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SCHOOL OF BIO AND CHEMICAL ENGINEERING

DEPARTMENT OF CHEMICAL ENGINEERING

UNIT – I – MASS TRANSFER II – SCH1302

Introduction

Humidification is the process of constituting the water-vapor content in a gas. The reverse of the operation is called dehumidification. Both are important for many industrial operations such as air conditioning, gas cooling, controlled drying of wet solids, comfort heating etc. When a gas is brought in contact with a pure liquid in which it is essentially insoluble, interphase mass and heat transfer takes place.

Humidification operations: In this operation, water transfers from liquid phase to gas phase. Hence, moisture content of air increases. Air with particular moisture content is useful for drying of a solid under controlled condition.

Dehumidification operations: It is the reverse phenomena of humidification. A portion of water vapor from moist warm air is condensed by contacting cold water in air conditioning.

Four major applications of humidification operations are as follows:

a) Humidification of gases for controlled drying of wet solids
b) Dehumidification and cooling of gas in air conditioning
c) Gas cooling with the help of water
d) Cooling of liquid (e.g. water) before reuse

Terminologies and definitions:

Dry-bulb temperature: It is true temperature of air measured by a thermometer whose bulb is dry.

Wet-bulb temperature: It is the steady-state temperature attained by a small amount of evaporating water in a manner such that the sensible heat transferred from the air to the liquid is equal to the latent heat required for evaporation.

Relative humidity: It is the ratio of partial pressure of water vapour (pA) in air at a given temperature to the vapor pressure of water (pA°) at the same temperature.

$$\%relative\ humidity = \frac{p_A}{p_A^0} \times 100$$

Relative humidity does not 'explicitly' give the moisture content of a gas, but gives the 'degree of saturation' of the gas at a given temperature.

Absolute humidity (simply humidity): It is the direct measurement of moisture content in a gas. The mass of water vapor per unit mass of dry gas is called absolute humidity; it is occasionally called 'Grosvenor humidity' after the name of the inventor.

$$Y' = \left(\frac{p_A}{P - p_A} \right) \frac{18.02}{28.97}$$

Cooling tower principle and operation

A cooling tower is a special type of heat exchanger in which the warm water and the air are brought in direct contact for ‘***evaporative cooling’***. It provides a very good contact of air and water in terms of the contact area and mass transfer co-efficient of water vapor while keeping air pressure drop low. Enthalpy of air is lower than enthalpy of water. Sensible heat and latent heat transfer take place from water drop to surrounding air. Thus, cooling is accomplished by sensible heat transfer from water to air and evaporation of a small portion of water. A generalized cooling tower system is shown in Figure 6.4. The hot water which is coming from heat exchanger is sprayed at the top of the cooling tower. Air enters through the louvers at the two opposite walls of the cooling tower. During cooling process of water, around 2% water is evaporated. Make water is used to compensate the water loss due to evaporation. Blowdown is there to drain a part of water containing solid deposit. The exit cold water from the cooling tower is used in the heat exchanger or other unit operation.

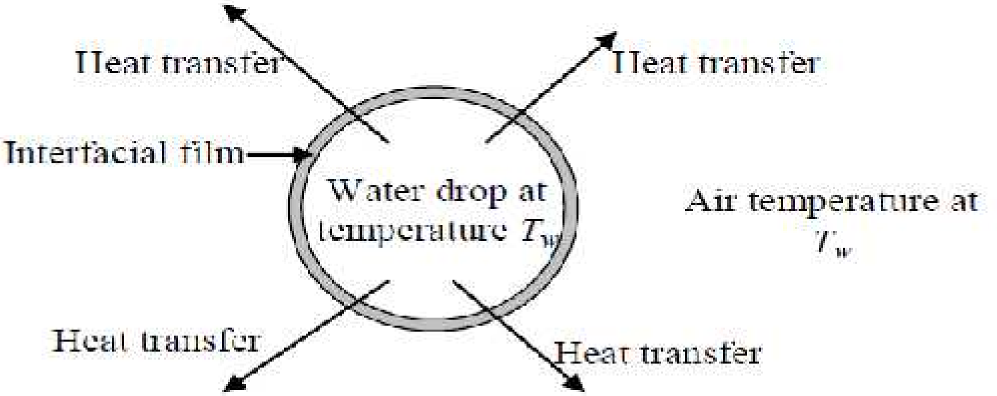


Figure 1.1 Wet Bulb temperature

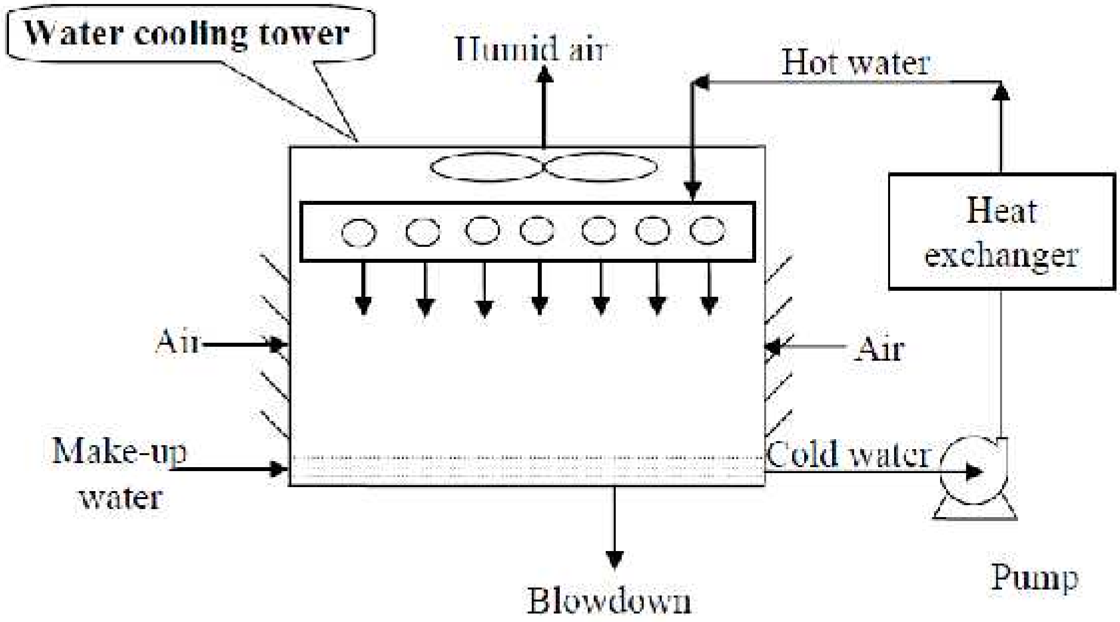


Figure 1.2 Cooling tower

Factors govern the operation of cooling tower

- The dry-bulb and wet-bulb temperatures of air
- Temperature of warm water
- The efficiency of contact between air and water in terms of volumetric mass transfer coefficient.
- Contact time between air and water
- The uniformity of the distribution of the phases within the tower
- Air pressure drop
- Desired temperature of cooled water

