MARRI LAXMAN REDDY INSTITUTE OF PHARMACY
(Approved by AICTE & PCI, New Delhi and Affiliated to JNTUH)
Dundigal - Gandimaisamma (V) &(M), Medchal (Dt), Hyderabad, Telangana - 500 043.

PHARMACEUTICAL ENGINEERING

Lab Manual

B. Pharmacy II Year I Semester

About MLRIP

VISION

To be an educational Institute of par excellence and produce competent pharmacy professionals to serve the community through research and the ever-increasing needs of Industry.

MISSION

1. Imparting quality education and innovative research for various career opportunities.
2. Creating conducive academic environment to produce competent pharmacy professionals.
3. Indoctrination of students adorned with high human values and make them aware of their responsibility as health care professionals.

Program Educational Objectives

PEO 1: To produce graduates with sound theoretical knowledge and technical skills required for their career opportunities in various domains.

PEO 2: To incite the students towards research and to address the challenges with their innovative contributions for the benefit of the mankind.

PEO 3: To instill the essence of professionalism, ethical commitment to become a health care professional with sound integrity and adherence to the core human values in the service of the society.

PROGRAM OUTCOMES

1. **Pharmacy Knowledge:** Possess knowledge and comprehension of the core and basic knowledge associated with the profession of pharmacy, including biomedical sciences; pharmaceutical sciences; behavioral, social, and administrative pharmacy sciences; and manufacturing practices.
2. **Planning Abilities:** Demonstrate effective planning abilities including time management, resource management, delegation skills and organizational skills. Develop and implement plans and organize work to meet deadlines.
3. **Problem analysis:** Utilize the principles of scientific enquiry, thinking analytically, clearly and critically, while solving problems and making decisions during daily practice. Find, analyze, evaluate and apply information systematically and shall make defensible decisions.
4. **Modern tool usage:** Learn, select, and apply appropriate methods and procedures, resources, and modern pharmacy-related computing tools with an understanding of the limitations.
5. **Leadership skills:** Understand and consider the human reaction to change, motivation issues, leadership and team-building when planning changes required for fulfillment of practice, professional and societal responsibilities. Assume participatory roles as responsible citizens or leadership roles when appropriate to facilitate improvement in health and well-being.
6. **Professional Identity:** Understand, analyze and communicate the value of their professional roles in society (e.g. health care professionals, promoters of health, educators, managers, employers, employees).
7. **Pharmaceutical Ethics:** Honour personal values and apply ethical principles in professional and social contexts. Demonstrate behavior that recognizes cultural and personal variability in values, communication and lifestyles. Use ethical frameworks; apply ethical principles while making decisions and take responsibility for the outcomes associated with the decisions.
8. **Communication:** Communicate effectively with the pharmacy community and with society at large, such as, being able to comprehend and write effective reports, make effective presentations and documentation, and give and receive clear instructions.
9. **The Pharmacist and society:** Apply reasoning informed by the contextual knowledge to assess societal, health, safety and legal issues and the consequent responsibilities relevant to the professional pharmacy practice.
10. **Environment and sustainability:** Understand the impact of the professional pharmacy solutions in societal and environmental contexts, and demonstrate the knowledge of, and need for sustainable development.
11. **Life-long learning:** Recognize the need for and have the preparation and ability to engage in independent and life-long learning in the broadest context of technological change. Self-assess and use feedback effectively from others to identify learning needs and to satisfy these needs on an ongoing basis.

BP308P - PHARMACEUTICAL ENGINEERING (Practical) 4 Hours/week

I. Determination of radiation constant of brass, iron, unpainted and painted glass.

II. Steam distillation – To calculate the efficiency of steam distillation.

III. To determine the overall heat transfer coefficient by heat exchanger.

IV. Construction of drying curves (for calcium carbonate and starch).

V. Determination of moisture content and loss on drying.

VI. Determination of humidity of air – i) From wet and dry bulb temperatures –use of Dew point method.

VII. Description of Construction working and application of Pharmaceutical Machinery such as rotary tablet machine, fluidized bed coater, fluid energy mill, de humidifier.

VIII. Size analysis by sieving – To evaluate size distribution of tablet granulations –

+Construction of various size frequency curves including arithmetic and logarithmic probability plots.

IX. Size reduction: To verify the laws of size reduction using ball mill and determining Kicks, Rittinger's, Bond's coefficients, power requirement and critical speed of Ball Mill.

X. Demonstration of colloid mill, planetary mixer, fluidized bed dryer, freeze dryer and such othermajor equipment.

XI. Factors affecting Rate of Filtration and Evaporation (Surface area, Concentration

and Thickness/ viscosity

XII. To study the effect of time on the Rate of Crystallization.

XIII. To calculate the uniformity Index for given sample by using Double Cone

Blender.

1. DETERMINATION OF RADIATION CONSTANT OF BRASS

AIM: -To determine the radiation constant of Brass cylinder.

REQUIREMENT:-

Brass Cylinder with hole or cavity.

Thermometer (360°C)

Burner

Weighing Balance

Stop watch

Screw gauge

Graph Paper

PRINCIPLE: - Heat transfer is a major unit operation of pharmacy. Heat flows from a region of higher temperature to a region of low temperature. Heat may flow by one or more of the three basic mechanisms.

a) Conduction is a process in which heat flow in a body is achieved by the transfer of the momentum of the individual atoms or molecules without mixing.
b) Convection is a process in which heat flow is achieved by actual mixing of warmer portions with cooler portions of the same materials.
c) Radiation is a process in which heat flows through spaces by means of electromagnetic waves. It is also called as thermal radiation.

In this system, the heat loss through convection is neglected, since movement of particles is negligible. As the metal cylinder is freely suspended without any contact with the metal, the heat loss through conduction is considered minimum. Thus heat loss by radiation is highlighted. Stefan-Boltzmann law gives the rate of radiation emitted by a body.

$$q= bAT^4$$

Where, q = Energy radiated per second, W (or J/s)

A = Area of radiating surface, m^2

T = Absolute temperature of the radiating surface, K

B = Constant, $W/m^2.K^4$

The difference in the temperature of hot body and ambient is the temperature gradient for the heat loss by radiation. The radiation constant (α) is calculated using the following equation:

$$Ms\ dq/dt = \alpha A\ [(T_1/100)^4 - (T_1/100)^4)] + \beta A\ (T_1-T_2)^{1.23}$$

Where,

M = Mass of the metal cylinder, w g

s = Specific heat of the metal, J/Kg.K

(dq/dt) = Rate of heat loss by metal cylinder, W/s

T_1 = Temperature of the metal body, K

T_2 = Temperature of the ambient (room temperature), K

α = Radiation constant, $W/m^2.K^4$

β = Convection factor

A = Surface area for heat transfer, m^2

PROCEDURE:-

1) Take a Brass cylinder whose surface is smooth.
2) Measure the weight, area and radius of the cylinder.
3) Place the cylinder on a tripod stand and heat it for about 300^0C
4) Now hold the cylinder with the longs and place on a non conducting surface (wood/glass) without touching any surface.
5) Note the temperature reading for every five minutes, using stop watch.
6) Plot a graph between time on X-axis and temperature on Y axis.
7) Find out the slopes dq/dt at various arbitrary temperature.
8) Calculate radiation constant.

OBSERVATIONS AND CALCULATIONS:

Time, mins	Temperature, °C	Time, mins	Temperature, °C	Time, mins	Temperature, °C

Weight of the brass cylinder =

Height of the brass cylinder =

Diameter of the brass cylinder =

Radius of the brass cylinder =

Surface area of the brass cylinder =

REPORT:-

2. DETERMINATION OF RADIATION CONSTANT OF IRON

AIM: -To determine the radiation constant of Iron cylinder.

REQUIREMENT:-

Iron Cylinder with hole or cavity.

Thermometer (360°C)

Burner

Weighing Balance

Stop watch

Screw gauge

Graph Paper

PRINCIPLE: - Heat transfer is a major unit operation of pharmacy. Heat flows from a region of higher temperature to a region of low temperature. Heat may flow by one or more of the three basic mechanisms.

a) Conduction is a process in which heat flow in a body is achieved by the transfer of the momentum of the individual atoms or molecules without mixing.
b) Convection is a process in which heat flow is achieved by actual mixing of warmer portions with cooler portions of the same materials.
c) Radiation is a process in which heat flows through spaces by means of electromagnetic waves. It is also called as thermal radiation.

In this system, the heat loss through convection is neglected, since movement of particles is negligible. As the metal cylinder is freely suspended without any contact with the metal, the heat loss through conduction is considered minimum. Thus heat loss by radiation is highlighted. Stefan-Boltzmann law gives the rate of radiation emitted by a body.

$$q = bAT^4$$

Where, q = Energy radiated per second, W (or J/s)

A = Area of radiating surface, m^2

T = Absolute temperature of the radiating surface, K

B = Constant, $W/m^2.K^4$

The difference in the temperature of hot body and ambient is the temperature gradient for the heat loss by radiation. The radiation constant (α) is calculated using the following equation:

$$Ms\ dq/dt = \alpha A\ [(T_1/100)^4 - (T_1/100)^4)] + \beta A\ (T_1-T_2)^{1.23}$$

Where,

M = Mass of the metal cylinder, w g

s = Specific heat of the metal, J/Kg.K

(dq/dt) = Rate of heat loss by metal cylinder, W/s

T_1 = Temperature of the metal body, K

T_2 = Temperature of the ambient (room temperature), K

α = Radiation constant, $W/m^2.K^4$

β = Convection factor

A = Surface area for heat transfer, m^2

PROCEDURE:-

1. Take a Iron cylinder whose surface is smooth.
2. Measure the weight, area and radius of the cylinder.
3. Place the cylinder on a tripod stand and heat it for about 300^0C
4. Now hold the cylinder with the longs and place on a non conducting surface (wood/glass) without touching any surface.
5. Note the temperature reading for every five minutes, using stop watch.
6. Plot a graph between temperature on Y axis and time on X-axis.
7. Find out the slopes dq/dt at various arbitrary temperature.
8. Calculate radiation constant.

OBSERVATIONS AND CALCULATIONS:

Time, mins	Temperature, °C	Time, mins	Temperature, °C	Time, mins	Temperature, °C

Weight of the iron cylinder =

Height of the iron cylinder =

Diameter of the iron cylinder =

Radius of the iron cylinder =

Surface area of the iron cylinder =

REPORT:-

3. DETERMINATION OF RADIATION CONSTANT OF COPPER

AIM: -To determine the radiation constant of Copper cylinder.

REQUIREMENT:-

Copper Cylinder with hole or cavity.

Thermometer (360°C)

Burner

Weighing Balance

Stop watch

Screw gauge

Graph Paper

PRINCIPLE: - Heat transfer is a major unit operation of pharmacy. Heat flows from a region of higher temperature to a region of low temperature. Heat may flow by one or more of the three basic mechanisms.

d) Conduction is a process in which heat flow in a body is achieved by the transfer of the momentum of the individual atoms or molecules without mixing.

e) Convection is a process in which heat flow is achieved by actual mixing of warmer portions with cooler portions of the same materials.

f) Radiation is a process in which heat flows through spaces by means of electromagnetic waves. It is also called as thermal radiation.

