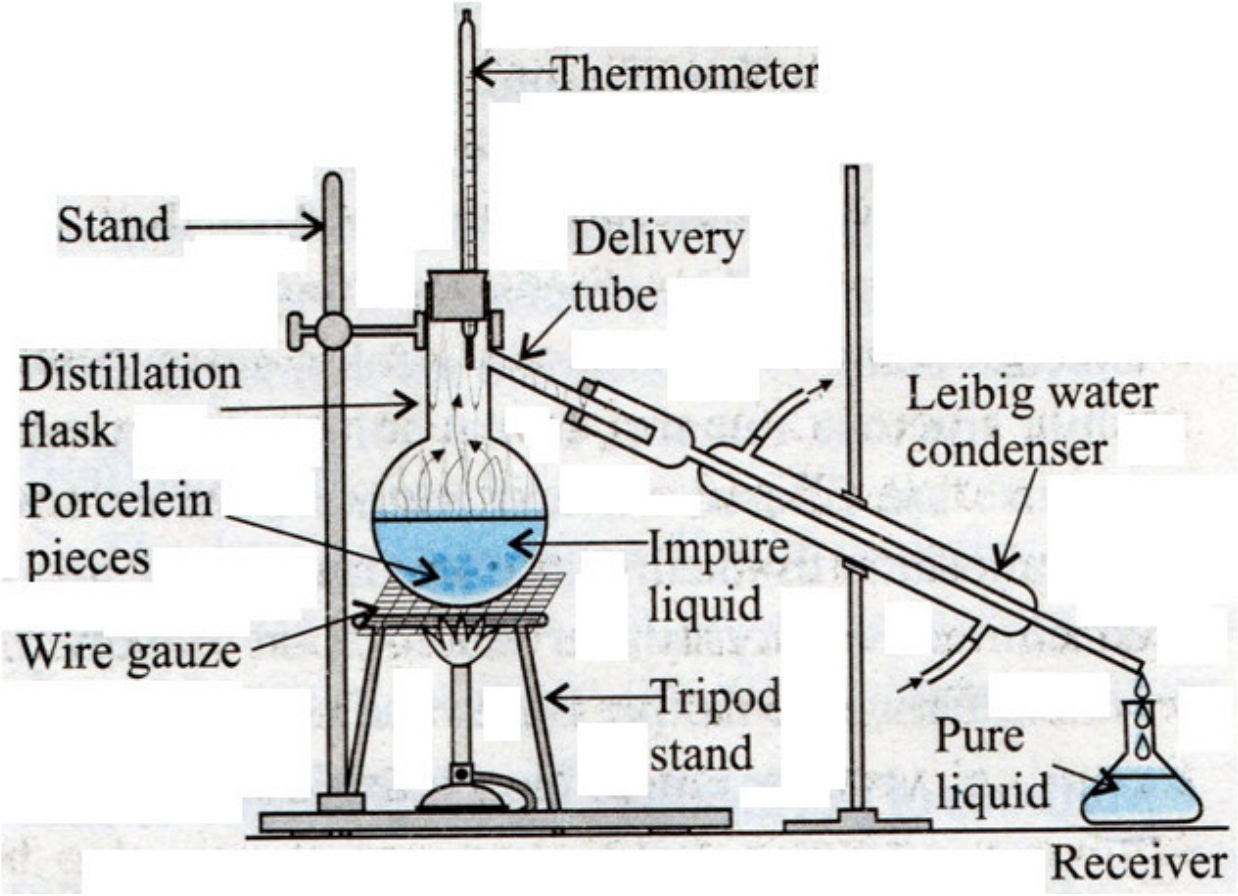


Fig. Simple Distillation

e) Procedure:

1) Impure liquid is taken into the distillation flask fitted with Leibig water condenser, thermometer and receiver as shown in fig.
2) A few pieces of broken porcelain are added into the flask to avoid bumping of solution.
3) Distillation flask is then heated slowly.
4) Liquid distils at its B.P. and is collected in pure form in the receiver.
5) When temperature starts rising above B.P. of given liquid, heating of flask is then stopped and receiver is detached.

f) Disadvantages:

Simple distillation is **not effective for** separating & purifying;

i) liquids which have close boiling point (B.P.)
ii) individual liquid from their mixture containing two or more liquids.

4.2) Fractional Distillation: (Not in syllabus)

Definition: The process of separating and purifying the components of a mixture of **two or more miscible (soluble) liquids** having different boiling point (B.P.) is known as **'Fractional Distillation'**

Principle: The liquid which is **more volatile distills out first** leaving behind the less volatile liquid in the distillation flask.

