LABORATORY MANUAL
IN
PHARMACEUTICAL ENGINEERING

For Third semester B.Pharm

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CONTENTS

Experiment – 01

DETERMINATION OF RADIATION CONSTANT OF METAL CYLINDERS

Aim: To determine the radiation constant of metal cylinders made of brass, iron, unpainted and painted glass

Requirements: Metal cylinder, hot plate, thermometer, weighing balance

Principle

        Radiation is the process of transfer of heat through space by electromagnetic waves. Example: Microwave oven.

        Heat can travel from one place to another by three mechanisms-*Conduction, convection* and *radiation*.

        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. Ex: Heating of a metal shell or boiler

        Convection is a process in which heat flow is achieved by actual mixing of warmer portions with cooler portions of the same material. Ex: *Coil type of water heater* (Immersion Coil)

        Heat always transfer from a higher gradient to lower gradient. Heat transfer is dynamic process. A black body is an object that radiate maximum amount of energy at a given temperature. The total amount of radiation emitted by black body can be explained by Stefan Boltzmann's law.

$Q=bAT^4$

Where,

q = energy radiated per second (J/s)

A= area of radiating surface (m^2)

T=absolute temperature of radiating surface (K)

b=constant (W/ m^2·K^4)

For black body, the value of b= 5.67 x 10 ^{4}W/m^2 K^{-4}

Applications in Pharmacy

1. In heat exchangers and interchangers

2. In crystallization

3. In evaporation

4. In distillation

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Procedure

1. Take a metal cylinder (iron) and clean it

2. The diameter (d) and height (h) of the cylinder are measured. The radius (r) is calculated. The surface area (A) of the cylinder is also calculated and recorded.

3. Place it on a hot plate and switch on. (Bunsen burner can also be used)

4. Heat the metal cylinder to maximum temperature.

5. Remove metal cylinder with the help of a long tong carefully and place it over a glass tripod stand.

6. A thermometer (360°C) is placed in the centre of the cylinder and fixed to a stand using a thread if necessary.

7. Allow the cylinder to cool and note the temperature at intervals of 5 minutes until it reaches to room temperature

8. A graph is plotted by taking time (min) on x-axis and temperature (^{o}C) on y-axis. Normally a curve is observed.

9. Depending on the temperature at which the radiation constant is to be determined, a tangent is drawn at that temperature. The slope of the tangent is calculated, which represents the rate of fall of temperature. The parameter is related to rate of loss of heat (dq/dt) by the hot body.

10. Radiation constant alpha is determined at that temperature of convection

11. Repeat same procedure with metals made of other materials.

Important Precautions: (1) The hot metal cylinder should not be touched with hands. (2) Care should be taken while transferring hot metal cylinder from metal tripod to glass tripod.

Observations & Calculations

Heat loss by convection, ß =0

Specific heat of metals

Iron, s   = 106 J/kg.K

Copper, s   = 385 J/kg.K

Brass, s   = 370 J/kg.K

Aluminium, s = 913 J/kg.K

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S.No.	Parameters of the metal cylinder	
1	Wt. *(w) g*	
2	Diameter*(d)*cm	
3	Radius*(r)*cm	
4	Height *(h)*cm	
5	Surface area*(A)*	

Heat loss by convection, ß =0

Specific heat of metals

Iron, s   = 106 J/kg.K

Copper, s   = 385 J/kg.K

Brass, s   = 370 J/kg.K

Aluminium, s   = 913 J/kg.K

Report

The radiation constant of metal cylinders of

i.   Brass   =   iii. Unpainted glass =

ii.   Painted glass =   iv. Iron   =

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Expreiment-02

DETERMINATION OF OVERALL HEAT TRANSFER COEFFICIENT

Aim: To determine overall heat transfer coefficient of a heat exchanger (glass cylinder/insulated glass cylinder)

Requirements

Distillation flask

Thermometer 110 ^{o} C

Water Condenser (insulated & non-insulated)

Bend tubes(short)

Steam generator

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. When the hot fluid and cold fluid enter the apparatus from the same end, the flow is parallel to each other. This arrangement is known as parallel flow. When the feed of hot fluid is passed through one end of the apparatus, while the cold fluid is passed through the other end, the arrangement is called as countercurrent or counter flow method. These types of flow are observed in heat exchangers.

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 = Q/A x Δtav……………………….(1)

Where Q = Amount of heat transferred(J/s)

A= Surface area of the glass tube, m2

Δtav= Temperature gradient

U = Overall heat transfer coefficient/m2.K

In equation (1), the term Q is represented as

Q= Q1+Q2……………………….(2)

Where Q1 is heat loss by steam,W (J.s)

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Q2 heat gain by cold body,W(J.s)

Heat loss by hot body may be expressed as

Q1= M1L+M1.s.t1……………………….(3)

Heat gain by the cold body may be expressed as

Q2=M2.s.t2

Where m1 = mass of condensed steam,kg

M2= Mass of circulating water,kg

S= Specific heatv of steam/kg.K

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

t1= temperatrure drop on steam ,K

t2= temperature rise on the circulating water side,K

The temperature gradient Δtav is expressed as,

Δtav = Δt1+Δt2/2

Where Δt1= Difference in temperature on steam side,K

Δt2= Difference in temperature on cold side,K

 The water condenser used in the laboratory for distillation is an example for the counter current flow of liquids and heat transfer. Thus overall heat transfer coefficient is determined using water condenser. Glass water condenser without any insulation and an insulated condenser with cotton and insulation tape can be used for this purpose. The differences reflect the heat transfer efficiency of a condenser. Cold water is circulated through the jacket of the water condenser. As a result, the steam in the condenser gets condensed due to heat transfer by conduction through the glass wall, followed by convection. Thus the condensed water is collected at the other end of the condenser.
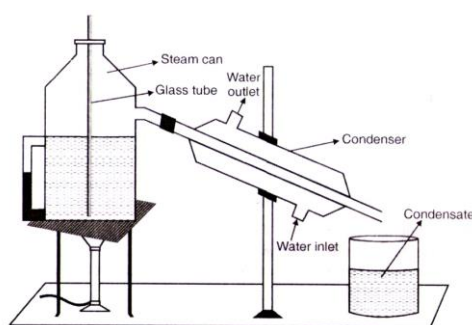
Arrangement of apparatus and counter current flow of liquids

Applications in Pharmacy

1. In the preparation of starch paste for granulation

2. In crystallization

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3. In evaporation

4. In distillation

Procedure

1. The length and diameter of the plain water condenser is determined and reported in the observations. Based on this value, surface area of the condenser is estimated.

2. The apparatus is assembled as shown in the figure

3. The inlet of water condenser is connected to the tap. The outlet is placed in a 2 litre beaker.

4. The temperature of the water at the inlet of the condenser is noted. This will be same as the temperature of tap water.

5. The steam generator is heated so that steam will be generated, After some time, the thermometer shows constant temperature. This temperature is noted and experiment is started from this point. The steam passes through the water condenser and gets condensed in the condenser due to circulation of water in the jacket.

6. The condensate is collected in a 50 ml beaker.

7. Allow the experiment for two minutes after attaining constant temperature of steam.

8. The quantity of condensate water is noted and its temperature is noted.

9. The quantity of circulated water is collected at the outlet of the condenser and its temperature is noted.

10. The entire experiment is repeated with an insulated condenser in place of plain glass condenser

Precautions

1. The rate of flow of water through the jacket of the water condenser must be kept constant.

2. The joints of the assembly must be as short as possible to prevent heat loss at these points.

Observation with Plain water condenser

Diameter of the condenser = ………..,,…cm = ………….m

Radius of the condenser   = ……………cm =……………m

Length of the condenser   = ……………cm = ………….m

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

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

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

The following calculations are made. Recall equations (2) and (3)

Heat loss by steam, $Q_1 = M_1L + M_1s\Delta t_1$

Heat gained by tap water, $Q_2 = M_2s\ \Delta t_2$

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Heat transferred Q = Q1+Q2/2

Recall equation (1),

U = Q/A x Δ tav

Similar observations can be obtained by conducting the experiment using insulated water jacket condenser.

Parameters for counter current flow

	Water condenser circulating water		On steam side of the condenser	
Temperature ^{o} C	Tap water	Water outlet	Steam	Condenser
Temperature K				
Difference in temperatures	Δt_1		Δt_2	
Average temperature Δt_{av}	$\Delta t_1 + \Delta t_2 / 2$			
Volume of water in 2 minutes	-	M_1	-	M_2
Mass of water in 2 minutes,*kg*				
Mass of water per second,*kg/s*				

Report

Overall heat transfer coefficient of plain glass tube (Water condenser)   =

Overall heat transfer coefficient of insulated glass tube (Water condenser) =

Inference: The heat transfer coefficient of the insulated glass tube is higher/lower than the plain glass tube

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Experiment – 03

STEAM DISTILLATION

Aim: To separate turpentine oil from water-turpentine oil mixture by steam distillation

Requirements

Steam Can (Steam Generator)	Two holed rubber cork
Distillation flask, 250 ml	Measuring cylinder 100 ml
Balance	Thermometer 110 °C
Bunsen Burner	Water condenser
Safety tube	Receiving Condenser
Stands	Distilled water
Turpentine oil	Beaker 100 ml

Principle

Steam distillation is a process of distillation carried out with the aid of steam and is used for the separation of high boiling substance from non-volatile impurities. Steam distillation is used to separate turpentine oil from a mixture. A mixture of immiscible liquids begins to boil when the sum of their vapour pressure is equal to the atmospheric pressure. In case of a mixture of water and turpentine oil, mixture boils below the boiling point of pure water. though turpentine boils at a much higher temperature than that of water. As a result, high boiling substances may be distilled at a temperature much below its boiling point, when water(steam)is used. The turpentine is distilled along with water. These liquids are immiscible and they are separated using a separating funnel. Thus it is possible to separate and purify one liquid from a mixture.
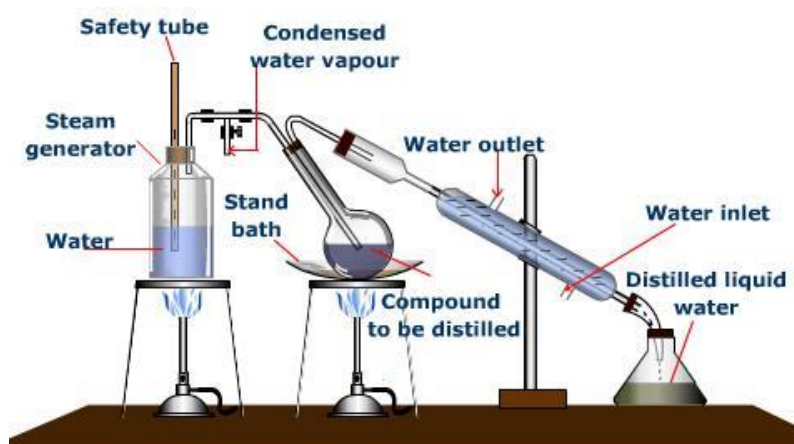
Assembly of apparatus for steam distillation

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Applications in Pharmacy

1. Steam distillation is used for the separation of immiscible liquids. Ex: Toluene and water

2. This method is used for extracting volatile oils such as clove, anise and eucalyptus

3. It is useful in the purification of liquids with high boiling point. Ex: Essential oil of almond

4. In the distillation of camphor

5. Aromatic waters are prepared by this method.

Advantages: Volatile oils can be separated at a lower temperature in steam distillation, without any decomposition and loss of aroma. If a substance has low solubility, 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

The apparatus is assembled as shown in the figure.

