East Point College of Pharmacy

East Point Campus, Jnana Prabha, Virgo
Nagar PostBengaluru – 560049, Karnataka

Approved
by
Pharmacy Council of India, New Delhi

Affiliated
to
Rajiv Gandhi University of Health SciencesKarnataka
Bengaluru – 560041 India

LAB MANUAL

PHARMACEUTICAL ENGINEERING

B. PHARM 3rd SEMESTER

B Pharmacy

Program Outcomes (PO's)

PO 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.

PO 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.

PO 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

PO 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.

PO 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 wellbeing.

PO 6- Professional Identity

Understand, analyse and communicate the value of their professional roles in society (e.g. health care professionals, promoters of health, educators, managers, employers, employees).

PO 7- Pharmaceutical Ethics

Honor personal values and apply ethical principles in professional and social contexts. Demonstrate behaviour 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

PO 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

PO 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.

PO 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.

PO 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-access and use feedback effectively from others to identify learning needs and to satisfy these needs on an ongoing basis.

Programme Specific Outcomes (PSO's)	
PSO 1	Acquire a thorough foundational knowledge in pharmaceutical sciences, including pharmacology, pharmaceutics, medicinal chemistry, and pharmacognosy, to excel in further academic pursuits
PSO 2	Gain expertise in the application of contemporary pharmaceutical techniques and technologies, enhancing employability across various sectors including the pharmaceutical industry, academia, and research institutions.
PSO 3	Equip with entrepreneurial skills and knowledge of pharmaceutical business management, including market analysis, product development, regulatory affairs, and financial planning, to initiate and run successful ventures in the pharmacy sector

Course Outcomes (CO's)	
Code: BP308P PHARMACEUTICAL ENGINEERING	
CO 1	Ability to determine particle size of polydispersed powder by using sieve analysis. Ability to apply the concept of size reduction using various size reduction techniques
CO 2	Understand the significance of various factors affecting filtration, evaporation and crystallization
CO 3	Understand the construction, working and application of various equipment's by practical demonstration
CO 4	Ability to determine end point of drying, loss of drying and moisture content of wet sample by constructing drying rate curve.

EAST POINT COLLEGE OF PHARMACY

East Point Campus, Jnana Prabha, Virgo Nagar Post,
Bengaluru – 560049, Karnataka

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Experiment No. 01

**FACTORS AFFECTING RATE OF FILTRATION - SURFACE AREA,
CONCENTRATION, THICKNESS / VISCOSITY**

Aim: To study the various factors such as surface area, concentration, thickness and viscosity on rate of filtration

Requirements: Filters papers, Buchner funnels of different sizes, Calcium carbonate, glass funnels, Carboxymethyl cellulose sodium.

Principle:

Filtration is defined as a process of separation of a solid form a fluid by means of a porous medium that retains the solid but allows the fluid to pass through. The rate at which the filtrate is obtained depends on the physical conditions of the operation and the materials which make up the slurry. If the resistance offered by the filter is assumed to be negligible when compared with the resistance offered by a non-compressible cake, the rate of filtration can be considered to be directly proportional to the available filtering area and the pressure difference across the filter cake. As the cake resistance is directly proportional to the cake thickness, the rate of filtration will be inversely proportional to the cake thickness. The above statements can be represented mathematically as follows:

$$dv/d\Theta = PA/CL$$

where, $dv/d\Theta$ = rate of filtrate flow. P = pressure drop across the cake V = volume of filtrate collected upto any time Θ

A = total filtering area. L = thickness of cake

C = proportionality constant.

The resistant offered by the filter medium though significant in laboratory operation, is negligible when compared with the cake resistance encountered in large – scale filtration. This resistance offered by the cake will increase proportionality as more and more of the suspended solids settle on the filter medium. The cake resistance is affected by the properties of the solid like surface area of the particles, their rigidity or compressibility and the porosity of the cake.

For achieving high filtration rates by increased filter areas, a large number of small filter units can be connected in parallel (filter press) or the cake can be removed continuously (drum filter). The pressure drop across the filter medium can be increased by employing gravity, reduced

pressure, positive pressure or centrifugal forces. The rate of filtration is found to increase with decrease in viscosity. This decrease in the viscosity can be attained by dilution or by raising the temperature, provided the slurry is not thermolabile.

Some of the equations that govern the rate of filtration are:

1.Poiseuille's Equation	2. Darcy's equation	3. Kozeny- carman equation
$V=\pi\Delta Pr^4/8L\eta$	$V = KA\Delta P/L\eta$	$V = A\Delta P\varepsilon^3 / \eta S^2 KL$
Where	Where	$(1-\varepsilon)^2$
V = Rate of flow (m3/s) ΔP = Pressure difference across the filter bed (Pa)	V = Rate of flow (m^3/s) ΔP= Pressure difference across the filter bed (Pa)	Where V = Rate of flow (m^3/s) S = Specific surface area of particles of the cake (m^2/s)
r = radius of the capillary in the filter bed (m)	K = Permeability coefficient of the cake (m^2)	K= Kozeny constant E = Porosity of the cake ΔP
L = Thickness of the filter cake(m)	L = Thickness of the filter cake(m)	= Pressure difference across the filter bed (Pa)
η = viscosity of the filtrate (Pa.s)	η = Viscosity of the filtrate (Pa.s)	η = viscosity of the filtrate (Pa.s)

Procedure:

Prepare ml of 1% calcium carbonate suspension using 0.5% or 1% of CMC as suspending agent. This shall be used as slurry for the experiment.

1. Area of filter media and rate of filtration

Arrange Buckner funnels of suitable sizes along with filter papers. Pass 10ml of slurry to each of them and note the time taken for filtration. Also calculate the area of the filter paper and note the recordings.

Plot a graph of rate of filtration v/s area of filter media.

2. Thickness of filter bed and rate of filtration

Carry out the filtration of 10ml of slurry through a Buchner funnel (suitable size) using a single filter paper and note the time taken for filtration and calculate the rate of filtration.

Repeat the same procedure by using Buckner funnels containing 2,4,6 and 8 filter papers and calculate rate of filtration in each case.

Plot a histogram with rate of filtration on y axis and thickness of filter bed in terms of filter papers on x axis. (This portion of the experiment can also be carried out by using sand as filter bed)

3. Influence of cake formation on rate of filtration

Pass 10ml of slurry through a Buckner funnel with a single filter paper on it. Calculate the rate of filtration. After the filtration, pass another 10ml of slurry through the same assembly and note the time taken for filtration, calculate the rate of filtration. Continue this process 4 to 5 times and calculate the rate of filtration in each case. Interpret the result in terms of rate of filtration and time.

4. Concentration of slurry and rate of filtration

Prepare 50ml of 2,4,6 and 8% w/v suspensions of calcium carbonate with 1% CMC as suspending agent.

Arrange a filtration assembly with a single filter paper.

Using 10ml of suspension carry out the filtration of each of the above concentration. Note the time taken to filter 10ml of suspension and calculate the rate of filtration for each concentration Plot a histogram with rate of filtration on y axis and concentration on x axis.

5. Pressure and rate of filtration

Carry out the filtration of 10ml of the slurry (1% calcium carbonate) using an ordinary glass funnel and also with the Buckner assembly with suction pump. Note the time taken for filtration in each and calculate the rate of filtration.

Observations & calculations:

Area of filter media and rate of filtration

Sl. No	Area of filter media	Amount of the slurry filtered	Time taken for filtration (min)	Rate of filtration ml/min
1				
2				
3				
4				

Thickness of filter media and rate of filtration

Sl. No	Thickness of filter bed	Amount of the slurry filtered	Time taken for filtration (min)	Rate of filtration (ml/min)
1				
2				
3				
4				
5				

Pressure and rate of filtration

Sl. No	Type	Time in min	Rate of filtration ml/min
1	Gravity		
2	Negative pressure		

Concentration and rate of filtration

Sl. No	Concentration of the slurry	Amount of the slurry filtered (ml)	Time taken for filtration (min)	Rate of filtration (ml/min)
1				
2				
3				
4				

Report:

The various factors such as surface area, concentration, thickness and viscosity on rate of filtration was studied. The rate of filtration was found to be (due to various factors):

- Area of filter media
- Thickness of filter media
- Filtration pressure
- Concentration of the slury

Experiment No. 02

FACTORS AFFECTING RATE OF EVAPORATION - SURFACE AREA, CONCENTRATION, THICKNESS / VISCOSITY

Aim: To study the various factors such as surface area, concentration, and viscosity on rate of evaporation.

Requirements: Beaker(50ml,100ml,250ml), Water bath, balance, Weight box, Measuring cylinder.

Principle:

Evaporation is a surface phenomenon where the mass transfer takes place below boiling point of the liquid. The rate of evaporation depends on several factors such as temperature, viscosity concentration of slurry, vapour pressure, surface area, time of evaporation, films and deposits, moisture content of feed, types of production and economic factors.

The higher the temperature, greater will be the rate of evaporation. The greater the surface area of liquid, greater will be evaporation for this reason evaporation is conducted in evaporators with large heating surface area. This is verified by taking beakers of different surface area. These 50ml,100ml,250ml, capacity. Same quantity of slurry is maintained and exposed to same time, same temperature. The difference is initial and final weight permits the verification of factor surface. Expose time is longer, greater will be evaporation.

Higher the concentration of dissolved solids, lower the rate of evaporation the working concentration of Nacl do not alter the viscosity factor.

Procedure:

1. **Effect of surface area.**
   - Exactly 25ml measured quantity of water is taken in a cleaned beakers measuring 50ml,100ml,250ml.
   - Weight of each beaker is recorded in column no 3
   - All the beakers containing water are heated in water bath constant temperature for 30min
   - After heating, all beakers are weighed again. The weights are recorded in coloumn 4
   - The difference between weights is determined. The difference in the amount of water evaporated during 30min is noted.
   - Radius of beaker is noted using the radius, surface area of beakers is calculated using the below mentioned formula and recorded n column 2
   - Surface area of beaker = πr^2
   - Rate of evaporation= quantity of water evaporated

o Time of heating (min)

o The graph is plotted by taking rate of evaporation on y axis and surface area on x axis.