Use of fractional distillation:

Fractional distillation is **effective for** separating & purifying;

i) liquids which have **close boiling point** (B.P.)
ii) liquids which have close volatilities.
iii) volatile solid from non-volatile oils /liquids
iv) volatile liquid from non-volatile oils /liquids

Example: A mixture of two miscible liquids like acetone and water can be separated by using this method.

Disadvantages:

Fractional distillation is **not effective for** separating and purifying;

i) Compounds which decompose before their M.P. or B.P.
ii) Compounds which are heat sensitive or high boiling liquids
iii) Compound which is highly viscous fluids

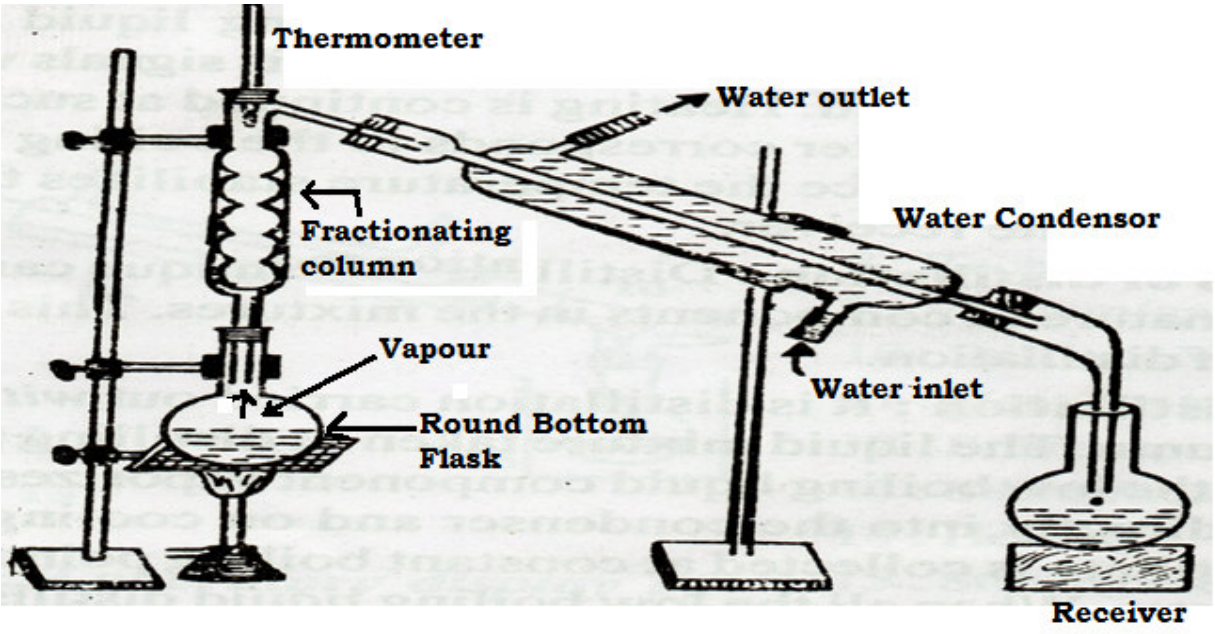


Fig. Fractional Distillation

Q. Write short note on i) Fractionating columns
ii) Types of water condenser

i) <u>Fractionating Columns</u>:

1) It is inserted between distillation flask and water condenser.
2) The column consists of a **number of obstructions** due to which the **cooling area for the hot vapours** increases.
3) **Separation of vapours of different components in the column:** When vapours of both the liquids rise in the column, they come into contact with large area of obstructions. Vapours of liquids having **lower b.p. rise up the column and pass over into the condenser,** whereas the vapours of liquid having **higher b.p. are condensed** in the column itself and condensed liquid trickles down back into the distillation flask. Thus, automatically the separation of two vapours takes place in the column.
4) Fractionating columns are glass columns and are available in various shapes and designs as shown in fig.