In this system, the heat loss through convection is neglected, since movement of particles is negligible. As the metal cylinder is freely suspended without any contact with the metal, the heat loss through conduction is considered minimum. Thus heat loss by radiation is highlighted. Stefan-Boltzmann law gives the rate of radiation emitted by a body.

$$q = bAT^4$$

Where, q = Energy radiated per second, W (or J/s)

A = Area of radiating surface, m^2

T = Absolute temperature of the radiating surface, K

B = Constant, $W/m^2.K^4$

The difference in the temperature of hot body and ambient is the temperature gradient for the heat loss by radiation. The radiation constant (α) is calculated using the following equation:

$$Ms\ dq/dt = \alpha A\ [(T_1/100)^4 - (T_1/100)^4)] + \beta A\ (T_1-T_2)^{1.23}$$

Where,

M = Mass of the metal cylinder, w g

s = Specific heat of the metal, J/Kg.K

(dq/dt) = Rate of heat loss by metal cylinder, W/s

T_1 = Temperature of the metal body, K

T_2 = Temperature of the ambient (room temperature), K

α = Radiation constant, $W/m^2.K^4$

β = Convection factor

A = Surface area for heat transfer, m^2

PROCEDURE:-

1) Take a Copper cylinder whose surface is smooth.
2) Measure the weight, area and radius of the cylinder.
3) Place the cylinder on a tripod stand and heat it for about 300^0C
4) Now hold the cylinder with the longs and place on a non-conducting surface (wood/glass) without touching any surface.
5) Note the temperature reading for every five minutes, using stop watch.
6) Plot a graph between time on X-axis and temperature on Y axis.
7) Find out the slopes dq/dt at various arbitrary temperature.
8) Calculate radiation constant.

OBSERVATIONS AND CALCULATIONS:

Time, mins	Temperature, °C	Time, mins	Temperature, °C	Time, mins	Temperature, °C

Weight of the copper cylinder =

Height of the copper cylinder =

Diameter of the copper cylinder =

Radius of the copper cylinder =

Surface area of the copper cylinder =

REPORT:-

4. DETERMINATION OF RADIATION CONSTANT OF UNPAINTED GLASS

AIM:- To determine the radiation constant of un-painted glass.

REQUIREMENT:-

Round bottomed non painted flask

Beaker

Thermometer

Cork

Stand with clamp

Stop watch

PRINCIPLE:- Heat transfer by radiation involves the transfer of energy in the form of electromagnetic waves. All solid bodies radiate energy when their temperatures are above absolute zero. The principle form of radiant energy is thermal energy for industrial applications. The radiant energy emitted by a hot body is expressed by Stefan-Boltzmann law as given below:

$$q = bAT^4$$

Where, q = Energy radiated per second, W (or J/s)

A = Area of radiating surface, m^2

T = Absolute temperature of the radiating surface, K

B = Constant, $W/m^2.K^4$

The difference in the temperature of hot body and ambient is the temperature gradient for the heat loss by radiation. The radiation constant (α) is calculated using the following equation:

$$(M_1s_1 - M_2s_2)\, dq/dt = \alpha A\, [(T_1/100)^4 - (T_1/100)^4)] + \beta A\, (T_1-T_2)^{1.23}$$

Where,

M_1 = Mass of water, w g

M_2 = Mass of round bottom unpainted flask, kg

S_1 = Specific heat of the metal, J/Kg.K

S_1 = Specific heat of the glass, J/Kg.K

(dq/dt) = Rate of heat loss by metal cylinder, W/s

T_1 = Temperature of the metal body, K

T_2 = Temperature of the ambient (room temperature), K

α = Radiation constant, $W/m^2.K^4$

β = Convection factor

A = Surface area for heat transfer, m^2

PROCEDURE:-

1. Take a round bottom flask, measure the diameter, average radius and then surface area in determined whose heat loss to be calculated.
2. The Flask is hanged in air by tying one end for neck with a thread, and other end to a clamp of stand.
3. Boil the water upto its boiling point and taken in to the flask up to the neck level.
4. The flask is fitted with a rubber cork having one hole, which is fitted with thermometer.
5. The temperature is noted for every 5 min. till it reaches to room temperature.
6. A graph is plotted between temperature on Y-axis and time on X-axis.
7. Calculate radiation constant.

OBSERVATIONS AND CALCULATIONS:

Time, mins	Temperature, °C	Time, mins	Temperature, °C	Time, mins	Temperature, °C

Weight, M_1 =

Diameter of the flask, D =

Radius of the flask, R =

Diameter of the neck, d =

Radius of the neck,r =

Surface area,A =

Surface area of the iron cylinder =

REPORT:-

5. DETERMINATION OF RADIATION CONSTANT OF PAINTED GLASS

AIM:- To determine the radiation constant of painted glass.

REQUIREMENT:-

Round bottomed painted flask

Beaker

Thermometer

Cork

Stand with clamp

Stop watch

PRINCIPLE:- Heat transfer by radiation involves the transfer of energy in the form of electromagnetic waves. All solid bodies radiate energy when their temperatures are above absolute zero. The principle form of radiant energy is thermal energy for industrial applications. The radiant energy emitted by a hot body is expressed by Stefan-Boltzmann law as given below:

$$q = bAT^4$$

Where, q = Energy radiated per second, W (or J/s)

A = Area of radiating surface, m^2

T = Absolute temperature of the radiating surface, K

B = Constant, $W/m^2.K^4$

The difference in the temperature of hot body and ambient is the temperature gradient for the heat loss by radiation. The radiation constant (α) is calculated using the following equation:

$$(M_1s_1 - M_2s_2)\, dq/dt = \alpha A\, [(T_1/100)^4 - (T_1/100)^4)] + \beta A\, (T_1-T_2)^{1.23}$$

Where,

M_1 = Mass of water, w g

M_2 = Mass of round bottom unpainted flask, kg

S_1 = Specific heat of the metal, J/Kg.K

S_1 = Specific heat of the glass, J/Kg.K

(dq/dt) = Rate of heat loss by metal cylinder, W/s

T_1 = Temperature of the metal body, K

T_2 = Temperature of the ambient (room temperature), K

α = Radiation constant, $W/m^2.K^4$

β = Convection factor

A = Surface area for heat transfer, m^2

PROCEDURE:-

1. Take a round bottom flask, measure the diameter, average radius and then surface area in determined whose heat loss to be calculated.
2. The Flask neck is covered with a black carbon paper and is hanged in air by tying one end for neck with a thread, and other end to a clamp of stand.
3. Boil the water upto its boiling point and taken in to the flask up to the neck level.
4. The flask is fitted with a rubber cork having one hole, which is fitted with thermometer.
5. The temperature is noted for every 5 min. till it reaches to room temperature.
6. A graph is plotted between temperature on Y-axis and time on X-axis.
7. Calculate the radiation constant.

OBSERVATIONS AND CALCULATIONS:

Time, mins	Temperature, °C	Time, mins	Temperature, °C	Time, mins	Temperature, °C

Weight, M_1 =
Diameter of the flask, D =
Radius of the flask, R =
Diameter of the neck, d =
Radius of the neck,r =
Surface area,A =
Surface area of the iron cylinder =

REPORT:-

VIVA VOCE Questions:

1) What are the mechanisms of heat transfer?
2) Define Radiation, Conduction and Convection.
3) What is Stefan-Boltzmann law?
4) What is the application of radiation (thermal) energy?
5) Define black body.

6. STEAM DISTILLATION- Separation of Turpentine Oil

AIM: To study the process of steam distillation.

PRINCIPLE: Steam distillation is a process of distillation carried with the aid of steam and is used to separate high-boiling substances from nonvolatile materials.

A mixture of immiscible liquids begins to boil when the sum of their vapour pressures is equal to the atmosphere pressure. Incase of a mixture of water and turpentine oil, mixture boils below the boiling point of pure water, though turpentine boils at much higher temperature than that of water. The net result, high boiling substances may be distilled at a temperature much below its boiling point, when water (steam) is used. The turpentine oil is distilled along with water. These liquids are immiscible and separated using a separating funnel. Thus it is possible to separate and purify one liquid from a mixture.

APPLICATIONS:

- It is used for the separation of liquids immiscible with water, like toluene and water.
- This method is used for extracting volatile oils like clove, anise.
- It is useful in purification of liquid with high boiling point, ex- essential oil of almond.
- Aromatic waters are prepared by this method.

ADVANTAGES:

- Volatile oils can be separated at a lower temperature in steam distillation, without any decomposition and aroma.
- If a substance has low volatility, it can be satisfactorily distilled, provided its molecular weight is considerably higher than water.

DISADVANTAGES:

- Steam distillation is not suitable when immiscible liquid and water react with each other.

ASSEMBLY OF APPARATUS FOR STEAM DISTILLATION:

The assembly of apparatus for steam distillation on laboratory scale consists of a metallic (Copper) steam can fitted with a rubber cork having two holes. Through one of the hole, a long tube is passed so as to reach almost the bottom of the steam generator.

This tube acts as a safety tube, so that in case the pressure inside the steam generator becomes too much, water will be forced out of it and the pressure will be relieved. Moreover, when steam starts coming out from the safety tube; it indicates that the steam can is almost empty.

Through another hole, a bent tube is passed. The other end of the bent tube is connected to the flask containing non aqueous liquid through a rubber bung. This tube should reach almost the bottom of the flask.

Through another hole of the rubber bung, a delivery tube is inserted which connects the flask and the condenser. The condenser is connected to a receiver flask using an adaptor.

PROCEDURE:

Simple Distillation

- 50 ml of turpentine oil is placed in a 250 ml round bottom flask.
- The cork carrying thermometer is fitted to the flask. The tip (mercury) should be in front of the side tube of the flask.
- The flask is heated by Bunsen burner. The turpentine oil gets heated and after some time starts boiling.
- The temperature (T_1) is noted at which turpentine oil distills. This is boiling point of turpentine oil and remains constant.
- Simple distillation is continued to collect 25ml of condensate.

Steam Distillation

- 30 ml of turpentine oil is placed in 250 ml round bottom flask.
- 100 ml of water is added to the above flask
- The steam can (or round bottom flask) is filled with water and assemble the remaining.
- Both the steam can and flask are heated simultaneously, so that steam flows uniformly through the mixture.
- The mixture gets heated. After some time, it starts boiling.
- The temperature (T_2) at which boiling occurs is noted.

- Steam carries the vapour of oil and passes into the condenser where condensation takes place.
- Condensate is collected and oil is separated from water by using separating funnel.
- The weight of turpentine oil (w_2) and water (w_1) layers are noted.
- The percent efficiency is calculated and reported.