2. Effect of viscosity

- Different concentration of Glycerin, water mixtures are prepared in different beakers.

- The beakers containing Glycerin. Water mixture are weight viscosities of these mixtures at same temperature are given in column 3

- All the beakers are heated in water bath at constant temperature for 30min.

- Difference between weight is determined.

- Rate of evaporation is calculated.

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

3. Effect of concentration

- Prepare 2,4,6,8% w/v of NaCl by dissolving 1,2,3,4 of NaCl in 50ml water

- The beakers containing NaCl are weighed and recorded

- All the beakers are heated for 30min in a water bath at constant temperature.

- Beakers are weighed again

- Difference between weights are determined

Rate of evaporation is calculated.

Observations & calculations:

Effect of surface area

Sl No	Capacity of beaker	W_2 (g)	W_1 (g)	W= w_1-w_2	Surface area	Rate of evaporation
1	50ml					
2	100ml					
3	250ml					

Effect of viscosity on rate of evaporation

Sl No	Quantity of glycerin	Quantity of water	Concentration	W_1	W_2	W=w_1-w_2	At 70^{0}C viscosity	Rate of evaporation
1								
2								
3								
4								
5								

Report:

The various factors such as surface area, concentration, thickness and viscosity on rate of evaporation was studied. The rate of filtration was found to be (due to various factors):

- Surface area of beaker
- Viscosity
- Concentration

Experiment No. 03

CONSTRUCTION OF DRYING CURVES

Aim: Construction of drying rate curve

Requirements: Hot Air Oven, Thermometer, Weighing balance

Procedure:

1. A sample is weighed on the analytical balance and the wet weight is thus determined.
2. Then the water is removed through heating.
3. The residual weighing gives the dry weight, and so the difference between the intial weight and the residual weight tells us the mass of the water in sample.
4. Then calculate the moisture content and loss on drying by using following formula

$$\% \text{Moisture content} = \frac{\text{Weight of water in sample}}{\text{Weight of dry sample}} \times 100$$

$$\text{Loss on drying (\%)} = \frac{\text{Weight of water in sample}}{\text{Total Weight of Wet Sample}} \times 100$$

Observations & calculations:

Sl. No	Weight of wet sample	Weight after drying or dry weight	Weight of water in sample (weight of wet sample -Weight after drying)
1			
2			
3			

Report:

The % Moisture content and Loss of Drying was found to be:

Experiment No. 04

DETERMINATION OF MOISTURE CONTENT AND LOSS ON DRYING

Aim: Determination of moisture content and loss on drying
Requirements: Hot Air Oven, Thermometer, Weighing balance

Procedure:

1. A sample is weighed on the analytical balance and the wet weight is thus determined.
2. Then the water is removed through heating.
3. The residual weighing gives the dry weight, and so the difference between the intial weight and the residual weight tells us the mass of the water in sample.
4. Then calculate the moisture content and loss on drying by using following formula

$$\% \text{Moisture content} = \frac{\text{Weight of water in sample}}{\text{Weight of dry sample}} \times 100$$

$$\text{Loss on drying (\%)} = \frac{\text{Weight of water in sample}}{\text{Total Weight of Wet Sample}} \times 100$$

Observations & calculations:

Sl. No	Weight of wet sample	Weight after drying or dry weight	Weight of water in sample (weight of wet sample -Weight after drying)
1			
2			
3			

Report:

The % Moisture content and Loss of Drying was found to be:

Experiment No. 05

DETERMINATION OF HUMIDITY OF AIR – I) FROM WET AND DRY BULB TEMPERATURES – USE OF DEW POINT METHOD

Aim: To determine the humidity, % humidity, relative humidity, humid heat and humid volume by using dry and wet bulb temperature and due point method.

Requirements: Simple thermometer, Dry and wet bulb thermometer, glass beaker, ice, absorbent cotton, humidity chart, steam table and calculator.

Principle:

Humidification is a unit operation, which involves the transfer of liquid water in to mixture of air and water vapour. Though the definition describes the transfer of liquid in to vapour, the reverse process is also possible. The process in which the moisture(humidity) is decreased (transfer of vapour to liquid state) in the air is known as dehumidification.

The basic definitions of different terms involved in this experiment are discussed below.

1. **HUMIDITY:** Thenumber of kilogram or pound of water vapour carried by 1 kilogram or pound of dry air is known as humidity of the air.

   Generally, the amount of vapour is measured in the terms of pressure not in weight so mathematically,

   H= P_A / (P-P_A)

   Where H= mass of vapour present in air

   P_A= partial pressure of water in air P= total pressure= 760mm Hg.

   Since molecular weight of water and air is 18 and 29 respectively, therefore.

   H= 18P_A /[29(P-P_A)]

   Humidity can be found out by wet bulb and dry bulb temperature using humidity chart.

2. **Percentage Humidity (% H or Hp):** It is the 100 times ratio of the actual humidity of air to the humidity of saturated air at the same temperature and pressure.

   % Humidity = 100 (Actual Humidity)/ (Saturated air Humidity)

   % H = 100 H/Hs

   Where H= Humidity

   Hs = Saturated humidity

3. **Relative Humidity (H_R):** Relative humidity is expressed as percentage. It is defined as 100 times ratio of actual partial pressure of water vapour in the air- water mixture (p) to partial pressure of water vapour at saturation (ps).

$H_R = 100 \, p/ps$

4. **Humid Volume (V_H):** It is the volume in cubic foot occupied by 1 pound of dry air plus any moisture that air may contain.

$V_H = V_0 + Hp \, (V_S - V_0)/\, 100$

Here, V_0= Volume occupied by unit mass of dry air, it is obtained from

Humidity Chart =..cubic foot / dry air.

Hp, = percentage humidity from above calculation (2) =
%
So, V_H =.. cubic ft/ dry air.

5. **Humid Volume (V_H):**

$V_H = V_0 + Hp \, (V_S - V_0)/\, 100$

Where V_H = Humid volume
V_0 = dry air volume
V_S = saturated air volume Hp= %Humidity
V_S and V_0 can be obtained from humidity chart using dry bulb temperature.

6. **Humid heat (H_h or s):** It is the heat required to raise unit mass of dry air and its associated vapour through one-unit difference in temperature at constant pressure.

Mathematically it is expressed as $S = C_a + H \, C_w$

Where S= humid heat (Btu/IbUF)

C_a = specific heat capacity of dry air = 0.24 BTu/Ib°F

C_w =specific heat capacity of water vapour = 0.45 Btu/Ib°F therefore, s= 0.24+ 0.45H

7. **Dry bulb temperature:** It is the temperature of moist air when it is measure at rest by any instrument. This is not affected by the moisture content of air by radiation.

8. **Wet bulb temperature:** It is the dynamic equilibrium temperature attained by a water surface when exposed to air under adiabatic condition.

Dew point: It is the temperature to which a mixture of air-water vapour must be cooled (at constant humidity) to become saturated (i.e., to be in equilibrium with liquid at the dew point)

Procedure:

PART -I: Take Dry and Wet bulb thermometer in which water is present in the wet arm of the thermometer. Allow to stand for 10 minutes and note down the temperature of dry and wet bulb in both arm and consider as dry bulb temperature and wet bulb temperature respectively. Finally refer Humidity chart (Psychometric chart) for finding out the humidity, relative humidity, humid heat and humid volume.

OR

Take two thermometers and fix them in stand separately. Cover the bulb of one thermometer with wet absorbent cotton. Allow to stand for 10 minutes and noting down the temperature of dry and wet bulb in both arm and consider as dry bulb temperature and wet bulb temperature respectively. Finally refer Humidity chart (Psychometric chart) for finding out the humidity, relative humidity, humid heat and humid volume.

PART -II: Take a clean beaker; fill it with water up to half of its volume. Add crushed ice to water and stir it well. When fogginess appeared outside the beaker not down the temperature as dew point. Finally refer Humidity chart for finding out humidity and humid heat.

Observations & Calculations:
Part I
Thermometer data:

Dry bulb temperature (DBT): ....°F

Wet bulb temperature (WBT): .... °F

Humidity-chart data:
Humidity (H):...Ib water vapour/Ib dry air

% RH: …………………………..

V_0..cubic foot/dry air

Vs: ....cubic foot / dry air

H_H:... BTu/°F* Ib dry air

Calculation (Part I):

1. Partial vapour pressure of water in air (Pa):

H= 18 Pa / [29(P-Pa)]

Here, H = Humidity value obtained from Humidity chart P= 1 atm = 14.7 Ibf/in^2

By putting value of H and P find out value of Pa So, Pa=   bf/in^2

2. Percentage Humidity (% H or Hp):

%H= 100 × Pa /Ps × (P-Ps)/(P-Pa)

Here, Pa= from the above calculation (1).................................. bf /in^2

Ps = Partial pressure of water vapour in saturation obtained

using Steam table by considering Dry bulb temperature.

P= 14.7Ibf /in^2

So, %H =.. %

3. Relative Humidity (H$_R$):

HR= 100 × Pa /Ps
So, H$_R$=.. %

4. Humid Heat (H$_H$):

H$_H$ = 0.45H + 0.24
Here H = humidity from Humidity chart
So, H$_H$=.. BTu/°FIb dry air.

Part II

Thermometer data:

Due point temperature (DPT)..°F

Humidity chart data:

Humidity (H):...Ib water vapour / Ib dry air

Humid heat (H$_H$)...BTu/°FIb dry air.