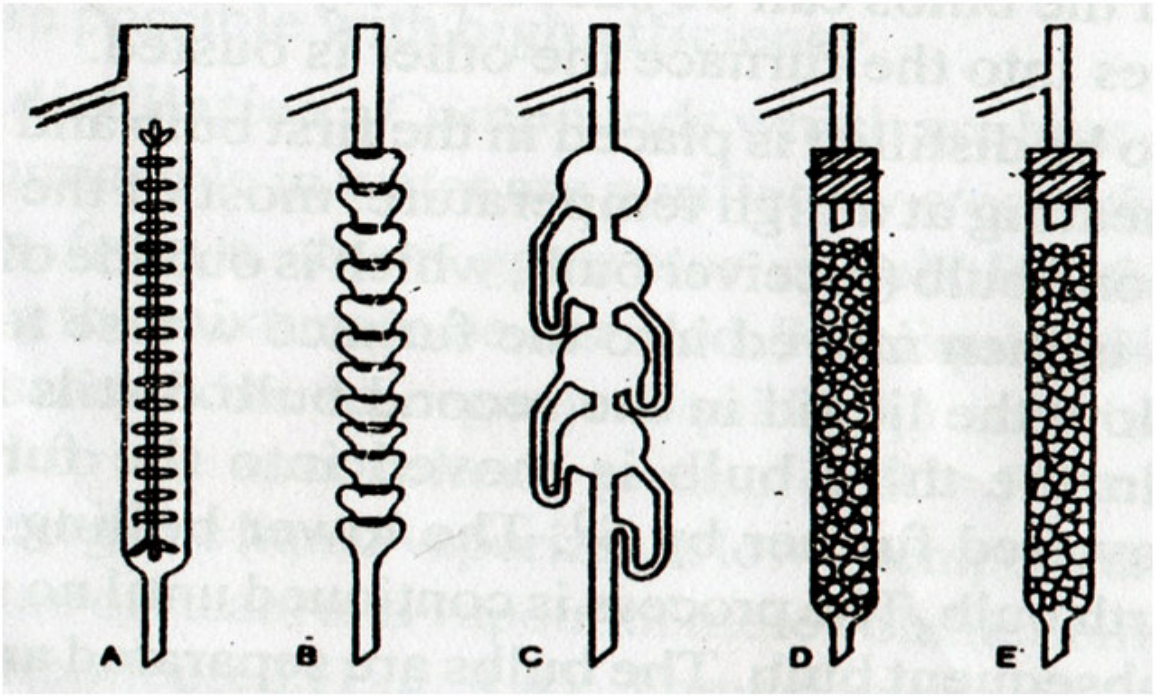


Fig. Types of fractionating columns

Types of condensers: Condensers are used to **cool to condensate the vapour emerging from the column** while passing it.

The different condensers are shown below;

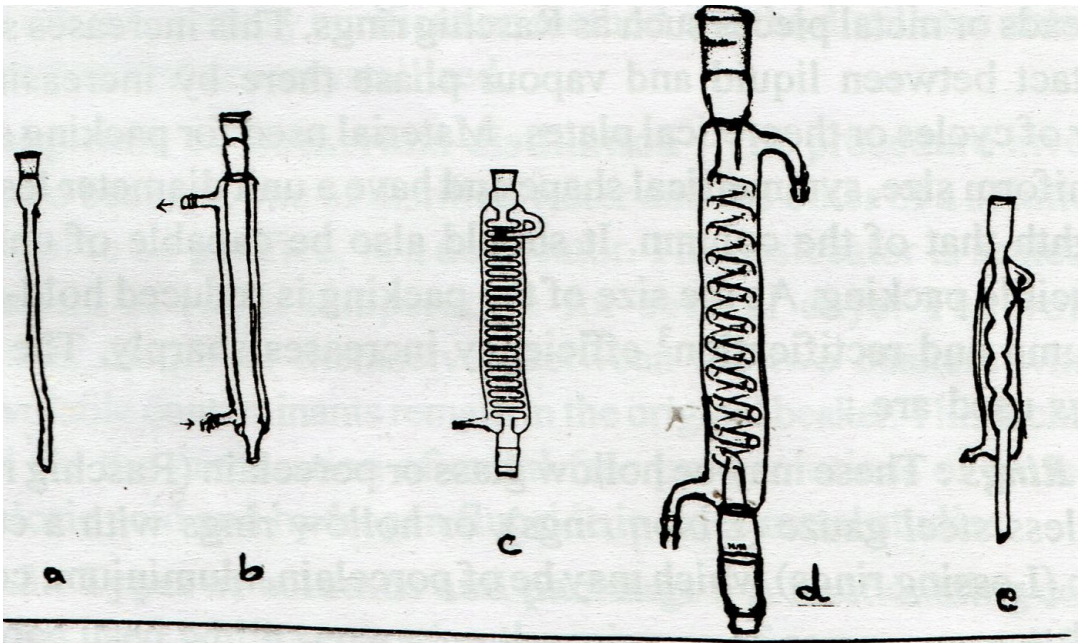


Fig. 4.6 : *Types of distillation condensers*

Where,

a = air condenser which is simple glass tube

b= Liebig condenser which is inner glass tube surrounded by a glass jacket through which water is circulated.

c= coil condenser is an open tube consists of glass coil or spiral tube through which water is circulated.

d = double surface condenser is a tube in which the vapour is condensed between an outer and inner water-cooled jacket.