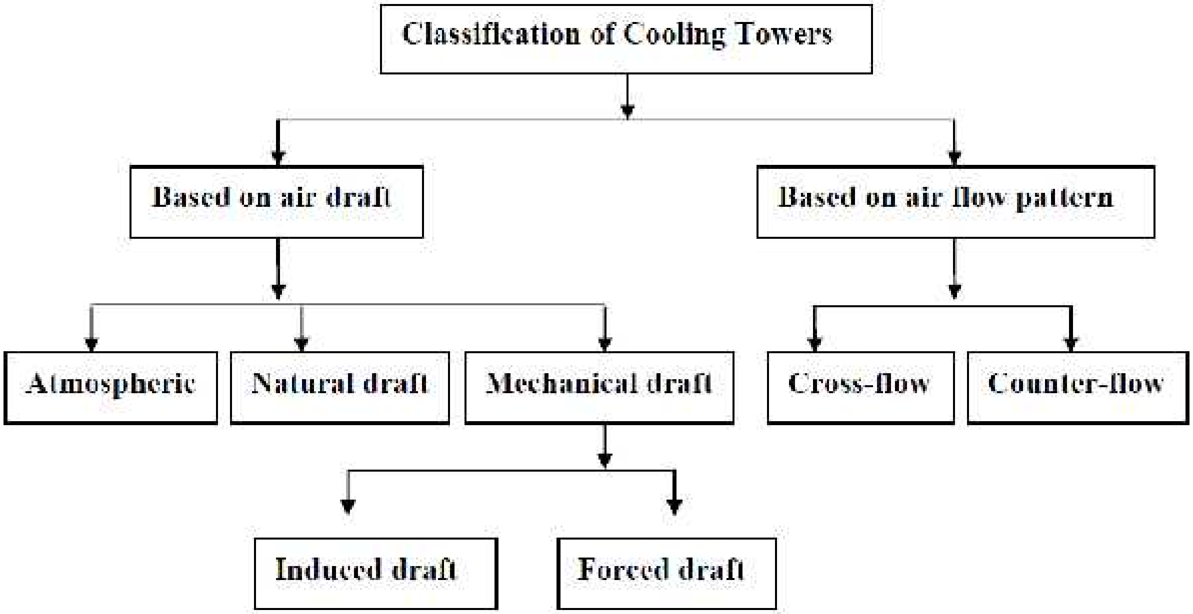


Figure 1.3 Classification of Cooling tower

It is a big rectangular chamber with two opposite 'louvered' walls. Tower is packed with a suitable 'tower fill'. Atmospheric air enters the tower through louvers driven by its own velocity. Direction and velocity of wind greatly influence its performance.

Natural Draft Towers

A natural draft cooling tower has a large reinforced concrete shell of hyperbolic shape (also called 'hyperbolic tower'). Natural flow of air occurs through the tower; hence it is called natural draft.

Factors responsible for creating natural draft

- A rise in temperature and humidity of air in the column reduces its density
- Wind velocity at the tower bottom

Fan is used to enhance the air flow rate in fan assisted natural draft tower. The typical diameter of tower is 150 m and capacity is 5, 00,000 gpm.

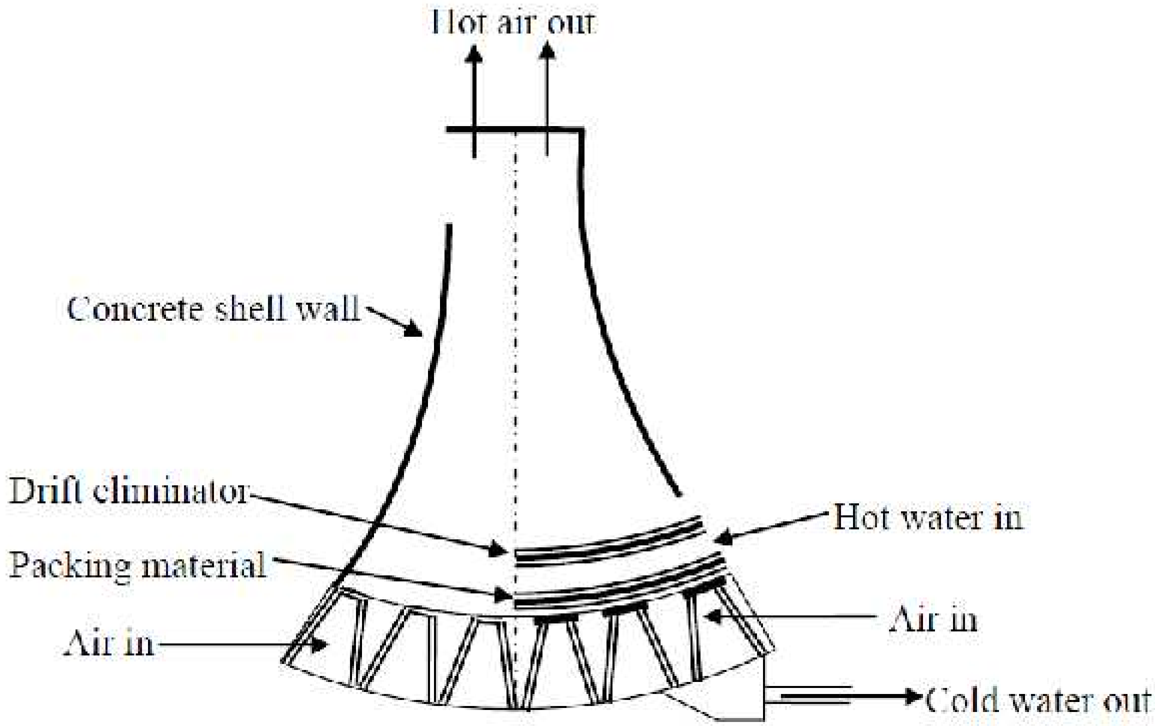


Figure 1.4 Natural Cooling tower

Advantages of hyperbolic shape

- More packing materials can be placed at the bottom
- The entering air gets smoothly directed towards the centre
- Greater structural strength and stability

Forced draft towers:

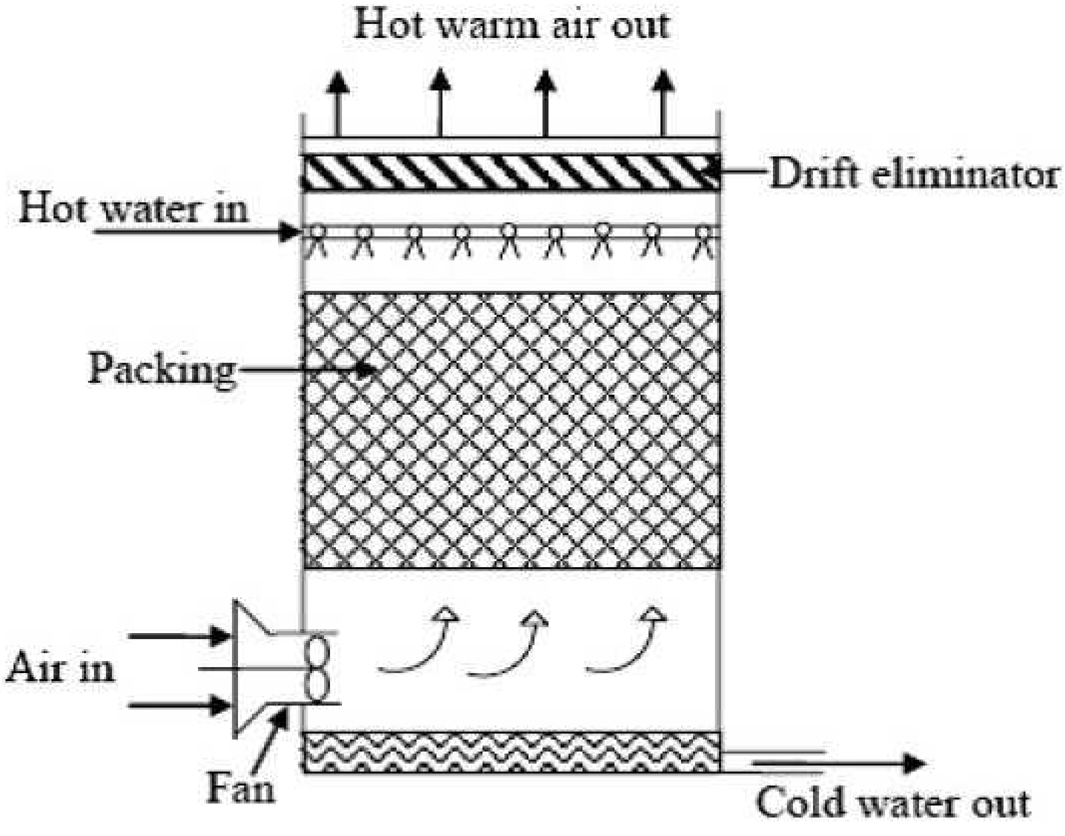
It can be seen from Figure below; that it has one or more fans located at the tower bottom to push air into tower.