Results:

Part I: The value of various parameters from humidity chart and calculation for Dry bulb and Wet bulb temperature method (PART-I) are shown Table-2.1 as below;

Table 2.1: Results obtained from Humidity Chart and calculation

Parameters humidity chart	value form	Calculated Value
Partial v.p. of water in air (Pa)		
Humidity (H)		
Percentage Humidity (%)		
Relative Humidity (H_R)		
Humid Heat (H_H)		
Humid Volume (V_H)		
Dry air Volume (V_0)		
Saturated air Volume (Vs)		

Part II: The value of Humidity(H) and Humid heat (H_H) for Due point method was … …. and respectively**.**

Report: The values of relative humidity and humid heat obtained from Humidity Chart and calculation did not show any significant difference.

Experiment No. 06

SIZE ANALYSIS BY SIEVING – TO EVALUATE SIZE DISTRBUTION OF TABLET GRANULATION

Aim: To determine the average particle size and particle size distribution of the given sample.
Requirements: Standard sieves set 20, 40, 60, 80, 100, 120, and Mechanical shaker, and bottom pan.

Principle:

Sieving or shifting is one of the simplest and most widely used methods for size separation of different grades. The sieves in the pharmacy consist of circular frame made up of wood or metal fitted with wire made up of copper alloys, stainless steel, horse hair, silk, nylon, or Terylene having uniform circular cross section. The sieves are designated by numbers. The number mentioned for the sieve is the number of openings in a length of 25,4mm or 1 inch in each direction. sieve no 10 means 10 meshes in 25.4mm in each direction and total 100 openings in 1 sq. Inch area termed as sieve no.10.

1	2	3	4	5	6	7	8	9	10
2									
3									
4									
5									
6									
7									
8									
9									
10									

Nominal size of the aperture:

This is the distance between the wires, so that it represents the length of the side of the square aperture. While it is the diameter of the largest sphere that it would pass the mesh, it is not necessarily the maximum dimension particle to pass. the plate like particles passes through diagonally and long fibrous particles require only suitable orientation.

Nominal diameter of the meshes:

The dimension and the number of meshes from the basic standards of the sieve. The wire diameter has been to give a suitable aperture size and also to have sufficient strength to avoid distortion.

Approximate screening area:

This standard expresses the area of the meshes as a percentage of total area of the sieve.

Aperture tolerance average:

Some variation in the aperture size is unavoidable and this variation is expressed as a percentage, is known as the aperture tolerance average.

Standards for powders:

The degree of coarseness or fineness of the powder is differentiated and expressed by size of the mesh of the sieve through which the powder is able to pass.

• Coarse powders: (10/44): A powder of which all the particles passes through a sieve no.1 (1.7mm aperture) and not more than 40% pass through sieve no.44 (355µm aperture).

• Moderately coarse powders (22/60): A powder of which all particles pass through sieve no.22 (710µm) but not more than 40% through sieve no.60 (250µm aperture).

• Moderately fine powders (44/85): A powder of which all the particles pass through sieve no.44 (355µm) but not more than 40% should pass through sieve no.85 (180µm).

• Fine powder (85/120): A powder which all particles passes through sieve no.85 and not more than 40% pass through sieve no.120 (125µm).

• Very fine powder (120): powder which all particles passes through sieve no.120 is known as fine powders.

• Micro fine powder: A powder of which not less than 90% of the particles should pass through sieve no. 350 (45µm)

• Super fine powder: A powder of which not less than 90% of the particles are less than (10µm).

Types of sieves:

a. **Woven wire sieves:** They are general purpose sieves and widely used. The type of woven sieves are of plain woven or twilled wave. And for fine sieving metal wire woven sieves are used these are used these are attached to mills and the wire is given with double crimp to preserve the alignment of the wire.

b. **Blotting cloth sieves:** silk, cotton, nylon, are generally woven from twisted multistand fibres. Nylon clothes are designated by their micrometr opening and available in different grades. These are used for separation of fine powders.

c. **Bar screens:** These are used in handling large and heavy pieces of materials The bars are fixed in parallel position and held by cross bars.

d. **Punched perforated plates**: (perforated screens): These sieves are used for coarse sieving and prepared by using a sheet metal of varying thickness with perforated holes. which they may be round, oval, square or rectangular these screens are used in the hammer mill.

Sieve shaker machine:

Principle: 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, so that the size separation is rapid and fast.

Construction: Standard sieves of different mesh numbers are available commercially as per the specifications of I.P and U.S.P. these sieves are fixed in mechanical shaker apparatus.

Working: Sieves are arranged in a nest with the coarsest at the top. A sample of powder is placed on the top sieve this sieve is set fixed to the mechanical shaker apparatus and shaken for certain period of time (20 minutes) the powder retained on each sieve is weighed.

Advantages: It is in expensive, simple and rapid with reproducible results.

Disadvantages: Lower limit of the particle is 50μm, if the powder is not dry, apertures get clogged with particles and leading to improper sieving. During shaking attrition occurs causing size reduction of particles this leads to error in the estimation.

Procedure:

Weigh 100grams of the given sample powder and is placed on the top most mesh of the sieve machine and operated for 30minutes. the particles retained on each sieve is collected separately and weighed the under size and the oversize particles is calculated. A graph of arithmetic mean aperture size vs. cumulative weight and arithmetic mean aperture size vs. % weight retained is plotting.

Sl. No	Mesh No.	Nominal aperture size	A.M of Nominal apertures	Wt. of particle retained	%wt.	Wt. size	Cumulative %over size	Cumulative % under size
1								
2								
3								
4								
5								
6								
7								
8								
9								

Observations & Calculations:

Arithmetic means = Sum of nominal aperture size × Individual sieve aperture size

Total number of sieves

Report:

The Average particle of the given sample powder was found to be …….

Experiment No. 07

TO STUDY THE EFFECT OF TIME ON THE RATE OF CRYSTALLIZATION

Aim: To study the process of crystallization of acetic acid

Requirements: Beaker, oxalic acid, Microscope, electronic balance

Principle:
The mechanism of crystallization of a substance from solution is explained by 3 steps
1. **Super saturation**

2. **Nucleus formation**

3. **Crystal growth**

Super saturation: When the solubility of a compound is a solvent exceeds the saturation solubility., the solution becomes super saturated and the compound may precipitate or crystallize. Super saturation can be achieved through.

1. Evaporation of solvent from the salt
2. Cooling of solution if the solute has a positive heat of solution
3. Formation of new solute as a result of chemical reaction.
4. Addition of a substance with is more soluble in solvent then the solid to be crystallized.

In the absence of seed crystal, significant super saturation is necessary to initiate of nuclei. The reaction rate of separation, particle size, uniformity, and distribution depends on two successive largely independent process namely nuclei growth of nuclei.

Nucleation: Nucleation refers to the birth of new phase with thin homogeneous super saturated liquid phase it is a consequence of rapid local flocculation at the molecular level when molecules or ions or atoms are in random motion in any small volume. Initially several molecules or atoms or ions associate to form nucleolus these clusters. They are losing however when enough particles associate to form an embryo, there is a beginning of the lattice arrangement and formation of new solid phase. In most of the cases embryo, the embryo lives very shortly and dissolves as soon as they form, an embryo may grow to such a size that it is thermodynamically equilibrium with the solution. The initially formed crystals are of molecular size which is termed as nuclei on certain occasion, the nucleus on certain occasion grown in dimension that are limited by the amount of materials available and thus form crystals.

Several methods are available for nucleation they are

1. Soft or weak crystals colloid with the moving parts of the crystallizer and these break and form into fragments which acts as nuclei.

2. In super saturated solution because of poor mixing, the needle like crystals are formed these needle-like structures grow faster and a poor quality of crystals are formed.

Procedure:

1. Take 50ml 0f water in 100ml beaker

2. Add 32 gms of oxalic acid

3. Agitate for about 15min

4. Heat the solution on a water bath for 10min and cool for about 15min.

5. After the formation of the crystals dry and calculate the percentage yield obtained

6. Observe the crystals under microscope.

Observations & Calculations:

Percentage yield $= \frac{Practical\ Yield}{Theoritical\ Yield} \times 100$

Report:

The % yield was found to be …….

Experiment No. 08

TO CALCULATE THE UNIFORMITY INDEX FOR GIVEN SAMPLE BY USING DOUBLE CONE BLENDER

Aim: To calculate uniformity or mixing index of given sample using double cone blender.

Requirements: Salicylic acid, Lactose, 0.1 M sodium hydroxide, phenol red solution, Beaker, spatula etc

Procedure:

1. Place 10gm salicylic acid and 90gm lactose in 250ml plastic beaker.
2. Arrange the beaker in double cone blender.
3. Set bender at 30 rpm and operate it for 5,10,15,20,25 and 30 minutes.
4. Withdraw sample of 1.2g after each mixing operation.
5. Determine the content of salicylic acid in blender mixture after various time intervals.

Assay for Salicylic acid content-

Dissolve 1.2gm of sample (containing approximately 0.120gm of salicylic acid) in 30ml of alcohol and 20 ml Of water. Titrate with 0.1 M sodium hydroxide using 0.1 ml of phenol red solution as indicator.

Observations & Calculations:

Concentration of salicylic acid in blender mixture				
Time in min	Amount of sample withdrawn(gm)	Fill volume of the blender 40%	Fill volume of the blender 60%	Fill volume of the blender 80%
5				
10				
15				
20				
25				

Calculation

$$\sigma_0^2 = x(1-x) \quad \sigma_r^2 = x(1-x)/N$$

Where X = fraction of component in the original mixture σ_0^2 is the variance of unmixed state and σ_r^2 is the variance of completely mixed random state.

$$\sigma^2 - 1/(n-1) \sum_{i=1}^{n} (y1-y)^2$$

Report:

The unfromity index was found to be …….

21

Experiment No. 09

DETERMINATION OF RADIATION CONSTANT OF BRASS & IRON

Aim: Determine of radiation constant of metal cylinder made of brass & iron

Requirements: Metal cylinder, Thermometer, weighing box, gas cylinder, match box etc.

Principle:

When a brass cylinder is heated to a temperature and suspended in air, the cylinder loses its heat by radiation and conversion. Heat transmission takes place by means pf energy transfer using air as electromagnetic waves.