1. It consists of a metallic copper steam can (steam generator) fitted with a cork having two holes.

2. Through one hole of rubber cork, a long tube is passed so as to reach almost the bottom of the steam can.

3. This tube act as a safety tube so that in case the pressure inside the steam can becomes too much, water is forced of it and the pressure will be relieved.

4. More over when steam starts coming out from the safety tube, it indicates that the steam can is almost empty.

5. Through another hole, a bent tube is passed

6. The other end of the bent tube is connected to the flask containing non aqueous liquid through a rubber bung (stopper). This tube should reach almost bottom of the flask.

7. Through the other hole of the rubber bung, a delivery tube is inserted which connects the flask and condenser.

8. The condenser is connected to a receiver flask using an adaptor. Bunsen burner/heating mantle is used to heat the steam can and flask.

Calculation of recovery of immiscible liquid: As long as the immiscible liquid is present in the still, the temperature remains constant. The number of moles of liquid distilling will be proportional to its vapour pressure. This can be obtained from the following equations.

Let the volume of vapour before being condensed = Vm^3

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Let the moles of water = n_1

Let the moles of turpentine oil (immiscible liquid) = n_2

Let the partial pressure of turpentine oil = P_2kPa

Let the partial pressure of water = P_1kPa

$W_2/W_1 = M_2P_2/M_1P_1 = 136 \times 15.06$

   18.0 x 86.245

= 2048.16/1552.41

= 1.3193 = 1.32

Where,

w_1= Weight of water

M_1= Molecular mass of water

w_2=Weight of immiscible liquid in g

M_2= Molecular mass of immiscible liquid, g/mol

Generally, liquids with high boiling points have high molecular mass. The molecular mass of water is less than that of the liquid. From equation, it can be inferred that the weight of high boiling liquid that distills over it is sufficiently high, in spite of its low vapour pressure.

Procedure

Careful attention is required while handling all glass distillation apparatus.

1. 30 ml of turpentine oil is placed in 250 ml round bottom flask.

2. Round bottom flask is connected to the condenser and receiver.

3. Turpentine oil present in the flask is heated directly using Bunsen burner (heating

Mantle)

4. Boiling point of turpentine is noted in the thermometer.

5. To 30 ml turpentine oil taken in the round bottom flask in step 1, add 200 ml of water.

6. The steam can is filled with water, and the remaining assembly is done as in the figure.

7. Both the steam can and flask are heated simultaneously so that steam flows uniformly through the mixture. (the mixture gets heated and after some time boiled)

8. The temperature at which boiling occurs is noted. Steam carries the vapour of oil and passes into the condenser where condensation takes place

9. Condensate is collected into the receiver.

10. Oil is separated from water using separating funnel

11. The weights of turpentine oil(W2) and water(W1) layers are noted.

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12. The practical recovery ratio of oil and water(W2/W1) is calculated and reported.

<u>**Observations & Calculations**</u>

Boiling temperature of turpentine oil by simple distillation = ^oC

Boiling temperature of the mixture = ^oC

Weight of turpentine oil obtained = g

Weight of water obtained = g

Theoretical recovery ratio of turpentine oil to water = 1.32:1

Practical recovery ratio of turpentine oil to water =

Percentage efficiency of steam distillation = $\left(\dfrac{\text{Practical recovery}}{\text{Theoretical recovery}} \right)$ X 100

<u>Report</u>

1. During steam distillation, the temperature at boiling (................ ^oC) is less than the boiling point of turpentine oil

2. The practical recovery of turpentine oil = ... %

3. The percentage efficiency of steam distillation for turpentine -water mixture = ...

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Experiment – 04

CONSTRUCTION OF DRYING RATE CURVE

Aim: To dry calcium carbonate slurry and plot the drying rate curve

Requirements

Hot air Oven

Electronic Balance

Petri dish or Tray or SS plate

Beaker

Measuring cylinder

Calcium carbonate powder

Principle

Drying is defined as the removal of small amounts of water or other liquid from a material by application of heat. Drying involves both heat and mass transfer operations. Heat must be transferred to the material to be dried in order to supply the latent heat required for vaporization of the moisture. Mass transfer involves the diffusion of water through the material to the evaporating surface and subsequent evaporation of the water from the surface.

Unbound water is the amount of water held by the material that exerts an equilibrium vapour pressure equal to that of pure water at the same temperature.

Equilibrium moisture content (EMC) is the amount of water present in the solid that exerts a vapour pressure equal to the vapour pressure of the atmosphere surrounding it. *Free moisture content* (FMC) is defined as the amount of water that is free to evaporate from the solid surface.

FMC = Total water content — Equilibrium moisture content

Drying is an oldest and most common operation in chemical engineering. Drying occurs by effecting vaporization of the liquid by supplying heat to the wet feed stock. Heat may be supplied by convection (direct dryers) by conduction (contact dryers) radiation or volumetrically by placing the wet material in a microwave or radio frequency electromagnetic field.

Rate of drying in this experiment 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

Rate of drying   = $\dfrac{\text{weight of water removed}}{\text{Weight of dry powder X time of drying X Surface area exposed}}$ g/g.h.cm2

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A graph is plotted by taking percentage moisture content on x-axis and drying rate on y-axis. The curve so obtained is called as drying rate curve. It represents different changes occurring during drying, where "AB" is constant rate period, "BC" is first falling rate period, and "CD" is second falling rate period. The FMC at the end of constant rate period is known as critical moisture content (CMC).

TYPICAL DRYING RATE CURVE
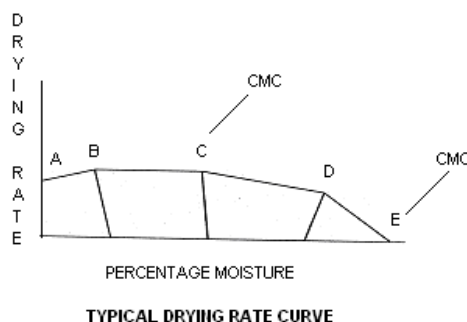
AB- Constant rate period
BC- First falling Rate period
CD-Second falling rate period

Applications

1. In the manufacture of bulk drugs, drying is the final stage of processing. Example is dried aluminium hydroxide gel.

2. Drying is necessary in order to avoid deterioration, for example, crude drugs of animal and vegetable origin

3. Granules are dried to improve the fluidity and compression characteristics.

4. Drying of viscous and sticky materials modifies the flow characteristics.

5. Removal of moisture makes the material light in weight and reduces the bulk.

Procedure

1. Take a clean Petri dish without lid and determine its weight (W_1 g)

2. Determine the area of the Petri dish "A"

3. Take 10 g of Calcium Carbonate in the Petri dish. Find out the weight of powder
and dish (w_2 g)

4. Add about 20 to 25 ml water and make slurry. Let its weight be 'W_3' g.

5. Keep Petri dish in a hot air oven with temperature maintained at 70° C

6. 6. Continue drying and determine the weight of the sample at every 15 minutes.

Stop drying, when there is no change in the weight of the sample.

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7. Determine the percentage of moisture content and drying rate for each time interval using the following equation

Percentage moisture content $= \left(\dfrac{W_3 - W_2}{W_3 - W_1} \right)$ X 100

Drying rate $\qquad = \left(\dfrac{W_3 - W_2}{\text{Area of tray "A"}} \right)$ X time

8. Plot a graph of Drying curve by taking percentage moisture content on 'x' axis and drying rate on 'y' axis.

Observations & Calculations

1. Area of plate in square cm available for drying (A) $= \pi r^2$) $=$…………..cm^2

2. Weight of empty Petri dish 'W_1' $\qquad\qquad\qquad$ =…………….g

3. Weight of Petri dish + sample 'W_2' $\qquad\qquad$ =…………….g

4. Weight of Petri dish + sample + water 'W_3' $\qquad$ = …………….g

5. Weight of sample W $\qquad\qquad\qquad\qquad$ = …………….g

6. (After drying at different time intervals) 'W_4' $= W_3 - W_2$ = …………….g

7. Weight of slurry sample after drying it for 30 minute $\quad = \ W_5$ or W_{n+1}

Data

S.No.	Time(min)	Weight of slurry+petri dish (g)	*Wn-Wn+1* (g)	Rate of drying
01	0			
02	30			
03	60			
04	90			
05	120			

Rate of drying up to 30 minutes (R_1) $\qquad\qquad = \quad W_3 - W_5 / \Delta t.A$

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Rate of drying between 30—60 minutes (R_2)   = W_5-W_6 / $\Delta t.A$

Average drying rate (R avg)   = R_1+R_2+……Rn /n

Report

1.Average drying rate for the given sample from above observations was found to be……………………g/minute

2.Plotted a graph of Drying curve by taking percentage moisture content on 'x' axis and drying rate on 'y' axis.

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Experiment-05

DETERMINATION OF MOISTURE CONTENT AND LOSS ON DRYING

Aim: To determine moisture content and loss on drying

Requirements: Hot air Oven, thermometer, weighing balance, and sample like calcium carbonate.

Principle

Moisture content can be defined as the ratio of weight of dry sample to ratio of weight of water in a given substance. *Equilibrium moisture Content*(EMC)is the moisture content of the solid when in equilibrium with the given partial pressure of vapor in the gas phase. EMC data are helpful in predicting final moisture content in drying operations. *Bound water* is the moisture content of a substance that excerts an equilibrium vapour pressure lower than that of the pure liquid at same temperature. Unbound water is the moisture content of a substance that exerts an equilibrium vapor pressure equal to that of pure liquid at same temperature.

Other methods to determine moisture content in a product include Karl-Fischer titration. KF method measures only the water content in a product while LOD measures the total change in weight of a material as a result of drying.