Advantages:

- A part of the velocity head of air thrown by the blower is converted to pressure head on entering into the tower. It makes energy efficient than induced draft.
- Less susceptible to vibrations as fans are installed near the ground.

Disadvantages:

- Air flow through the packing may not be uniform
- Some of the warm and humid air may be recirculated back. Recirculation rate becomes low if the wind velocity is high. It is not popular except for small capacities.



Schematic of forced draft towers.

Figure 1.5 Natural Cooling tower

Induced draft towers:

One or more fans are installed at the top of the tower. Depending on the air inlet and flow pattern, induced draft towers are of two types, cross-flow and counter flow towers.

Major advantages of countercurrent induced draft cooling tower

- Relatively dry air contacts the coldest water at the bottom of the cooling tower
- Humid air is in contact with the warm water and hence maximum average driving force prevails for both heat and mass transfer.

Disadvantage of induced draft towers compared to forced draft towers

- It consumes more horse power. Cross-flow induced draft cooling tower requires less motor horse power than countercurrent induced draft cooling towers.

(B) Cross-current and counter-current

Cross-flow induced draft cooling tower supplies horizontal air flow along the packed height and requires less motor horse power than the counter-flow type. Additional cells may be added to raise the capacity.

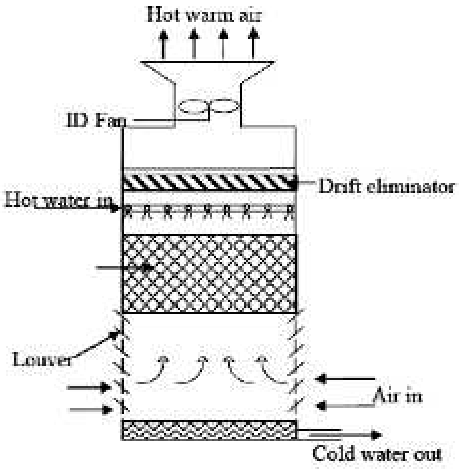
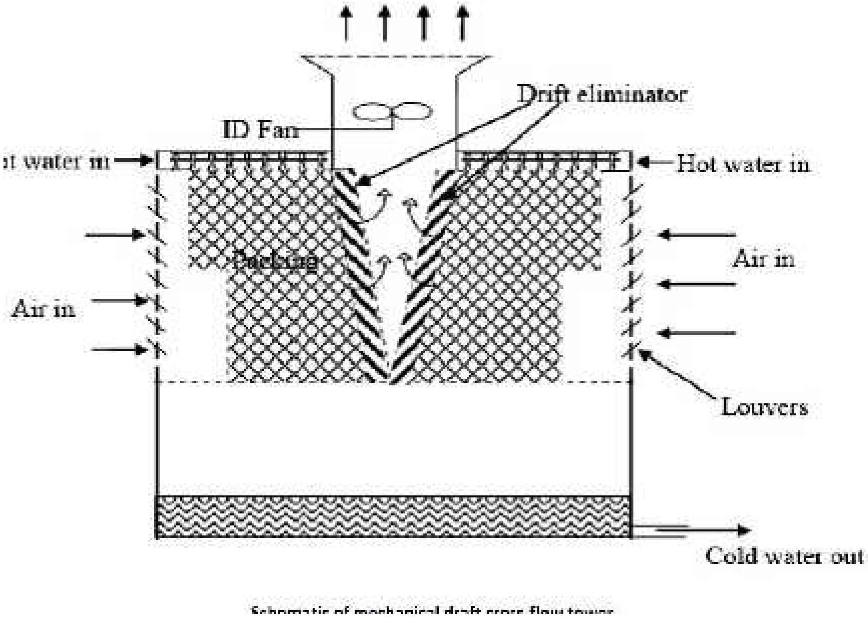


Figure 1.6 Cross flow Cooling tower



SPRAY POND:

A **spray pond** is a reservoir in which warmed water is cooled for reuse by evaporation of water discharged from nozzles in spray or mist form over the pond.

Figure 1.7 Spray pond

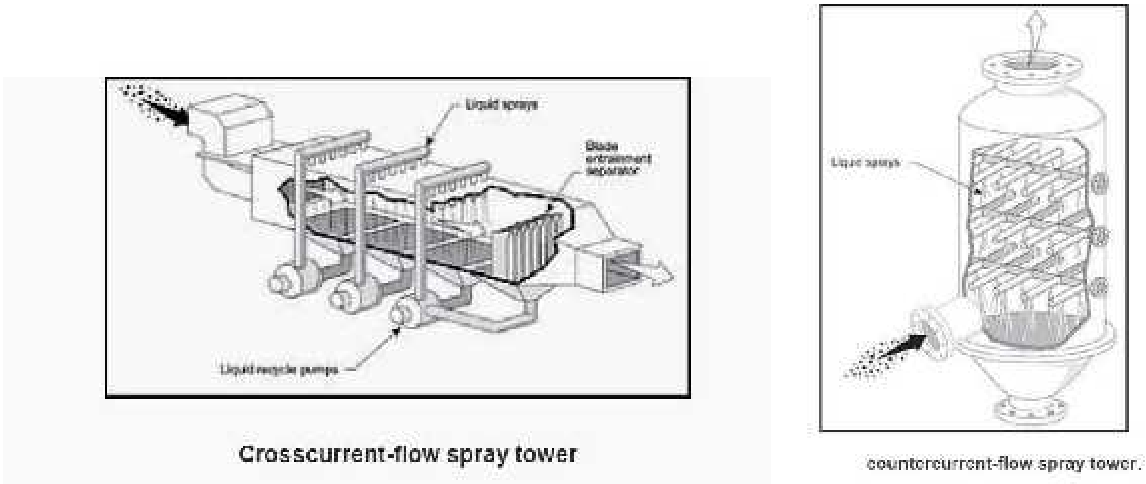
The warm water is sprayed into cooler air, cooling down as it reaches the water. The spray pond is the predecessor to the natural draft cooling tower, which is much more efficient and takes up less space but has a much higher construction cost. A spray pond requires between 25 to 50 times the area of a cooling tower. However, some spray ponds are still in use today.

SPRAY TOWER:

A **spray tower** (or **spray column** or **spray chamber**) is gas-liquid contactor used to achieve the mass and heat transfer between a continuous gas phase (that can contain dispersed solid particles) and a dispersed liquid phase. It consists of empty cylindrical vessel made of steel or plastic and nozzles that spray liquid into the vessel. The inlet gas stream usually enters the bottom of the tower and moves upward, while liquid is sprayed downward from one or more levels. This flow of inlet gas and liquid in the opposite direction is called countercurrent flow. This type of technology can be used for example as a wet scrubber for air pollution control. Countercurrent flow exposes the outlet gas with the lowest pollutant concentration to the freshest scrubbing liquid. Many nozzles are placed across the tower at different heights to spray all of the gas as it moves up through the tower. The reason for using many nozzles is to maximize the number of fine droplets impacting the pollutant particles and to provide a large surface area for absorbing gas.

Theoretically, the smaller the droplets formed, the higher the collection efficiency achieved for both gaseous and particulate pollutants. However, the liquid droplets must be large enough to not be carried out of the scrubber by the scrubbed outlet gas stream. Therefore, spray towers use nozzles to produce droplets that are usually 500–1000 µm in diameter. Although small in size, these droplets are large compared to those created in the venturi scrubbers that are 10–50 µm in size. The gas velocity is kept low, from 0.3 to 1.2 m/s (1–4 ft/s) to prevent excess droplets from being carried out of the tower.