A black body is a perfect emitter and absorbs for the total art of radiation fall on it. The total amount of radiation emitted by s black body may be calculated using Stephan Boltzman law :

$$q = bAT^4$$

in condition, the energy transfer occurs by transmission of momentum of individual molecules.

Radiation constant is calculated using the following equation:

$$MS \, d\theta/dt = \alpha \, A \, \{ (T1/100)^4 - (T2/100)^4 \}^{1.23} + \beta \, A \, (T1\text{-}T2)$$

Procedure:

1. Take a metal cylinder and clean it.
2. Determine surface area (s) and weight (M)of cylinder.
3. Placed it on hot plate and switch on the hot plate.
4. Heat the metal cylinder to maximum temperature (T).
5. Remove metal cylinder with the help of tong.
6. Place it over hot pad to reduce heat loss by conduction.
7. Note the temperature reading after interval of 2 minutes. continue note the reading
a. until it reaches to room temperature.
8. Find out dT/dt.
9. Draw cooling curve by taking time in minutes on X axis and temperature on Y axis.
10. Also draw graph bt taking temperature on X axis and dT/dt(rate of change of a. Temperature) on Y axis.
11. Find out the slope of the plot(h1 s/c).
12. Calculate coefficient of convection.
13. Substitute this values in following equation on get radiation constant (h).
$$M \, dT/dt = h_1 S/C(T\text{-}T_{air}) + h_2 S/C(T^4\text{-}T^4 S)$$
14. Repeat the same procedure for metal made of other material.

Observation and calculations:

1. Diameter of the cylinder (d)=..........

2. Radious of the cylinder (r)=D/2=..........

3. Height of cylinder (h)=.........

4. Surface area of cylinder = $2\prod r(r+h)$=.........

5. Mass of the body =.........

6. Room temperature =........

7. Specific heat of metal = ...C^0

Table: Date for construction of cooling curve

Sl. No	Time (min)	Temperature (c ^{0})

Sl. No	Temperature (c ^{0})	Radiation constant (α)

Report:

The radiation constant was found to be …….

Experiment No. 10

DETERMINATION OF RADIATION CONSTANT OF UNPAINTED AND PAINTED GLASS

Aim: Determine of radiation constant of metal cylinder made of unpainted and painted glass

Requirements: RBF, Thermometer, weighing box, gas cylinder, match box etc.

Principle:

When a brass cylinder is heated to a temperature and suspended in air, the cylinder loses its heat by radiation and conversion. Heat transmission takes place by means pf energy transfer using air as electromagnetic waves.

A black body is a perfect emitter and absorbs for the total art of radiation fall on it. The total amount of radiation emitted by s black body may be calculated using Stephan Boltzman law :

$$q = bAT^4$$

in condition, the energy transfer occurs by transmission of momentum of individual molecules.

Radiation constant is calculated using the following equation:

$$MS \, d\theta/dt = \alpha A \{ (T1/100)^4 - (T2/100)^4 \}^{1.23} + \beta A (T1-T2)$$

Procedure:

1. Take two RBF, clean it and Paint one of the RBF.
2. Determine surface area (s) and weight (M)of the RBFs.
3. Placed it on water bath and switch on the water bath and place the water filled RBFs (Painted and Unpainted) .
4. Heat the RBFs until the water is boiling.
5. Remove the RBFs with the help of tong.
6. Place it over hot pad to reduce heat loss by conduction.
7. Note the temperature reading after interval of 10 minutes. continue note the reading
a. until it reaches to room temperature.
8. Find out dT/dt.
9. Draw cooling curve by taking time in minutes on X axis and temperature on Y axis.
10. Also draw graph bt taking temperature on X axis and dT/dt(rate of change of a. Temperature) on Y axis.
11. Find out the slope of the plot (h1 s/c).
12. Calculate coefficient of convection.
13. Substitute this values in following equation on get radiation constant (h).
$$M \, dT/dt = h1S/C(T-T_{air}) + h2S/C(T^4-T^4S)$$
14. Repeat the same procedure for metal made of other material.

Observation and calculations:

1. Diameter of the cylinder (d)=..........

2. Radius of the cylinder (r)=D/2=..........

3. Height of cylinder (h)=.........

4. Surface area of cylinder = $2\prod r(r+h)$=.........

5. Mass of the body =.........

6. Room temperature =........

7. Specific heat of metal = ...C^0

Table: Date for construction of cooling curve

Sl. No	Time (min)	Temperature (c⁰)

8.

Sl. No	Temperature (c⁰)	Radiation constant (α)

Report:

The radiation constant was found to be …….

Experiment No. 11

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

Aim: 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: Metal balls, Calcium carbonate, weighing balance, sieves etc.

Principle:

Size reduction is the operation carried out for reducing the size of bigger particles into smaller one of desired size and shape with the help of external forces.

Mechanism of size reduction

➢ Impact —particle concussion by a single rigid☐ force (hammer).

➢ Compression—particle disintegration by two☐ rigid forces (nutcracker).

➢ Shear —produced when the particle is☐ compressed between the edges of two hard surfaces moving tangentially.

➢ Attrition —arising from particles scraping against☐ one another or against a rigid surface (a file).

Size reduction theories

The energy requirement for particle size reduction is a☐ function of input and output of particle size, hardness, strength and other properties of solids. Various theories for energy requirement are:

Rittinger's theory

Rittinger's theory suggests that energy required in a size reduction process is proportional to the new surface area produced.

where, E = energy required for size reduction K_R = Rittinger's constant

S_i = initial specific surface area S_n = final specific surface area

Application: It is most applicable in size reducing brittle materials undergoing fine milling.

Kick's theory

Kick's theory states that the energy used in deforming (or fracturing) a set of particles of equivalent shape is proportional to the ratio of change of size, or:Assumes that the energy required to reduce a material in size was directly proportional to the size reduction ratio dDs/Ds. where,

E = energy required for size reduction K_K = Kick's constant

d_i = initial diameter of particles d_n = final diameter of particles

Application: For crushing of large particles Kick's theory most useful.

Bond's theory

Bond's theory states that the energy used in crack propagation is proportional to the new crack length produced.

where, E = energy required for size reduction K_B = Bond's work index

d_i = initial diameter of particles d_n = final diameter of particles

Application: This law is useful in rough mill sizing. The work index is useful in comparing the efficiency of milling operations.

The ball mill consists of a cylindrical shell slowly turning about a horizontal axis and filled to about half its volume with a solid grinding medium.

At very high speeds the balls are carried right round in contact with the sides of the mill and little grinding takes place, the mill is then said to be centrifuging. The speed of the mill at which centrifuging occurs is called Critical speed. Centrifugal force keeps the ball in contact with the wall and with each other during the upward movement.

The critical speed of the rotation can be determined by the following equation.

$$Cs=76.63/\sqrt{D}$$

Procedure:

1. The metal ball to be used in mill should be washed and cleaned
2. Take 100 gm Calcium carbonate and loaded into ball mill.
3. Weigh the given sample and obtain the initial size distribution by sieving.
4. Grind the sample in the ball mill for 30 minutes noting the energy consumed during the process.
5. Sieve the powder through sieve no. 10. Measure the size distribution by sieving
6. The remaining sample is again transferred into the container

7. Again operate the ball mill for 60 minutes

8. The powder is again sieve through sieve no 16

9. Determine average particle size

Observation and calculations:

Determination of effect of number of balls on grinding operation of ball mill

Sl No	Initial weight of the powder(gm)	Number of balls	Weight of fines(gm)	% weight of fines
1.	100	8		
2.	100	12		
3.	100	16		
4.	100	20		
5.	100	204		

Critical speed of rotation of the ball mill $Cs=76.63/\Box D$

Where, Cs=Critical speed of rotation in rpm

D =Inner diameter of the ball mill in inch

D =……cm =…. inch

Report:

Percentage of fines increases with increases in the number of balls, hence size reduction is increased by increasing the number of balls.

- The critical speed of rotation of the ball mill=

- The optimum speed of rotation of ball mill=

Experiment No. 12

STEAM DISTILLATION – TO CALCULATE THE EFFICIENCY OF STEAM DISTILLATION

Aim: To Perform steam distillation of clove oil. To determine the percentage recovery of the clove oil during steam distillation and to determine the % efficiency of steam distillation.

Requirements: Steam distillation assembly (Round bottom flask, measuring cylinder, Steam can, Adaptors, Bend tubes, Burette stand, Thermometer), Weighing balance, Beakers (250 ml), Separating funnel, clove oil, water

Principle:

Steam distillation refers to a process in which live steam is in direct contact with the distilling system in either batch or continuous operation. The basis of steam distillation rest on the fact that with most organic substances and these mixtures will boil at temperature below that of either water especially if the organic compound has a high boiling point at which it may be unstable or decom- pose. As long as liquid water is present, the high boiling component will vaporize at a temperature well below its normal boiling point. Steam distillation is mainly used for:

1. Separating relatively small amounts of volatile impurity from a large amount of material.
2. Separating appreciable quantities of higher boiling materials.
3. Recovering high-boiling materials from small amounts of impurity which have a higher boiling point.
4. Distilling thermally unstable or components react with other components associated with it at the boiling temperature.
5. The materials cannot be distilled by indirect heating even under low pressure because of the high boiling temperature.
6. Distillation where direct fired heaters cannot be used because of fire hazards.