Applications in Pharmacy

1. In the manufacture of bulk drugs, drying is the final stage of processing. Example is dried aluminium hydroxide gel.

2. Drying is necessary in order to avoid deterioration, for example, crude drugs of animal and vegetable origin

3. Granules are dried to improve the fluidity and compression characteristics in tablet manufacturing.

4. Drying of viscous and sticky materials modifies the flow characteristics.

5. Removal of moisture makes the material light in weight and reduces the bulk.

Procedure

1. A sample (CaCo3) is weighed and weight noted

2. Water is removed through heating in an oven

3. The residual weighing gives the dry weight, and so the difference between the initial weight and residual weight will give the weight of water in the sample.

4. Calculate the moisture content and loss on drying using the following formula

Percentage Moisture content = Weight of water in sample/Weight of sample x *100*

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$$\text{Loss on drying (\%)} = \left[\frac{\text{Weight of water in sample(kg)}}{\text{Total weight of wet sample(kg)}} \right] \times 100$$

Observations and calculations

S.No.	Weight of wet sample	Weight after drying	Weight of water in sample(wt. of wet sample-Wt. after drying)

Report

1. Percentage moisture content =…………….%

2. Percentage Loss on drying   =…………….%

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Experiment-06

DETERMINATION OF ATMOSPHERIC HUMIDITY BY PSYCHROMETRIC MEHOD

Aim: To determine humidity and relative humidity of the ambient environment by psychrometric method

Requirements

1.Psychrometer with wet and dry bulbs

2.Distilled water

3.Psychrometric chart

Principle

Humidity is defined as the ratio of mass of water present in air to the mass of fry air. Humidity has units of kg/kg OR pound/pound. The concentration of water vapour in a gas is called as the humidity of gas. When expressed in percentage it is known as relative humidity. Relative humidity is defined as the ratio of actual humidity to the saturation humidity at a given temperature.

Dry bulb temperature is the temperature of moist air when it is measured at rest by any instrument, which is not affected by the moisture content of air by radiation.

Wet bulb temperature is the dynamic equilibrium temperature attained by a water surface when exposed to air under adiabatic condition

Adiabatic: A condition in which heat does not enter or leave the system

Psychrometer: Psychrometer (hygrometer) consists of two similar thermometers that are fixed to a metal or wood frame. The bulb of one thermometer is kept open to ambient conditions. This thermometer gives dry bulb temperature. The bulb of second thermometer is kept moist by a wet cotton wick immersed in water reservoir. This thermometer gives wet bulb temperature. When the psychrometer is kept for a while, the wet bulb temperature reaches a minimum and remains constant. The temperatures of two thermometers are noted.

Psychrometric charts (Humidity charts)

For engineering calculations, the properties of mixture of air and water vapour are necessary. Humidity charts or psychrometric charts are helpful for this purpose. A typical psychrometric chart contain the following parameters.

1.   Humidity vs. temperature

2.   Humid heat vs. humidity

3.   Specific volume vs. temperature

4.   Adiabatic cooling lines or constant wet bulb temperature lines

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5.   Saturation humidity curve

Saturation humidity is absolute humidity at which the partial pressure of water vapour in the air is equal to the vapour pressure of free water at the same temperature. These plots are considered for the determination of existing conditions of environment at constant pressure. Based on these values, the desirable environmental conditions are established suitable adjustment of the equipment.
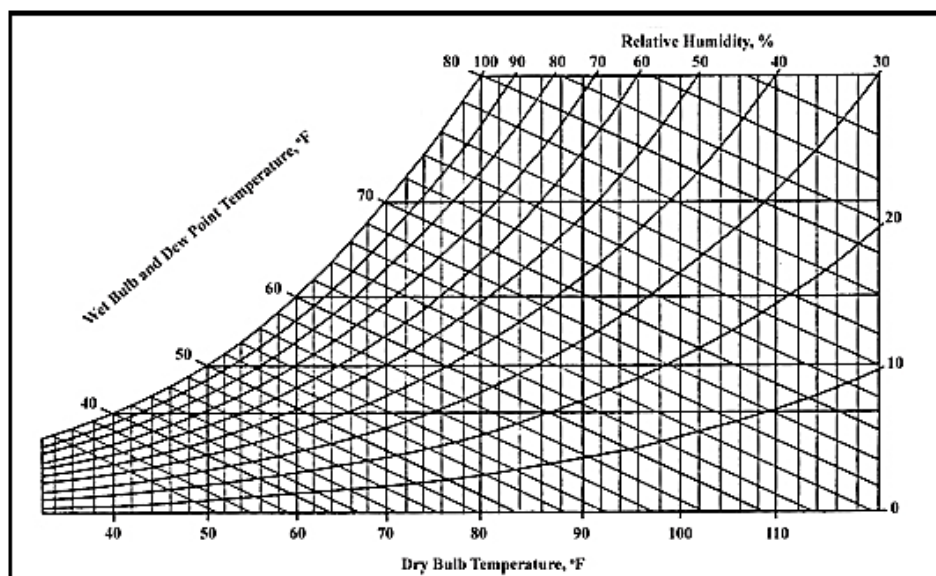
Humidity chart

Applications in pharmacy

1. The humidity conditions of air is important for determining operating conditions for drying of medicinal substances

2. Based on humidity needs, condensers, cooling towers, dehumidifier and air conditioners are designed. Air conditioning is a critical factor in the manufacturing of dosage forms, tablets, capsules, sterile products etc.

The differences between the wet bulb and dry bulb temperatures are used for calculating the relative humidity and humidity of air.

$$\text{Percent relative humidity} = \left(\frac{\text{Humidity of air}}{\text{Humidity of saturated air}}\right) \times 100$$

The intersection point is identified at which the saturated curve (100%) crosses the wet bulb temperature. From this point, the horizontal line towards the y-axis (humidity scale) is drawn and humidity values are noted.

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Procedure

Determination of humidity inside the Laboratory

1. The psychrometer is taken and verified for the dry bulb thermometer, which is exposed to open air. The wet bulb thermometer is verified for its cotton wick dipping in water present in the plastic container (fill the container with water before starting the experiment)

2. The psychrometer is kept in the air of laboratory to get constant value.

3. The dry bulb and wet bulb temperatures are noted at five minutes intervals. Once the temperatures remain constant, these are considered as dry bulb and wet bulb temperatures for calculations

4. The humidity and relative humidity is determined using humidity chart.

Humidity of open air outside the Laboratory

Observations and Calculations

Dry bulb and wet bulb temperatures at different locations

S.No	Location	Av.dry bulb temp.		Av.wet bulb temp.		Humidity	Percentage relative humidity
01	Inside the laboratory	ºC	ºF				
02	Outside the laboratory						
	Average						

Report

1. Humidity of air   =………………lb pound/lb water vapour

2. Percentage relative humidity =………………. %

_____

Illustration

The wet bulb and dry bulb temperatures of air at atmospheric pressure are 26º C and 32º C respectively. Calculate the humidity and percentage relative humidity.

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Solution

Humidity: From the wet bulb temperature, the corresponding adiabatic cooling line is identified. Along this line, proceeded to the 100% saturated curve (green in colour) The intersect point is identified and the coordinates x and y are noted. From the psychrometric chart, this point is 299 K or 0.022 kg/kgThe y co-ordinate, that is 0.022 kg/kg is the humidity.

Percentage relative humidity: From the above intersect point, move horizontally up to dry bulb temperature,32o C or 305 K. The intersect point on the vertical line is identified. The curve passes through this intersect represent the percentage relative humidity. From the above data, the percentage relative humidity is 70% R.H.

Alternatively, dry bulb temperature is considered on x axis. From this point, moved vertically up to 100% saturation curve (Green line) and intersect point is obtained. From psychrometric chart, the coordinates are 305 and 0.031. The saturated humidity is 0.031

Percentage relative humidity = $\underline{\text{Humidity of air}}$ =   0.022/0.31 = x 100 = 71%

                                                    Humidity of saturated air

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Experiment-07

DETRMINATION OF HUMIDITY OF AIR BY DEW POINT METHOD

Aim: To determine the humidity of air from wet and dry bulb temperatures-using dew point method

Requirements

Round bottom long neck flask 100 ml

110°C Thermometer

Humidity chart

Water

Ice

Stand

Principle

Dew point is defined as the temperature to which a mixture of air-water vapour must be cooled at constant humidity to become saturated (That is to be in equilibrium with liquid)

Formation of mist and disappearance of mist are considered and dew point is determined. Dew point temperature is noted on the x axis and moved vertically on the psychrometric chart. The intersect point at saturated curve (100%) is identified. The coordinates at the point are noted. ie, temperature and humidity. The point which intersect y axis is the humidity of air. These values are substituted in the equation

Percent relative humidity $= \left(\dfrac{\text{Humidity of air}}{\text{Humidity of saturated air}} \right) \times 100$

Percentage humidity of air is directly read from the psychrometric chart.

Psychrometric charts (Humidity charts)

For engineering calculations, the properties of mixture of air and water vapour are necessary. Humidity charts or psychrometric charts are helpful for this purpose. A typical psychrometric chart contain the following parameters.

6. Humidity vs. temperature

7. Humid heat vs. humidity

8. Specific volume vs. temperature

9. Adiabatic cooling lines or constant wet bulb temperature lines

10. Saturation humidity curve

Saturation humidity is absolute humidity at which the partial pressure of water vapour in the air is equal to the vapour pressure of free water at the same temperature. These plots are considered

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for the determination of existing conditions of environment at constant pressure. Based on these values, the desirable environmental conditions are established suitable adjustment of the equipment.
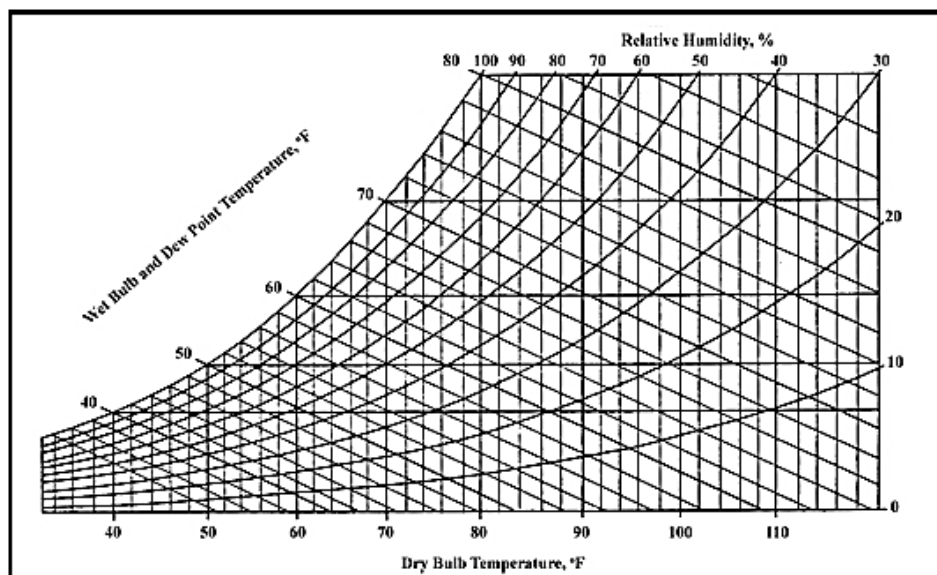
Humidity Chart

Applications in pharmacy

1. The humidity conditions of air are important for determining operating conditions for drying of medicinal substances

2. Based on humidity needs, condensers, cooling towers, dehumidifier and air conditioners are designed. Air conditioning is a critical factor in the manufacturing of dosage forms like tablets, capsules, sterile products etc.