In order to maintain low gas velocities, spray towers must be larger than other scrubbers that handle similar gas stream flow rates. Another problem occurring in spray towers is that after the droplets fall short distances, they tend to agglomerate or hit the walls of the tower. Consequently, the total liquid surface area for contact is reduced, reducing the collection efficiency of the scrubber.



Crosscurrent-flow spray tower

countercurrent-flow spray tower.

Figure 1.8 Spray tower

In cocurrent-flow spray towers, the inlet gas and liquid flow in the same direction. Because the gas stream does not "push" against the liquid sprays, the gas velocities through the vessels are higher than in countercurrent-flow spray towers. Consequently, cocurrent-flow spray towers are smaller than countercurrent-flow spray towers treating the same amount of exhaust flow. In crosscurrent-flow spray towers, also called horizontal-spray scrubbers, the gas and liquid flow in directions perpendicular to each other.

1. Definition of humidity. The humidity H of an air–water vapor mixture is defined as the kg of water vapor contained in 1 kg of dry air. The humidity so defined depends only on the partial pressure p_A of water vapor in the air and on the total pressure P (assumed throughout this chapter to be 101.325 kPa, 1.0 atm abs, or 760 mm Hg). Using the molecular weight of water (A) as 18.02 and of air as 28.97, the humdiity H in kg H_2O/kg dry air or in English units as lb H_2O/lb dry air is as follows:

$$H \frac{\text{kg } H_2O}{\text{kg dry air}} = \frac{p_A}{P - p_A} \frac{\text{kg mol } H_2O}{\text{kg mol air}} \times \frac{18.02 \text{ kg } H_2O}{\text{kg mol } H_2O} \times \frac{1}{28.97 \text{ kg air/kg mol air}}$$

Saturated air is air in which the water vapor is in equilibrium with liquid water at the given conditions of pressure and temperature. In this mixture the partial pressure of the water vapor in the air–water mixture is equal to the vapor pressure p_{AS} of pure water at the given temperature. Hence, the saturation humidity H_S is

$$H_S - \frac{18.02}{28.97} \frac{p_{AS}}{P - p_{AS}}$$

2. Percentage humidity. The percentage humidity H_P is defined as 100 times the actual humidity H of the air divided by the humidity H_S if the air were saturated at the same temperature and pressure.

$$H_P = 100 \frac{H}{H_S}$$

Dew point: Dew point is a temperature at which a vapor-gas mixture must be cooled (at constant humidity) to become saturated. The dew point of a saturated gas equals the gas temperature. If a vapor-gas mixture is gradually cooled at a constant pressure, the temperature at which it just becomes saturated is also called its dew point.

Humid volume: The humid volume, is defined as the volume of unit mass of dry air with accompanying water vapor at a given temperature and pressure.

$$\upsilon_H \text{ m}^3\text{/kg dry air} = \frac{22.41}{273} T \text{ K} \left(\frac{1}{28.97} + \frac{1}{18.02} H \right)$$
$$= (2.83 \times 10^{-3} + 4.56 \times 10^{-3} H) T \text{ K}$$

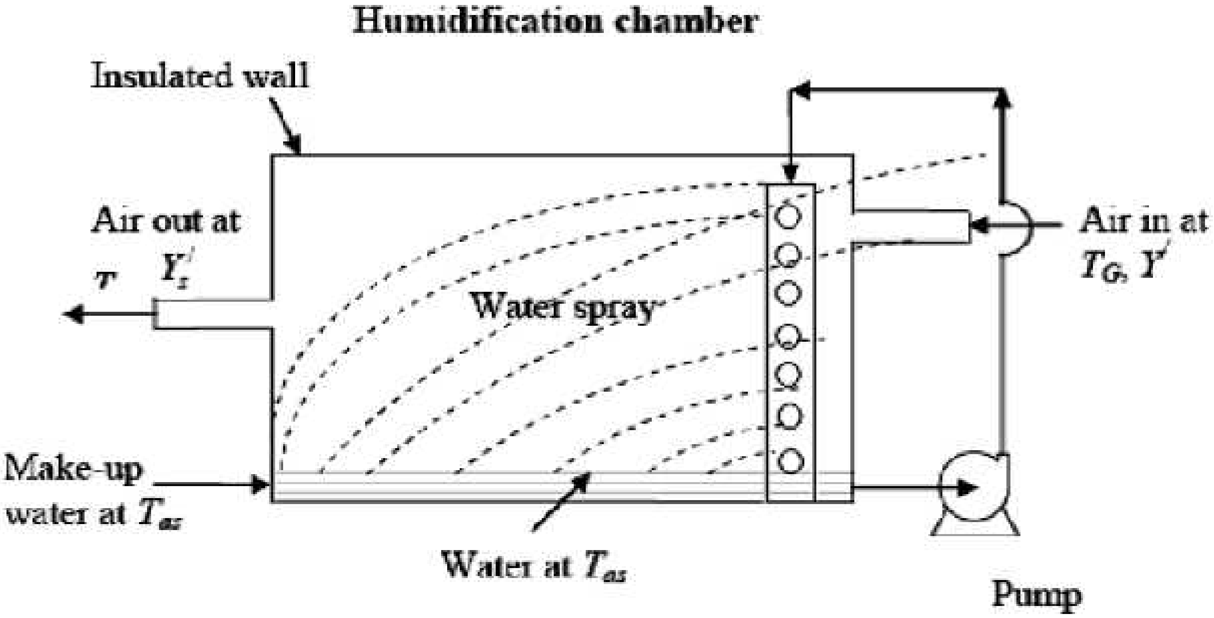
Humid heat: The humid heat, is the heat energy required to raise the temperature of unit mass of dry air with the accompanying water vapor by one (1) degree.

$$c_S \text{ kJ/kg dry air} \cdot \text{K} = 1.005 + 1.88H$$

Enthalpy: The enthalpy of a vapor-gas mixture is the sum of the relative enthalpies of gas and vapor content.

$$H_y \text{ kJ/kg dry air} = c_S(T - T_0) + H\lambda_0 \Rightarrow (1.005 + 1.88H)(T - T_0\,^\circ\text{C}) + H\lambda_0$$

Adiabatic Saturation temperature



Schematic representation of adiabatic saturation of air

Figure 19 AST

The air stream attains thermal equilibrium with water at temperature *Tas* and also gets saturated with water vapor at that temperature before it leaves. A small quantity of water at the temperature *Tas* is fed to the humidification chamber continuously in order to compensate for the vaporization loss of water. The chamber operates adiabatically, wall is well-insulated. The temperature *Tas* attained by air (same as water) is called "***adiabatic saturation temperature, Tas***".