OBSERVATION AND CALCULATION:

Boiling point of turpentine oil by simple distillation, T_1 °C=

Boiling point of the mixture by steam distillation, T_2 °C=

Decrease in boiling point, $T_3= T_1-T_2=$

Theoretical recovery ratio of turpentine oil to water=

Weight of water obtained, $w_1=$

Weight of turpentine oil obtained, $w_2=$

Practical recovery ratio of turpentine oil to water, $w_2/w_1=$

Percent efficiency of steam distillation, (Practical recovery/ Theoretical recovery) X 100 =

REPORT:

VIVA VOCE Questions:

1) What is distillation?
2) Define steam distillation.
3) In which condition organic liquid vaporize in steam distillation?
4) What are the basic applications of steam distillation?
5) What type of substances can be extracted by steam distillation?
6) Mention one example of purification by steam distillation.

7. DETERMINATION THE OVERALL HEAT TRANSFER COEFFICIENT BY HEAT EXCHANGER

AIM: -to determine the overall heat transfer coefficient by heat exchanger.

REQUIREMENT:-

Steam generator, Beaker, bent tube, water condenser, Thermometer,

PRINCIPLE: Heat transfer by convection is involved between two liquids, when these are separated by glass wall. The differences in the modes of feeding largely determine the efficiency of a heat process. Heat exchangers are the devices used for transferring heat from one fluid (hot gas or steam) to another fluid (liquid) through a metal wall. When the feed of hot fluid is passed through one end of the apparatus and the cold fluid is passed through the other end, this arrangement is known as counter-current or counter flow method. The overall heat transfer coefficient of a glass tube is mathematically expressed for a counter current flow as:

$$U = \frac{Q}{A \times \Delta t_{av}}$$

Where, Q = amount of heat transferred, W (J/s)

A = Surface area of the glass tube, m^2

Δt_{av} = temperature gradient, K

U = overall heat transfer coefficient, W/ m^2·K

In the above equation Q represented as:

$$Q = (Q_1+Q_2) / 2$$

$Q_1 = M_1.L+M_1.s.\ \Delta t_1$

$Q_2 = M_2.s.\ \Delta t_2$

Where,

M_1 = Mass of condensed steam, kg

M_2 = mass of circulated water, kg

S= specific heat of steam, J/kg.K

L= latent heat of vaporization of water, J/kg

t_1 = temperature drop on steam, K

t_2 = temperature rise on the circulating water side, K

The temperature gradient, Δt_{av} is expressed as:

$$\Delta t_{av} = (\Delta t_1 + \Delta t_2)/2$$

Δt_1= difference in temperature on steam side, K

Δt_2 = difference in temperature on cold water side, K

The water condenser used in the laboratory or distillation is an example or the counter current flow of liquids and heat transfer. Thus, overall heat transfer coefficient is determined using water condenser.

PROCEDURE:-

1. The length and diameter of the plain water condenser is determined.
2. Using the plain water condenser, the distillation apparatus is assembled.
3. The inlet of water condenser is connected to the tap. The outlet of the condenser is placed in the (2 lit) beaker.
4. The temperature of the tap water inlet is noted.
5. The steam generator is heated so that steam is produced. As the steam is generated, the steam thermometer shows constant temperature. This temperature is noted.
6. As the process continues, a steady state situation is obtained. At this stage, heat transfer measurements are made.
7. The condensate begins to collect into an empty beaker. At the same time, the water is collected from the outlet into an empty vessel.

8. After a lapse of time (i.e 5 or 10 or 15 mins) , collecting of condensate is stopped by removing the beaker from the condenser.
9. Exactly at the same time, collecting of tap water from the outlet is also stopped, by removing the bottle from the rubber tubing.
10. The condensate is swirled and the temperature is noted. The quantity of condensate is measured and recorded.
11. The tap water collected into the bottle is also swirled and temperature is noted. The quantity of the tap water is measured and recorded.

OBSERVATIONS AND CALCULATIONS:

Diameter of the condenser, d=

Radius of the condenser, r=

Length of the condenser, l=

Area of the condenser, $A=2\pi rl$

Latent heat of vaporization of water, L= 226.1 J/kg

Specific heat of steam, s= 4190 J/kg.K

Heat loss by steam, Q_1=

Heat gain by tap water, Q_2=

Heat transferred, Q=

	Steam temp. A	Tap water temp. (outlet) b	Condensate temp. c	Tap water temp. (inlet),d
Temperature, °C				
Temperature, K				
Difference in temperatures	Δt_1 = (a-b) =		Δt_2 = (c-d) =	
Average temperature, Δt_{av}	$(\Delta t_1 + \Delta t_2)/2$			

REPORT:

VIVA VOCE Questions:

1) What are the processes of heat transfer?
2) Define convection with example.
3) What is counter-current or counter flow method?
4) What is the application of heat transfer in pharmacy?
5) What is the equation of overall heat transfer?

8. CONSTRUCTION OF DRYING CURVE(Calcium carbonate)

AIM:- To dry calcium carbonate slurry and plot the rate of drying curves.

REQUIREMENTS:- Calcium carbonate, hot air oven, balance, petri plate, beaker etc.

PRINCIPLE:- Drying is defined as removal of small amounts of water or other liquid from a material by application of heat. Drying rate relationship can be studied considering a simple model which mimics the conditions of a dryer. In this model, the wet slab to be dried is placed in a tray whose bottom end sides are insulated. The air is blown over the solid under the constant drying conditions (like temperature, humidity, pressure etc). the superficial water diffuses through the surrounding air film and is carried away rapidly by the moving air stream. Then water diffuses from the interior of the solid to the surface. This process continues until bound water gets evaporated. Then the material attains equilibrium moisture content.

Rate of drying of this process can be determined by periodically weighing the calcium carbonate slurry. The difference in the weights of two successive weighing gives the loss of moisture content, i.e., amount dried. The following equation is used to calculate rate of drying:

$$\text{Rate of Drying} = \frac{\text{Weight of water removed}}{\text{Weight of dry powder X time of drying X surface area exposed}} \quad g/g.h.cm^2$$

PROCEDURE:-

1. The petri plate is weighed and the weight is recorded as W_1.
2. 15.0 g of calcium carbonate is transferred into a beaker. Water (about 30 ml) is added slowly to prepare slurry.
3. The calcium carbonate slurry is transferred into the petri plate.
4. Filling must be done in such a way that $3/4^{th}$ of the volume of the stainless steel plate is filled with slurry.
5. The weight of petri plate plus slurry is taken and recorded as W_2.
6. The plate containing slurry is placed in hot air oven, whose temperature must be maintained at 60°C.
7. The time is noted soon after placing plate containing slurry in hot air oven.
8. After 15 mins, the wt. of plate with slurry is taken. The weight is recorded in table.

9. Again the petri plate containing slurry is placed in the dryer. (petri plate should be immediately placed back into the dryer , otherwise temperature decreases enormously and results will be erroneous.
10. Step 8 and 9 are repeated until constant weight is obtained.
11. The rate of drying is calculated.
12. A graph is plotted by taking free moisture content (weight of water) on x-axis and rate of drying on y-axis.

OBSERVATION AND CALCULATIONS:-

SN	**Time, mins**	**Wt. of empty petridish (W_1) gm**	**Wt. of petridish + $CaCo_3$ (W_2) gm**	**Wt. of petridish + $CaCo_3$ + Water (W_3) gm**	**Wt. of petridish + $CaCo_3$ + Water after drying (W_4) gm**	**Moisture evaporated in 15 mins time interval (W_3- W_4)**	**Total moisture content (W_4- W_2)**	**Moisture content on drying basis (W_4- W_2)/ (W_2- W_1)**	**Average moisture content**	**Rate of drying gm/min/cm^2 (W_3 - W_4) / Time X Area**
1										
2										
3										
4										
5										
6										
7										
8										
9										

1. Wt. of empty petridish, W_1 =
2. Wt. of petridish + $CaCo_3$, W_2 =
3. Wt. of petridish + $CaCo_3$ + Water ,$W_3 = W_2 - W_1$ =
4. Diameter of petridish, d = cm

5. Radius of petridish, r = d/2 = cm
6. Area of petridish, $A = \pi r^2$ = cm^2
7. Moisture content at '0' time =
8. Moisture content at '15' mins =
9. Average moisture content =

REPORT:-

VIVA VOCE QUESTIONS:

1) Define drying.
2) What is bound water and unbound water?
3) Define EMC and FMC.
4) What is constant rate drying period?
5) What is falling rate period?
6) How can we calculate rate of drying?

9. CONSTRUCTION OF DRYING CURVE(Starch)

AIM:- To dry starch slurry and plot the rate of drying curves.

REQUIREMENTS:- starch, hot air oven, balance, petri plate, beaker etc.

PRINCIPLE:- Drying is defined as removal of small amounts of water or other liquid from a material by application of heat. Drying rate relationship can be studied considering a simple model which mimics the conditions of a dryer. In this model, the wet slab to be dried is placed in a tray whose bottom end sides are insulated. The air is blown over the solid under the constant drying conditions (like temperature, humidity, pressure etc). the superficial water diffuses through the surrounding air film and is carried away rapidly by the moving air stream. Then water diffuses from the interior of the solid to the surface. This process continues until bound water gets evaporated. Then the material attains equilibrium moisture content.

Rate of drying of this process can be determined by periodically weighing the calcium carbonate slurry. The difference in the weights of two successive weighing gives the loss of moisture content, i.e., amount dried. The following equation is used to calculate rate of drying:

$$\text{Rate of Drying} = \frac{\text{Weight of water removed}}{\text{Weight of dry powder X time of drying X surface area exposed}} \quad g/g.h.cm^2$$

PROCEDURE:-

1. The petri plate is weighed and the weight is recorded as W_1.
2. 15.0 g of starch is transferred into a beaker. Water (about 30 ml) is added slowly to prepare slurry.
3. The starch slurry is transferred into the petri plate.
4. Filling must be done in such a way that $3/4^{th}$ of the volume of the stainless steel plate is filled with slurry.
5. The weight of petri plate plus slurry is taken and recorded as W_2.
6. The plate containing slurry is placed in hot air oven, whose temperature must be maintained at 60°C.
7. The time is noted soon after placing plate containing slurry in hot air oven.
8. After 15 mins, the wt. of plate with slurry is taken. The weight is recorded in table.
9. Again the petri plate containing slurry is placed in the dryer. (petri plate should be immediately placed back into the dryer , otherwise temperature decreases enormously and results will be erroneous.
10. Step 8 and 9 are repeated until constant weight is obtained.
11. The rate of drying is calculated.
12. A graph is plotted by taking free moisture content (weight of water) on x-axis and rate of drying on y-axis.

