S.A.RAJA PHARMACY COLLEGE

VADAKKANGULAM 627 116

SUBJECT:

PHARMACEUTICAL ENGINEERING

III SEMESTER B.PHARM

PRACTICAL LAB MANUAL

PHARMACEUTICAL ENGINEERING

(PRACTICAL MANUAL) SECOND YEAR (III SEMESTER)

1. CONSTRUCTION OF DRYING CURVE FOR CALCIUM CARBONATE

AIM:

Todetermine the construction of drying curve for calcium carbonate.

REQUIRMENTS:

- ❖ Petridish
- ❖ hot air oven
- ❖ calcium carbonate
- ❖ weighing balance
- ❖ spatula

PRINCIPLE:

To behaviour of drying of solid is explained by drying curve. The time required for drying a batch of weight of martial in a dry air can be estimated with the help of drying curve.Drying is a mass transfer process consists of the removal of water or other solvent by evaporations from a solid, semisolid or liquid. This process is obtained used as final production steps before, packing products.

PROCEDURE:

- ❖ Take a clean Petridish without Lid and consider it's weight as W_1 gm
- ❖ Note the area of petridish
- ❖ Take 10gm calcium carbonate in a clean petridish and consider its weight as W_2 gm
- ❖ Prepare slurry by adding water consider it's weight as W_3 gm
- ❖ Heat petridish in hot air oven at temperature of 70°C
- ❖ Note down the weight of the sample after every 15 minutes.
- ❖ Continue drying until there is no change in weight of the sample is obtained
- ❖ Determine percentage moisture content and drying rate by using following formula
- ❖ Percentage moisture = W3-W2/W3-W1 $\times$ 100

❖ Drying content =W3-W2/area of petridish ×100

REPORT:

❖ The construction of drying curves of calcium carbonate was determined

❖ The percentage moisture content is =

Calculation:

Weight of empty petridish (w_1) gm =

Weight of empty petridish + sample (w_2) gm =

Weight of empty petridish + sample + water (w_3) gm =

Weight of empty petridish + sample + water + (w_4) gm =

(After drying at different time interval)

MC_1 (moisture content at 0 time) $= \dfrac{W3-W1}{W2-W1}$

MC_2 (moisture content at 15 minutes time) $= \dfrac{W4-W2}{W2-W1}$

Average moisture content $= \dfrac{MC1+MC2}{2}$

Rate of drying $= = \dfrac{W3-W2}{area\ (A)petridish} \times$ time

2. DETERMINATION OF MOISTURE CONTENT AND LOSS ON DRYING

AIM:

To determine the moisture content and loss on drying.

REQUIRMENTS:

- ❖ Petridish
- ❖ Measuring cylinder
- ❖ Pipette
- ❖ Tray dryer
- ❖ Calcium carbonate
- ❖ Spatula
- ❖ Weighing balance

PROCEDURE:

- ❖ Take clean dry petridish and weight it (x)
- ❖ Weigh 10g of calcium carbonate powder and transfer to petridish and weight it
- ❖ Add sufficient amount of water with the help of pipette to the powder until slurry form.
- ❖ Again weigh the petridish and note down weight (y)
- ❖ Place the petridish in tray dryer and weigh it at every 5 min
- ❖ Allow it to dry until it's constant weight the down the constant dry weight (z)
- ❖ Calculate the percentage loss on drying and percentage moisture content for the given sample.

REPORT:

- ❖ The moisture content and loss on drying was determined
- ❖ The percentage of moisture content is =
- ❖ The loss on drying is =

Calculation:

Weight of empty petridish (x) =

Weight of empty petridish + sample + water (y) =

Weight of empty petridish + sample + water

(After drying at every 5 minutes) =

$$\text{Loss of drying} = \frac{weight\ of\ moisture\ in\ the\ sample}{total\ weight\ of\ wet\ sample}$$

$$\text{Percentage moisture content} = \frac{weight\ of\ moisture\ in\ the\ sample}{weight\ of\ dry\ sample}$$

3. RADIATION CONSTANT OF UNPAINTED GLASS

AIM:

To determine the radiation constant of unpainted glass.

PRINCIPLE:

Flow of heat takes place from the high temperature region towards the low temperature region. This is based upon the following three mechanisms.

A) CONDUCTION

It is considered as one of the efficient modes of heat transfer this mechanism of heat transfer occurs through this mechanism by heat transfer occurs through transfer of the momentum of each atoms or molecules without mixing conduction is limited to solids and fluid.

B) CONVECTION:

In this made of heat transfer, the warmer parts mix with the colour parts of the same substance heating of water by using coil type water heater is one of the examples of convections here, the energy is transferred as heat to a flowing fluid by a hot surface convection is limited to the flow of heat in fluids(i.e, liquids and gases).

C) RADIATION:

Mechanism of heat transfer through apace by means of electromagnetic waves is called radiation. A good examples of radiation is black body radiation which occurs by absorbing all energy incidents upon it, at the same time the quantitatively transferred into heat the radiant thermal energy expressed by **"STEFFAN BALOZMANN"** equation as given below.

$$q = bAT^4$$

Where,

q = energy radiated per second (w)

A= Area of radiating surface (m^2)

T= absolute temperature of the radiating surface (k)

b= constant w/m^2 x k^4

Radiation constant is calculated by using following equation,

$$M_1 S_1 - M_2 S_2 \, dq/dt = \alpha \, A \, [(T_1/100)^4 - (T_2-100)^4] + b \, A \, (T_1 - T_2)$$

REQUIREMENTS:

- ❖ Round bottom flask (unpainted)
- ❖ Thermometer (110^0)
- ❖ Hot plate or burner
- ❖ Stain with clamp
- ❖ Stop clock, Tripod stand
- ❖ Weighing balance
- ❖ Purified water

PROCEDURE:

- ❖ A round bottom flask is cleaned and dried.
- ❖ The weight of the flask is determined(M_2/kg)
- ❖ The diameter(d) of neck of flask is determined
- ❖ Boil hot water is prepared and measured volume of hot water is transferred to the flask (M_1). The volume of water is external surface of the round bottom flask is thoroughly dried the flask with hot water is placed on the tripod stand.
- ❖ Thermometer (110^0) is dipped to centre of the flask and tied at the top to and iron stand.
- ❖ Slowly the temperature of the hot body decreases.
- ❖ The decrease in temperature is noted every minute.
- ❖ The data are recorded in table.