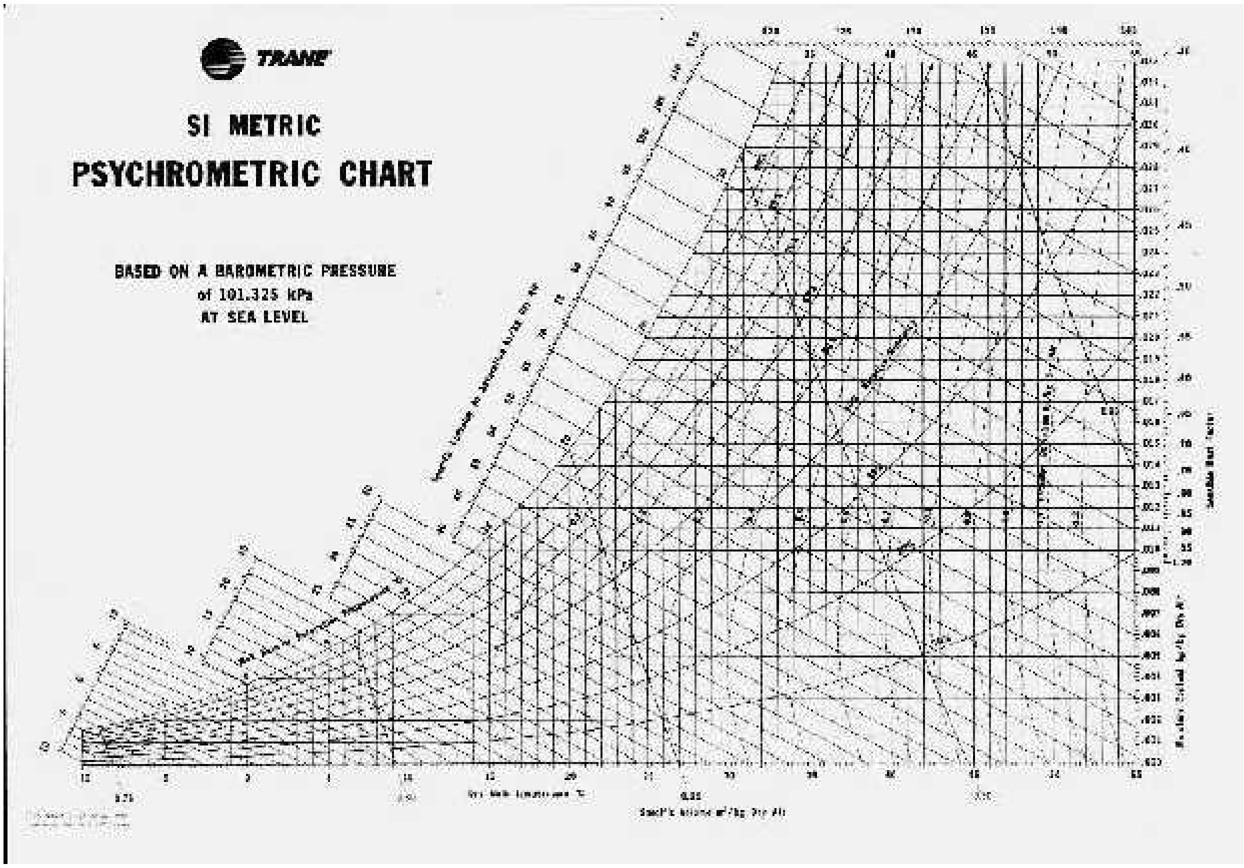


Figure 1.10 Psychrometric chart

Problem 1

The air in a room is at 26.7°C (80°F) and a pressure of 101.325 kPa and contains water vapor with a partial pressure $p_A = 2.76$ kPa. Calculate the following.

(a) Humidity, H.
(b) Saturation humidity, H_S, and percentage humidity, H_P.
(c) Percentage relative humidity, H_R.

From the steam tables at 26.7°C, the vapor pressure of water is $p_{AS} = 3.50$ kPa (0.507 psia). Also, $p_A = 2.76$ kPa and $P = 101.3$ kPa (14.7 psia). For part (a), using Eq. (9.3-1),

$$H = \frac{18.02}{28.97}\frac{p_A}{P - p_A} = \frac{18.02(2.76)}{28.97(101.3 - 2.76)} = 0.01742 \text{ kg H}_2\text{O/kg air}$$

For part (b), using Eq. (9.3-2), the saturation humidity is

$$H_S = \frac{18.02}{28.97}\frac{p_{AS}}{P - p_{AS}} = \frac{18.02(3.50)}{28.97(101.3 - 3.50)} = 0.02226 \text{ kg H}_2\text{O/kg air}$$

The percentage humidity, from Eq. (9.3-3), is

$$H_P = 100\frac{H}{H_S} = \frac{100(0.01742)}{0.02226} = 78.3\%$$

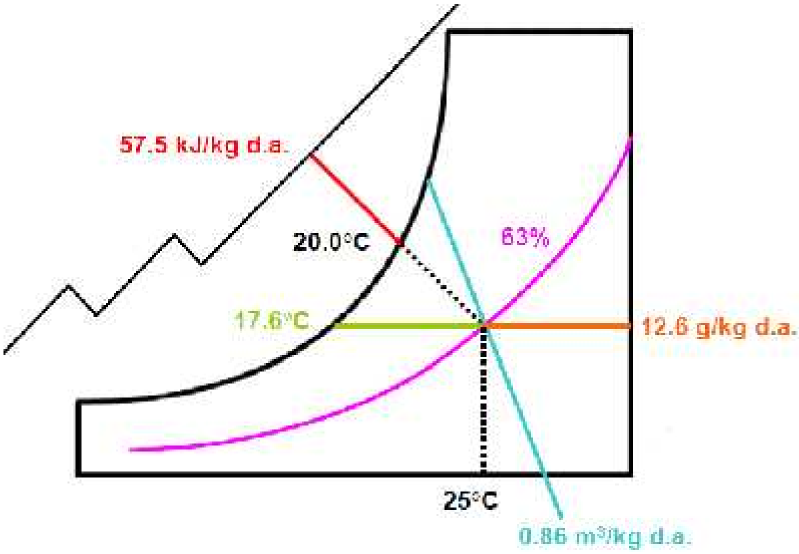
For part (c), from Eq. (9.3-4), the percentage relative humidity is

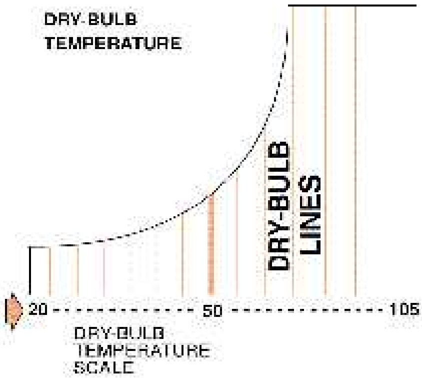
$$H_R = 100\frac{p_A}{p_{AS}} = \frac{100(2.76)}{3.50} = 78.9\%$$

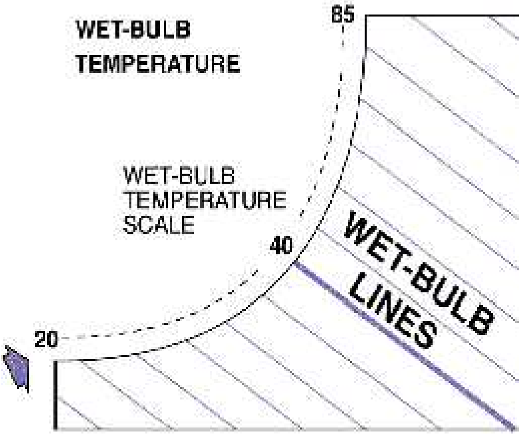
Problem 2

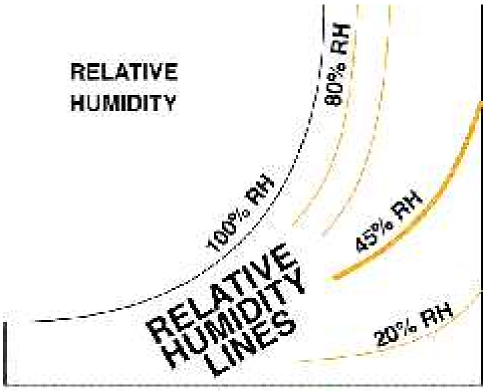
Given: DBT = 25°C WBT =20°C

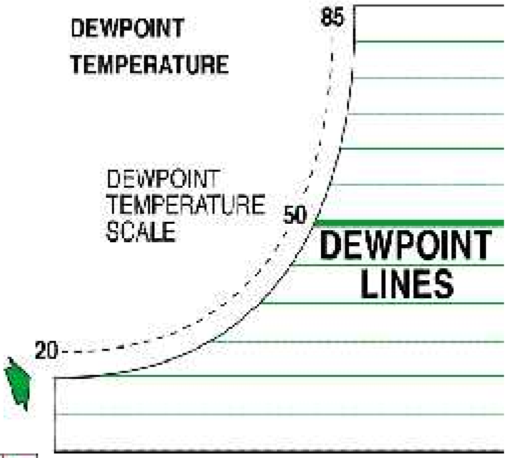
Required: (a) RH, (b) Tdp, (c) HR, (d) v, (e) h