In this present experiment the separation of clove oil from water is done by steam distillation. The clove oil (eugenol) is having a molecular weight of 164.20 and boiling point of 254^0C.when this is mixed with water the boiling point is reduced and the mixture starts boiling at fairly low temperature. Steam is widely used because of its energy level, cheapness, and availability. The

weight of clove oil which distills along with water can be determined with the help of the equation,

$$W2=W1(M2P2)/(M1P1)$$

Where, W2=Weight of clove oil (g); W1=Weight of water (g); M1=Molecular mass of water; M2=Molecular mass of clove oil; P1=Partial pressure of water (mm Hg); P2=Partial pressure of clove oil(mm Hg)

The partial pressure of the clove oil at the boiling point can be determined by,

Ptotal=Pwater+Pclove oil

The efficiency of the distillation=Amount recovered/Amount used ×100

Procedure:

1. Measure 15ml of clove oil and 50ml of water

2. Add both the liquids in the round bottom flask.

3. Arrange the steam distillation assemble as shown in the figure (Fig. 40.1).

4. Fill the steam can with water and heat it so that a continuous flow of steam is generated

5. As the steam entered into the still the mixture is heated.

6. Continuously monitor the temperature in the thermometer.

7. The temperature is noted when the mixture starts to boil.

8. The mixture starts boil and distils off to the receiver via condenser.

9. Note down the temperature as well as the amount collected at an interval of 5 min.

10. Distillation is carried out till ≤5mlof mixture is remaining in the RB flask.

11. It is advised not to distil the content in the flask to dryness.

12. Separate the contents in the receiver with the help of a separating funnel.

13. Find out the weight of the clove oil as well as water individually

14. Find out the practical recovery of clove oil.

Observation and calculations:

Determination of Rate of Distillation

Determination of Amount of Distillate Collected with time

Sl No	Time (min)	Temperature(ºC)	Amount collected (ml)	Rate of distillation (ml/min)

Determination of Theoretical Recovery

Weight of clove oil (Initial)- 15x Weight per ml of clove oil

---- --- -- = × gm

Weight of clove oil distilling with 1 gm of water $W_2 = W_1 (M_2 P_2) / (M_1 P_1)$

$$W_2 = 1 \times (164.20x \text{ c}) (18xb)$$

$$= ---- gm$$

Weight of water separated(distilled)=--------d ------------------------ gm

Weight of clove oil distilled (W2x d) = ---------------------------gm

Determination of Practical Recovery

Weight of clove oil separated (Distilled)=------e---gm Weight of water separated= gm

Practical recovery of clove oil=e/x*100

The efficiency of the distillation= Practical recovery/Theoretical recovery

--------≡------

Report:

The steam distillation is performed.

The weight of clove oil distilled with 1gm of water=--------- The efficiency of the distillation=

---%

The average rate of distillation= -----------------------------ml/min

Experiment No. 13

TO DETERMINE THE OVERALL HEAT TRANSFER COEFFICIENT BY HEAT EXCHANGER

Aim: To determine the overall heat transfer coefficient based on the inner and outer surface of condenser.

Requirements: Round bottom flask, Condenser, Conical flask, Heating source (burner or mantle), Burette stand, Beaker250ml, Thermometer, stop watch, Measuring cylinder

Principle:

The overall heat transfer coefficient U is a measure of the overall ability of a series of conductive and convective barriers to transfer heat. It is commonly applied to the calculation of heat transfer in heat exchangers.

A condenser used in the laboratories is considered as a simple heat exchanger and the demonstration of overall heat transfer coefficient can be done with the help of this. For a condenser, the rate of heat transfer can be determined by simple equation based on the flow rate of the cold liquid.

$$q = m_c c_{pc}(T_{cb} - T_{ca})$$

Q=Rate of heat transfer; M_C=Mass flow rate of cold water (liter/sec); C_{pc}=Specific heat of cold water (J/kg); T_{cb}=Temperature of the leaving cold water (°C); T_{ca} =Temperature of the entering cold water (C); Rate of heat transfer, based on the area of heating surface (tube) can be determined as follows,

$$dq/da = U\Delta T \quad \text{-(Eq:3.2)}$$

dq/da-over all local temperature difference (Th-Tc, where T_h is the average temperature of the hot fluid, Tc is the average temperature of the cold fluid)

U=Local overall heat transfer coefficient

Since ΔT can vary considerably from point to point along the tube, the heat flux is proportional to ΔT. To apply this equation for the total surface area, the equation must be integrated. This can be done with some simple assumption that, the overall coefficient U is a constant, the specific heat of hot and cold fluids is constant, the flow is steady in both parallel or counter current.

After integration and proper arrangement, the equation becomes

$$q = \frac{UA\Delta T2 - \Delta T2 - \Delta T1}{\ln(\frac{\Delta T2}{\Delta T1})} = UA\Delta T1 \quad \text{-(Eq:3.3)}$$

Where, q-Rate of heat transfer, U=overall heat transfer coefficient, A=The total surface area of heat transfer

$$\Delta TL = \frac{\Delta T2 - \Delta T1}{\ln(\Delta T2 / \Delta T1)} \quad \text{-(Eq: 3.4)}$$

When AT1 and AT2 are nearly equal the arithmetical average can be used instead of ΔTL Thus the overall heat transfer coefficient can be written as

$$U = q/A\Delta T_L \quad \text{-(Eq:3.5)}$$

In the condenser the actual heating surface is glass tube of certain thickness, so the heat transfer is based on either the inner surface area or the outer surface area.
So,

$$q = U_i A_i \Delta T_L$$
$$q = U_0 A_0 \Delta T_L$$

From this we can determine the overall heat transfer coefficient based on inner and outer surface

$$U_i = q/A_i \Delta T_L$$

$$U_0 = q/A_0 \Delta T_L$$

So $U_0/U_i \ A_i/A_0 \ D_i/D_0$

Since ΔTL and q are independent of the choice of area we can write, $U_0 A_0 = U_i A_i$

Thus, $$U_0 = U_i A_i / A_0$$

$$U_i = U_0 A_0 / A_i$$

In plate type b heat exchanger, the area on the side S are same so there is only one value of U.

Procedure:
1. Inlet of the condenser is attached to the tap, and the outlet is placed to a 250 ml beaker
2. Arrange the assembly in such a way that it can be heated with a suitable
heating medium. (either burner or mantle) (Temperature 100 °C).
3. Steam is generated by heating.
4. When steam is steady through the condenser, start collecting the liquid from the
outlet of the condenser and time is noted.
5. Collect 200 ml of water and corresponding time to collect 200 ml is also noted.
6. experiment is repeated for different water velocity by adjusting the knob of the tap.
7. For each velocity inlet and outlet temperature of the cold water is
noted (Water circulating, through the condenser)
8. The inner and outer diameters of the condenser tube are determined.
9. The length of the inner tube of the condenser is determined.

10.   Find out the mass flow rate

11.   Find out the heat transfer and overall heat transfer coefficient.

Report:

The overall Heat transfer coefficient based on inner and outer surface area is calculated and reported.

Uo=   ------------------

Uo=

Experiment No. 14

DESCRIPTION OF CONSTRUCTION WORKING AND APPLICATION OF PHARMACEUTICAL MACHINERY SUCH AS ROTARY TABLET MACHINE, FLUIDIZED BED COATER

Aim: To describe of Construction working and application of Pharmaceutical Machinery such as rotary tablet machine, fluidized bed coater.

A. Rotary tablet Punching machine-

Principle:

In the tablet compression machine main principle is compressing of the upper and lower punch in a die hole, the hydraulic pressure plays a key role. This pressure is transmitted unreduced through the static fluid. Any externally applied pressure is transmitted via static fluid to all the direction in same proportion. It also makes it possible to multiply the force as needed. If we increase the hydraulic pressure more compressing force on tablet, then it becomes more hard.

Construction:
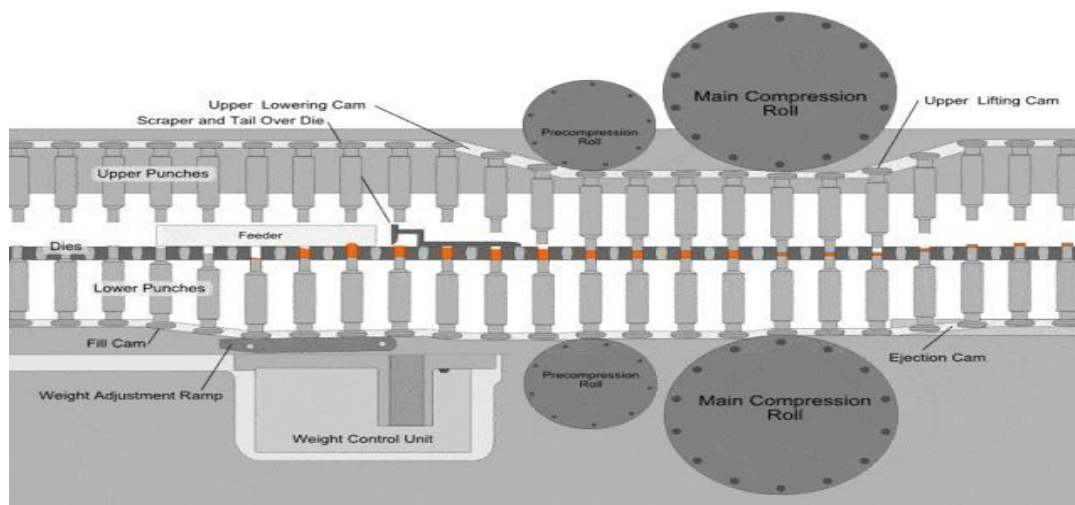
Construction of the tablet punching machine is given as per the above diagram. It consists of various main parts such as

1. Hopper
2. Upper and lower punches
3. Cam track
4. Die table
5. Compression rollers
6. Ejection cam
7. Weight adjustment unit

Working:

Different Stages of Tablet Compression Process:

Tablet compression process is divided into four distinct stage. These stage including filling, metering, compressing and ejection.

Tablet compressing stage:

Filling	Formulation is overfilled at the compressing station
Metering	Overfill is removed
Compression	Tablet is formed by pressure of punches within die
Ejection	Tablet is ejected from die

1. **Filling:** The filling stage of tablet compression process involves transfer of raw materials into position for tablet compression. These raw materials have undergone prior processing by wet granulation, dry granulation (roller compaction), sizing or other process. The final formulation is then blended to yield a homogeneous blend. The blend then flows to the compressing machine punch-die cavity. The punch die cavity is composed of punch die and lower punch. The position of lower punch within the die determines the volume of the punch-die cavity. This volume must be appropriately sized for the weight of granulation to be compressed into tablets. The granulation is overfilled on the die table (turret) to ensure complete filling of the punch-die cavity volume.