- ❖ A graph is plotted by taking time (minutes) or X-axis and temperature on Y-axis normally is a curve is obtained.
- ❖ Depending on the temperature at which radiation constant is determined, a tangent is draw at that temperature the slope is calculated (dq/dt).
- ❖ Radiation constant (α) is determined at the temperature.

Calculations:

Diameter of round bottom flask (d) =

Radius of round bottom flask (r) =

Diameter of neck of round bottom flask (d) =

Radius of neck of round bottom flask (r) =

Surface area of round bottom flask = $4\Pi r^2 - \Pi r^2$

Empty weight of round bottom flask (M_2) =

Volume of heat water with flask (M_1) =

Room temperature (t_2) =

Derived room temperature (t_1) =

Specific heat of water (r_1) =

Specific heat of glass (r_2) =

$$M_1 S_1 - M_2 S_2 \ dq \ /dt \ = \alpha \ A \ [(T_1/100)^4 - (T_2 - 100)^4] + b \ A \ (T_1 - T_2)$$

REPORT:

The radiation constant of unpainted glass α =

4. DETERMINATION OF PARTICLE SIZE DISTRIBUTION BY SIEVING METHOD

AIM:

To determine the average particle size and find out their distribution pattern for the given granules by sieve analysis method.

PRINCIPLE:

Sieve method gives sieve diameter, sieve diameter is defined as the diameter of the sphere that possess through the sieve aperture as the asymmetric particle sieve method directly give weight distribution. Particles having size range from 50 and 1500µm are estimated by sieving method. In this method, the size is expressed as d_{sieve}. The sieving method finds application in dosage and development of tablets and capsules. Normally 15 percent of fine powder (passed through mesh 100) should be present in granulated material to get a proper flow of material and achieve good compaction in tableting. Therefore, percent of coarse and fine can be quickly estimated. Sieves for pharmaceutical testing are constructed from wire cloth with square meshes, woven from wire of brass, bronze, stainless steel or any other suitable material.

Designations and Dimensions of I.P specification sieves

Sieve Number	Aperture Size Micrometer	Sieve Number	Aperture Size Micrometer
10	1700	44	325
12	1400	60	250
16	1000	85	35
22	710	100	36
25	600	120	34
30	500	150	36
36	425	170	35

Advantages of sieving method

1. It is in expensive, sample and rapid with reproducible results.
2. Sieving method is useful when particles are having size range between 50 and 1500µm.

Disadvantage of sieving method

1. Lower limit of the particle size is 50µm.
2. If the powder is not dry, apertures become clogged with particles leading to improper sieving.
3. During shaking, attrition occurs causing size reduction of particles. This leads to errors in estimation.

Factors influencing the sieving method

Factors influencing sieving are weight of sample, duration of shaking and type of motion. The types of motion influencing sieving are vibratory motion, (most efficient), side tap motion, bottom pat motion, rotary motion with tap and rotary motion. The type of motion standardized. Care should be taken in order to get reproducible results.

PROCEDURE:

1. Standard sieves set is selected (sieve no: 10, 22, 36, 44, 65, 80, 100,120) arrange them in such manner that the coarsest remains at the top and finest at the bottom.

2. Weigh approximately 50g of sample, place the sample on the coarsest sieve no.10.

3. Fix the above sieves set on hand sieve shaker and shaken for 20 minutes.

4. Collect the Sample retained on each sieve into a paper, weigh all the ample.

5. Report the weights retained on each sieve in the table against corresponding sieve number.

Sieve No	Aperture Size As Per I.P (μm)	Arithmetic Mean Size of opening (d) (μm)	Weight of Granules Retained on Sieve over Size (gm)	% Wt of Granules Retained (Under Size) N	Cumulative % Granules retained (Under Size)	Weight Size (n×d)	% Under Size	% Over Size (100-% Under Size)
10	1700	1205	2.1	2.1%	2.1	2530.5	2.1	97.9
22	710	922.5	71.5	71.5%	73.6	65958.75	73.6	26.4
36	425	602.5	13.6	13.6%	87.2	8194	87.2	12.8
44	355	302.5	2.5	2.55%	89.7	756.25	89.7	10.3
60	250	215	3.0	3.0%	92.7	645	92.7	7.3
85	180	165	1.7	1.7%	94.4	280.5	94.4	5.6
100	150	137.5	2.3	2.3%	96.7	316.25	96.7	3.3
120	125	62.5	1.0	1.0%	97.7	62.5	97.7	2.3
pan	0	0	1.1	1.1%	98.8	0	98.8	1.2
			$\sum_n=98.8$			$\sum(n×d)=78743$		

Average diameter $= \dfrac{\sum(\mathbf{nxd})}{\sum\mathbf{n}}$

$= \dfrac{\mathbf{78743.75}}{\mathbf{98.8}}$

$= \mathbf{797.001}\mu m$

Report:

The average diameter of the given granules was found to be = μm

5.FACTORS AFFECTING RATE OF FILTERATION

AIM:

To study the effect of various factors on the rate of filtration.