OBSERVATION AND CALCULATIONS:-

SN	Time, mins	Wt. of empty petridish (W_1) gm	Wt. of petridish + $CaCo_3$ (W_2) gm	Wt. of petridish + starch + Water (W_3) gm	Wt. of petridish + starch + Water after drying (W_4) gm	Moisture evaporated in 15 mins time interval (W_3- W_4)	Total moisture content (W_4- W_2)	Moisture content on drying basis (W_4- W_2)/ (W_2- W_1)	Average moisture content	Rate of drying gm/min/cm^2 (W_3 - W_4) / Time X Area
1										
2										
3										
4										
5										
6										
7										
8										
9										

1. Wt. of empty petridish, W_1 =
2. Wt. of petridish + starch , W_2 =
3. Wt. of petridish + starch + Water ,$W_3 = W_2 - W_1$ =
4. Diameter of petridish, d = cm
5. Radius of petridish, r = d/2 = cm
6. Area of petridish, $A = \pi r^2$ = cm^2
7. Moisture content at '0' time =
8. Moisture content at '15' mins =
9. Average moisture content =

REPORT:-

10. DETERMINATION OF HUMIDITY OF AIR BY DEW POINT METHOD

AIM: -To determine humidity of Air by using Dew point method.

REQUIREMENT:-

Round bottom flask having polished surface.

Thermometer

Tripod Stand

Stirrer

Humidity Chart

PRINCIPLE: - The **Dew point temperature** (DPT) is the temperature to which a mixture of air-water vapour must be cool (at constant pressure and constant water vapor content) in order to reach saturation. Formation of mist and disappearance of mist are considered and dew point is determined. Dew point temperature is noted on the temperature axis (x-axis) and moved vertically on the psychrometric chart. The intersect point at saturated curve (100%) is identified. The coordinates of the point (temperature, K, humidity) are noted. The y-axis point is the humidity of air. These values are substituted in the equation:

Percent relative humidity= (humidity of air/ humidity of saturated air) X 100

PROCEDURE:-

1. Take a polished round bottom flask (100 ml) and fill water upto 2/3 of its volume
2. Place the flask on tripod stand and fix it.
3. Hang a thermometer hanging from the main stand such that the thermometer's bulb is dipping into the water in the vessel.
4. Drop small pieces of ice cubes into the vessel one by one slowly, under continuous stirring of the water with the help of glass rod or magnetic stirrer. Continue the stirring until a film of moisture (mist) is firmed on the polished surface of flask.
5. Note the temperature of this stage which is dew point and record it.
6. Denote humidity of air by using dew point with the help of humidity.

OBSERVATION AND CALCULATION:-

Trails	**Dew point, °C**		**Humidity**
	Mist appearance	**Average value**	

From humidity chart, humidity of saturated air dew point =

Percent relative humidity =

REPORT:-

11. DETERMINATION OF HUMIDITY OF AIR BY WET AND DRY BULB TEMPERATURE

AIM: -To determine humidity of air by wet and dry bulb temperature.

REQUIREMENT:-

Thermometer

Humidity Chart

PRINCIPLE: - Humidity is defined as the amount of water vapor present in a unit volume of air, usually expressed in kilograms per cubic meter. It can also defined as the ratio of mass of water present in the air to the mass of dry air. **Relative humidity** is defined as the ratio of actual humidity to the saturation humidity at a temperature. The **dry-bulb temperature** (DBT) is the temperature of air measured by a thermometer freely exposed to the air but shielded from radiation and moisture. Temperature is usually measured in degrees Celsius (°C), Kelvin (K), or Fahrenheit (°F). The **Wet bulb temperature** (WBT) is the temperature of air measured by a thermometer having a wick moistened with distilled water. Temperature is usually measured in degrees Celsius (°C), Kelvin (K), or Fahrenheit (°F). Wet-bulb temperature is largely determined by both actual air temperature (dry-bulb temperature) and the amount of moisture in the air (humidity). At 100% relative humidity, the wet-bulb temperature equals the dry-bulb temperature.

PROCEDURE: - Dry bulb temperature is the temperature, which we will get, if a thermometer is placed in a sample of air. In a sample of air, if we place the thermometer, then we shall see somewhere there is the mercury level and that gives the temperature of the sample. So that is dry bulb temperature or simply we can call it temperature of the sample. For wet bulb temperature we have a similar thermometer but the bulb of this thermometer is wrapped with the help of a layer of cotton and then, this cotton is kept moist with the help of distilled water. We shall get two readings- one from the dry bulb thermometer and another from wet bulb thermometer. These readings are used for finding humidity from adiabatic cooling line in the psychometric charts.

OBSERVATIONS AND CALCULATIONS:-

Average dry bulb temperature		Average dry bulb temperature		Humidity	Percent relative humidity
°C	°F	°C	°F		

REPORT:-

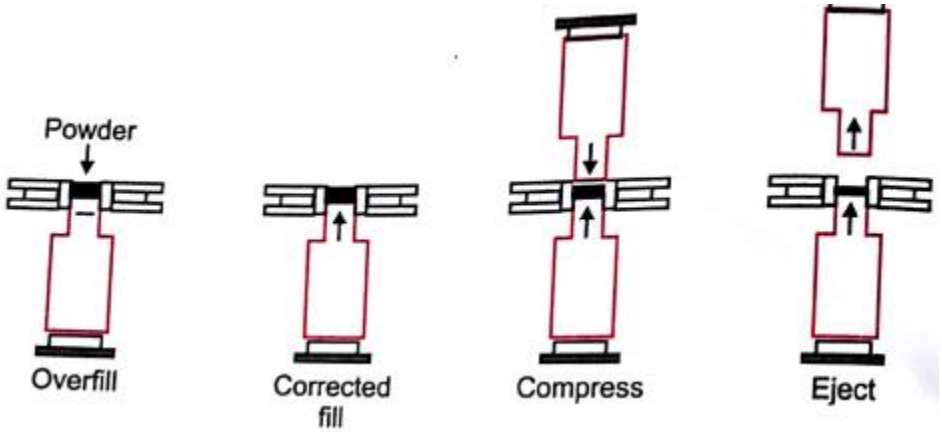
VIVA VOCE Questions:

1) Define humidity and relative humidity.
2) What is wet bulb temperature and dry bulb temperature?
3) What is dew point temperature?
4) What is psychrometry?
5) What are the different parameters of psychrometric chart?
6) How can we calculate percentage relative humidity?

12. DESCRIPTION OF CONSTRUCTION, WORKING AND APPLICATION OF PHARMACEUTICAL MACHINERY (ROTARY TABLET MACHINE, FLUIDIZED BED COATER, FLUID ENERGY MILL, DE HUMIDIFIER)

I.ROTARY TABLET MACHINE

It is also called multi station tablet press. It is called rotary machine because the head of machine that holds the upper punches, dies and lower punches in places rotates. Steps involved in manufacturing of tablet.



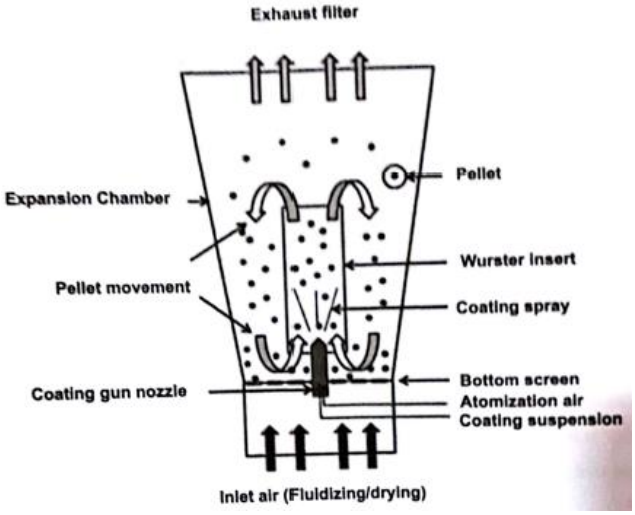
- The material to feed through hopper.
- The fill cam pulls the lower punches down to affixed distance and the dies are filled with material.
- The quantity of the material filled is larger than the actual amount required. Remove excess amount with the help of Spatula.
- After that, upper punch is lower and inserted into the dies.
- The material is compressed and the tablets are formed.
- After the compression, pulls the upper punches into their top position and simultaneously lifts the lower punches until the tablets are ejected from the dies.
- Then tablet is passed through the discharge chute.

APPLICATIONS

- It is operated continuously
- Used for large scale production
- A single rotary press produce 1150 tablets in a minute while double rotary press can produce 10,000 tablets in a minute.

II.FLUIDIZED BED COATER:

Three types of air suspension coater are available, namely, top spray coater, wurster or bottom spray coater and tangential spray coater. In top spray coater, there is a counter (opposite direction) movement of powder particles or pellets and liquid spray. In wurster or bottom spray coater, there is a concurrent (same direction) movement of powder particles or pellets and liquids spray in tangential spray coater the powder particles or pellets move in a helical fashion due to spinning rotor disk on the bottom of the equipment. Steps involved in wurster or bottom spray coater



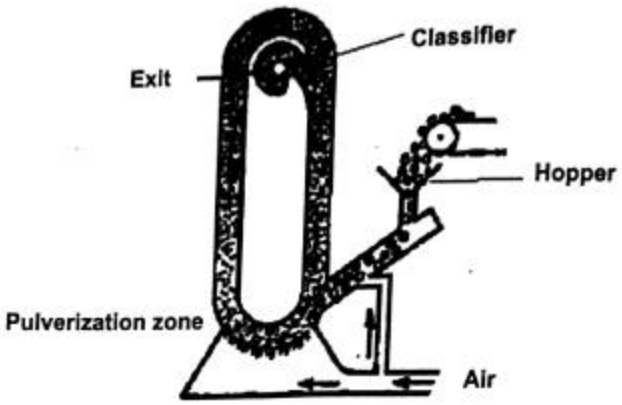
- The drying inlet air is passed upwards through the bottom perforated plate into the fluid bed chamber.
- This air passes to wurster column, in which a spray gun perpendicular to bottom plate and parallel to the wurster column.
- The air is passed out from the exhaust filters situated at the top of the equipment.
- The material to be coated is loaded in the fluid bed chamber and fluidized.

- The inlet air cause fluidization of the material as well as its drying during the coating operation.
- The pellets are passing through the liquid spray of coating solution from the spray gun positioners parallel to the column.
- After coating the coated particles falls by gravity at the bottom of wurster column and recycled to coating zone.

APPLICATIONS:

- It is used to coat pharmaceutical dosage form with polymeric material to mask objectionable taste or odor and also to protect an unstable ingredient and to improve appearance.
- Fluidized bed coaters are used for coating of powders, granules, tablets, pellets etc by column of air.
- Fluid bed coating equipment is popular for coating multi particulate systems such as beads and non-parcel seeds.

III.FLUID ENERGY MILL

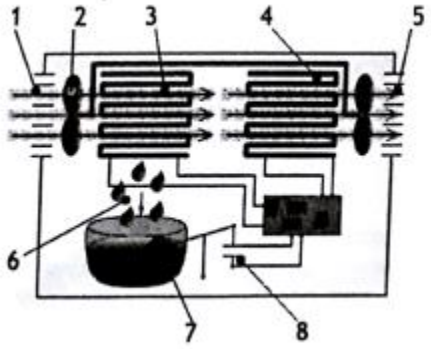


- A fluid usually air, is injected at very high pressure through nozzles at bottom of the loop. As a results turbulence produce.
- Solids are introduced into the stream through hopper.
- Due to this turbulence occur and impacts and attrition occur between the particles.
- A classifier is fitted at the exist so that only finer size particles are collected as products.
- The larger size particles are again sent to the stream of air for futher size reduction.