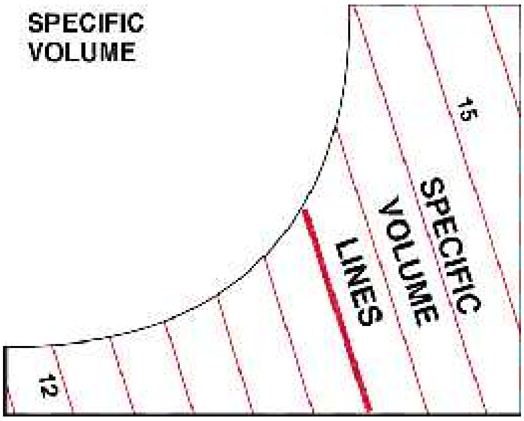



DRY-BULB TEMPERATURE
DRY-BULB LINES
20
50
105
DRY-BULB TEMPERATURE SCALE


WET-BULB TEMPERATURE
85
WET-BULB TEMPERATURE SCALE
40
WET-BULB LINES
20


RELATIVE HUMIDITY
80% RH
100% RH
45% RH
RELATIVE HUMIDITY LINES
20% RH


DEWPOINT TEMPERATURE
85
DEWPOINT TEMPERATURE SCALE
50
DEWPOINT LINES
20


SPECIFIC VOLUME
15
SPECIFIC VOLUME LINES
12

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UNIT – II – MASS TRANSFER - II – SCH1302

Introduction

The term drying refers generally to the removal of moisture from a substance. It is one of the oldest, most commonly used and most energy consuming unit operation in the process industries. Drying is often necessary in various industrial operations particularly in chemical process industries to remove moisture from a wet solid, a solution or a gas to make it dry and choice of drying medium is depends on the chemical nature of the materials. Three basic methods of drying are used today

1) Sun drying, a traditional method in which materials dry naturally in the sun,
2) Hot air drying in which materials are exposed to a blast of hot air and
3) freeze drying, in which frozen materials are placed in a vacuum chamber to draw out the water.

The fundamental nature of all drying process is the removal of volatile substances (mainly moisture) from mixture to yield a solid product. In general drying is accomplished by thermal techniques and thus involves the application of heat, most commonly by convection from current of air. Throughout the convective drying of solid materials, two processes occur simultaneously namely, transfer of energy from the local environment in the dryer and transfer of moisture from within the solid. Therefore this unit operation may be considered as simultaneous heat and mass transfer operation. Drying processes and equipment may be categorized according to several criteria, including the nature of material and the method of heat supply and the method of operation. For example in the sugar industry washed and centrifuged sugar crystals are dried to get finished product for packing. Drying is an important operation in food processing. Milk is dried in a spray chamber to produce milk powder. All the above examples indicate that wet material loses moisture in direct contact with hot air/gas. The hot air/gas supplies the energy required for drying and also carries away the moisture released by the solid. For heat sensitive materials much of the resistance to drying resides within the material. Unduly high heat and mass transfer rates applied at the surface only result in overheating or over drying of the surface layer resulting in quality problems without major increase in the drying kinetics. The rate of migration of the moisture from within the solid to the evaporation front often controls the overall drying rate. Therefore, drying may be defined as an operation in which the liquid, generally water, present in a wet solid is removed by vaporization to get a relatively liquid free solid product. Drying of a solid does not demand or ensure complete removal of the moisture. Sometimes it is desirable to retain a little moisture in the solid after drying. Dryer and drying process selection for a specific operation is a complex problem, and many factors have to be taken into account. Though, the overall selection and design of a drying system for a particular material is dictated by the desire to achieve a favourable combination of a product quality and process economics. In general, with respect to the rate and total drying time, dryer performance is dependent on the factors such as air characteristics, product characteristics, and equipment characteristics. But despite the many commercially available drying techniques at present most dehydrated products (i.e. fruits and vegetables) are still produced by the method of hot air drying. There are other water/liquid removal processes such as filtration, settling, centrifugation, supercritical extraction of water from gels etc. In all these operations liquid is removed by mechanical means but a considerable amount of liquid is still retained in the solid. This residual liquid can be removed by drying. One such example is the production of condensed milk involves evaporation, but the production of milk powder involves drying. The phase change and production of a solid phase as end product are essential features of the drying process. Drying is an essential operation in chemical, agricultural, biotechnology, food, polymer, pharmaceutical, pulp and paper, mineral processing, and wood processing industries.

Drying does not mean only removal of the moisture but during the process, physical structure as well as the appearance has to be preserved. Drying is basically governed by the principles of transport of heat and mass. When a moist solid is heated to an appropriate temperature, moisture vaporizes at or near the solid surface and the heat required for evaporating moisture from the drying product is supplied by the external drying medium, usually air or a hot gas.

Drying is a diffusional process in which the transfer of moisture to the surrounding medium takes place by the evaporation of surface moisture, as soon as some of the surface moisture vaporizes; more moisture is transported from interior of the solid to its surface. This transport of moisture within a solid takes place by a variety of mechanisms depending upon the nature and type of the solid and its state of aggregation.

Different types of solids may have to be handled for drying crystalline, granular, beads, powders, sheets, slabs, filter-cakes etc. The mechanism of moisture transport in different solids may be broadly classified into (i) transport by liquid or vapour diffusion (ii) capillary section, and (iii) pressure induced transport. The mechanism that dominates depends on the nature of the solid, its pore structure and the rate of drying.

Different mechanisms may come into play and dominate at different stages of drying of the same material. The following term are commonly used in designing of drying systems.

Moisture content of a substance which exerts as equilibrium vapour pressure less than of the pure liquid at the same temperature is referred to as ***bound moisture.***

Moisture content of the solid which exerts an equilibrium vapour pressure equal to that of pure liquid at the given temperature is the ***unbound moisture.***

The moisture content of solid in excess of the equilibrium moisture content is referred as free *moisture.*

During drying, only free moisture can be evaporated. The free moisture content of a solid depends upon the vapour concentration in the gas.

The moisture contents of solid when it is in equilibrium with given partial pressure of vapour in gas phase is called as ***equilibrium moisture content***. Similarly, the moisture content at which the constant rate drying period ends and the falling rate drying period starts is called ***critical moisture content***. During the ***constant rate drying period***, the moisture evaporated per unit time per unit area of drying surface remains constant and in ***falling rate drying period*** the amount of moisture evaporated per unit time per unit area of drying surface continuously decreases.

CLASSIFICATION

Drying equipment is classified in different ways, according to following design and operating features.It can be classified based on mode of operation such as batch or continuous, In case of batch dryer the material is loaded in the drying equipment and drying proceeds for a given period of time, whereas, in case of continuous mode the material is continuously added to the dryer and dried material continuously removed. In some cases vacuum may be used to reduce the drying temperature. Some dryers can handle almost any kind of material, whereas others are severely limited in the style of feed they can accept. Drying processes can also be categorized according to the physical state of the feed such as wet solid, liquid, and slurry. Type of heating system i.e. conduction, convection, radiation is another way of categorizing the drying process. Heat may be supplied by direct contact with hot air at atmospheric pressure, and the water vaporized is removed by the air flowing. Heat may also be supplied indirectly through the wall of the dryer from a hot gas flowing outside the wall or by radiation. Dryers exposing the solids to a hot surface with which the solid is in contact are called adiabatic or direct dryers, while when heat is transferred from an external medium it is known as non-adiabatic or indirect dryers. Dryers heated by dielectric, radiant or microwave energy are also non adiabatic. Some units combine adiabatic and non adiabatic drying; they are known as direct-indirect dryers.