Apparatus:

- ✓ Beaker
- ✓ Filter paper
- ✓ Measuring cylinder

Principle:

Filtration is a process of where by solid particles present in a suspension are separated from the liquid or gas employing a porous medium. Which retains the solid but allows the fluid to pass through the volume of the filtrate obtained through the filter paper per unit time is called "**rate of filtration"** can be given by mathematical equation.

$$dv/dt = K.A . \triangle P/\mu L - \text{darcy's law}$$

where,

A = area of filter

$\triangle P$ = pressure drop across the filter medium and cake

μ = viscosity of filtrate

L = thickness of cake

V = volume of the filtrate

T = time taken for filtration

K = constant for the filter medium and filter cake or resistance

Procedure:

a) Effect of thickness of cake:

Prepare two solutions of calcium carbonate using water as the solvent the concentration of the solutions are 5% and 10% respectively. Filter them and note the time taken for filtration to calculate the rate of filtration and compare them.

b) Effect of viscosity:

Effect of viscosity causing two solutions one with water and other with mixture of glycerine and water (20:80) ratio respectively. Prepare two different solutions of 5% calcium carbonate ($CaCo_3$) using above prepared water and glycerine mixture. Filter them and note the time for filtration to calculate the rate of filtration and compare them.

c) Effect of area:

This can be determined by using funnel of large (big) and small area for the same concentration of the solutions (5% $CaCo_3$) time taken for filtrations are noted. Calculate the rate of filtration and compare them.

d) Effect of pressure:

Prepare two solutions of calcium carbonate (5% $CaCo_3$) using water as a solvent filter one of the solutions through Buchner funnel. Which is connected to a suction pump and the other one is filtered through without suction pump and note the time taken for filtrate. Calculate the rate of filtration and compare them

Calculation:

Effect of thickness of cake:

Sample: 50 ml of 5% CaCo3

 50 ml of 10% CaCo3

Sample	Time Taken For Filtration	Volume of Filtrate	Rate of Filtration
5% CaCo3	5 minutes (300 seconds)	34 ml	34/300 = 0.1134 ml/sec
10% CaCo3	7 minutes (420 seconds)	40 ml	40/420 = 0.0952 ml/sec

Effect of viscosity:

Sample: 50 ml of 5% CaCo3

50 ml of 10% CaCo3

(Glycerine and water mixture 20:80)

Sample	Time Taken For Filtration	Volume of Filtrate	Rate of Filtration
5% CaCo3	8 minutes (480 seconds)	35 ml	35/480 = 0.0729 ml/sec
5% CaCo3 with glycerin and water mixture	6 minutes (360 seconds)	44 ml	44/360= 0.1222 ml/sec

Effect of area of funnel:

Sample: 50 ml of 5% CaCo3

Sample	Time Taken For Filtration	Volume of Filtrate	Rate of Filtration
5% CaCo3 filter in small funnel	7 minutes	42 ml	0.1 ml/sec
10% CaCo3 filter in big funnel	7 minutes	40 ml	0.09 ml/ sec

Effect of pressure:

Sample: 50 ml of 5% CaCo3 (glycerine water mixture 20:80)

Sample	Time Taken For Filtration	Volume of Filtrate	Rate of Filtration
5% CaCo3 filter with pressure	36 seconds	46 ml	1.27 ml/sec
10% CaCo3 filter without pressure	6 minutes	42 ml	0.116 ml/ sec

Report:

Rate of filtration in thickness

$\qquad$ 5% caco3 $\qquad$ =

$\qquad$ 10% caco3 $\qquad$ =

Rate of filtration in viscosity

$\qquad$ 5% caco3 $\qquad$ =

$\qquad$ 5% caco3 with glycerine $\qquad$ =

$\qquad$ Water mixture

Rate of filtration in area

$\qquad$ 5% caco3 in small funnel $\qquad$ =

$\qquad$ 5% caco3 in big funnel $\qquad$ =

Rate of filtration in pressure

$\qquad$ 5% caco3 with pressure $\qquad$ =

$\qquad$ 5% caco3 without pressure $\qquad$ =

6. FACTORS AFFECTING RATE OF EVAPORATION

AIM:

To study the effect of various factors on the rate of evaporation.

Principle:

Evaporationis the process by which liquid water goes directly to the vapour phase due to an increase in temperature. The main objective of evaporation is to get a concentrated product by vaporizing volatile liquid.Evaporation is conducted by non-volatile solute and volatile solvent to produce thick liquor. Rate of evaporation is controlled by rate of heat transfer.

Objectives of evaporation:

Evaporation is done by following,

- ➢ To get a concentrated product
- ➢ To remove water from aqueous solution
- ➢ To evaporate sea water for developing drinking water
- ➢ To get solid from water which is used in boiler for chemical process

Factors influencing evaporation:

1. **Temperature**

   The evaporation rate directly related to the temperature. As the temperature increases the rate of evaporation also increases because the temperature is rising the water molecules begin to move faster.

2. **Surface area**

   The rate of evaporation is directly proportional to the surface area of the vessel exposed to evaporation.

3. **Agitation**

   Agitation is necessary for evaporation.

4. **Vapour pressure**

   Liquids with low boiling point evaporate quickly due to high vapour pressure.

5. **Types of product required**

   The selection of the method and apparatus to be used for evaporation depends upon type of product required for example open pan produce liquid or dry concentrate while film evaporator yield liquid concentrates.

6. **Density**

   As the density increases, the rate of evaporation decreases.

7. **Time of evaporation**

   Exposure to a relatively high temperature for a short time may be loss destructive of the active ingredients than a lower temperature with exposure for a longer period.

8. **Economic factors**

   When selecting the method and apparatus the economic factors are important. Evaporators are designed to give maximum heat transfer to liquid.

9. **Moisture content**

   Some drug constituents decompose more rapidly in the presence of moisture especially as raised temperature.