4.3) Distillation under reduced pressure: MIMP 5Marks

It is also known as 'Vacuum distillation' IMP MCQ

Definition: The process of separating and purifying liquids under reduced pressure which decompose at their boiling point (B.P.) or below their B.P. is known as **'Vacuum Distillation Or Distillation under reduced pressure'**

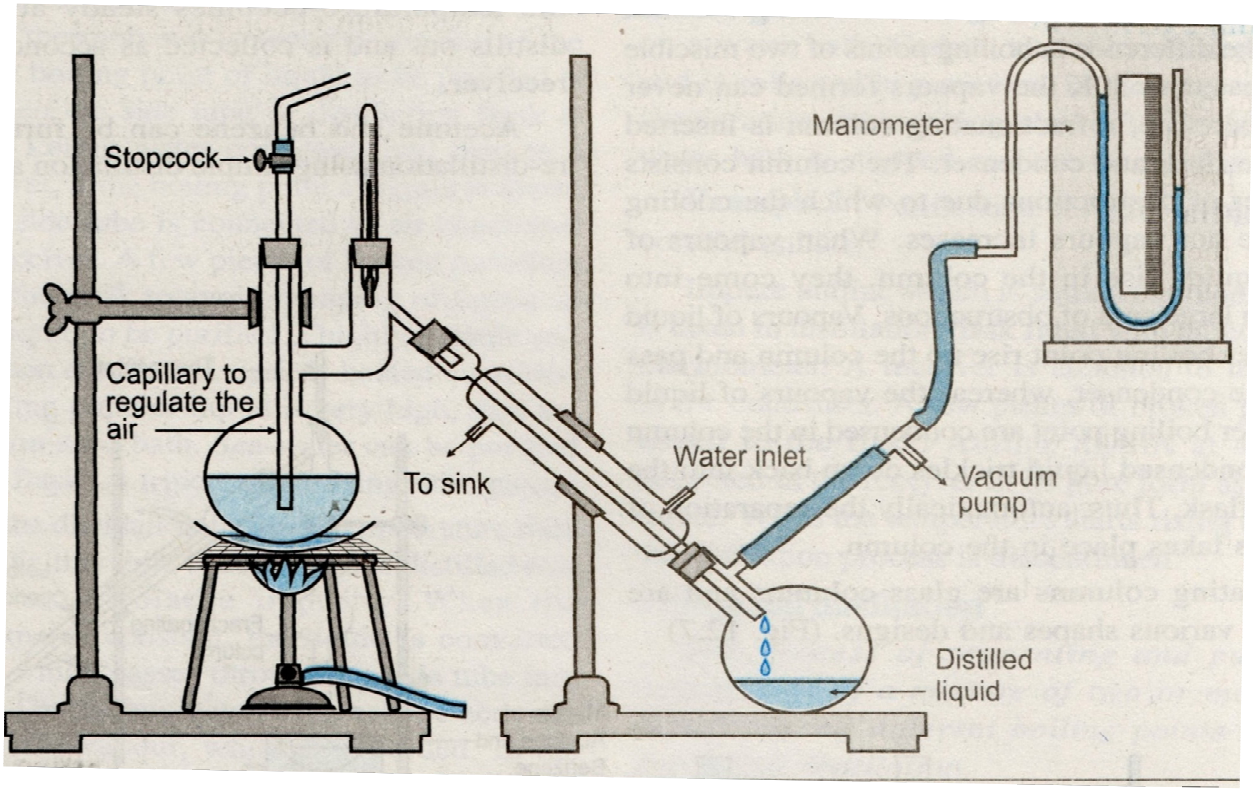
Principle: In this method, the liquid is made to **boil at a temperature lower than its normal boiling point** (B.P.) by **reducing the pressure** on its surface.