APPLICATIONS:

- The particle size of the product is smaller when compared to other method of size reduction.
- No chance of contamination of the product.
- This method is suitable where fine powders are required like micronization of griseofulvin.

DEHUMIDIFIER



- Warm moist air is sucked in through one side of the machine.
- An electric fan is used to draws the air inward.
- The warm air passes through cold pipes through which a coolant circulated. Due to cooling of air the moisture it contains turns back into liquid water.
- Then the air passes over a heating element and warms back up to its original temperature.
- Warm dry air blows back into the room through another side of machine.
- The moisture that was in the air drips down into collecting tray (or bucket) at the bottom of the machine.
- As the collecting tray fills up a plastic float in the machine rises upward.
- When the tray is full the float trips an electric switch that turns off the fan and switches on an indicator light which indicates that the machine needs emptying.

APPLICATIONS

- A dehumidifier is used to reduce the level of humidity in the air.
- Large dehumidifiers are also used in commercial buildings such as indoor ice rinks to control the humidity level.

VIVA VOCE Questions:

1) What are the steps involved in Rotary tablet machine in manufacturing of tablets?
2) What are the basic parts of Rotary tablet machine instrument?
3) What is the basic principle involved in fluidized bed coater?
4) What are the applications of fluidized bed coater?
5) What is the basic principle involved in fluid energy mill?
6) What are the applications of fluid energy mill?
7) What is the application of dehumidifier?

13. SIZE ANALYSIS BY SIEVING

AIM:-To determine the particle size distribution of powder by sieving method.

REQUIREMENTS:-

Sieve set (sieve no. 30, 45, 60, 100, 140, and200), Electromagnetic laboratory sieve machine or electrical sieve shaker

CHEMICAL / REAGENTS:-

Calcium carbonate/aspirin/ calamine powder/any power substances

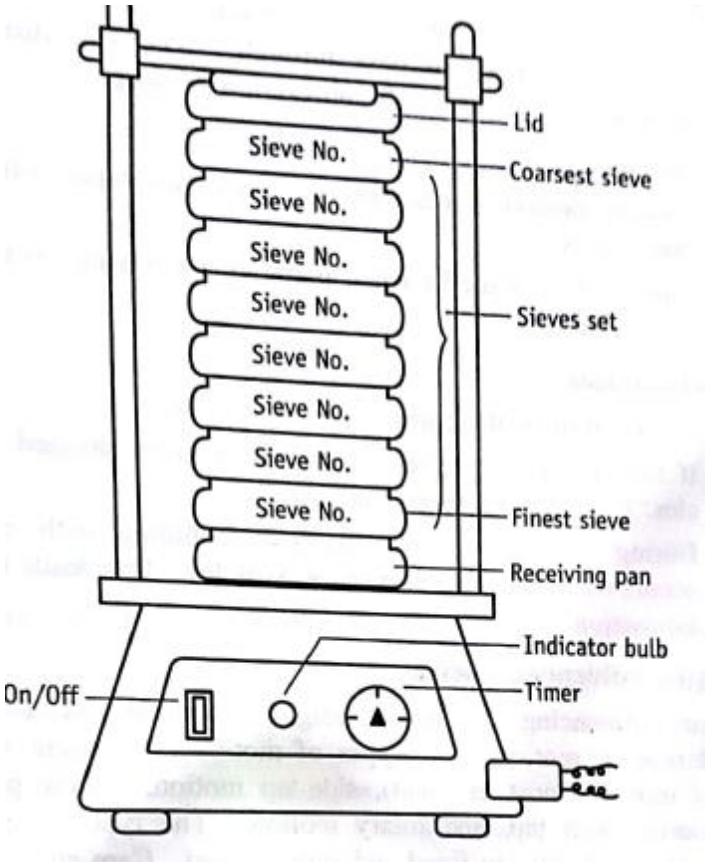
PRINCIPLE:

The basic principle involved in this method is size separation using standard sieves or screens

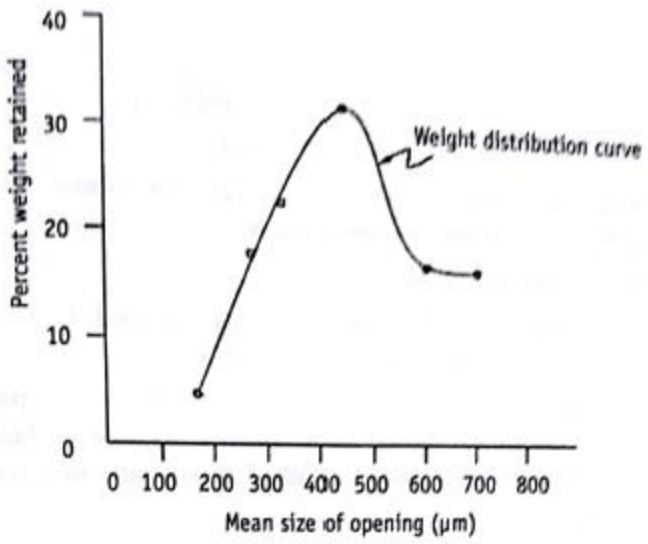
Size separation is unit operation that involves the separation of various sizes of particles into two or more portions by means of screening surfaces. size separation is also known as sieving, sifting, classifying or screening.

Designations and dimensions of IP and USP specification sieves

Sieve number		**Nominal mesh aperture size, mm**		**Sieve number**		**Nominal mesh aperture size, µm**	
IP	**USP**	**IP**	**USP**	**IP**	**USP**	**IP**	**USP**
4	8	4000	2380	36	50	425	297
8	10	2000	2000	44	60	355	250
10	20	1700	840	60	70	250	210
12	25	1400	710	85	80	180	177
16	30	1000	595	100	100	150	149
22	35	710	500	120	120	125	125
25	40	600	420	150	140	106	105
30	45	500	350				



The powdered drug is separated according to its particle size using a number of sieves in a nest. These are subjected to different types of agitation in sieve shaker, so that size separation is rapid. Sieves are arranged in anest with the coarsest at the top. A sample of the powder or granules is placed on the top sieve. This sieve sets is filed to the mechanical gyratory shaker and shaken for a period of time. The powder retained on each sieve is weighed. Then normal weight distribution curve is constructed.



The average particle diameter of a powder (d_{sieve}) is calculated using the following equation.

$$d_{sieve} = \frac{\Sigma(n \times d)}{\Sigma(n)}$$

Where n= frequency of particles in a particle size range, g;or percent weight of powder undersize, g

d = average particle diameter of particular sieve (sieve diameter), μm

PROCEDURE:-

1. Arrange set of sieves in the descending order.
2. Weighed amount of sample is tube placed in the sieve at the top of the sieve set.
3. Start the sieving machine. The length of time and speed of vibration can be controlled by semiautomatic attachment in the machine.
4. Collect the powder material retained on the various sieves.
5. Weigh the powder material retained on the sieves.
6. Calculate percent frequency of each size of particle and plot the graphs.
7. Determine the geometric mean weight diameter and geometrical standard deviation.

OBSERVATIONS :-

(a) Weight of substance, W_1 = g

(b)Time of shaking = min

(c) Speed of electrical shaker = rpm

S. no.	Sieve number (passed/retained)	Arithmetic Mean size Of opening (μm)	Weight Retained on a sieve (g)	Percent Weight Retained (undersize)	Cumulative Percent Retained
1	30/45	470			
2	45/60	300			
3	60/80	213			
4	80/100	163			
5	100/140	127			
6	140/200	90			

CALCULATION :-

Calculation of percent weight retained on screen.

Percent weight retained on screen = $\frac{\text{Weight retained on screen}}{\text{Total weight of powder}} \times 100$

1. Plot frequency distribution curve taking particle size on X axis and percent weight retained on the screen on Y axis.
2. The logarithm of the particle size is plotted against the cumulative percent frequency on a probability scale. It showed a linear relationship.

3. The geometrical mean weight diameter dg and geometrical standard deviation can be obtained from the straight line.

REPORT:

VIVA VOCE Questions:

1) Why sieve analysis is done?
2) What is the range of analysis of sieving method?
3) What is the basic principle involved in sieve analysis?
4) What ate the disadvantages of sieving method?

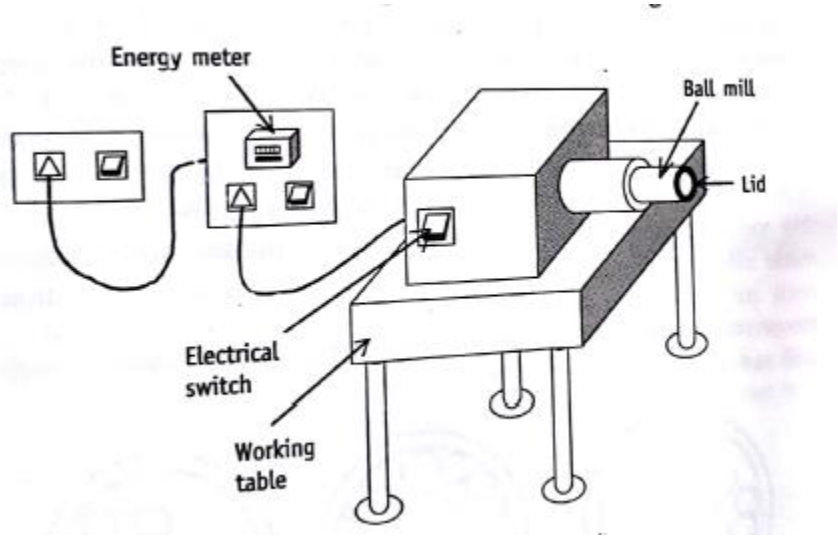
14. SIZE REDUCTION BY BALL MILL

AIM: Size reduction by ball mill and to verify the laws of size reduction using ball mill and determining Kicks, Rittinger's, Bond's coefficients, power requirement and critical speed of Ball Mill.