Tray Drier

Schematic of a typical batch dryer is shown in figure 4.1. Tray dryers usually operate in batch mode, use racks to hold product and circulate air over the material. It consists of a rectangular chamber of sheet metal containing trucks that support racks. Each rack carries a number of trays that are loaded with the material to be dried. Hot air flows through the tunnel over the racks. Sometimes fans are used to on the tunnel wall to blow hot air across the trays. *Even b*affles are used to distribute the air uniformly over the stack of trays. Some moist air is continuously vented through exhaust duct; makeup fresh air enters through the inlet. The racks with the dried product are taken to a tray-dumping station.These types of dryers are useful when the production rate is small. They are used to dry wide range of materials, but have high labor requirement for loading and unloading the materials, and are expensive to operate. They find most frequent application for drying valuable products. Drying operation in case of such dryers is slow and requires several hours to complete drying of one batch. With indirect heating often the dryers may be operated under vaccum. The trays may rest on hollow plates supplied with steam or hot water or may themselves contain spaces for a heating fluid. Vapour from the solid may be removed by an ejector or vacuum pump.

Pan Dryer

The atmospheric pan drier has a jacketed round pan in which a stirrer or mill revolves slowly, driven from below. The slow moving stirrer exposes fresh surfaces and thereby raises the rate of evaporation and, hence, of drying. The pan drier is a batch machine and is limited to small batches. Pan driers may be used first to evaporate a solution to its crystallizing concentration and then can function as a crystallizer by sending cold water instead of steam into the jacket. The effect of the stirrer during crystallization prevents the growth of large crystals and promotes formation of small, uniform crystals. The mother liquor is then drained off and the crystals dried in the same apparatus.

Agitated Vacuum Dryer

The agitated vacuum dryer is one of the most versatile in the range and is similar in principle to a pan dryer. The dryer essentially consists of a jacketed cylindrical vessel arranged for hot water, steam or a suitable thermal fluid flow through the jacket for heating. Doors are provided on the shell, at the top for loading the feed material and at the bottom for discharging. The dryers are available in variety of sizes. The entire drying chamber is well machined to insure small clearance with the agitator blade. Thus ensures proper shuffling of the material and avoids localized overheating. Due to the agitation of the product in the agitated vacuum dryer the drying time is substantially reduced.

Rotary Dryer

The rotary drier is basically a cylinder, inclined slightly to the horizontal, which may be rotated, or the shell may be stationary, and an agitator inside may revolve slowly. In either case, the wet material is fed in at the upper end, and the rotation, or agitation, advances the material progressively to the lower end, where it is discharged. Figure shows a direct heat rotary drier. Typical dimensions for a unit like this are 9 ft diameter and 45 ft length. In direct-heat revolving rotary driers, hot air or a mixture of flue gases and air travels through the cylinder. The feed rate, the speed of rotation or agitation, the volume of heated air or gases, and their temperature are so regulated that the solid is dried just before discharge. The shell fits loosely into a stationary housing at each end. The material is brought to a chute that runs through the housing; the latter also carries the exhaust pipe. The revolving shell runs on two circular tracks and is turned by a girth gear that meshes with a driven pinion. The inclination is one in sixteen for high capacities and one in thirty for low ones. As the shell revolves, the solid is carried upward one-fourth of the circumference; it then rolls back to a lower level, exposing fresh surfaces to the action of the heat as it does so. Simple rotary driers serve well enough when fuel is cheap. The efficiency is greatly improved by placing longitudinal plates 3 or 4 in. wide on the inside of the cylinder. These are called lifting flights. These carry part of the solid half-way around the circumference and drop it through the whole of a diameter in the central part of the cylinder where the air is hottest and least laden with moisture.

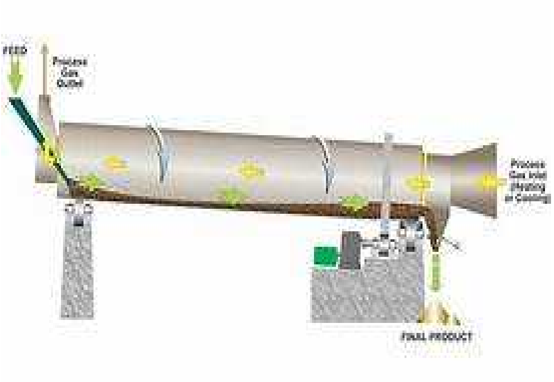


Figure 2.1 Rotary Drier

Applications :

Pharmaceutical industry

Food industry

Biochemical industry

Chemical

Petrochemical

Drum Dryer

In drum dryers a liquid containing dissolved solids or slurry carrying suspended solids forms a thin layer on the outside surface of a large rotating drum. For a single drum unit thickness of the film can be controlled by an adjustable scraping blade. In case of a double drum unit thickness can be controlled by the gap between the drums. A gas, normally air may be blown over the surface for rapid removal of moisture. The rotation of the drum adjusted so that all of the liquid is fully vaporized and a dried deposit can be scrapped off with the help of flexible or adjustable knife. This type of dryer mainly handles the materials that are too thick for a spray dryer and too thin for a rotary dryer. The solid collects on an apron in front of the knife and rolls to a container or to a screw conveyor. The operation of the drum drier is continuous. The drum is rotated continuously by a gear driven by a pinion that receives its motion through a belt, a chain, or a reduction gear from. The speed of the drum may be regulated by a variable-speed drive to adopt the speed to any slight variation in the feed quality. The speed of the drum regulated depending upon the nature of materials (i.e wet or dry), if the product material is wet/dry quite a distance before the knife is reached, the speed should be decreased/increased. The design of the components is similar to that of drum filter. The knife may be held just against the surface. It may be brought closer by turning the adjusting wheels. The knife supports may be turned through part of a circle so that the angle of the blade of the knife relative to the drum surface may be selected for the greatest shearing effect.

Flash Dryer

The flash driers also called pneumatic dryers, are similar in their operating principle to spray dryer. The materials that are to be dried (i.e. solid or semisolid) are dispersed in finely divided form in an upward flowing stream of heated air. These types of dryer are mainly used for drying of heat sensitive or easily oxidizable materials. The wet materials that are to dried can be passed into a high- temperature air stream that carries it to a hammer mill or high-speed agitator where the exposed surface is increased. The drying rate is very high for these dryers (hence the term *flash dryers*), but the solid temperature does not rise much because of the short residence time. A flash dryer is not suitable for particles which are large in size or heavy particles. The special advantage of this type of dryer is that no separate arrangement is required for transporting the dried product. The fine particles leave the mill through a small duct to maintain the carrying velocities (drying gas) and reach a cyclone separator. A solid particle takes few seconds to pass from the point of entry into the air stream to the collector.