If external pressure is reduced then boiling point (B.P.) of liquid is lowered. External pressure can be reduced with the **help of vacuum pump.** Hence this method is also known as **'Vacuum distillation'.**

Use of vacuum distillation: It is **effective for** separating & purifying;

i) Compounds which decompose before their M.P. or B.P.
ii) Compounds which are heat sensitive or high boiling liquids
iii) Compound which is highly viscous fluids

Example: Glycerol can be separated from spent-lye in soap industry by using this method.



Disadvantages:

Vacuum distillation is **not effective for** separating & purifying;

i)liquids which have very close boiling point (B.P.)

ii) Individual liquid from their mixture containing more than two liquids.

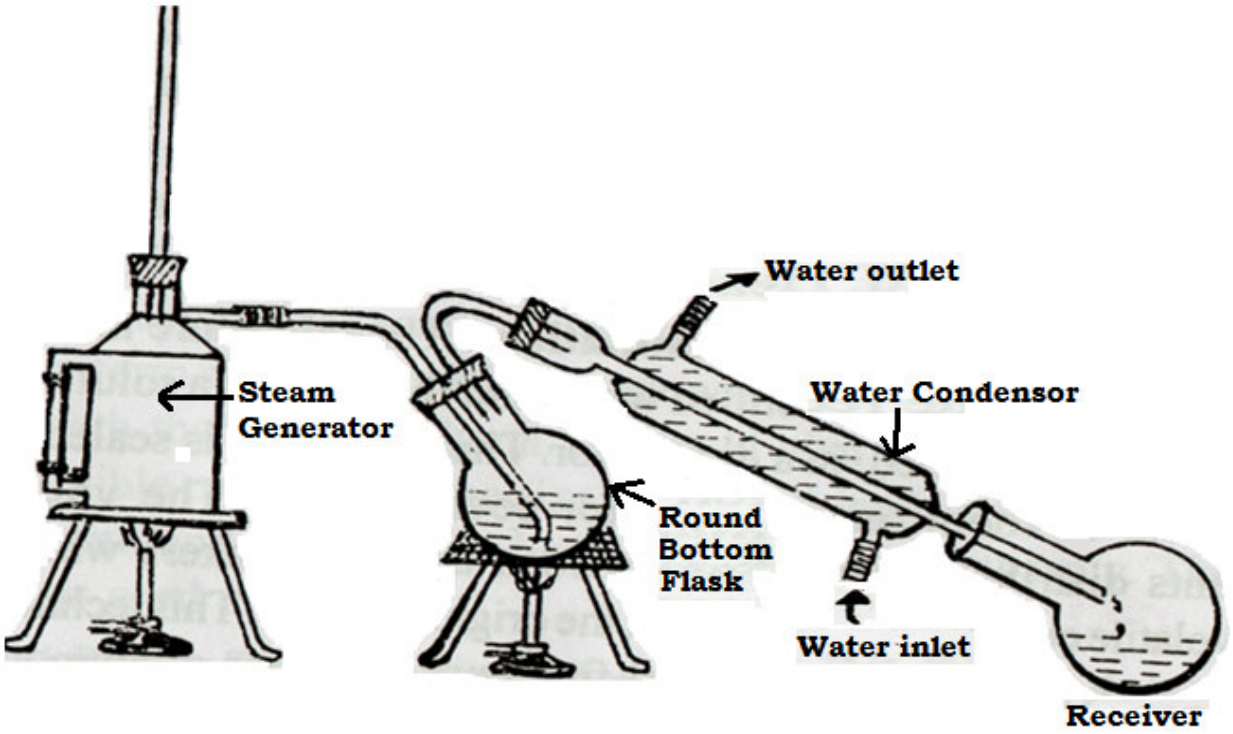
4.4) Steam Distillation: MIMP 5Marks

Definition: The process of separating and purifying liquids which are immiscible (insoluble) with water but are steam volatile is known as 'Steam Distillation' (1/2Mark)

Principle: The liquids which are immiscible with water but are steam volatile are separated by this method. (1/2Mark)

Example: Aniline is separated from a mixture of aniline and water by the method of steam distillation. (1Mark)

Diagram : (2Mark)



Procedure: Steam from a steam generator is passed through heated flask containing the impure liquid. When the sum of vapour pressures of the liquid(P_1) and water(P_2) becomes equal to the atmospheric pressure(P) i.e. when $P = P_1 + P_2$, the impure liquid boils. Since P_1 is less than P, the liquid vapourizes at lower temperature than its boiling point.

The mixture of steam and liquid is distilled put. T liquid is separated from the distillate by using a separating funnel.