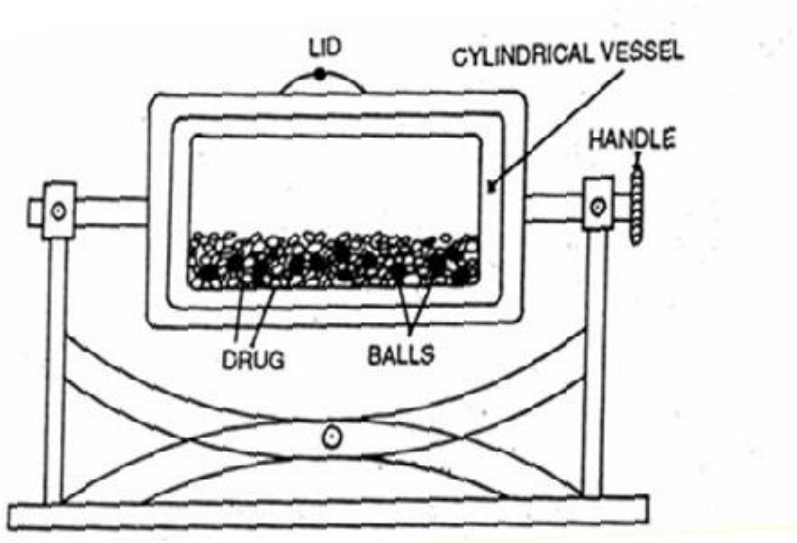
REQUIREMENTS: Ball mill, energy meter, granules, balance, sieves, sieve shaker.

PRINCIPLE:

Size reduction is a process of reducing large soild masses (vegetable and chemical substances) into small unit masses, coarse particles or fine particles.

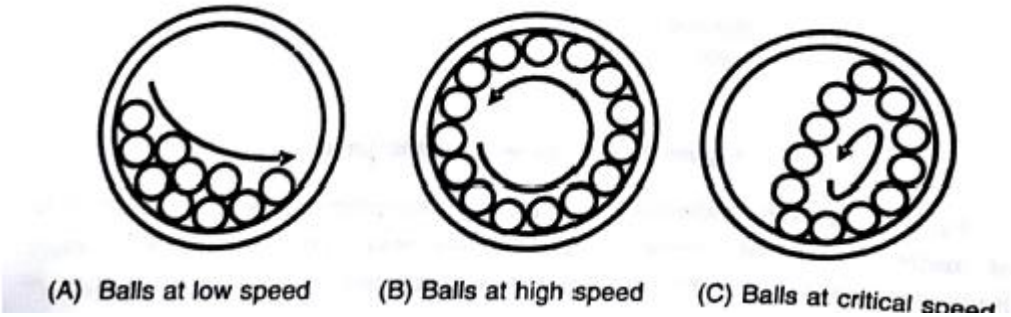


Ball mill with electric circuit



Ball mill

Ball mill are also known as tumbling mills or pebble mills. The ball mill works on the principle of impact between the rapidly moving balls and the material. Itconsists of hallow cylinder with metal ball acting as a grinding medium. Balls occupy 30-50% of mean volume. The hollow cylinder rotates around the longitudinal axis for size reduction of material placed around it. Ball mill is used especially for reducing properties of both wet and dry powder which can able to produce desired results .the speed with which the mill rotates should be optimum. At this speed the ball will fall and strikes the bottom of mill crushing the powder into small pieces.This producing an impact stress on the material to be grinded.



(A) Balls at low speed (B) Balls at high speed (C) Balls at critical speed

KICK'S LAW:

It states that the energy required for size reduction is proportional to the logarithm of the ratio between the initial and final size .For crushing (compression) of large particles, Kick's equation is more useful.

$$E = K_K \ln \left(\frac{d_i}{d_n}\right)$$

Where, E= amount of energy required to produce a change in unit mass,

K_K=Kick's constant energy per unit mass

d_i=initial particle size of sample (before size reduction), µm.

d_n= final particle size of sample (after size reduction), µm.

RITTINGER'S LAW:

It states that the energy consumed in the size reduction of solids is directly proportional to the new surface created. It is mostly applicable to brittle materials undergoing fine milling

$$E = K_R \left(\frac{1}{d_n} - \frac{1}{d_i}\right)$$

Where E=amount of energy required to produce a change in unit mass.

K_r=Rittinger's constant energy per unit area.

BOND'S WORK INDEX:

It states that the energy used for deforming a set of particles of equivalent shape is proportional to the change in particle dimensions. The bond's work index is a useful way for comparing the efficiency of milling operation. This is useful for rough mill sizing.

$$E = 2K_B\left(\frac{1}{\sqrt{d_n}} - \frac{1}{\sqrt{d_i}}\right)$$

Where, K_B=Bond's work index, energy per unit mass

PROCEDURE:

1. The initial dial reading of energy meter is noted as N_1.
2. The cleaned ball mill is taken with sufficient number of balls.
3. The ball mill is operated without load for 10 min.
4. The reading (revolution) in energy meter is noted down as N_2, (The difference, i.e., $N_3 = N_2-N_1$ gives the energy required for running the ball mill without feed).
5. Hundred grams of sample is weighed and subjected to sieve analysis. The average particle size of the sample is calculated.
6. Hundred grams of feed, which was subjected to sieve analysis is transferred into the ball mill.
7. The ball mill is operated for 10 minutes.
8. The reading (revolutions) is noted as N_4. (The difference, i.e., $N_5 = N_4-N_2$ gives the energy required for running the ball mill and size reduction of material).
9. The difference i.e., $N_6 = N_5-N_3$, gives the energy actually consumed for the size reduction of material.
10. The product is unloaded on to a tray and subjected for sieve analysis.
11. The average particle size of the product after size reduction is determined.

12. The data is substituted in equations of Kick's constant, Rittinger's constant, and Bond's work index to determine them.

OBSERVATIONS AND CALCULATIONS:-

Table No: Weight distribution of sample after size reduction.

S. No.	**Sieve No.**	**Nominal mesh aperture size, µm**	**Aperture Size(passed/retained), µm**	**Mean size of opening* (d), µm**	**Weight of powder undersize (n), gm**	**Percent weight retained on smaller sieve, (d)**	**Weight size n x d (4) x (6)**
	(1)	(2)	(3)	(4)	(5)	(6)	(7)
	Pan		-	-	-	-	-
1	120	125	125/Pan	125.0			
2	100	150	150/125	137.5			
3	85	180	180/150	165			
4	60	250	250/180	215			
5	44	355	355/250	302.5			
6	22	840	710/355	532.5			
7	10	1700	1700/710	1205.5			
						Σ(n) =	Σ(nd)=

Mean diameter of the powder sample before size reduction, di = Σnd / Σn

CALCULATIONS:

1. Initial reading of energy meter, N_1 =
2. Energy meter reading after the use of ball mill without feed, N_2 =
3. Energy consumed by the ball mill, $N_3 = N_2 - N_1$
4. Energy meter reading after the use of ball millwith feed, N_4 =
5. Energy consumed for size reduction plus the ball mill, $N5 = N_4 - N_2$
6. Energy consumed for the size reduction, $N_6 = N_5 - N_3$ =
7. Weight of the sample taken, w = 100 g (0.1 kg).

8. Calculation of energy meter constant , E (on the energy meter, the relationship between the revolution and energy is given. Use that relationship for calculations).

   750 units reading (revolutions) the energy = 3600 kW.s (1 kW.h)

   1 unit reading

   1 unit reading = 3600/750 = 4.8 kJ (E)

9. Energy on no load, $E_1 = N_3$ X E = kJ.
10. Energy on load, $E_2 = N_5$ X E = kJ.
11. Net energy required per unit mass = $\{(E_2 – E_1)/w\}$ = kJ.
12. Average particle diameter of feed, di = µm.
13. Average particle diameter of product, dn = µm.

REPORT: Rittinger's constant =

Kick's constant =

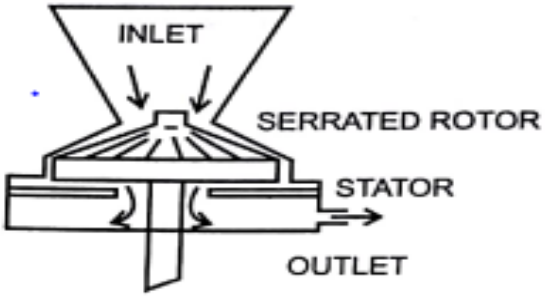
Bond's work index =

VIVA VOCE Questions:

1) Define size reduction.
2) What is the basic principle involved in ball mill?
3) How speed of ball mill is affecting the size reduction?
4) What are the advantages and disadvantages of ball mill?
5) What is Kick's law?
6) What is Rittinger's law?
7) What is Bond's law?

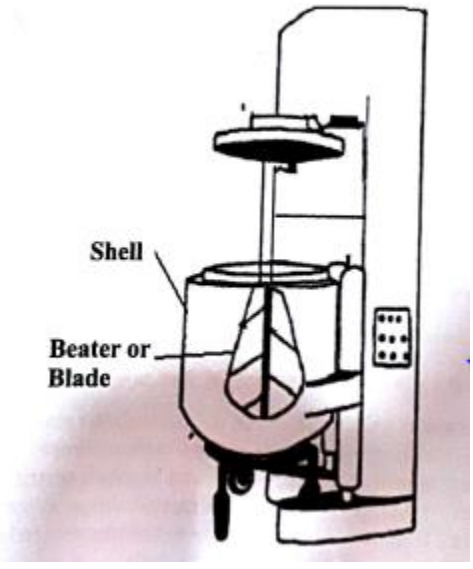
14. DEMONSTRATION OF COLLOID MILL, PLANETARY MIXER, FLUIDIZED BED DRYER, FREEZE DRYER

COLLOID MILL:



- The colloid mill used to reduce the size of the suspended droplets.
- The materials is feed in though the inlet hopper and placed into the mill
- It is then move through the narrow gap between the rotor and stator to reduced the particle size
- Then final product is removed through the outlet.

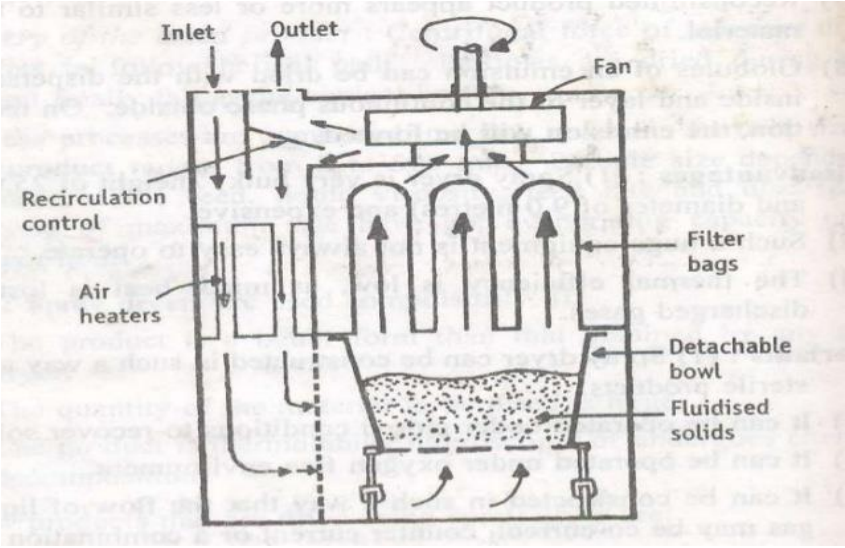
PLANETARY MIXER:



- The material to be mixed is loaded into mixing bowl or shell.