The differences between the wet bulb and dry bulb temperatures are used for calculating the relative humidity and humidity of air.

$$\text{Percent relative humidity} = \left(\frac{\text{Humidity of air}}{\text{Humidity of saturated air}} \right) \times 100$$

The intersection point is identified at which the saturated curve (100%) crosses the wet bulb temperature. From this point, the horizontal line towards the *y*-axis (humidity scale) is drawn and humidity values are noted.

Procedure

1. The round bottom flask is thoroughly cleaned and dries the external surface.

2. Water is filled up to 2/5 rd volume

3. The above flask is kept on an appropriate stand.

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4. A thermometer (110ºC) is dipped in the water

5. Crushed ice is slowly added to the water in the flask and stirred thoroughly with the help of a glass rod or magnetic stirrer.

6. As the temperature of water in the flask is lowered, mist begins to form on the outer bottom surface of the flask. At this point, the temperature is noted. This is the dew point.

7. In psychrometric chart, from the temperature (X axis), moved vertically up until the saturation curve and identified the intersecting point. The coordinates at the point are noted. ie, temperature, K and humidity. The point which intersect y axis is the humidity of air.

Observations and calculations

Dew point determination

S.No.	Dew Point, oC		Humidity
	Mist appearance	Average value	
01			
02			

Report

1. Humidity of air   = ……….. lb water vapour/lb dry air
2. Percentage relative humidity   =…………..%

Illustration

The dew point of air is identified as 17ºC. Calculate humidity of air:

Solution

The dew point of air is 17°C or 17+/- 273012 K = 290 K. This temperature is identified on *x* axis. From this point, moved vertically up to 100% RH (saturated humidity) curve. The intersect point on the curve is identified and coordinates are noted. From the psychrometric chart, the coordinate of the intersect is 290 K, 0.014 *kg/kg*) The Y coordinate is the humidity.ie, 0.014 *kg/k.*

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Experiment-08

PARTICLE SIZE ANALYSIS BY SIEVING

Aim: To determine particle size by sieving method and to construct various size frequency curves

Aim: To determine the average particle size and construction of size distribution frequency plot of a given sample (powder or granules) by sieving method.

Requirements

Set of standard sieves

Sieve shaker

Weighing balance

Calcium carbonate/Sodium chloride/Sodium bicarbonate/citric acid etc.

Principle

Size separation is a 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.* From the theoretical considerations, screen may be ideal or actual.

Sieving is widely used method for particle size analysis because it is economical (in expensive), simple and rapid. Sieving or sifting is a process by which different grades of powders are separated from each other. In the laboratory, different grades of sieves are used for sifting the powders and preparing the granules of required particle size. After size reduction, any material is passed through a specific grade sieve to get the required size particles.

The B.P specifies five grades of powders and these are shown in the table.

Powder classification in British Pharmacopoeia

S.No	Grade of powders	Sieve through which all particles must pass	Sieve through which NMT 40% of particles pass
01	Coarse	10	44
02	Moderately coarse	22	60
03	Moderately fine	44	85
04	Fine	85	Not specified
05	Very fine	120	Not specified

Sieving method directly gives weight distribution analysis. From the data, d_{sieve} can be calculated. Sieve number is defined as the diameter of a sphere that passes through the sieve aperture as asymmetric particle. Sieve number is applied to those particles that pass through a sieve.

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It is useful for establishing size distribution analysis. On the other hand, powder is expressed by average particle diameter.
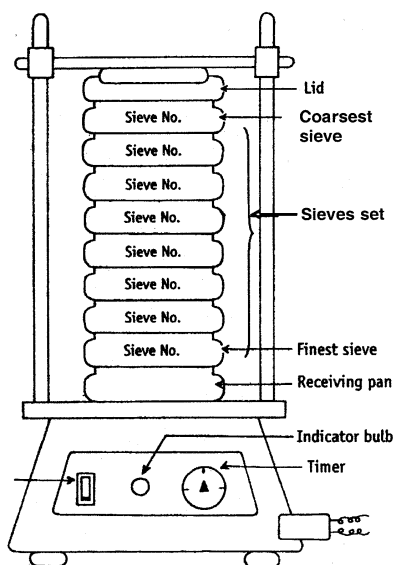
Laboratory gyratory sieve shaker and arrangement of sieves

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 a nest with the coarsest at the top. A sample of powder (100 g) is placed on the top sieve. The sieve set is fixed to the gyratory mechanical sieve shaker and shaken for a period of time (05 minutes). The powder retained on each sieve is weighed and the normal weight distribution curve is constructed.
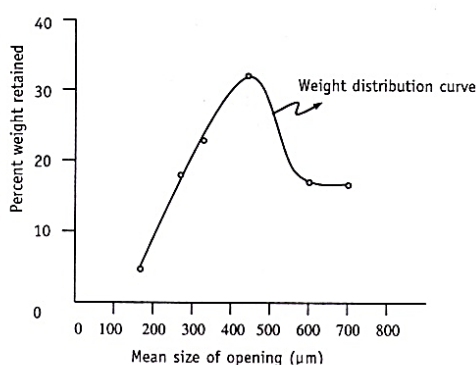
Normal weight distribution curve

The average particle diameter of a powder (*d average*) is calculated using the following equation.

d average = Σ (n x d)/ Σ (n)……………………………………… (1)

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Where *n*= Frequency of particles in a particle size range, *g* or percentage weight of powder undersize *g*

d= Average particle diameter of a particular sieve (sieve diameter) *mcm*

Applications in Pharmacy

1. In the production **of** tablets and capsules. Normally, 15% of fine powder (passed through 100-mesh sieve) should be present in a granulated material to get a proper flow of material and achieve good compaction in tab letting. Therefore, per cent of coarse and fine powder can be quickly estimated using sieving method.

2. Milled material is subjected to size separation in order to obtain the Powder of desired size or size distribution.

3. Size distribution analysis is important in different areas as mentioned below:

 i. Quality control tool for the analysis of raw materials.

 ii. Testing the efficiency of a size reduction equipment or process.

 iii. Optimizing the process conditions such as method of agitation, time of screening etc.

 iv. The size distribution in powdered drug also influences the absorption of drug.

Procedure

1.Standard sieve set is selected. The set consists of various sizes (10,22,44,60,85,100 and 120 mesh sieves)

2.Sieves are arranged in such a manner that coarsest remains at the top and finest at the bottom. The pan is kept below the sieve set (Figure)

3.100g of the given sample (granules/powder) is weighed.

4.The sample is placed on the coarsest sieve. The lid is placed.

5.The above sieve set is fixed on mechanical shaker and clamped tightly.

6.The timer is adjusted for 05 minutes and the mechanical shaker is switched on. (Each sieve shaker must be standardized for the time of shaking)

7.When the shaker automatically stops, the sample retained on each sieve is collected into a paper.

8.All samples are weighed.

9.The weights retained on each sieve are recorded in the table.

10.The data is analyzed for normal weight distribution pattern. A plot is drawn by taking mean size of opening (column 04) on x axis and percent weight retained on smaller sieve (column 06) on y axis and the average particle size is calculated.

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Observations & Calculations

Particle size distribution of given sample

Sieve No	Normal mesh aperture size(mcm)	Aperture size (Passed/retained) μ*m*	Mean size of opening* *d.* μ*m*	Weight of powder undersize *n.g*	%weight retained on smaller sieve	weight size n x d *(4)x (6)*
(1)	(2)	(3)	(4)	(5)	(6)	(7)
Pan	-----	-----	-----	**	-----	-----
120	125	125/pan	125.0			
100	150	150/125	137.5			
85	180	180/150	165.0			
60	250	250/180	215.0			
44	355	355/250	302.5			
22	840	840/355	597.5			
10	1700	1700/840	1270.0	***	Σ *(n)=*	Σ*(nd)=*

*Mean size of the opening may be calculated as follows. For example, a powder passed through 22 mesh sieve and retained on 44 mesh sieve is assigned an arithmetic mean diameter of [(840+355)/2] of 597.5 mcm. This value is reported as undersize.

**The sample reached pan represents the weight of powder that passed through 120 mesh sieve. therefore, this value is written in the column 5 against 120.Similarly in other cases.

***Sample retained on 10 mesh sieve is not considered for calculation of undersize, as this value represents oversize.

Report

1. The average particle size of the given powder   =……….*μm*

2. Normal weight distribution curve is plotted.

Illustration

Calculate d average for the following data. The following observations are made by sieve analysis of particle size distribution of a powder.

Mean size, d (μm)   Frequency weight, n

213   25.0

300   41.2

470   13.0

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Solution: The given data are processed as follows

Mean size, d (µm)	Frequency weight, n	Weight size, n x d
213	25.0	5325
300	41.2	12360
470	13.0	6110
Σ (n)= 79.2		Σ(nxd)= 23795

d average = Σ (n x d)/ Σ (n) = 23795/79.2 = 300.44 µm

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Experiment -09

DETERMINATION OF PARTICLE SIZE BY BEAKER DECANTATION METHOD

Aim: To determine the particle size distribution of a powder by beaker decantation method

Requirements: Beaker (500 ml) 3 numbers, glass rod, hot air oven, weighing balance, liquid ammonia, distilled water.

Principle

Particle size in the sub sieve range can be obtained by gravity sedimentation explained by Stokes law.

Rate of settling = h/t d2 (ρs- ρ1) g /18 $\eta\rho$

Where, d- diameter of the particle

ρs- Density of solid dispersed in the liquid

ρ1-Density of the liquid in which solid is dispersed

g-Acceleration due to gravity

η-Viscosity of the medium

h-Height of capillary

t-Time of falling

The diameter can be calculated by rearranging the Stoke's equation.

d=η10h/(ρs-ρ1) *gt*

This equation holds good for spheres falling freely but can also be used for particles with irregular shape too. The particles should not form aggregates. Hence a deflocculating agent is added so that the particles in the suspension remain free and correct results can be obtained.