Use of Steam distillation: It is **effective for** separating liquids which are immiscible with water but are steam volatile. (1/2Mark)

Disadvantages: (1/2Mark)

Vacuum distillation is **not effective for** separating & purifying;

i) liquids which have very close boiling point (B.P.)

ii) Individual liquid from their mixture containing more than two liquids.

4.5) Spinning Band Distillation: (5Marks)

Definition: The process of separating and purifying close boiling mixtures or liquids by using spinning band columns is known as 'Spinning Band Distillation' (1Mark)

Principle: Spinning band distillation is one of the methods to separate close boiling mixtures by using spinning band columns. (1Mark)

Diagram : (Draw diagram here => 2Mark)

Explanation:

1) Spinning band distillation can be effective as it does not require any solvent or other component for the separation.
2) The separation of close boiling mixtures can be achieved by this with good efficiency.
3) Spinning band distillation creates intimate contact between vapors and condensate in a dynamic process. This speeds up number of condensation-vaporization cycles.
4) The helical pumping action of band forces refluxing liquid down with an intimate contact of the vapor going up the column which results in better efficiency in short distillation column.

Applications: (1Mark)

1) It is used to recycle waste solvents which contain different solvents and other chemical compounds.
2) Spinning band distillation is one of the methods to separate close boiling mixtures by using spinning band columns.