2. **Metering:** The metering stage of the tablet compressing process involves removal of excess granulation from the compressing machine. This stage enables the exact weight (volume) of granulation to be compressed into tablets. The exact weight of granulation is controlled by the height of the lower punch in the die. The height of the lower punch is controlled by the metering cam (also called the dosage cam). The lower punch is raised to the appropriate level in the die to provide the exact weight of granulation in the punch-die cavity. The excess granulation is scraped from the surface of the die table. The metering stage is similar to the method used to measure flour when baking a cake. A measuring cup is first over-filled with flour; then a knife is used to scrape off the excess. The exact amount of flour is then left in the measuring cup.

3. **Compression:** The compression stage of the tablet compressing process forms the tablet. This stage involves bringing together the upper and lower punches under pressure within the die

to form the tablet. As the punches enter the compressing stage, the upper and lower punches move between two large wheels called pressure rolls. These pressure rolls push the punches together to form the tablet. The distance between the upper and lower punches determines the thickness and the hardness of the tablet. When the punches are close together, a thin and hard tablet is created. When the punches are farther apart, the tablet made is softer and thicker. The proper balance of thickness and hardness determines the optimum roll distance for any specific product. These adjustments are made while keeping the tablet weight constant.

4. **Ejection:** The ejection stage of the tablet compressing process involves removal of the tablet from the lower punch-die station. In this stage, the upper punch retracts from the die cavity and rises above the turret table. Then the lower punch rises in the die, which in turn pushes the tablet upward to the top surface of the die table and out of the die cavity. A scraper (also called take off scraper or tablet rake- off) then pushes the tablet off the die table away from the compressing machine into the collection container.

B. **Fluidized Bed Coater-**
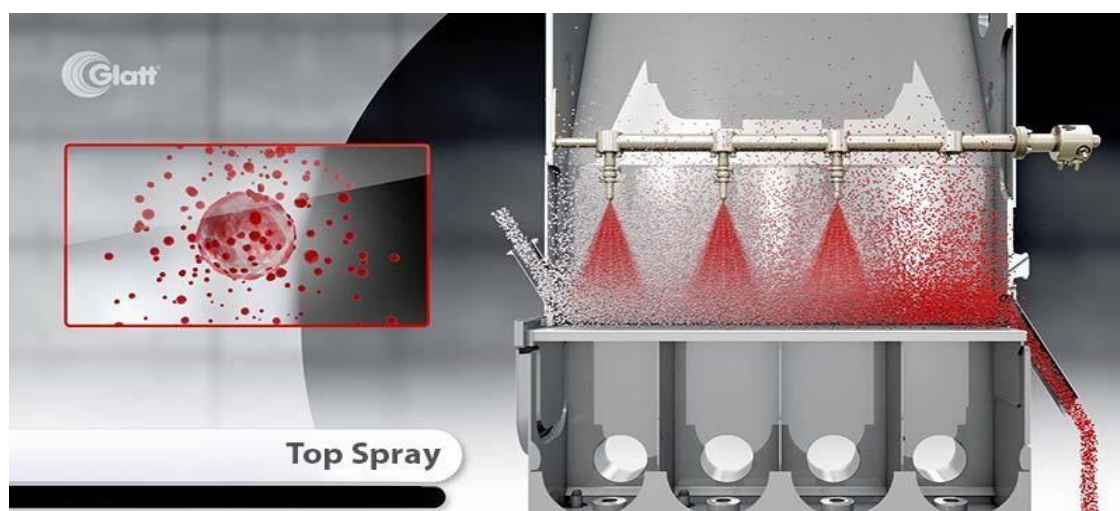
Bottom Spray

Report:

The construction, working and application of Pharmaceutical Machinery such as rotary tablet machine, fluidized bed coater was studied.

Experiment No. 15

DESCRIPTION OF CONSTRUCTION WORKING AND APPLICATION OF PHARMACEUTICAL MACHINERY SUCH AS FLUID ENERGY MILL, DEHUMIDIFIER

Aim: To describe the construction working and application of pharmaceutical machinery such as fluid energy mill, dehumidifier.

A. Fluid Energy Mill-

Principle:

It works on the principle of impact and attrition.

Construction:

Fluidized Energy mill, also known as micronized or jet mill is a type of mill that consists of a hollow toroid which has a diameter of 20-200mm depending on the height of the loop which can be 2 m.

FLUID ENERGY MILL
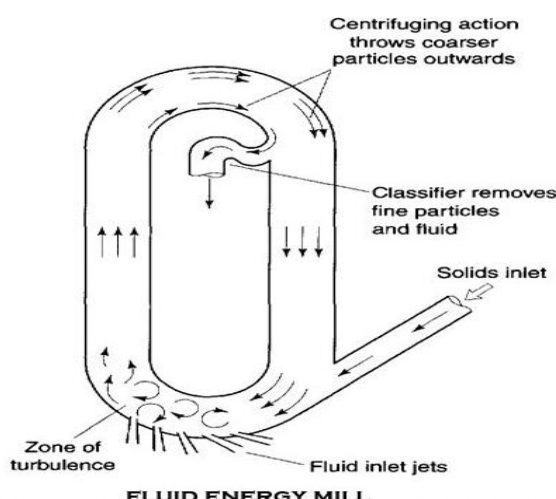
Working:

It operates by particles impaction and attrition. A fluid or milling gas, usually air or inert gas is injected as a high-pressure jet through nozzles at the bottom of the loop. The powder particles in the mill are accelerated to high velocity. The kinetic energy of the air plus the turbulence created causes interparticle (particle-particle collision) and particle-wall contact resulting in particle size between 2 and 10 micrometres. The fluidized effect transports the particles to a classification zone where the size classifier retains the particles until sufficiently fine to be removed. The particle size and share are determined by:

- The speed of air/inert gas

- Feed rate and size

- The configuration of the mill

- Design of the classifier

- The position of the nozzle

- The impact between the feed and air

Fluidized energy mill subclasses have no moving parts and primarily are distinguished from

one another by the configuration and/or shape of their chambers, nozzles and classifiers. They

include;

- Tangential jet

- Loop/oval

- Opposed jet

- Opposed jet with dynamic classifiers

- Fluidized bed

- Moving target

- Fixed target

- High-pressure homogenizers.

Uses:

- Fluidized energy is used in milling thermolabile materials

- It is the choice of mill when higher degree of drug purity is required

- Fluidized energy mill is used for the fine grinding of frits, Kaolin, Zircon,

titanium and calcium, alumina.

Advantages of Fluidized Energy mill:

- The machine has no moving parts and thus the tendency of contamination due to wear of

parts is minimized.

- The equipment is easily sterilized.

- Small particle size (between 2 and 10) is usually obtained at the end of milling.

- Thermolabile materials can be milled with little degradation since the heat produced by the

process is nullified by the cooling effect of the expansion of the compressed gas.

Disadvantages of Fluidized Energy mill:

- Tendency of forming aggregates or agglomerates after milling.

- Generation of amorphous content due to high energy impact.

- Formation of ultra-fine particles.

B. Dehumidifier-
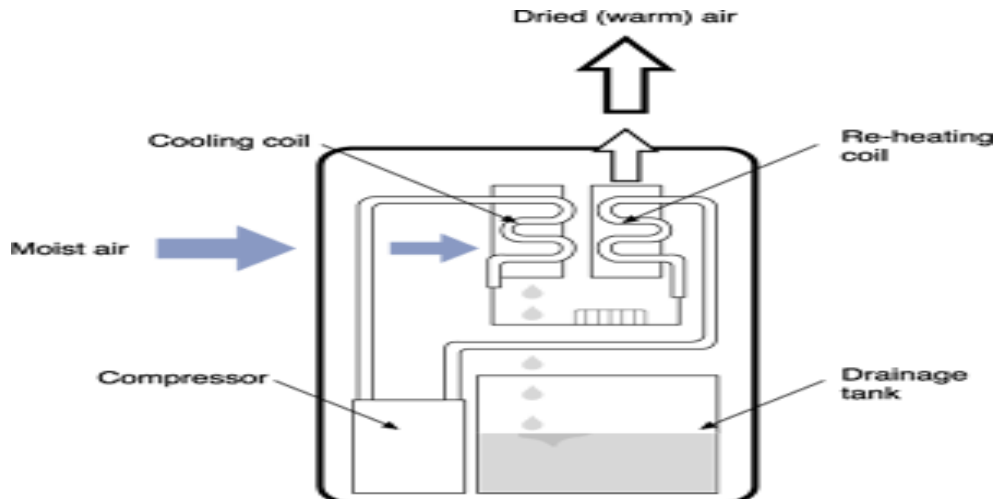
Construction:

A dehumidifier is generally a household appliance which reduces the level of humidity in the air, usually for health or comfort reasons, or to eliminate musty odor. Large dehumidifiers are also used in commercial buildings such as indoor ice rinks to control the humidity level. By their operation, dehumidifiers extract water from the conditioned air. This collected water (usually called condensate) is not normally used for drinking, and is often discarded.

Working:

Working on the same principle as a refrigerator, moisture-filled air is drawn into the dehumidifier and passed over a cold, 'refrigerated' coil. The damp air condenses on the cold coil into water droplets which are collected in the water tank. The air is then passed over a warm coil and sent out from the dehumidifier, producing clean, dry air. In cold conditions the water that is passed over the cold coil may freeze. Dehumidifiers utilize a hot gas defrost system, enabling hot gas to be transferred into the cold coil, reversing the freezing process.

1. Warm, moist air is sucked in.
2. An electric fan draws the air inward.
3. The warm air passes over freezing cold pipes.
4. The air passes over a heating element.
5. Warm, dry air blows back into the room.
6. The moisture that was in the air originally drips down into a collecting tray (or bucket) at the bottom of the machine.