Applications in Pharmacy

Size distribution analysis is important in different areas as mentioned below:

  i.   Quality control tool for the analysis of raw materials.
 ii.   Testing the efficiency of a size reduction equipment or process.
iii.   Optimizing the process conditions such as method of agitation, time of screening etc.
 iv.   The size distribution in powdered drug also influences the absorption of drug.

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Procedure

1. Weigh 5 g of calcium carbonate and make slurry with distilled water.
2. Take three 500 ml beaker and mark them as A,B,C and make a mark up to 10 cm on each beaker.
3. Take the slurry in beaker A and make up the volume up to 10 cm mark with distilled water and add a drop of liquid ammonia.
4. The contents are stirred with a glass rod and allow to settle for 1. 275 minutes so that the particles greater than 40 mcm will be settled
5. The supernatant solution from beaker A was transferred to beaker B without disturbing the settled solid particles. The solution is allowed to stand for 5 minutes, for settling of particles more than 20 mcm.
6. The above supernatant solution was then transferred to a beaker and allowed to stand for indefinite time so that particles of less than 20 mcm settle at the bottom.
7. The residue from each beaker was filtered, dried, weighed and calculated the percentage weight

Observations and Calculations

Size in microns	Time in minutes	Weight of filter +paper residue	Weight of filter paper after remaining residue	Weight of residue (a-b)	Percentage weight (5/5 x 100)
		A	**B**	**C**	**D**
Greater than 40 mcm					
Between 20 & 40 mcm					
Lower than 20 mcm					

Initial weight of calcium carbonate taken =   5   g

Distance of falling time = 10 cm

Density of calcium carbonate= 2.5 g/cc

Acceleration due to gravity = 980 cm/s^2

Density of water = 1 g/cc

Viscosity of water = η = 0.01 poise

$V = h/t = d^2_{st} (\rho s - \rho 0) g / 18 \eta 0$

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$\eta 1/\tau 1 = d^2_{st} (\rho s - \rho 0) g / 18 \eta \times h$

$t_1 = 18 \eta 0 \times h/d^2((\rho s - \rho 0)g$

$t_2 = \rho^2 (\rho s - \rho 0) g$

Report

The size distribution of particle was found to be

S.No	Particle size	Particle weight
01	Greater than 40 mcm	
02	Between 20 & 40 mcm	
03	Lower than 20 mcm	

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Experiment-10

SIZE REDUCTION-Verification of laws

Aim: To verify the laws of size reduction using ball mill and determining Kick's, Rittinger's, Bond's coefficients, power requirement and critical speed of a ball mill.

Requirements
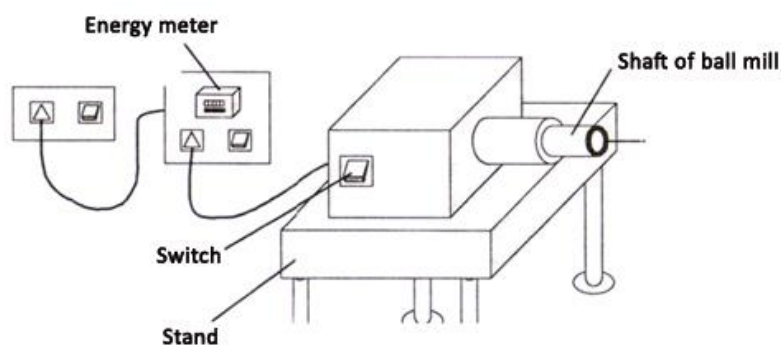
Ball mill   1   Powder sample

Gyratory sieve shaker 1

Sieves set   1

Balance (triple beam) 1

Energy meter   1

Assembly of a ball mill (Lab model)

Principle

Size reduction is a process of reducing large solid masses into small unit masses, coarse particles, or fine particles. Ball mills are also known as tumbling mills or pebble mills. The ball mill works on the principle of impact and attrition between the rapidly moving balls and the material. Both the balls and the powder material are enclosed in a hollow cylinder. Speed at which the balls rotate is important. At low speeds, the balls roll over each other and attrition takes place (Figure). At critical speed, the centrifugal force just occurs, as a result the balls are picked up by the mill wall and carried nearly to the top, where they break contact with the wall and fall to the bottom to be picked up. In this manner, impact stress will also be induced and the size reduction is made effective. (Figure) Thus in the ball mill, impact or attrition or both are responsible for size reduction. At high speeds the balls are thrown out to the wall by centrifugal force (Figure). Hence, grinding will not occur. The energy required to reduce the size of the particles can be calculated by Rittinger's

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equation. Kick's equation and Bond's equation. The principle involved the determination of mean particle size of the material before and after size reduction. These changes are related to the energy consumed for size reduction.
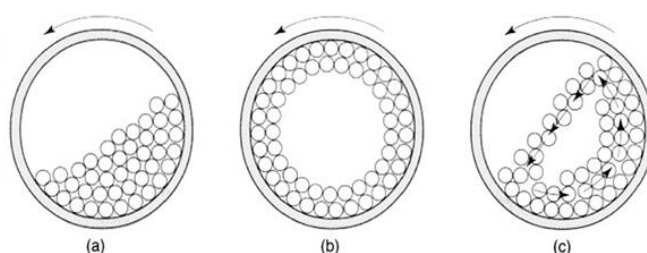

CORRECT CASCADE OFACTION OF A BALL MILL IN OPERATION

PHOTO CREDIT: AULTON'S PHARMACEUTICS:THE DESIGN AND MANUFACTURE OF MEDICINE

Kick's law: It states that the energy required for size reduction is proportional to the logarithm of the ratio between the initial and the final size.

$$E = K_k \ln (d_i/d_n) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots.(1)$$

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

Kk = Kick's constant, energy per unit mass, kW h

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

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

For crushing (compression) of large particles, Kick's equation is more useful.

Rittinger's law : It states that the energy consumed in the size reduction of solids is directly proportional to the new surface created.

$$E = K_R (1/d_n - 1/d_i) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots.(2)$$

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

KR - Rittinger's constant, energy per unit area, kW-h/μm3

It is mostly applicable to brittle materials undergoing fine milling. Rittinger's theory ignores particle deformation before fracture.

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. According to Bond's theory, the equation can be written as:

$$E = 2K_B (1/\sqrt{d_n} - 1/\sqrt{d_i}) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots.(3)$$

where KB = Bond's work index, energy per unit mass, kWh√μm

The Bond's work index is a useful way for comparing the efficiency of milling operation. This is useful for rough mill sizing.

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Procedure

1. The initial dial reading of energy meter is noted as N1

2 The cleaned metal chamber (of ball mill) is taken with sufficient number of balls.

3. The ball mill is operated without load for 10 min.

4. The reading (revolutions) in energy meter is noted down as N2. (The difference, i.e., N3 = N2 – N1 gives the energy required for running the ball mill without feed).

5. 100 grams of sample is weighed and subjected to sieve analysis (Procedure is given in this manual). The average particle size of the sample is calculated.

6. 100 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 down as N4. (The difference, i.e., N5 = N4 - N2 gives the energy required for running the ball mill and size reduction of material).

9.The difference, i.e., = N5 - N3, 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 (1), (2), and (3) for obtaining Kick's constant, Rittinger's constant, and Bond's work index, respectively.

Sieve analysis of sample prior to and after size reduction

Mean size of opening, μm d	Weight of powder retained on smaller sieve, undersize, g	Per cent weight retained on smaller sieve, n	Wt. size in (n x d) (1) x (3
(1)	(2)	(3)	(4)
125.0			
137.5			
165.0			

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215.0			
302.5			
597.5			
1270.0			
		$\sum n = 100$	$\sum nd =$

Weight distribution analysis of sample before Size Reduction

Sieve number	Nominal mesh aperture size *μm*	Aperture size (passed/ retained), *μm*	Mean size of opening *d*, *μm*	Before size reduction Weight of powder retained on smaller sieve, undersize *n, g*	After size reduction Weight of powder retained on smaller sieve, undersize n, g
(1)	(2)	(3)	(4)	(5)	(6)
Pan	—	—	—	—	—
120	125	125/pan	125.0		
100	150	150/125	137.5		
85	180	180/150	165.0		
60	250	250/180	215.0		
44	355	355/250	302.5		
22	840	840/355	597.5		
10	1700	1700/840	1270.0		

Mean diameter of the powder sample (before size reduction), $di = \sum nd / \sum n$

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Weight distribution analysis of sample after Size Reduction

Mean size of opening, μm d	Weight of powder retained on smaller sieve, under size, g	Per cent weight retained on smaller sieve, n	Wt. size in $(n \times d)$ $(1) \times (3)$
(1)	(2)	(3)	(4)
125.0			
137.5			
165.0			
215.0			
302.5			
597.5			
1270.0		$\sum n = 100$	$\sum nd =$

Mean diameter of the powder sample (after size reduction), $dn = \sum nd / \sum n$

Observations and 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 mill with feed, $N_4 =$

5. Energy consumed for size reduction plus the ball mill, $N_5 = N_4 =$

6. Energy consumed for size reduction, $N_6 = (N_5 - N_3) =$

7. Weight of the sample taken, $w = (100\ g)\ 0.1\ kg$

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8. Calculation of energy meter constant, *E* (On the energy meter, the relationship between the revolutions and energy is given. Use that relationship for calculations) 750 units reading   (revolutions)   the   energy   =   3600   *kWs*   (1kWh)

   1 unit reading   = ?

   I unit= 3600/750 =4.8 KJ(E)

9. Energy on no load(E1= N3 xE =………………*KJ*

10. Energy on load E2= N5 x E   = ………………*KJ*

11. Net energy required per unit mass= *{(E2-E1)/w}*=………………*KJ*

12. Average particle diameter of the feed= d_1 =…………………*µm*

13. Average particle diameter of the product= *dn* =…………………*µm*

Report

1.   Rittinger's constant = ………………

2.   Kick's constant   =………………

3.   Bond's work index = ………………

_____

Ilustration

Calculate the Rittinger's constant, Kick's constant and Bond's work index for the following data obtained from a size reduction of a material.