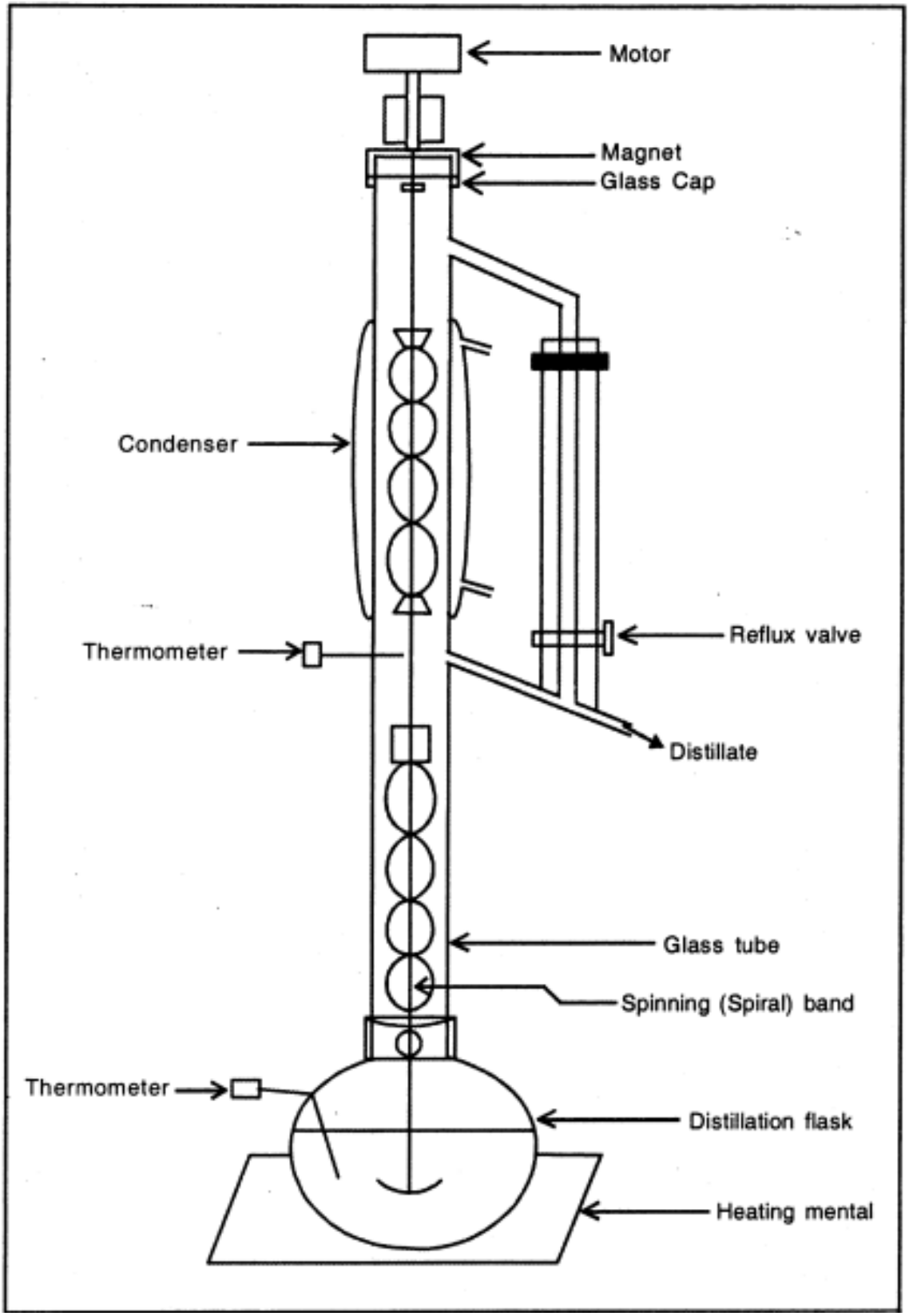


Fig (A) *(In exam, draw **any one** diagram for 2Marks)*

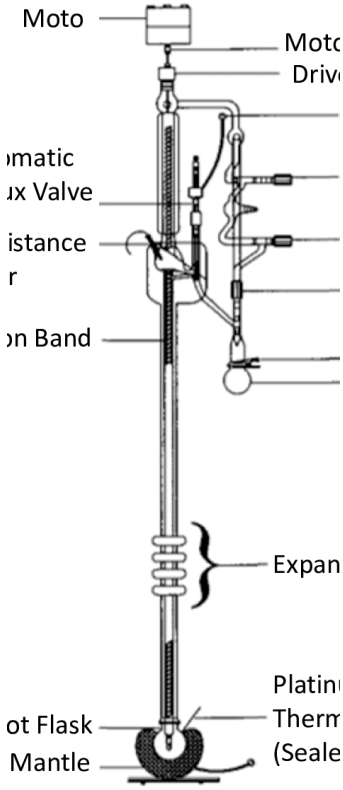


Fig.(B)

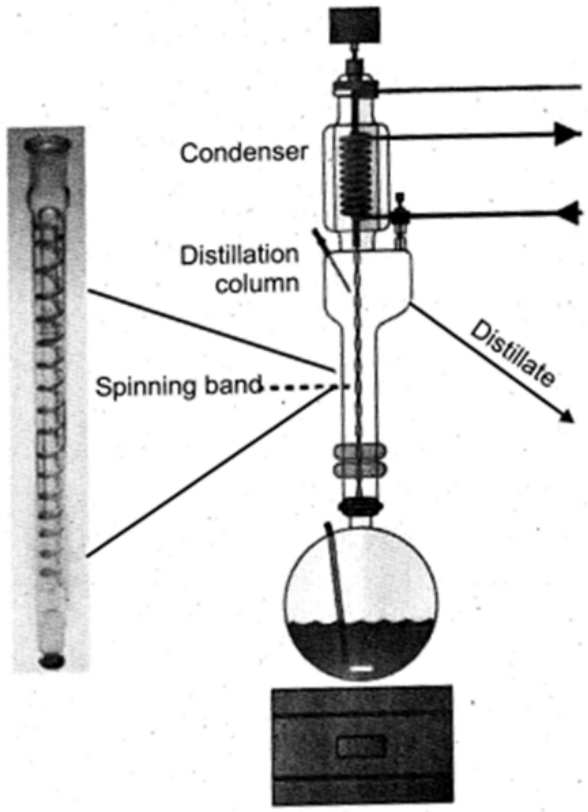


Fig.(C) (Nirali publication)

Previously Asked University Questions:

1) What is distillation? Mention different types of distillation. Explain Vacuum distillation and Steam distillation. 10M
2) With neat labelled diagram, explain following;
   a) Vacuum distillation (5M)
   b) Spinning band distillation (5M) 10M
3) Give different purposes of size reduction. Explain Jaw crusher and Ball mill in detail. 10M
   Marking Scheme: Purposes = 2M (Any 4 purposes)
   Jaw crusher = 4M & Ball mill = 4M
4) Give different purposes of size enlargement. Explain Pellet mill or Tumbling Agglomerators.
   Marking Scheme: Purposes = 2M (Any 4 purposes)
   Pellet mill = 4M & Tumbling Agglomerators =4M
5) Write short note on 'Froth Floatation Method' 5Marks

Multiple Choice Questions (MCQ)

1) Distillation **carried out under reduced pressure** is known as ___**IMP** M-14,O-15

a) Fractional distillation b) Simple distillation

c) Vacuum distillation d) Steam distillation

2) The components of mixture having **close B.P.** are separated by_____ M-14

a) Distillation b) **Fractional distillation**

c) Evaporation d) Sublimation

3) Froth Floatation process is used to concentrate _____ ore.