Effect of surface area on the rate of evaporation:

Requirements:

➢ Three petridishes of diameter 2.50 cm, 5 cm, 7.5 cm with cover, 10 ml of pipette and stop watch

Procedure:

- ❖ Clean and dry all petri plates and mark them as A, B, and C.
- ❖ Pipette out of 10 ml diethyl ether in each of the petri dishes A, B, and C and cover them immediately.
- ❖ All the petridish heated in water at constant temperature for 10 minutes

* ❖ Note the remaining volume after 10 minutes
* ❖ From the rate of evaporation is calculated

Effect of viscosity on the rate of evaporation:

Requirements:

➢ Glycerine, distilled water, beaker, measuring cylinder.

Procedure:

* ❖ Different concentration of glycerine and water are prepared in different beakers.
* ❖ Note the weight of beaker containing glycerine- water mixture.
* ❖ All the beakers are heated in water at constant temperature for 10 minutes.
* ❖ Again note the weight of the beakers after heating.
* ❖ Difference between the weights is measured. The difference indicated the amount of water evaporated during 10 minutes.
* ❖ From the rate of evaporation is calculated

Effect of concentration on the rate of evaporation:

Procedure:

* ❖ Clean all the glassware's
* ❖ Prepare different concentration (2%, 4%, 6%, 8%) solution of sodium chloride in 50 ml water in different beakers
* ❖ Weigh of the beaker containing sodium chloride solution
* ❖ All the beakers are heated in water at constant temperature for 10 minutes
* ❖ Again weigh the beakers after heating
* ❖ The difference between weights is measured. The difference indicated the amount of water evaporated during 10 minutes
* ❖ From this rate of evaporation is calculated.

Effect of surface area:

Petridish Marked	Diameter of Petridish	Volume Taken	Remaining Volume	The Volume of Liquid Evaporated	Rate of Evaporation (ml/sec)
A	2.5	10 ml			
B	5	10 ml			
C	7.5	10 ml			

Report:

Rate of evaporation in surface area

A =

B =

C =

Effect of viscosity:

Glycerin (ml)	Water (ml)	Concentration % V/V	Initial Weight of Solution (g)	Final Weight (g)	Weight of Water Evaporated	Rate of Evaporation g/sec
2	5	2%				
4	10	4%				
6	15	6%				
8	20	8%				
10	25	10%				

Rate of evaporation in viscosity

2 ml =

4 ml =

6 ml =

8 ml =

10 ml =

Effect of concentration:

Concentration % W/V	Initial Weight of Solution (g)	Final Weight of Solution (g)	Weight of Water Evaporated	Time of Heating	Rate of Evaporation (g/sec)
2%	20				
4%	20				
6%	20				
8%	20				

Rate of evaporation in concentration

$$2\% =$$

$$4\% =$$

$$6\% =$$

$$8\% =$$

7. DESCRIPTION OF CONSTRUCTION WORKING AND APPLICATION OF PHARMACEUTICAL MACHINARY SUCH AS ROTARY TABLET MACHINE, FLUIDIZED BED COATED, FLUID ENERGY MILL, DEHUMIDIFIER

AIM:

Description of Construction Working and application of Pharmaceutical machinery Such as Rotary Tablet Machine, Fluidized Bed Coated, Fluid Energy Mill, Dehumidifier.

1) ROTARY TABLET MACHINE:

It is also called multi station tablet press. It is called rotary machine 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 tablet:

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

Applications:

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

Tablet compression

Stage 1
- top punch
- powder
- bottom punch

Stage 2
- excess powder removed

Stage 3
- tablet pressed

Stage 4
- tablet ejected
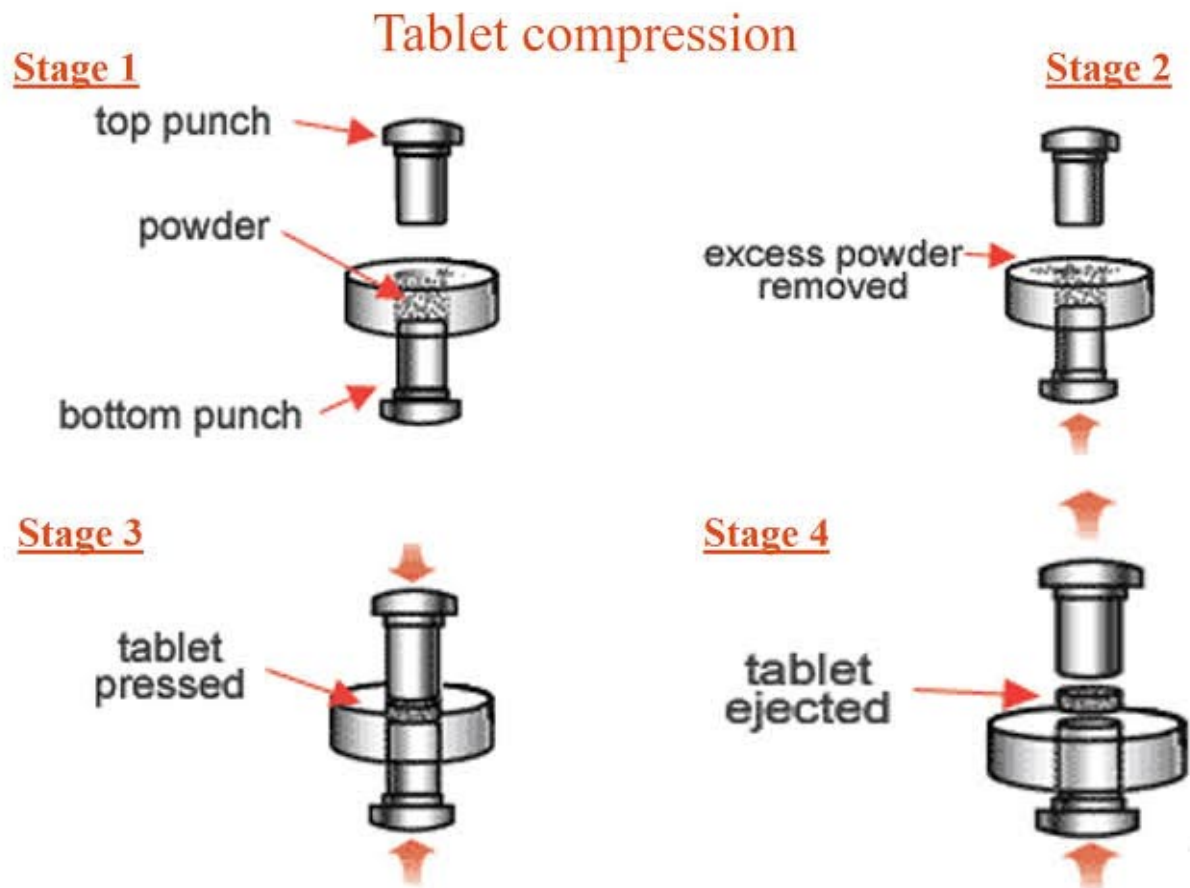
2) FLUIDIZED BED COATER:

Three types of air suspension coater are available, namely top spray coater, wurster or bottom spray coater, and tangential spray coater. In top spray coater, there is a counter current (opposite position) it 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:

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

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

Application:

- ❖ It is used to coat pharmaceutical dosage form with polymeric material to mark objectionable taste or odour and also to protect an unstable ingredient and to improve appearance
- ❖ Fluidized bed coaters are used for coating of powders, granules, tablets, pellets etc by column of air
- ❖ Fluidized bed coating equipment is popular for coating multiparticulate systems such as beads and non parallel seeds

Fluidized Bed Reactor Components

The material fluidized is a solid (catalyst).

The fluidizing medium is either a gas or a liquid.

Inlet to cyclone — product gas

freeboard — gas/solid separation

solids recycle

fluid bed of catalyst

heating/ cooling

Gas distributor

fluidizing gas/ educt — educt — windbox

**CHEMICAL REACTION ENGINEERING LABORATORY** — Washington
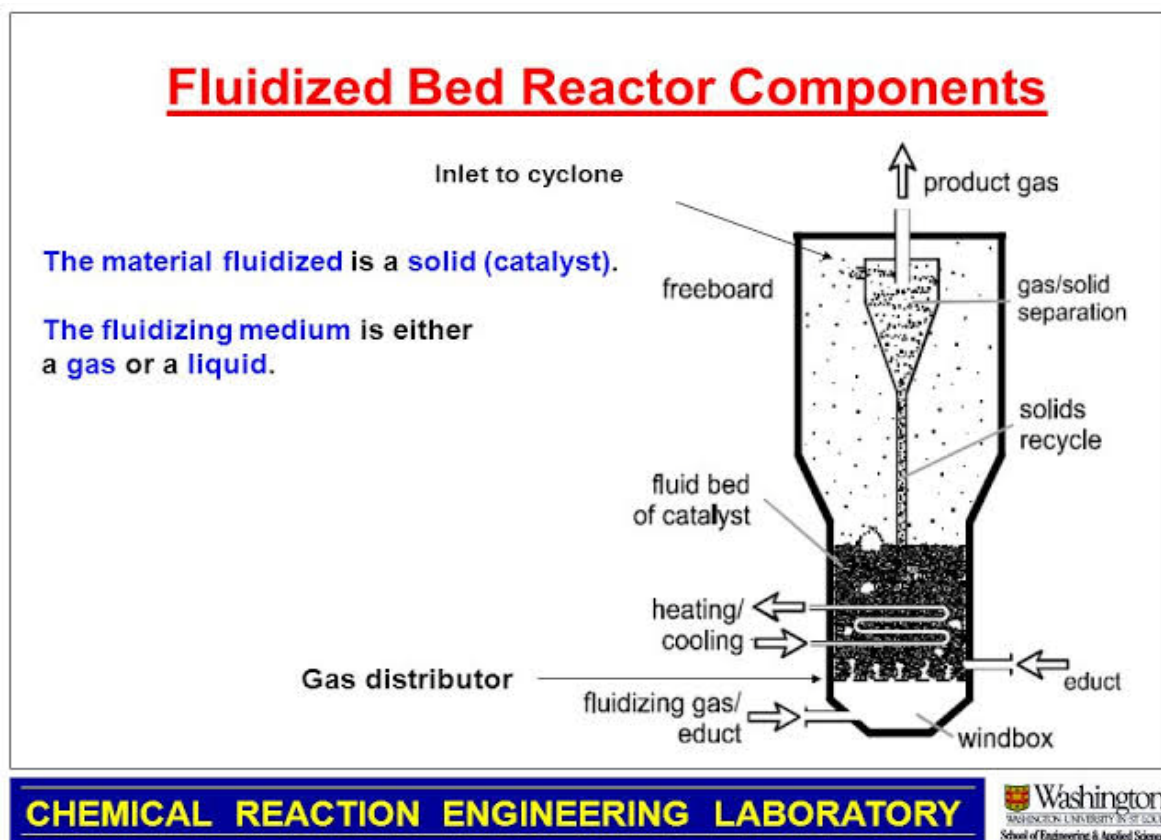
3) FLUID ENERGY MILL:

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

Application:

- ✓ The particle size of the product is smaller when compared to other method of size reduction
- ✓ No chance of contamination of the product
- ✓ This material is suitable where fine powders are required like micro ionization or griseofulvin
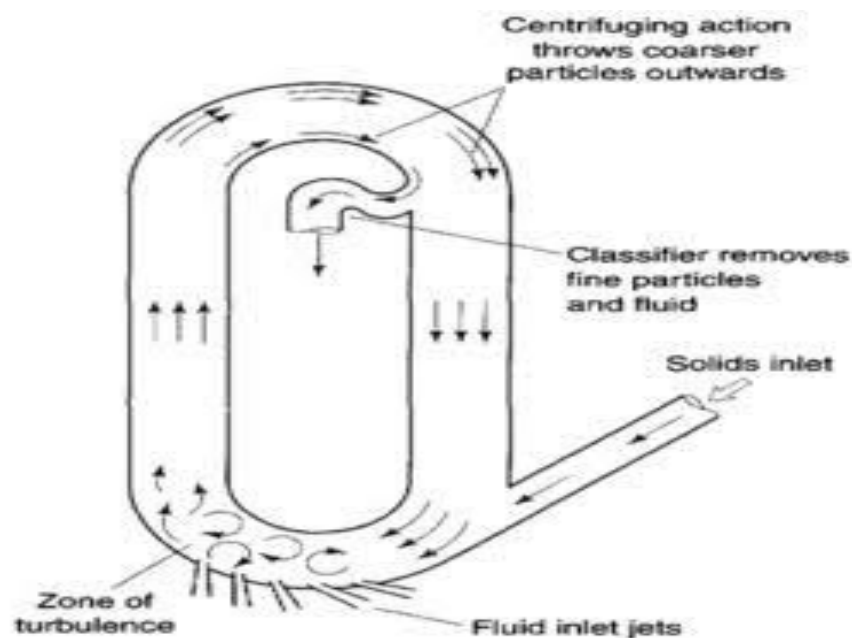
FLUID ENERGY MILL
PHOTO CREDIT: AULTON'S PHARMACEUTICS: THE DESIGN AND MANUFACTURE OF MEDICINE

4) DE-HUMIDIFIER:

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

Applications:

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

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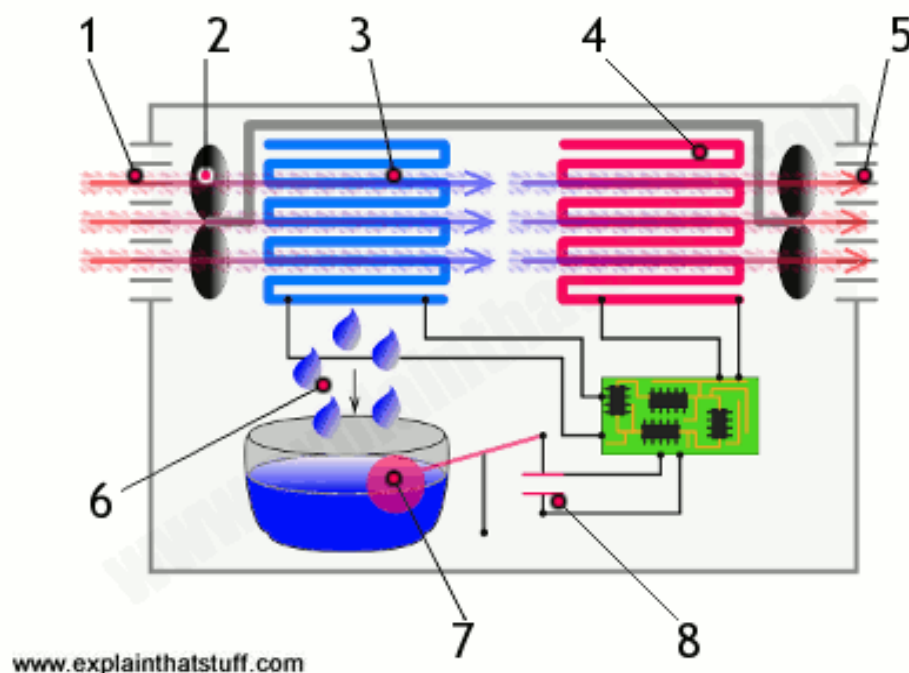
8. DEMONSTRATION OF COLLOID MILL, PLANETARY MIXER, FLUIDIZED BED DRYER, FREEZE DRYER

AIM:

Demonstrating Colloid Mill, Planetary Mixer, Fluidized Bed Dryer, Freeze Dryer.

1) COLLOID MILL:

❖ The colloid mill used to reduce the size of the suspended droplets

❖ The material is feed in through the inlet hopper and placed into the mill

❖ It is then move through the narrow gap between the rotor and stator to reduce the particle size

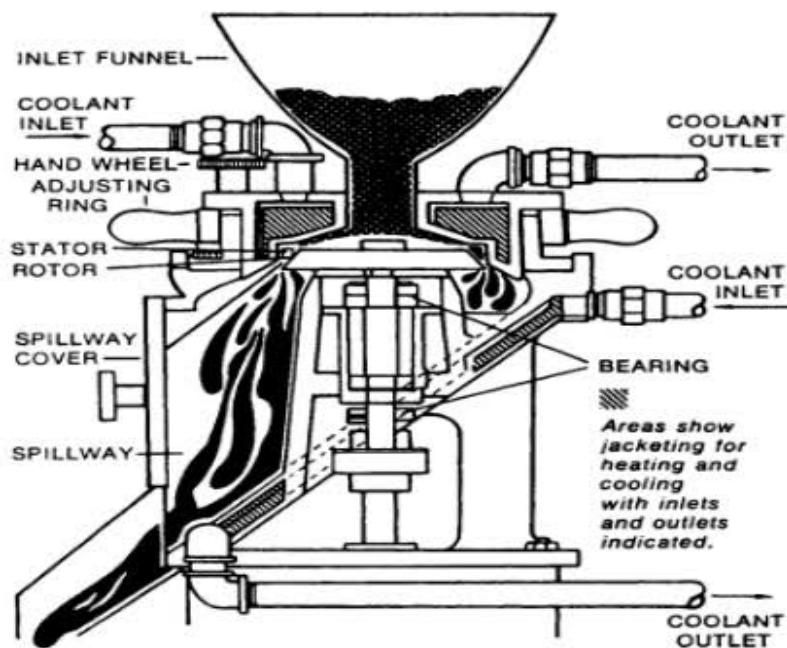❖ Then final product is removed through the outlet

Colloidal mill

Fig. 32   Cross-section of colloid mill. (From Ref. 29.)