- The blades rotate on their own axis when they orbit the mixing bowl on a common axis. Therefore there is no dead spot in the mixing and high shear is applied for mixing.
- After mixing, the material is discharged through a bottom valve, or by manual scooping of the material from the bowl.

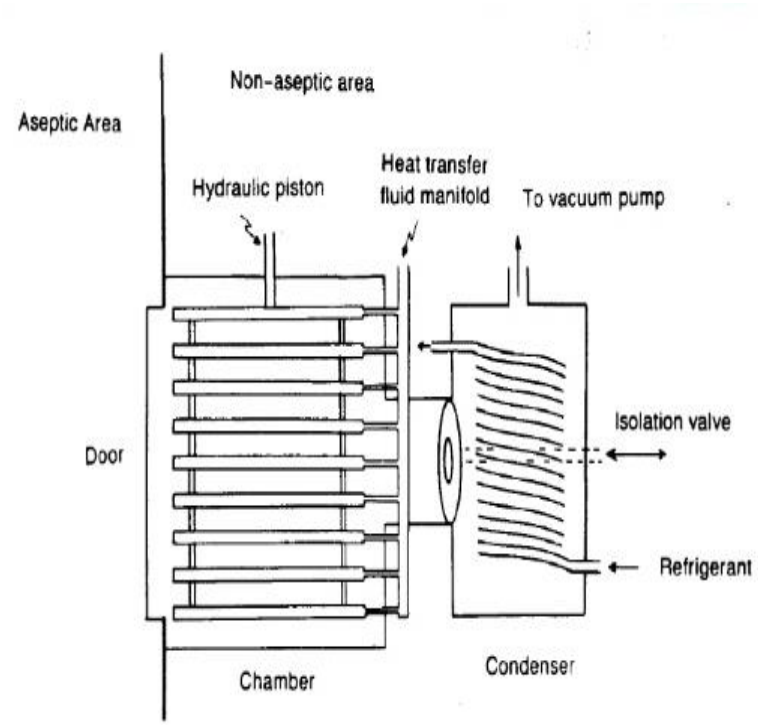
FLUIDIZED BED DRYER:



- The wet granules to be dried are placed in a detachable bowl. The bowl is inserted in the dryer.
- Fresh air can pass through a pre-filter, which is then heated when passing through a heat exchanger.
- Hot air flows through the bottom of the bowl. At the same time, the fan starts to rotate. The air speed increases gradually.
- After a specific times, a pressure point is reached in which the friction drag on the particles is equal to the force of gravity. The granules rise in the container. This condition is said to be fluidized.
- The gas surrounds each granule to dry them completely.The air comes out of the dryer passing through the filters in the bag.

- The entrained particles remain adhered to the interior of the surface of the bags. Periodically, the bags are shaken to remove entrained particles.
- The materials are left in the dryer to reach room temperature.
- The bowl is removed for unloading. The final product is free flowing.

FREEZE DRYER:



- The material is pretreated before freezing pretreatment methods include freezing concentration, solution phase concentration, formulation to preserve the appearance of the product, formulation to stabilize reactive products, formulation to increase the surface area and decreasing high vapor pressure solvent.
- The product should be frozen at a temperature low enough to solidify completely. The products are frozen in two ways, most of the products that are lyophilized consist mainly of water. It is very important in lyophilization process.

- After pre-freezing the product, conditions must be established in which the ice can be removed from the frozen product through sublimation, resulting in a dry, structurally intact product.
- After primary freeze- drying is complete, and all ice has submitted, bound moisture is still present in the product. The product appears dry, but the residual moisture content may be as high as 7-8% continued drying is necessary at warmer temperature to reduce the residual moisture content to optimum values. This process is called ‘isothermal desorption’.
- After vacuum is replaced by inert gas, bottle and vials are closed.

VIVA VOCE Questions:

1) What is the basic principle involved in colloid mill?
2) What are the uses of colloid mill?
3) What is the disadvantage of colloid mill?
4) Define mixing?
5) What is the basic principle involved in planetary mixer?
6) What are the advantages of planetary mixer?
7) What is the basic principle involved in fluidized bed dryer?
8) What are the applications of fluidized bed dryer?
9) What is the basic principle involved in freeze drying?
10) What are the applications of freeze drying?

15. DETERMINATION OF EFFECT OFCONCENTRATION, SURFACE AREA AND THICKNESS ON RATE OF FILTRATION

AIM: To determine the influence of concentration, surface area and thickness of filter medium on rate of filtration.

REQUIREMENTS: Measuring cylinder, Buckner funnel, filter paper, stop watch, calcium carbonate

PRINCIPLE: - Filtration may be defined as a process of separation of solids from a fluid by passing the same through a porous medium that retains the solids, but allows the fluid to pass through.

The rate of filtration depends on the concentration of solids. As the concentration of solids in the suspension increases, the thickness of the filter cake increases. As a result, rate of filtration decreases. The rate of filtration is directly proportional to surface area of the filter medium. Filter medium of sufficient thickness is used for filtration. But the thickness of the filter medium plays important role in determining the rate of filtration. Sometimes, increase in thickness decreases the rate of filtration. The rate of filtration also depends on the thickness of the filter cake formed.

The rate of filtration for practical purpose is calculated using the following equation:

$$\text{Rate of filtration} = \frac{\text{Volume of filtrate passed through the filter medium}}{\text{Time required for the filtrate to pass through}}$$

$$= m^3/\text{ sec}$$

PROCEDURE:

EFFECT OF CONCENTRATION

Preparation of calcium carbonate suspension (5%)

2.5 g of calcium carbonate is weighed and transferred to the mortar. 25 ml of water is added and triturated to get smooth paste. The contents are transferred to measuring cylinder (50 ml). The mortar and pestle are washed with 5 ml of water (2 to 3 times if necessary). The washings are transferred to measuring cylinder (50 ml) and make up to mark with water. The suspension is shaken thoroughly.

The same procedure is repeated for 10% and 15% suspensions using 10.0 and 15.0 g of calcium carbonate, respectively.

Method for studying the effect of concentration

1. The filter paper of appropriate size is placed into a Buckner funnel.
2. 50 ml of 5% calcium carbonate suspension is poured over the Buckner funnel.
3. Time required to collect 25 ml of the filtrate is recorded.
4. The experiment (step 2 to 4) is repeated for the same conc. of calcium carbonate suspension for two more trials.
5. The experiment is repeated for other conc. (10% and 15%)also.
6. A graph is plotted by taking conc. of calcium carbonate on x-axis and rate of filtration on y-axis.

OBSERVATIONS AND CALCULATIONS:

Conc. Of slurry	**Trial**	**Volume of filtrate collected (ml)**	**Time of collection of filtrate (Min)**	**Time of collection of filtrate (Sec)**	**Rate of filtration, ml/sec**	**Rate of filtration, m^3/sec**
1	**2**	**3**	**4**	**5**	**6 (3)/(5)**	**7 (6)X 10^{-6}**
5%	1	25				
	2	25				
	Mean	25				
10%	1	25				
	2	25				
	Mean	25				
15%	1	25				
	2	25				
	Mean	25				

EFFECT OF SURFACE AREA

Preparation of calcium carbonate suspension (5%):

2.5 g of calcium carbonate is weighed and transferred to the mortar. 25 ml of water is added and triturated to get smooth paste. The contents are transferred to measuring cylinder (50 ml). The mortar and pestle are washed with 5 ml of water (2 to 3 times if necessary). The washings are transferred to measuring cylinder (50 ml) and make up to mark with water. The suspension is shaken thoroughly.

Method for studying the effect of surface area on rate of filtration

1. The filter paper of appropriate size is placed into a Buckner funnel.
2. 50 ml of 5% calcium carbonate suspension is poured over the Buckner funnel.
3. Time required to collect 25 ml of the filtrate is recorded.
4. The experiment (step 2 to 4) is repeated for the same conc. of calcium carbonate suspension for two more trials.
5. The experiment is repeated for medium and bigger Buckner funnel also.
6. A graph is plotted by taking surface area on x-axis and rate of filtration on y-axis.

OBSERVATIONS AND CALCULATIONS:

Surface area of filter medium, m^2	Trial	Volume of filtrate collected (ml)	Time of collection of filtrate (Min)	Time of collection of filtrate (Sec)	Rate of filtration, ml/sec	Rate of filtration, m^3/sec
1	**2**	**3**	**4**	**5**	**6 (3)/(5)**	**7 (6)X 10^{-6}**
Small	1	25				
	2	25				
	Mean	25				
Medium	1	25				
	2	25				
	Mean	25				
Big	1	25				
	2	25				
	Mean	25				

EFFECT OF THICKNESS OF FILTER MEDIUM

Preparation of calcium carbonate suspension (5%):

2.5 g of calcium carbonate is weighed and transferred to the mortar. 25 ml of water is added and triturated to get smooth paste. The contents are transferred to measuring cylinder (50 ml). The mortar and pestle are washed with 5 ml of water (2 to 3 times if necessary). The washings are transferred to measuring cylinder (50 ml) and make up to mark with water. The suspension is shaken thoroughly.

Method for studying the effect of thickness of filter medium on rate of filtration

1. The filter paper of known thickness (one filter paper) is placed into a Buckner funnel.
2. 50 ml of 5% calcium carbonate suspension is poured over the Buckner funnel.
3. Time required to collect 25 ml of the filtrate is recorded.
4. The experiment (step 2 to 4) is repeated for the same conc. of calcium carbonate suspension for two more trials.
5. The experiment is repeated by taking two and three filter papers.
6. A graph is plotted by taking thickness of filter medium on x-axis and rate of filtration on y-axis.

OBSERVATIONS AND CALCULATIONS:

Thickness of filter medium	Trial	Volume of filtrate collected (ml)	Time of collection of filtrate (Min)	Time of collection of filtrate (Sec)	Rate of filtration, ml/sec	Rate of filtration, m^3/sec
1	**2**	**3**	**4**	**5**	**6 (3)/(5)**	**7 (6)X 10^{-6}**
1 filter paper	1	25				
	2	25				
	Mean	25				
2 filter paper	1	25				
	2	25				
	Mean	25				
3 filter paper	1	25				
	2	25				
	Mean	25				

REPORT:

16. DETERMINATION OF EFFECT OFCONCENTRATION, SURFACE AREA AND VISCOSITY ON RATE OF EVAPORATION

AIM: To determine the effect of concentration, surface area and viscosity on rate of evaporation.

REQUIREMENTS: Beaker, Water-bath, Measuring cylinder, Glycerin, Purified water.

PRINCIPLE: - Evaporation is a process of vaporizing large quantities of volatile liquid to get a concentrated product.