7. A plastic float in the machine rises upward as the collecting tray fills up.

8. The float trips an electric switch that turns off the fan and switches on an indicator light telling you the machine needs emptying.

Uses:

-   Use an extractor fan or open a window when taking a bath or shower.

-   Wipe down wet walls and floors in your bathroom after use.

-   Seal cracks in your walls with a caulk or sealant.

-   Place wet clothes to dry next to an open window.

-   If possible, dry your clothes outside.

-   When cooking on a hob, use your extractor fan hood.

Report:

The construction, working and application of pharmaceutical machinery such as fluid energy mill, dehumidifier was studied.

Experiment No. 16

DEMONSTRATION OF COLLOID MILL, PLANETARY MIXER, FLUIDIZED BED DRYER, FREEZE DRYER AND SUCH OTHER MAJOR EQUIPMENT

Aim: To demonstrate the construction, working and application of pharmaceutical machinery such as colloid mill, planetary mixer, fluidized bed dryer, freeze dryer.

A. Colloidal Mill-

Principle:

Colloidal mill consists of two steel discs having clearance between them. Equipment based on the combined impact and Attrition. When the material is passed through these discs, they get sheared. Thus coarse particles are broken down in small particles due to shear.

Construction:

It consists of high speed rotor and stator with conical milling surface. Milling surface may be rough or smooth surface and rough used for fibrous.

Working:

The product to be shared is passed through the inlet which is on top of the mill known as a hopper. It is then passed through to the narrow gap between the rotor and stator. The rugged edges shear the material in to the tiniest particles depending on the rotation speed. This mill uses a hydraulic system to shear the material into the final product. It mainly works on the material that will be suspended on the liquid or emulsions.

As you continue to add the heavier liquid, the suspension floats to the narrow gap and the shearing process continues. As the process continues, the two viscous elements continue to mix. There are models where both the stator and rotor together and there are other models where only the roto rotates and the stator remains still.

Uses:

- Colloidal mill is used for the preparing colloidal dispersion, suspension, emulsion and ointments.
- It is used for dry milling.
- Fibrous material can be milled using rough surface rotor and stator.

- Colloidal mill can be sterilized. So it is used in the production of sterile products.

- It helps to improving the stability of emulsions

Diagram:
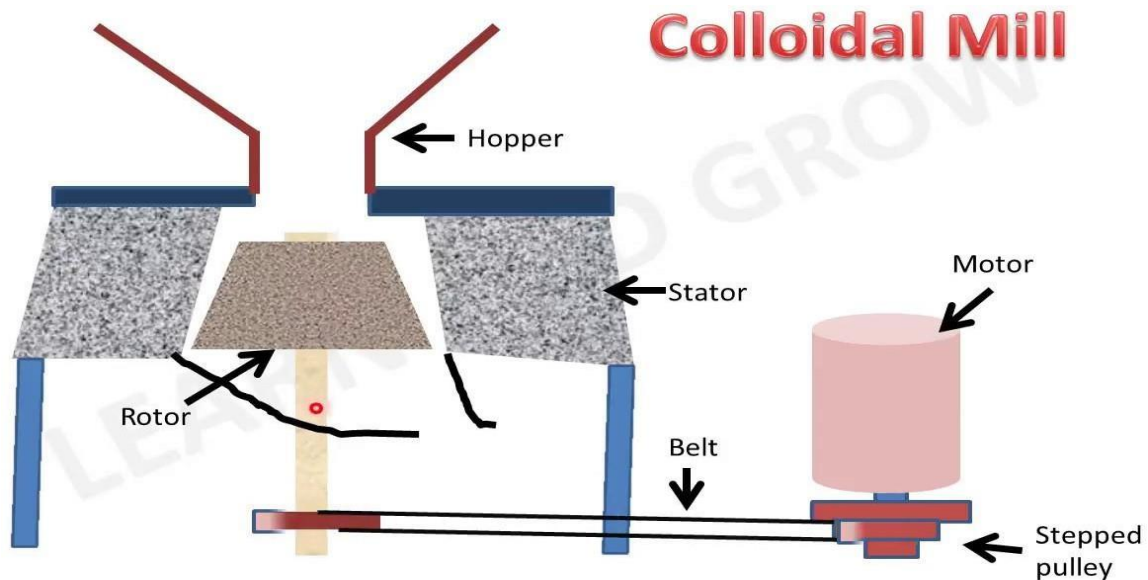
B. Planetary Mixers-

Principle:

Planetary mixers are one of the most widely used mixers in the pharmaceutical industry. In the pharmaceutical industry, the planetary mixer is often used for basic operations of mixing, blending, and low shear granulation. This machine is also used in other industries like cosmetics and personal care products, food, glass, cements, ceramics, metal industry etc.

Construction:

- The planetary mixer has two blades which rotate on their own axes, while they orbit the mix vessel on a common axis.
- The blades continuously advance along the periphery of the vessel, removing material from the vessel wall and transporting it to the interior.
- These mixers are ideal for mixing and kneading viscous pastes or putty like materials.

Working:

Planetary mixer is a new high efficiency and no dead-dead mixing equipment, it has a unique, novel mixing form, the kettle has two or 3 of multi-layer pulp leaf mixer and agitator in the body axis around the vessel revolution, and at different speeds around its own axis high speed rotation, so that the material in the kettle for complex movement, the efficiency of a strong times that of an ordinary mixer.

Currently common planetary mixers usually have two loss speed planetary agitator (usually multiple oblique blades, twist, frame), one or two high speed planetary agitator (dispensing plate) and scraping paddles.

Uses of planetary mixers:

- Planetary mixers are ideal for mixing of pharmaceutical creams, ointments, ceramics, colour and pigments, resin, ink, cosmetic creams, herbal creams etc.
- Planetary mixers are also ideal for mixing and kneading viscous pastes under atmospheric or vacuum conditions.
- Used in the mixing of viscous, heat sensitive and cohesive pastes, dough, and moist etc.

Diagram:

C. Fluidized Bed Dryers-

Principle:

- In fluidized bed dryer, hot air is passed at high pressure through a perforated bottom of the container containing granules to be dried.
- The granules are lifted from the bottom and suspended in the stream of air.
- This condition is called fluidized state.
- The hot gas surrounding every granule to completely dry them.
- Thus, material or granules are uniformly dried.
- The hot air/gas used for drying can be generated by either steam coils or a combustion furnace.
- The angle of response of gas solid mixture is zero and it assumes the shape of the vessel that contains it.
- In fluid bed drying uniform conditions of temperature, composition and particle size distribution is achieved throughout the bed because of complete mixing between the solids and gas is obtained.

Construction:

- The dryer is made of stainless steel or plastic.
- A detachable bowl is placed at the bottom of the dryer, which is used for charging and discharging.
- The bowl has a perforated bottom with a wire mesh support for placing materials to be dried.
- A fan is mounted in the upper part for circulating hot air.
- Fresh air inlet, prefilter and heat exchanger are connected serially to heat the air to the required temperatures.
- The temperature of hot air and exit are monitored.
- Bag filters are placed above the drying bowl for recovery of fines.

Working:

- When the velocity of the air is greater than the settling velocity of granules, the granules remain partially suspended in the gas stream.
- After some time, a point of pressure is reached at which frictional drag in the particles is equal to the force of gravity.
- The granules rise in the container because of high velocity gas and fell back in a random boiling motion.
- This condition is said to be fluidized state.
- Drying is achieved at constant rate and falling rate period is very short.

Advantages:

- It requires less time to complete drying these 20 to 40 mins compared
- To 24hrs of tray dryer. Handling time is also short. It is 15 times faster than the tray dryer.
- Hot spots are not observed in the dryer, because of its excellent mixing and drying capacities.
- It facilitates the drying of thermo labile substances, since the contact time is short.
- It can be used either as batch type or continuous type.

Disadvantages:

- Many organic powders develop electrostatic charges during drying which can be avoided by efficient electrical earthing of the dryer.
- The turbulence of the fluidized state of granules may cause attrition of some materials resulting in production of fines which can be avoided by using suitable binding agent.
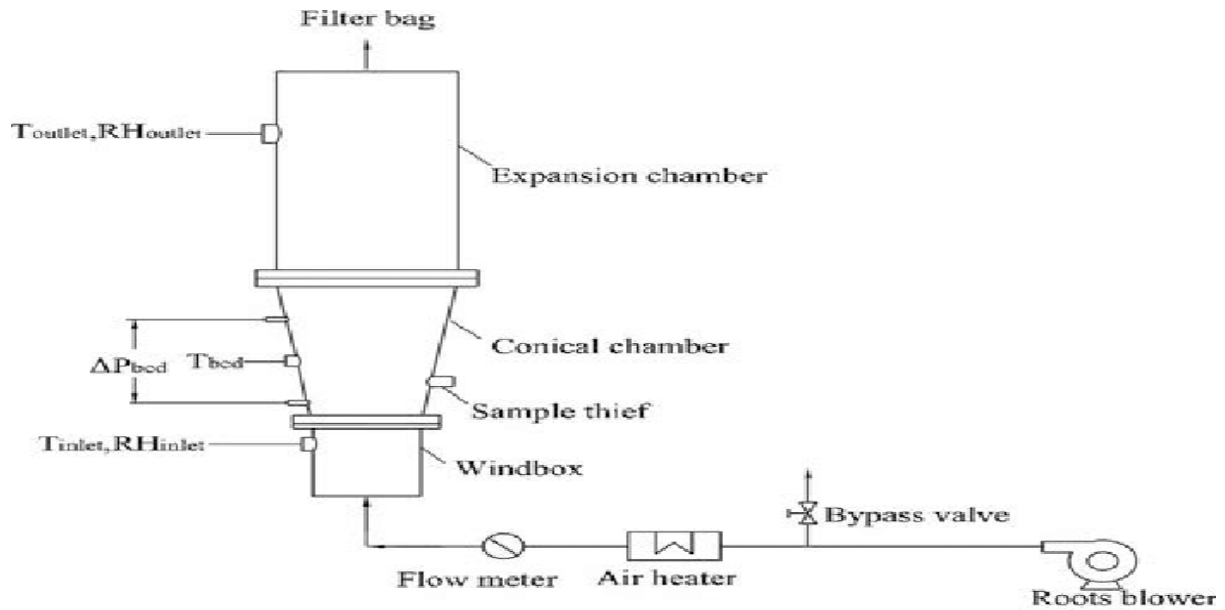
Toutlet,RHoutlet : Outlet air temperature and relative humidity
Tbed: Bed temperature
ΔPbed: Bed differential pressure
Tinlet,RHinlet : Inlet air temperature and relative humidity

D. Freeze Dryer-

Principle:

Freeze- drying is a process of drying in which water is sublimed from the product after it is frozen. It is a drying process applicable to manufacture of certain pharmaceuticals and biologicals that are thermolabile or otherwise unstable in aqueous solutions for prolonged storage periods, but that are stable in the dry state.