Mean particle diameter of the feed = 765 gm

Mean particle diameter of the product = 472 /zm

Energy consumed for this process per unit mass = 48 kJ

Solution

Rittinger's constant*: Recall equation (2)*

$E = K_R[1/d_n = 1/d_i]$
$48= K_R[1/472 − 1/765]= K_R (0.00219 - 0.00131) = K_R (0.00088)$

K_R= 48/0.00088=54545.4 kW h// µm ^{3}

Kick's constant: Recall equation (1)

$E= K_k \ln d_i/d_n$

$48=K_k In765/472= K_k In1.621=K_k \times 0.483$

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K_k=48/0.483=99.379 $kW.h$

Bond's work index: Recall equation (3)

$E =$ $\qquad\qquad 2K_B[1/\sqrt{d_n}-1/\sqrt{d_i}]$

48 =2K_B[1/$\sqrt{472}$-1/$\sqrt{765}$ = 2K_B[1/21.726-1/27.659]

48 = 2K_B(0.0460-0.0362)=2K_B (0.0098) = 0.0196 K_B ; K_B = 48/0.0196

= 2448.98 $kW.h\sqrt{}$ µm

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Experiment-11

FACTORS AFFECTING RATE OF FILTRATION

Aim: To determine the factors affecting rate of filtration and evaporation-surface area, concentration, and thickness/viscosity

Requirements: Beakers, Measuring cylinders, CaCo3 suspension, Acacia, Filter paper, funnel etc.

Principle

Filtration can be defined as a solid-liquid separation process in which solids are separated from a suspension (slurry) by passing it through a porous medium that accumulate the solids, but allows the passage of fluids.

Rate of filtration is proportional to the pressure difference across both the filter medium and filter cake. The pressure drop can be achieved by,

i.   Maintaining a head of slurry above the filter medium. The pressure developed will depend on the density of the slurry

ii.   The pressure below the filter medium can be reduced below atmospheric pressure by connecting the filter receiver to vacuum pump and developing a pressure difference across the filter.

iii.   Pumping the slurry in to the filter under pressure

The filtration rate is inversely proportional to the viscosity of the fluid. An increase in viscosity of the filtrate can increase the flow resistance. This problem can be avoided by two methods.

1. The filtration rate can be increased by raising the temperature of the slurry. But this is not practical in case of thermo labile and volatile substances.

2. Dilution is another method, but the rate must be doubled.

The rate of filtration through the filter cake is inversely proportional to the thickness of the cake.

Theories in filtration: Poiseuille's equation, Darcy's Law and Kozeny-Carman equation.

Applications in Pharmacy

1. In the production of sterile pharmaceutical dosage forms like injection, bacteria proof filters are used. This operation is essential when heat sterilization is not suitable on account of thermo labile nature of the contents.

2. Removal of impurities from reaction mixtures

3. In the clarification process of liquid oral preparations for obtaining clear solutions

4. In effluent treatment process.

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Effect of concentration of slurry on rate of filtration

Procedure

1. Prepare 100 ml of 2% calcium carbonate suspension in water.

2. Place filter paper over a funnel and arrange it on the measuring cylinder.

3. Pour the slurry on to filter paper and note down the time taken for the filtration of 100 ml of filtrate.

4. Repeat the whole procedure with 0% (ie, with water alone), 2%, 4% and 6% calcium carbonate suspension in water.

5. A graph is plotted by taking concentration of calcium carbonate on x - axis and rate of filtration on y- axis

Observations and Calculations

Conc.of slurry	Vol.of filtrate collected	Time in minutes	Time in seconds	Rate of filtration(ml/sec)
1	2	3	4	5
0%				
2%				
4%				
6%				

Effect of viscosity of slurry on rate of filtration

Procedure

1. Prepare 200 ml of calcium carbonate suspension in water.

2. Divide it into 4 equal parts

3. Add 0%, 2%, 4% and 6% acacia powder to it and triturate well to form a paste.

4. Take four measuring cylinders and pour slurry through filter paper and funnel.

5. Note the time taken for filtration of 50 ml of filtrate.

6. Plot a graph by taking concentration of calcium carbonate – acacia suspension on x-axis and rate of filtration on y axis.

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<div align="center">

Observations and calculations

</div>

Coc.of acacia in slurry	Vol.of filtrate collected	Time in minutes	Time in seconds	Rate of filtration(ml/sec)
1	2	3	4	5
0%				
2%				
4%				
6%				

Effect of Surface area of filter medium on rate of filtration

1. Prepare 200 ml of calcium carbonate suspension in water.

2. Small, medium and large funnels with filter papers equivalent o the size of the funnels are arranged over measuring cylinders and the suspension is passed through them, separately

3. A graph is plotted by taking surface area of filter medium on x-axis and rate of filtration on y-axis.

<div align="center">

Observation and calculations

</div>

Size of funnel (cm)	Vol.of filtrate collected	Time in minutes	Time in seconds	Rate of filtration(ml/sec)
1	2	3	4	5
Small				
Medium				
Large				

Report

1. As the concentration of calcium carbonate increases, the rate of filtration
   Increases /decreases.

2. As the viscosity of the vehicle used in suspension increases, the rate of filtration
   Increases/decreases.

3. As the surface area of the filter medium increases, the rate of filtration increases/decreases

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Experiment-12

FACTORS AFFECTING RATE OF EVAPORATION

Aim: To determine the factors affecting rate of evaporation-surface area, concentration, and thickness/viscosity

Requirements: Petridishes of different diameters, pipettes, stopwatch, beaker, measuring cylinder, glycerin, sodium choride etc.

Principle

The process of vaporizing large quantities of volatile liquid to get a concentrated product can be termed as evaporation. It is a surface phenomenon, where mass transfer is taking place from the suraface. Thus no boiling occurs.

The rate of evaporation depends upon many factors like temperature, viscosity, concentration of slurry, vapor pressure, surface area, time of evaporation, films and deposits, moisture content of the feed, nature of product required etc.

The higher the temperature, greater will be the rate of evaporation. Rate of evaporation is directly proportional to the vapour pressure of the liquid. Greater surface area also aid rate of evaporation. If the time of exposure will be longer, the evaporation rate will be faster. Viscosity of slurry increases, rate of evaporation decreases.

Applications in Pharmacy

In the manufacture of bulk drugs. This process is used in the manufacture of insulin, Penicillin-G and blood plasma.

Effect of surface area on rate of evaporation

Procedure

1. Clean and dry three petridishes of different diameters and mark them as A,B,C
2. Find out the radius (d/2) of petridishes.
3. Pipette out 25 ml di ethyl ether in each of the petridishes and cover them with the lids immediately
4. All petri dishes are kept at room temperature for 5/ 10 minutes.
5. Note the remaining volume after 10 minutes
6. From this rate of evaporation is calculated

***Extreme care should be exercised when heating diethyl ether.**

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Observations and calculations

Petridishes	Diameter of petridish(cm)	Volume taken(ml)	Remaining volume(ml)	Volume of liquid evaporated (Vol taken-remaining vol)	Rate of evaporation(The vol.of liquid evaporated/time)
A					
B					
C					

Effect of viscosity on rate of evaporation

Procedure

1. Different concentrations of glycerin and water are prepared in different beakers as shown in the following table.
2. Note the weight of beaker containing glycerin-water mixture.
3. All the beakers are heated in a water bath at constant temperature (70ºC) for 10 minutes.
4. Note down the weight of the beakers again. The difference between the weights indicates the amount of water evaporated during 10 minutes.
5. From the above data, rate of evaporation is calculated.

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

Observation and calculation

S.No	Glycerin (ml)	Water (ml)	Conc. (% v/v)	Initial wt. of solution (g)	Final wt. of solution (g)	Wt. of water evaporated (g)	Rate of evaporation (Wt.of water evaporated/time (e)
1							

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2						
3						

Effect of concentration on the rate of evaporation

Procedure

1. Prepare different concentration solutions of sodium chloride-2%,4%,6% and 8%in 50 ml beakers.

2. Weigh the beakers containing sodium chloride solution.

3. All beakers are heated on a water bath at constant temperature (70°C) for 10 minutes.

4. Weigh the beakers after heating.

5. The differences in weights indicate the amount of water evaporated during 10 minutes.

6. From the above data, rate of evaporation is calculated.

Observations and calculations

Conc. (% w/v)	Initial wt. of solution (g)	Final weight of solution(g)	Wt.of water evaporated	Time of heating	Rate of evaporation (Wt.of water evaporated/time)
2%					
4%					
6%					
8%					

Report

1. As the surface area increases, the rate of evaporation increases /decreases.

2. As the viscosity of the liquid increases, the rate of evaporation increases/decreases.

3. As the concentration of the solution increases, the rate of evaporation increases.

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Experiment-13

UNIFORMITY INDEX FOR GIVEN SAMPLE BY USING DOUBLE CONE BLENDER

Aim: To calculate the uniformity index (mixing index) for given sample using double cone blender.

Requirements

Volumetric flasks 100 ml -8

Pipette 10 ml -1

Pipette 5ml -1

Test tubes 20 ml

Cylindrical blender -1

Salicylic acid

4% Ferric nitrate solution

Lactose

Stop watch

Principle

Mixing is defined as a process that tends to result in a randomization of dissimilar particles within a system. The term mix means to put together in one mass or assemblage with more or less thorough diffusion of the constituents among one another. The term blending means to mix smoothly and inseparably together. During blending, a minimum energy is imparted to the bed. These terms are commonly used interchangeably in the industry.

The degree of mixing can be evaluated comparing standard deviation of sample under investigation with estimated standard deviation of sample from fully random mix.

In a double cone blender, mixing occurs due to tumbling motion. Mixing index refers to the degree of uniformity achieved during mixing. It is calculated by statistical procedure. With the data obtained, mixing index is calculated using the following formula:

$$M_s = \sqrt{\frac{\Sigma(y - \overline{y})^2}{n(1 - \overline{y})\overline{y}}}$$ ………………. (1)

Where Ms = mixing index

n = number of samples

 = true average composition of component A in the mixture

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y = actual composition of component A in a single sample.

The Ms Values are calculated at different time intervals. Based on the results, optimum time required for actual mixing can be estimate

Applications in Pharmacy

1. Double cone blender is efficient and versatile mixing equipment for homogenous mixing of dry powders and granules for the manufacture of tablets and capsules.

2. In food, chemical and cosmetic industries.

Procedure

Salicylic acid stock solution (1 mg/ml) : Accurately about 100 mg of salicylic acid is weighed and transferred into a 100 ml volumetric flask. Distilled water is added and the solution is shaken thoroughly to dissolve salicylic acid. Finally the volume is made up to the mark. Ferric nitrate solution (4%): Ferric nitrate (4.0 g) is weighed and transferred into a 100 ml measuring cylinder. By adding water the solid is dissolved. Finally, the volume is made up to the mark. The solution is transferred into an amber coloured bottle and labeled. Standard curve for salicylic acid: Standard plot is constructed using proven procedure.