a) oxide **b) sulphide**

c) magnetic d) non-magnetic

4) Compounds that **decompose at B.P.** are distilled by_____ **IMP**

a) Fractional distillation b) Isopiestic distillation

c) Vacuum distillation d) Steam distillation

e)

5) Unit operation used to purify **mixture of mixable liquids** is_____

a) Filtration b) **Distillation** **c)** Sublimation d)Crystallization

6) Simple distillation is effective for separating liquids,

a) Differing largely in volatilities b) from non-volatile liquids (oils)

c) from non-volatile solids d) **Each one of these**

7) Fractional distillation is effective for separating liquids,

a) Differing in volatilities b) Volatile solid from non-volatile liquids

c) Liquids having close B.P. **d) Each one of these mixtures**

8) Repeated condensation and vaporization occurs in_____

a) condenser b) reboil pot

c) fractionating column d) cold finger

9) _____ is fitted in between distillation flask & condenser.

a) Receiver b) reboil pot

c) fractionating column d) cold finger

(Hint: distillation flask is also called as pot or reboiler)

10) Distillation occurs **at lower than B.P. of liquid** in _____ distillation **IMP**

a) Fractional b) Steam c) Vacuum **d)Both b & c**

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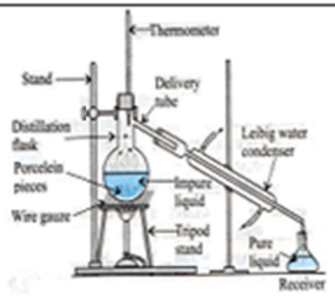
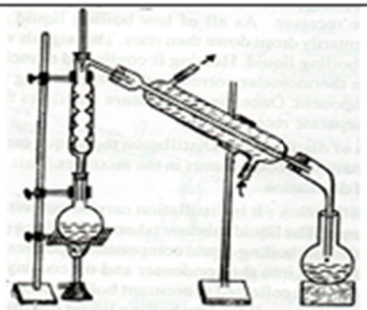
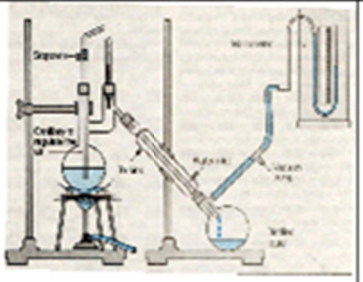
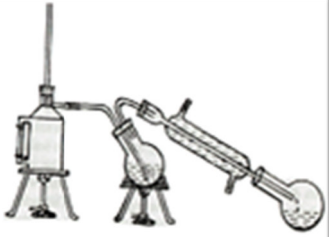
Points	Simple Distillation	Fractional Distillation	Vacuum Distillation (Distillation under reduced pressure)	Steam Distillation
1) Diagram				
2) Assembly	Distillation Flask + Condenser + Receiver	Distillation Flask **+Fractionating Column +** Condenser + Receiver	Distillation Flask + Condenser **+ Suction or Vacuum Pump +** Receiver	**Steam +** Distillation Flask + Condenser + Receiver
3) Effective for separating & purifying (i.e. **Advantages**)	i) two liquids which **differs largely** in their boiling point (B.P.) ii) liquid from non-volatile solids iii) **volatile liquid** from non-volatile oils /liquids	i)liquids which have **close boiling point (B.P.)** ii) **Individual liquid** from their mixture containing two or more liquids. iii) liquids which have close volatilities. iv) **volatile solid** from non-volatile oils /liquids v) **volatile liquid** from non-volatile oils /liquids	i) Compounds which decompose before their M.P. or B.P. ii) Compounds which are heat sensitive or high boiling liquids iii) Compound which is highly viscous fluids	It is **effective for** separating liquids which are immiscible with water but are steam volatile.
4) NOT Effective for separating & purifying (i.e. **Disadvantages**)	i)liquids which have **close boiling point (B.P.)** ii) **Individual liquid** from their mixture containing two or more liquids.	iii) Compounds which decompose before their M.P. or B.P. iv) Compounds which are heat sensitive or high boiling liquids v) Compound which is highly viscous fluids	i) liquids which have very close boiling point (B.P.) ii) Individual liquid from their mixture containing more than two liquids.	i) liquids which have very close boiling point (B.P.) ii) Individual liquid from their mixture containing more than two liquids.