The main principle involved in freeze drying is a phenomenon called sublimation, where water passes directly from solid state (ice) to the vapour state without passing through the liquid state. The material to be dried is first frozen and then subjected under a high vacuum to heat (by conduction or radiation or by both) so that frozen liquid sublimes leaving only solid, dried components of the original liquid.

Construction:

The process condenser is sometimes referred as just the condenser or the cold trap It is designed to trap the solvent, which is usually water, during the drying process. The process condenser will consist of coils or sometimes plates which are refrigerated to allow temperature. These refrigerated coils or plates may be in a vessel separate to the chamber, or they could be located

within the same chamber as the shelves. Hence there is designation "external condenser" and "internal condenser". Physically, the external condenser is traditionally placed behind the chamber, but it may be at the side, below or above.

The position of the condenser does not affect trapping performance. For an internal condenser the refrigerated coils or plates are placed beneath the shelves on smaller machines, and behind the shelves on larger machines, but again there is no performance constraint, only the geometry of the chamber.

- Shelf fluid system

The freeze-drying process requires that the product is first frozen and then energy in the form of heat is applied throughout the drying phases of the cycle. This energy exchange is traditionally done by circulating a fluid through the shelves at a desired temperature. The temperature is set in an external heat exchange system consisting of cooling heat exchangers and an electrical heater. The fluid circulated is normally silicone oil. This will be pumped around the circuit at a low pressure in a sealed circuit by means of a pump.

- Refrigeration system

The product to be freeze dried is either frozen before into the dryer or frozen whilst on the shelves. Compressors or sometimes-liquid nitrogen supplies the cooling energy. Most often multiply compressors are needed and the compressor may perform two duties, one to cool the shelves and the second to cool the process condenser.

- Vacuum system

To remove solvent in a reasonable time, vacuum must be applied during the drying process.

The vacuum level required will be typically in the range of 50 to 100µ bar.To achieve such a low vacuum, a two stage rotary vacuum pump is used. For large chambers, multiple pumps may be used.

- Control system

 Control may be entirely or usually fully automatic for production machines. The control elements required are as mentioned above, shelf temperature, pressure and time. A control

program will set up these values as required by the product or the process. The time may vary from a few hours to several days.

Working:

Sublimation in the freeze drying process can be described simply as: FREEZE - The product is completely frozen, usually in a vial, flask or tray. VACUUM - The product is then placed under a deep vacuum, well below the triple point of water. DRY – Heat energy is then added to the product causing the ice to sublime.

Diagram:
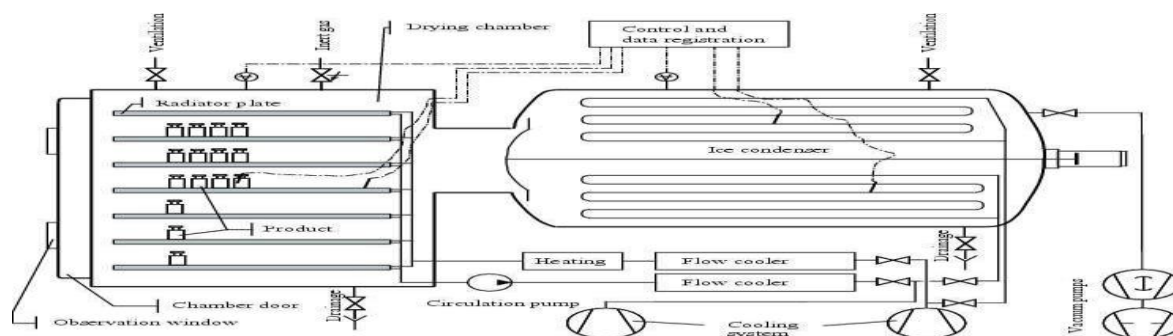
Advantage:

- Removal of water at low temperature.
- Thermoliable materials can be dried.
- Compactable with asceptic operations.
- Sterility can be maintained.

Disadvantage:

- Many biological molecules are damaged by the stress associated with freeze-drying or both.
- Cost may be an issue depending upon product.
- It is a long time process.

Uses:

- It is used in pharmaceuticals and biotechnology to increase the shelf life of the product.
- It is used in food industry to preserve food, produce essence or flavouring agents.
- In technical industry it is used in chemical synthesis and formation of stable product.

Applications:

- It is used in pharmaceuticals and biotechnology to increase the shelf life of the product.
- It is used in food industry to preserve food,produce essence or flavouring agents.
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- It is used in pharmaceuticals and biotechnology to increase the shelf life of the product.
- It is used in food industry to preserve food,produce essence or flavouring agents.
- In technical industry it is used in chemical synthesis and formation of stable product.
- In chemical synthesis, products are often freeze- dried to make them more stable, or easier to dissolve in water for subsequent use.
- In bio- separations, freeze-drying can be used also as a late-stage purification procedure, because it can effectively remove solvents.

E. Spray Dryer-

Principle:

In spray dryer, the fluid to be dried is atomized into fine dropelets,which are thrown radially into a moving stream of hot gas. The temperature of the droplets is immediately increased and fine droplets get dried instaneously in the form of spherical particles.this process completes in a few seconds before the droplets reach the wall of the dryer.

Construction:

- It consist of a large cylindrical drying chamber with a short conical bottom, made up of stainless steel.
- Diameter of 2.5-9 meters and height 25 meters or more.
- An inlet for hot air is placed in the roof of the chamber and another inlet carrying spray disk atomizer is set in the roof.
- Spray disk is about 30 mm in dia. and rotates at a speed 3000 to 50000 rpm.
- Capacity of spray dryer – 2000kg/hr.

Diagram:
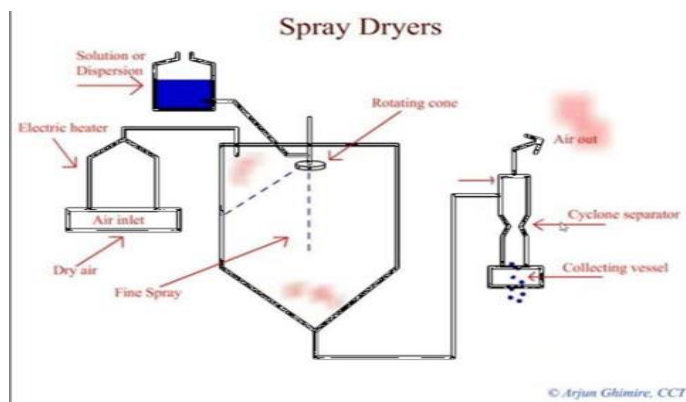
Working Procedure:

Liquid material is atomized through a special pressure nozzle with a high-pressure pump. Hot air enters the dryer after being adjusted by an air distributor. The material and hot air is mixed in the dryer, rapidly carries on heat and mass transfer. Powder or granular products are produced in a very short time. Products with larger particles size are discharged form the bottom of the dryer, smaller particles enter the cyclone with hot air. The exhaust gas is discharged into the air after dust removal in a scrubber. The dryers can be divided into high, middle and low pressure spray dryers according to different pressures of atomization, while taking into account different flow fields, they are divided into co-current, fountain mode and counter-current spray dryers.

Advantage:

- The dried powder will have a uniform particle size and shape.
- Because of good flow properties, the spray-dried powder can be easily compressed into the form of tablets.
- It is useful in the coating and encapsulation of both solids and liquids.
- Labor cost is low since the material is dried in a single operation with no handling.
- The droplets are small, giving a large surface area for heat and mass transfer so that evaporation is very rapid.
- It can be used for drying heat sensitive or oxidized materials without degradation.
- It can be designed for drying under sterile conditions. DISADVANTAGE:

- Solid materials cannot be dried.

- The equipment is very costly and bulky.

- Since the equipment is bulky, cleaning is time consuming.

- There is a lot of heat wasted. Applications:

- Spray dryers are used for the drying of liquid materials like emulsion, suspension, solution, slurries, thin pastes, etc.

- Spray drying can be used to dry materials that are sensitive to heat or oxidation without degrading them, even when high temperature air is employed.

- The liquid feed is dispersed into droplets, which are dried in seconds because of their high surface area and intimate contact with the drying gas.

Report:

The construction, working and application of pharmaceutical machinery such as colloid mill, planetary mixer, fluidized bed dryer, freeze dryer was studied.

Vision and Mission of the Institution

Vision

The East Point College of Pharmacy aspires to be a globally acclaimed institution, **recognized** for **excellence in** pharmaceutical education, research and nurturing students for **holistic development.**

Mission

M1 Create pharmacy graduates through **quality education**

M2 Promote innovation, **creativity**, and excellence **in teaching**, learning, and **research**

M3 **Inspire** integrity, teamwork, critical thinking**, personal** development, and ethics in **students** and lay **the** foundation for lifelong learning

M4 **Serve** the **healthcare, technological, scientific**, and **economic** needs of then **society**.