Method for Mixing

1. 1 g of salicylic acid and 50.0 g of lactose are weighed.

2. These two powders are placed in a double cone blender. (Ball mill chamber without balls can also be used for mixing. Place a glass rod of appropriate length diagonally inside the ball mill to enhance mixing efficiency).

3. The blender is allowed to rotate on its own axis for 15 minutes at 25 revolutions per minute (An appropriate speed suitable for your machine can be selected).

4. The samples each weighing 500 mg are drawn from three different places of the blender and placed in three different volumetric flasks (100 ml). Labeled them as 1A, IB, and 1C.

5. The blender is rotated to complete 30 minutes mixing.

6. Again three samples are drawn in a similar way as mentioned in step 4. These are transferred into three different volumetric flasks (100 ml) and labeled them as 2A, 2B, and 2C.

7. After 45 minutes of mixing, three samples are drawn and labeled them 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 flsks,10 ml solutions are transferred into 20 ml test tubes.

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10.5 ml ferric nitrate solution (4%w/v) is added to these test tubes. All solutions turn to purple colour.

11. The absorbance of the above solutions are measured at 547 nm, using a colorimeter /spectrophotometer.

12. The content of salicylic acid present in each sample is calculated and reported.

13. The data are substituted in equation and mixing index is calculated.

<u>**Observations and Calculations**</u>

Sampling time (Minutes)	Sample Number	Absorbance	Conc.of salicylic acid mg/ml	Conc.of salicylic acid in the sample (y mg) 4 x 100	$(y-\bar{y})$	$(y-\bar{y})^2$
1	2	3	4	5	6	7
After 15 min	1A					
	1B					
	1C					
	Average y=					
After 30min	2A					
	2B					
	2C					
	Average y=					
After 45 min	3A					
	3B					
	3C					
	Average y=					

<u>**Report**</u>

1. Mixing index after 15 minutes, M15 =

2. Mixing index after 30 minutes, M30 =

3. Mixing index after 45 minutes, M45 =

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Experiment-14

DESCRIPTION OF CONSTRUCTION WORKING AND APPLICATION OF PHARMACEUTICAL MACHINERIES

Aim: To describe the Construction, working and application of Pharmaceutical machineries such as rotary tablet machine, fluidized bed coater, fluid energy mill, de humidifier etc.

(1) Rotary Tablet Machine

Also called multi station tablet press. It is called rotary machine because the head of the machine that holds the upper punches, dies and lower punches in place rotates.

Steps involved in manufacturing of tablets

1. The material to be fed through hopper.

2. The fill cam pulls the lower punches down to a fixed distance and the dies are filled with material.

3. The quantity of the material filled is larger than the actual amount required. Remove excess amount with the help of spatula.

4. After that, upper punch is lowered and inserted into the dies.

5. The material is compressed and the tablet are formed

6. 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.

7. Then tablet is passed through the discharge chute.

Application

1. In the continuous manufacturing of tablets.

2. Used for large scale production

3. A single rotary press produce 1150 tablets in a minute while double rotary press can produce 1 0,000 tablets in a minute.


Rotary Tablet Machine

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(2) Fluidized Bed Coater

Three types of air suspension coaters are available, namely, Top spray coater, Wurster or bottom spray coater and Tangential spray coater. In top spray coater, there is a counter current (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 liquid 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

1. The drying inlet air is passed upwards through the bottom perforated plate into the fluid bed chamber.
2. This air passes to Wurster column, in which a spray gun perpendicular to bottom plate, and parallel to the Wurster column.
3. The air is passed out from the exhaust filters situated at the top of the equipment.
4. The material to be coated is loaded in the fluid bed chamber and fluidized.
5. The inlet air causes fluidization of the material as well as its drying during the coating operation.
6. The pellets are passing through the liquid spray of coating solution from the spray gun positioned parallel to the column.
7. After coating the coated particle falls by gravity at the bottom of Wurster column and recycled to coating zone.
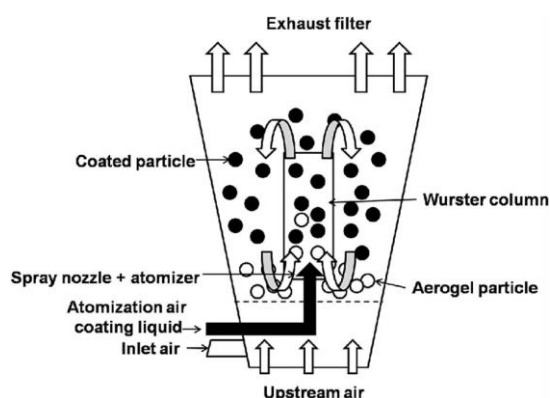
Fluidized bed coater

Applications

1. 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.

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2. Fluidized bed coaters are used for coating of powders, granules, tablets, pellets etc by column of air.
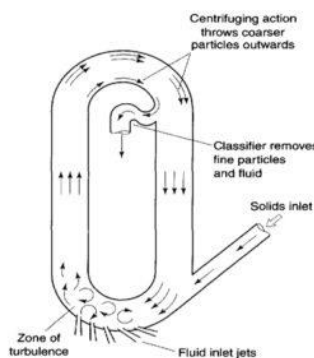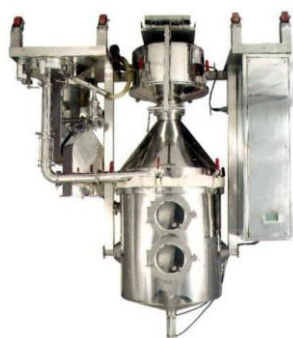
(3) Fluid Energy Mill

1.A fluid usually air, is injected at very high pressure through nozzles at the bottom of the loop. As a result, turbulence produce.

2. Solids are introduced into the stream through hopper.

3. Due to this turbulence occur and impacts and attrition occur between the particles

4. A classifier is fitted at the exit so that only finer size particles are collected as products.

5.The larger size particles are again sent to the stream of air for further size reduction.

Fluid Energy Mill

Applications

1.When we need small particle size which is not possible though other methods of size reduction.

2.No chance of contamination

3.This method is suitable where fine powders are required like micronization of griseofulvin.

(4) **Dehumidifier**

1. Warm moist air is sucked in through one side of the machine

2.An electric fan is used to draw the air inward.

3. The warm air passes through cold pipes through which a coolant circulates. Due to cooling of air, the moisture it contains turns back into liquid water.

4. Then the air passes over a heating element and warm backs up to its original temperature.

5. Warm dry air blows back into the room through another side of the machine

6. The moisture that was in the air drips down into a collecting tray (or Bucket) at the bottom of the machine.

7. As the collecting tray fills up, a plastic float in the machine raises upward.

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S. When the tray is full, the float trips an electric switch that turns of the fan and switches on an indicator light which indicates that the machine needs emptying.
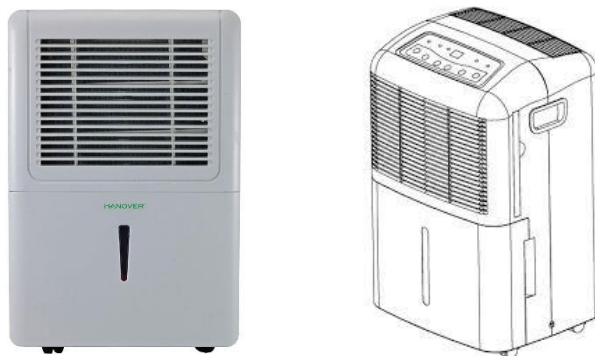

Dehumidifier

<u>Applications</u>

A dehumidifier is used to reduce the level of humidity in the air

2. Large dehumidifiers are also used in commercial buildings such as indoor ice, rinks to control the humidity level.

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Experiment-15

## DEMONSTRATION OF COLLOID MILL, PLANETARY MIXER, FLUIDIZED BED DRIER, FREEZE DRIER

Aim: To demonstrate colloid mill, planetary mixer, fluidized bed drier, freeze drier

Colloid Mill
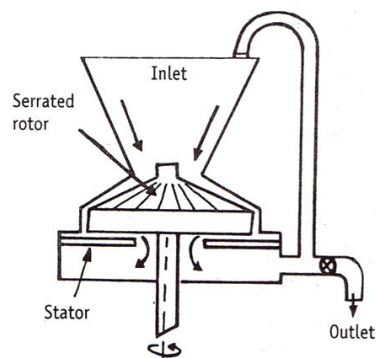
Colloid mill

Colloid mill is used to reduce the size of suspended droplets.

1. The material is feed through the inlet hopper and placed into the mill.
2. It is then moved through the narrow gap between the rotor and stator to reduce the particle size
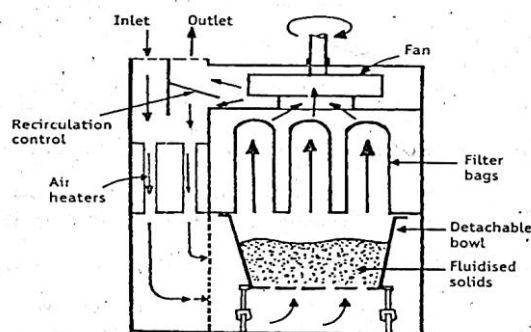3. The final product is removed through the out let.

Planetary mixer
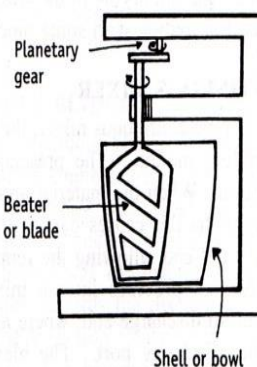
Planetary mixer

1. The material to be mixed is loaded into mixing bowl or shell.
2. The blades rotate on their own axis when they orbit the mixing bowl on a common axis. Therefore there won't be any dead spot in the mixing and high shear is applied for mixing.
3. After mixing the material is discharged through a bottom valve, or by manual scooping of the material from the bowl.

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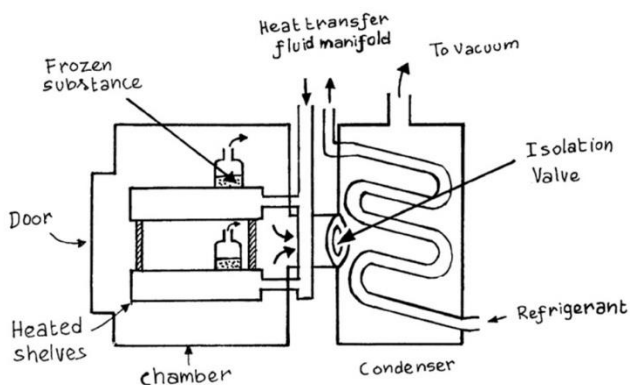
Fluidized Bed Drier

Fluidized bed dryer

The wet granules to be dried are placed in a detachable bowl. The bowl is inserted in the drier

1. Fresh air can pass through a pre filter, which is heated when passing through a heat exchanger.

2. Hot air pass through the bottom of the bowl. At the same time, the fans start to rotate. The airspeed increases gradually.