The rate of evaporation depends on several factors such as temperature, viscosity, concentration of the slurry, vapour pressure, surface area, time o evaporation, films and deposits. Higher the concentration of dissolved solids (like sodium chloride), the lower the rate of evaporation. Higher the viscosity of the slurry, the lower the rate of evaporation. This is verified by taking slurries of different viscosities and subjecting to evaporation at constant temperature and surface area. The greater the surface area of the liquid. The greater will be the evaporation. For this reason, evaporation is conducted in evaporators with large heating surface area. It is veriied by taking beakers of different surface area, i.e 50ml, 100ml and 250ml capacity. The rate of evaporation is calculated using the following formula:

$$\text{Rate of filtration} = \frac{\text{Quantity of water evaporated (w)}}{\text{Time of heating (min)}}$$

$$= \text{g/min}$$

PROCEDURE:-

EFFECT OF CONCENTRATION

Procedure:

1. 2, 4, 6, and 8% w/v solutions of NaCl are prepared by dissolving 1, 2, 3, and 4 g of NaCl in 50 ml of water in beakers.
2. The beaker containing sodium chloride solutions are weighed (W_1 g). Weights are recorded in table.
3. All the beakers are heated in a water bath at constant temp. (70°C) for 30 mins.
4. All the heaters are weighed again after heating (W_2 g).
5. The difference between the weights is determined. The diff. reflects the amount of water evaporated during 30 minutes.

6. Rate of evaporation is calculated using the formula.
7. A graph is plotted by taking conc. on x-axis and rate of evaporation on y-axis.

OBSERVATIONS AND CALCULATIONS:

S. No.	**Conc. of Sodium chloride solution, %w/v**	**Initial wt. of beaker + solution, W_1, g**	**Final wt. of beaker + solution, W_2, g**	**Wt. of water evaporated, w g**	**Time of heating, min**	**Rate of evaporation g/min.**
				(3)-(4)		**(5)/(6)**
(1)	**(2)**	**(3)**	**(4)**	**(5)**	**(6)**	**(7)**
1.	2 %					
2.	4 %					
3.	6 %					
4.	8 %					

EFFECT OF SURFACE AREA

PROCEDURE:

1. Beakers measuring 50, 100 and 250 ml are cleaned.
2. 25 ml quantity of water is taken in each beaker.
3. Beakers containing water are weighed (initial wt. of beaker, w_1 g).
4. All the beakers containing water are heated in a water bath at constant temp. (70°C) for 30 mins.
5. After heating all the beakers are weighed again (final wt. of beaker, w_2 g).
6. The difference between the weights is determined, w g. The difference reflects the amount of water evaporated during 30 mins.
7. Radius (half of the diameter) of the beaker is noted. Using the radius, surface area of beakers is calculated, using the formula given below :-

   Surface area of beaker = πr^2

8. Rate of evaporation is calculated using the formula
9. A graph is plotted by taking surface area on x-axis and rate of evaporation on y-axis.

OBSERVATIONS AND CALCULATIONS:

S. No.	**Surface area of beaker, cm^2**	**Initial weight of beaker, w_1 g**	**Final weight of beaker, w_2 g**	**Wt. of water evaporated, w g**	**Time of heating, min**	**Rate of evaporation g/min.**
				(3)-(4)		**(5)/(6)**
(1)	**(2)**	**(3)**	**(4)**	**(5)**	**(6)**	**(7)**
1.						
2.						
3.						
4.						

EFFECT OF VISCOSITY

PROCEDURE:

1. Different conc. of glycerin and water mixture are prepared in diff. beakers as shown in following Table:

Glycerin	Water	Conc.
5 ml	45 ml	10 %
10 ml	40 ml	20 %
15 ml	35 ml	30 %
20 ml	30 ml	40 %

2. The beakers containing glycerin-water mixtures are weighed (W_1 g), viscosities of these mixtures at room temp. are given in table. Weights are recorded in table.
3. All the beakers are heated in a water bath at constant temp. (70°C) for 30 mins.
4. All the heaters are weighed again after heating (W_2 g).
5. The difference between the weights is determined. The diff. reflects the amount of water evaporated during 30 minutes.
6. Rate of evaporation is calculated using the formula
7. A graph is plotted by taking viscosity on x-axis and rate of evaporation on y-axis.

OBSERVATIONS AND CALCULATIONS:

S. No	Conc. of glycerin water mix.	Viscosity of glycerin water mix	Initial wt. of beaker, W_1, g	Final wt. of beaker, W_2, g	Wt. of water evaporated, w g	Time of heating, min	Rate of evaporation g/min.
					(4)-(5)		(6)/(7)
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
1.	10 %	1.2823					
2.	20 %	1.8765					
3.	30 %	2.4020					
4.	40 %	2.9829					

REPORT :-

VIVA VOCE Question:

1) Define filtration?
2) What are the different factors effecting rate of filtration?
3) How we can calculate rate of filtration?
4) What is filter aid? Give examples.
5) How concentration and surface area effects rate of filtration?

17. EFFECT OF TIME ON THE RATE OF CRYSTALLIZATION

AIM:- To study the crystallization behavior of potassium nitrate.

REQUIREMENTS: potassium nitrate, water bath,

PRINCIPLE: Potassium nitrate crystals can be obtained using shock cooling technique. The solid is added to a solvent continuously until the solid is dissolved. Such a solution is called as saturated solution. The rate of dissolution process is enhanced by increasing the temperature and agitation. Then the undissolved solid also goes into solution, When some solid remained undissolved, then such a solution is called as supersatured solutions. When the temperature of supersatured solutions is decreased rapidly the solubility of solute decreases. As a result the dissolved solid gets crystals growth. The extent of crystallization depends on the time of contact in low temperature. The crystals are collected by filtration and weighed. Yield is expressed as per cent weight of crystals obtained. A graph is plotted taking time versus percent weight crystals.

PROCEDURE:

1. Seventy five grams of potassium nitrate is accurately weighed (W_1g).
2. Hundred ml of water is transferred into 250 ml beaker.
3. Beaker containing water is placed in constant temp. water bath maintained at 50°C.
4. Potassium nitrate is added into the water little by little, the solution is stirred with glass rod to dissolve the solute.
5. This process is continued until saturated solution (with little excess crystals) is formed.
6. Weight of potassium nitrate remained is weighed (W_2 g). Difference in the weights W g (W_1-W_2) gives weight of potassium nitrate added into 100 ml water.
7. From this, 10 ml quantities of saturated solution are transferred into 9 test tubes.
8. All the test tubes are placed in an ice bath at once. Temp. of the solution decreases suddenly due to shock cooling forming supersaturated solution (Rate of cooling can be maintained constant by keeping the test tubes either in constant temp. water bath maintained at 20°C or in refrigerator). Nucleation and crystal growth takes place.
9. After 10 mins. The solution of first test tube is filtered to collect crystals.

10. This is repeated after every 10 mins. Thereafter using the solution of other test tubes.
11. All the crystals collected on the filter paper separately are subjected to drying.
12. Weights of each sample of crystals are recorded in table.
13. A graph is plotted taking time on x-axis and % wt. on crystals on y-axis.

OBSERVATION AND CALCULATIONS:

Data for crystallization of potassium nitrate

S.No	Test tube No.	Time, mins	Weight of crystals formed, g (b)	% wt. of crystals b/a X 100
1.	1	10		
2.	2	20		
3.	3	30		
4.	4	40		
5.	5	50		
6.	6	60		
7.	7	70		
8.	8	80		
9.	9	90		

a = Weight of potassium nitrate present in 10 ml of water, g

REPORT: Percent crystals of pott. nitrate crystals formed in 90 min =

VIVA VOCE Questions:

1) Define crystallization.
2) How crystallization differs from precipitation?
3) What are the objectives of crystallization?
4) Define supersaturation.

18. MIXING INDEX

AIM: To determine the mixing index for the blending of salicylic acid and lactose in a blender.

REQUIREMENTS: Cylindrical Blender, Colorimeter and cuvettes, pipette (10 ml, 5 ml), test tube (20 ml), salicylic acid, Ferric nitrate solution (4% w/v), volumetric flask (100 ml), lactose.

PRINCIPLE: Cylindrical blender is used for mixing. In this experiment, dry powders of salicylic acid and lactose are mixed. During rotation of mixer, powders get mixed with each other. Sufficient time is allowed for mixing to get uniform blend.

During mixing, after every 10 mins, samples are drawn from three different places randomly. Each sample is subjected to determine the amount of ingredients present. Salicylic acid and lactose are soluble in water. Hence, to every sample dissolved in water, ferric nitrate is added to develop pink colour. Quantity of salicylic acid present is estimated by measuring absorbance of the colour colorimetrically at 547 nm. By knowing the amount of salicylic acid, the amount of lactose can be calculated.

Mixing index can be calculated using the following formula:

$$\text{Rate of Drying} = \sqrt{\frac{\Sigma(y-\breve{Y})^2}{n(1-\breve{Y})\,\breve{Y}}}$$

Ms = Mixing index.

N = number of samples.

$\breve{Y}$ = true average composition of component A in the mixture.

Y = Actual composition of component A in a single sample.

PROCEDURE:

1. One g of salicylic acid and 50 g of lactose are weighed.
2. These two powders are placed in a cylindrical blender.
3. The blender is allowed to rotate on its own axis for 15 mins at 25 rpm.
4. The sample (500 mg) are drawn from three diff. places of the blender and placed in three diff. conical flasks. Labelled them as 1A, 1B, and 1C.
5. The blender is again allowed to rotate for another 10 mind.

6. Again three samples are drawn in a similar way as mentioned in step 4. They are transferred into three diff. conical flask and labeled as 2A, 2B and 2C.
7. Repeat the steps 5 after 45 mins. The samples are labeled as 3A, 3B, and 3C.
8. The samples are dissolved in water with continuous shaking. Finally, the volume is made upto 100 ml in each case.
9. From the volumetric flasks, 10 ml solutions are transferred into 20 ml test tubes.
10. 5 ml of ferric nitrate solution (4% w/v) is added to the above test tubes. All the solutions turn to purple colour.
11. The absorbance o the above solutions are measured at 547 nm using a colorimeter or spectrophotometer and reported in the table.
12. The content of salicylic acid and mixing index is calculated.

OBSERVATIONS AND CALCULATIONS:

Sampling time	Sample number	Absorbance	Conc. Of salicylic acid	Conc. Of salicylic acid in the sample y, mg (4) X 100	(y-$\bar{y}$)	(y-$\bar{y}$)^2
(1)	(2)	(4)	(6)	(7)	(8)	
At 15 mins	1A					
	1B					
	1C					
			Average, $\bar{y}$ =		$\Sigma(y-\bar{y})^2$=	
At 30 mins	2A					
	2B					
	2C					
			Average, $\bar{y}$ =		$\Sigma(y-\bar{y})^2$=	
At 45 mins	3A					
	3B					
	3C					
			Average, $\bar{y}$ =		$\Sigma(y-\bar{y})^2$=	

REPORT:

1. Mixing index after 15 mins, M_{15} =
2. Mixing index after 30 mins, M_{30} =
3. Mixing index after 45 mins, M_{45} =

VIVA VOCE Questions:

1) Define mixing.
2) What are the different types of mixing?
3) What is mixing index?
4) What are the different equipments used in solid-solid mixing?