## 2) PLANETARY MIXER:

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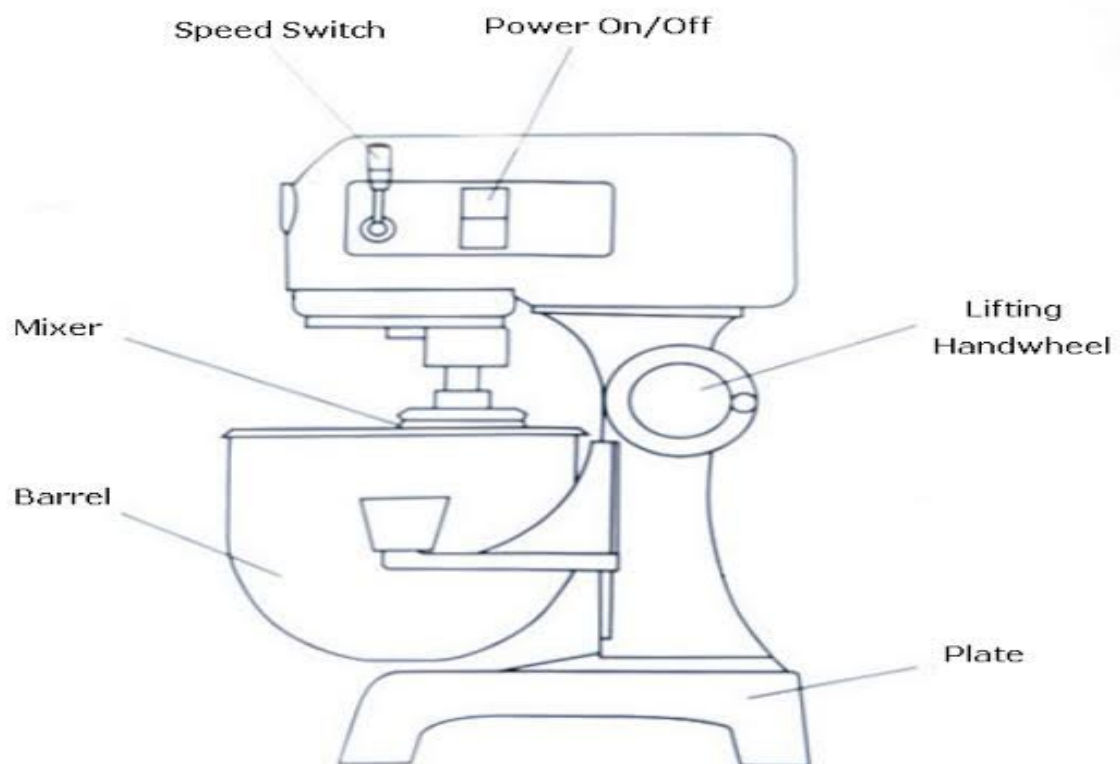

Planetary Mixer

3) FLUIDIZED BED DRYER:

- The wet granules to be dried are placed in a detachable bowl, the bowl is inserted in the dryer
- Fresh air can pass through a pre filter, which is then heated when passing through a heat exchanger
- Hot air flows through the bottom of the bowl at the same times, the fan starts to rotate the air speed increases gradually
- After a specific time a pressure point is reached in which the friction drag on the particles is equal to the force of gravity. The granules rise in the container. This condition is said to be fluidized state
- The gas surrounds each granule to dry them completely the air comes out of the dryer passing through the filters in the bag
- The entrained particles remain adhered to the interior of the surface of the bags. Periodically, the bags are shaken to remove entrained particles
- The materials are left in the dryer to reach room temperature
- The bowl is removed, the final product is free flowing

Figure 14-6. Fluidised bed dryer.
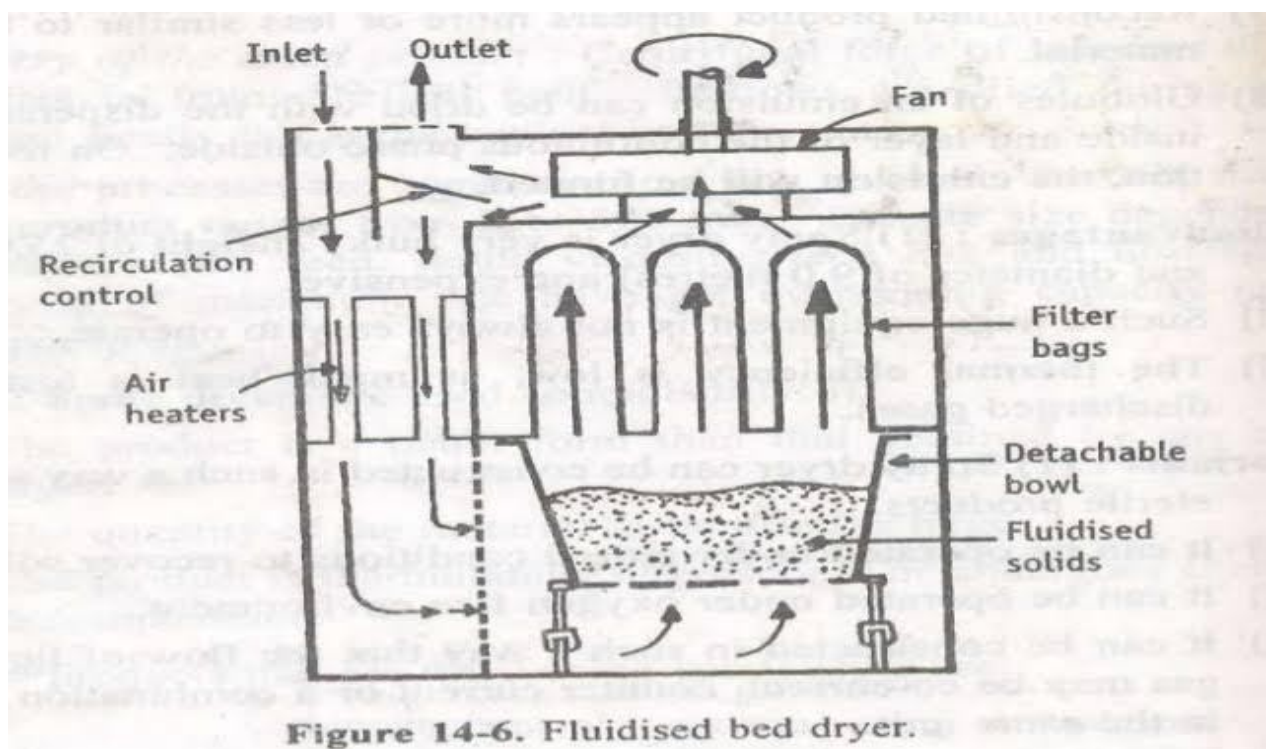
4) FREEZE DRYER:

❖ The material is pre-treated before freezing pre-treatment methods include freezing concentration, solution phase concentration and formulation to preserve the appearance of the product. Formulation to stabilize reactive products. Formulation to increase the surface area and decreasing high vapour pressure solvent

❖ The product should be frozen at a temperature low enough to solidify completely. The products are frozen in two ways, most of the products that are lyophilized consist mainly of water. It is very important in lyophilisation to pre-freeze the product below the eutectic temperature before beginning the lyophilisation process

❖ After the pre-freezing the product conditions must be established in which the ice can be removed from the frozen product through sublimation resulting in a dry, structurally intact product

❖ After primary freeze drying is completely and all the ice has sublimed, bound moisture is still present in the product. The product appears dry, but the residual moisture content may be as high as 7-8% continued drying is necessary at warmer temperature to reduce the residual moisture content to optimum value. This process is called "isothermal desorption" secondary drying is usually carried out for approximately 1/3 or 1/2 the time required for primary drying

❖ After vacuum is replaced by inert gas, bottle and vials are closed

11.Schematic diagram of freeze drier
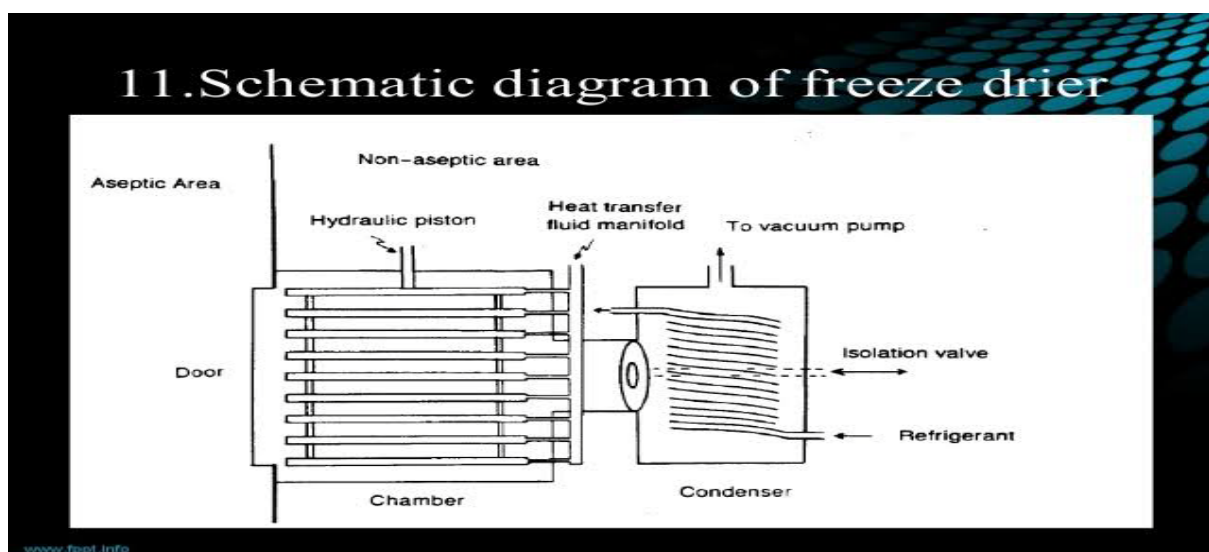
9. EFFECT OF TIME ON THE RATE OF CRYSTALIZATION

AIM:

To study the effect of time on the rate of crystallization.

REQUIREMENTS:

- ✓ Beakers
- ✓ Slide
- ✓ Cover slip
- ✓ Microscope

PROCEDURE:

- Refined bleached and deodorized palm oil are placed in a beaker which was initially heated in a thermally controlled water bath for 30 minutes at 70ºC to totally melt the oil.

- The temperature was then reduced to 30ºC within one hour followed by reducing to crystallization temperature of 14 or 22ºC within 30 minutes.

- Once the oil reached the desired temperature (14 or 22ºC), it was allowed to crystallize until 90 minutes where the analyses were made at 5,15,30,60 and 90 minutes to obtain the morphology of the crystals.

- The beaker content was constantly stirred at 90 rpm throughout the process using a stirring motor attached with two blades paddle propeller

- Samples of slurries were withdrawn at 5,15,30,60 and 90 minutes of crystallization and placed onto a slide which was then covered with a cover slip.

- Photograph of the crystals were taken at the magnification of 200x.

- The lengths of four longest dimensions of each crystal were recorded and an average of at least six crystals was measured during each observation.

Observation table:

Time (min)	Crystal Size (µm)
5	
15	
30	
60	
90	

Report:

Rate of crystallization of palm oil at the end of 90 minutes is = µm.