3. After a specific time period, 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 chamber, This condition is said to be "fluidized state".

4. The gas surround each granule allows them to dry completely. The air comes out of the drier passing through the filters in the bag

5. The entrained particles remain adhered to the interior of the surface of the bags. Periodically the bags are shaken to remove entrained particles

6. The materials are left in the drier to reach room temperature

7. The bowl is removed for unloading. The final product will be free flowing

Freeze Dryer

Freeze drier

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1. 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.

2. The product should be frozen at temperatures low enough to solidify completely. The products are frozen in two ways. most of the products lyophilized will contain water. It is very important in freeze drying to pre freeze the product below the eutectic temperature before the beginning of the process

3. 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.

4. After primary freeze drying is complete and all ice formed are sublimed, bound moisture can be found in the product at concentrations of 7-8%. Continued drying is necessary in warmer temperatures to reduce the residual moisture content to optimum values. The process is called "isothermal desorption". Secondary drying is usually carried out for approximately 1/3 or ½ the time required for primary drying.

5. After vacuum is replaced by inert gas, bottles and vials containing products are closed.

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Experiment-16

EFFECT OF TIME ON RATE OF CRYSTALLIZATION

Aim: To study the effect of time on rate of crystallization

Requirements: Beakers, glass slides, microscope etc.

Principle

      The process of formation of a crystalline structure from a liquid, gas or an amorphous solid. Crystals are formed in a super saturated solution and yield a defined crystal structure while precipitation is formed due to reaction of two substances to form sparingly soluble product which is something cloudy and ill defined.

The objectives of crystallization include

1. To obtain pure product
2. To obtain a product with better bioavailability
3. To obtain a more stable and attractive product
4. A crystalline substance possesses excellent flow property
5. Improved physical and chemical stability.

Super saturation can be defined as the concentration of a compound in its solution is more than the saturation solubility of the compound in that solvent.

Applications in pharmacy

In the manufacturing of solid dosage forms like tablets.

Procedure

1. Refined, bleached and de odorized palm oil is placed in a beaker which is initially heated in a thermally controlled water bath for 30 minutes at 70 º C to totally melt the oil.
2. The temperature is then reduced to 30ºC within one hour followed by reducing to crystallization temperatures of 14ºC or 22 ºC within 30 minutes.
3. Once the oil reached the desired temperatures (14 ºC or 22 ºC),it is allowed to crystallize until 90 minutes where the analysis were made at 5,10,15,30,60 and 90 minutes to obtain the morphology of the crystals.
4. The beaker content is stirred constantly at 90 rpm throughout the process using a propeller stirrer

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5. Samples of slurries are withdrawn at 5,15,30,60 and 90 minutes of crystallization and placed on to a glass slide covered with a cover slip.

6. Photographs of the crystals are taken at magnification of 200 x.

7. The length of four longest dimensions of each crystal are recorded and an average of at least six crystals are measured during each observation.

Observation and calculation

Time (min)	Crystal size (nm)
5	
10	
15	
30	
60	
90	

Report

The effect of time on rate of crystallization is studied and reported.

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Kerala University of Health Sciences

Syllabus (2017)

PHARMACEUTICAL ENGINEERING (Theory)

45 Hours

Scope: This course is designed to impart a fundamental knowledge on the art and science of various unit operations used in pharmaceutical industry.

Objectives: Upon completion of the course student shall be able:

1. To know various unit operations used in Pharmaceutical industries.

2. To understand the material handling techniques.

3. To perform various processes involved in pharmaceutical manufacturing process.

4. To carry out various test to prevent environmental pollution.

5. To appreciate and comprehend significance of plant lay out design for optimum use of resources.

6. To appreciate the various preventive methods used for corrosion control in Pharmaceutical industries.

Course content

UNIT-I   10 Hours

• Flow of fluids: Types of manometers, Reynolds number and its significance, Bernoulli's theorem and its applications, Energy losses, Orifice meter, Venturimeter, Pitot tube and Rotameter.

• Size Reduction: Objectives, Mechanisms & Laws governing size reduction, factors affecting size reduction, principles, construction, working, uses, merits and demerits of Hammer mill, ball mill, fluid energy mill, Edge runner mill & end runner mill.

• Size Separation: Objectives, applications & mechanism of size separation, official standards of powders, sieves, size separation Principles, construction, working, uses, merits and demerits of Sieve shaker, cyclone separator, Air separator, Bag filter & elutriation tank.

• Mixing: Objectives, applications & factors affecting mixing, Difference between solid and liquid mixing, mechanism of solid mixing, liquids mixing and semisolids mixing. Principles, Construction, Working, uses, Merits and Demerits of Double cone blender, twin shell blender, ribbon blender, Sigma blade mixer, planetary mixers, Propellers, Turbines, Paddles & Silverson Emulsifier.

UNIT-II   10 Hours

• Evaporation: Objectives, applications and factors influencing evaporation, differences between evaporation and other heat process. principles, construction, working, uses, merits and demerits of Steam jacketed kettle, horizontal tube evaporator, climbing film evaporator, forced circulation evaporator, multiple effect evaporator& Economy of multiple effect evaporator.

• Heat Transfer: Objectives, applications & Heat transfer mechanisms. Fourier's law, Heat transfer by conduction, convection & radiation. Heat interchangers & heat exchangers.

UNIT- III   10 Hours

• Drying: Objectives, applications & mechanism of drying process, measurements & applications of Equilibrium Moisture content, rate of drying curve. principles, construction, working, uses, merits and demerits of Tray dryer, drum dryer spray dryer, fluidized bed dryer,
vacuum dryer, freeze dryer.

• Distillation: Objectives, applications & types of distillation. principles, construction, working, uses, merits and demerits of (lab scale and industrial scale) Simple distillation, preparation of purified water and water for injection BP by distillation, flash distillation, fractional distillation, distillation under reduced pressure, steam distillation & molecular distillation.

UNIT-IV   08 Hours

• Filtration: Objectives, applications, Theories & Factors influencing filtration, filter aids, filter media. Principle, Construction, Working, Uses, Merits and demerits of plate & frame filter, filter leaf, rotary drum filter, Meta filter & Cartridge filter, membrane filters and Seitz filter.

• Centrifugation: Objectives, principle & applications of Centrifugation, principles, construction, working, uses, merits and demerits of Perforated basket centrifuge, Nonperforated basket centrifuge, semi continuous centrifuge & super centrifuge.

UNIT- V   07 Hours

• Materials of pharmaceutical plant construction, Corrosion and its prevention: Factors affecting during materials selected for Pharmaceutical plant construction, Theories of corrosion, types of corrosion and their prevention. Ferrous and nonferrous metals, inorganic and organic nonmetals.

• Material handling systems: Objectives & applications of Material handling systems, different types of conveyors such as belt, screw and pneumatic conveyors.

Recommended Books: (Latest Editions)

1. Introduction to chemical engineering – Walter L Badger & Julius Banchero, Latest edition.

2. Solid phase extraction, Principles, techniques and applications by Nigel J.K. Simpson-Latest edition.

3. Unit operation of chemical engineering – McCabe Smith, Latest edition.

4. Pharmaceutical engineering principles and practices – C.V.S Subrahmanyam et al., Latest

edition.

5. Remington practice of pharmacy- Martin, Latest edition.

6. Theory and practice of industrial pharmacy by Lachmann., Latest edition.

7. Physical pharmaceutics- C.V.S Subrahmanyam et al., Latest edition.

8. Cooper and Gunn's Tutorial pharmacy, S.J. Carter, Latest editi

Kerala University of Health Sciences

PHARMACEUTICAL ENGINEERING (Practical)

Syllabus (2017)

4 Hours/week

I. Particle size determination by beaker decantation method.

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

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

IV. Determination of moisture content and loss on drying.

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

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

VII. Size analysis by sieving – To evaluate size distribution of tablet granulations – Construction of various size frequency curves including arithmetic and logarithmic probability plots.

VIII. 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.

IX. Demonstration of colloid mill, planetary mixer, fluidized bed dryer, freeze dryer and such other major equipment.

X. Factors affecting Rate of Filtration and Evaporation (Surface area, Concentration and Thickness/ viscosity)

XI. To calculate the mixing Index for given sample by using Double Cone

Blender.

Third semester B. Pharm Degree examination

PHARMACEUTICAL ENGINEERING

Scheme for End semester practical examination

Max marks-35   Time:4 hrs

I. Synopsis (2.5 x 2 = 5 marks)

Principle and procedure of any two experiments mentioned in the syllabus

II. Major experiment (Any one from the following)   15 marks

a) Determine the effect of surface area/concentration/viscosity on the rate of evaporation.

b) Construct the drying rate curve for the given sample powder (determine the
   moisture content and loss on drying)

c) Perform the particle size analysis of the given sample powder by sieving.

d) Perform the particle size analysis of the given sample powder by beaker decantation.

e) **Det**ermine the mixing index of the given sample powder at different time intervals.

III. Minor experiment (Any one from the following)   10 marks

a) Determination of atmospheric humidity by psychrometric method.

b) Determination of atmospheric humidity by dew point method.

c) Determine the effect of surface area/concentration/viscosity on rate of filtration.

SPLIT UP OF MARKS

Sl No		Major(15)	Minor(10)
1	**Procedure with tabular column**	4	3
2	**Calculation including graph**	4	3
3	**Performance of the experiment**	4	2
4	**Report**	3	2
	Total	15	10

IV.   VIVA VOCE   (5 marks)

MODEL QUESTION PAPER

III SEMESTER B. PHARM END SEMESTER PRACTICAL EXAMINATION

[2017 SCHEME]

SUB: PHARMACEUTICAL ENGINEERING

Time: 4 hours
Maximum marks: 35

QUESTIONS

I. **Synopsis**   (2.5 X 2= 5 Marks)

   Write down the principle and procedure of,

   1. Steam distillation.

   2. Factors affecting filtration.

II. **Major experiment**   (15 marks)

   Study the factors affecting rate of evaporation and report- (surface area, concentration and viscosity)

III. **Minor experiment**   (10 marks)

   Find out the humidity of air inside the laboratory by dew point method and report.

IV. **Viva-voce**   (5 marks)

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