Applications :

Food industry
Chemical industry
Mineral industry
Polymer industry

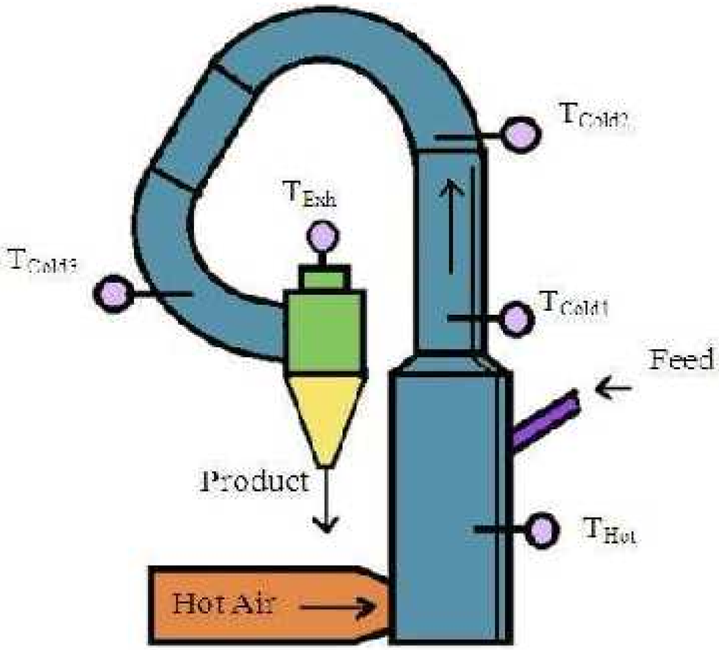


Figure 2.2 Flash Drier

Fluidised Bed Dryer

Fluidized bed dryer consist of a steel shell of cylindrical or rectangular cross section. A grid is provided in the column over which the wet material is rests. In this type of dryer, the drying gas is passed through the bed of solids at a velocity sufficient to keep the bed in a fluidized state. Mixing and heat transfer are very rapid in this type of dryers. The dryer can be operated in batch or continuous mode. Fluidized bed dryer are suitable for granular and crystalline materials. If fine particles are present, either from the feed or from particle breakage in the fluidized bed, there may be considerable solid carryover with the exit gas and bag filters.

The main advantage of this type of dryer are: rapid and uniform heat transfer, short drying time, good control of the drying conditions. In case of rectangular fluid-bed dryers separate fluidized compartments are provided through which the solids move in sequence from inlet to outlet. These are known as *plug flow dryers;* residence time is almost the same for all particles in the compartments. But the drying conditions can be changed from one compartment to another, and often the last compartment is fluidized with cold gas to cool the solid before discharge.

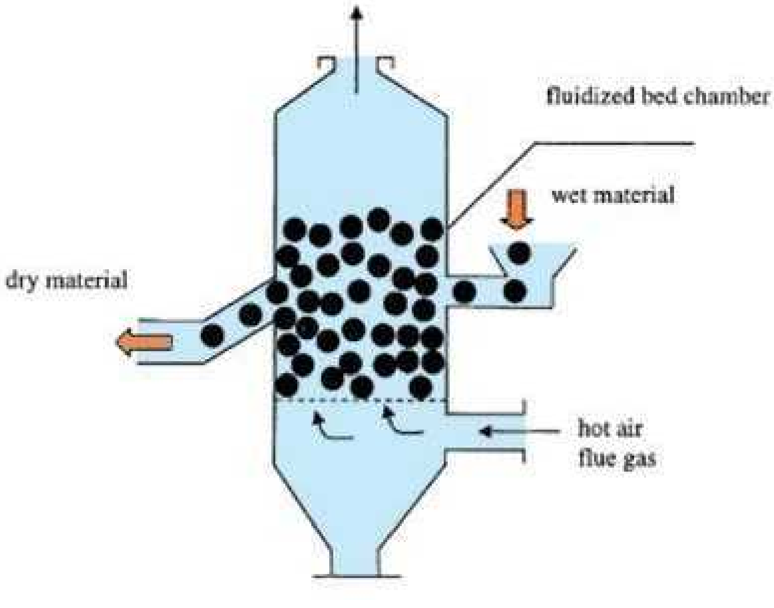


Figure 2.3: Continuous fluidized bed dryer

Screw Conveyer Drier

Screen conveyor dryer is also called a direct heat continuous type dryer. The solid to be dried are fed on to endless, perforated, conveyor belt through which hot air is forced. The belt is housed in a long rectangular drying chamber or tunnel (figure 4.6). The chamber is divided into series of separate sections, each with its own fan and air heater. Air may be recirculated through, and vented from each section separately or passed from one section to another counter current to the solid movement. The solid is carried through the tunnel and discharged at the opposite end. In order to prevent the higher flow rate of hot air through thinner regions of the bed a uniform feeding rate and distribution of the material over the conveyor is necessary. Coarse granular, flakey, or fibers materials can be dried by through circulation without any pretreatment and without loss of material through the screen. High drying rate can be achieved with good product quality control. Thermal efficiency of this type of dryer is high and with steam heating, the steam consumption for heating the drying gas can be as low as 1.5 kg per kg of water evaporated. Only disadvantage of this type of dryer are high initial cost and high maintenance cost due to the mechanical belt.

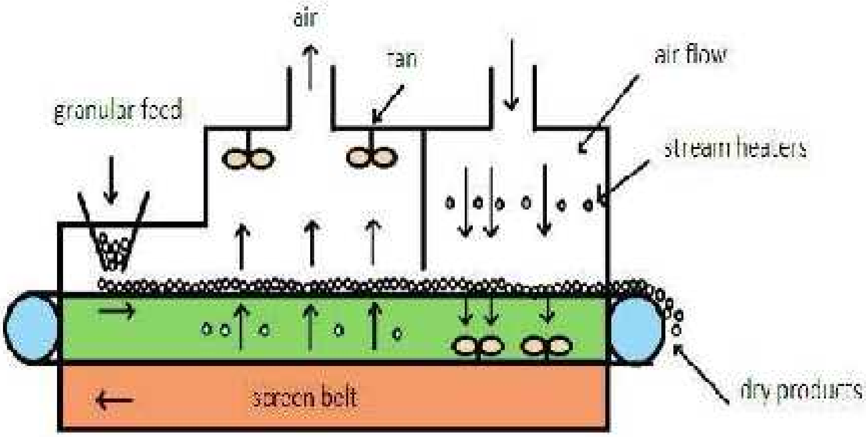


Figure 2.4: Screen conveyor dryer

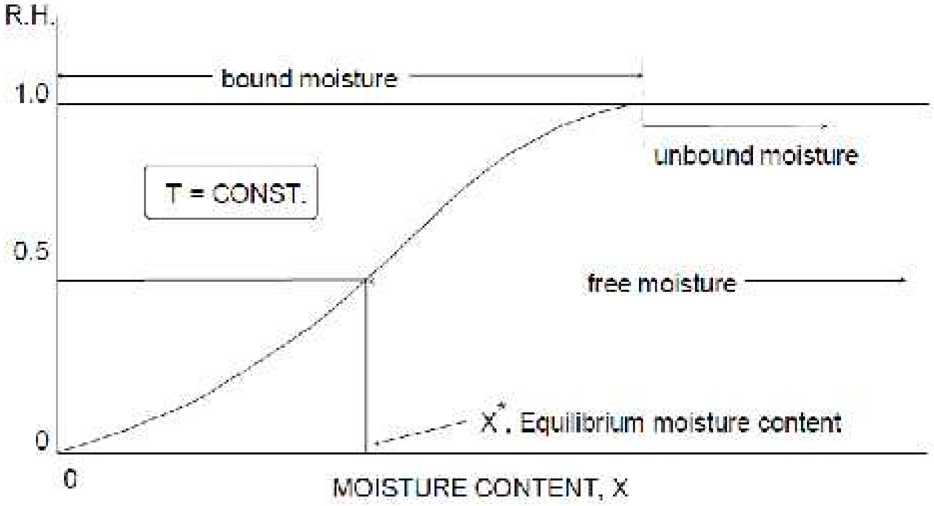


Figure 2.5: Drying curve

Calculation of Drying time:

- $\therefore \int_0^t dt = - \int_{X_i}^{X_f} \frac{S_S}{A} \frac{dX}{N} \rightarrow t = - \frac{S_S}{A} \int_{X_i}^{X_f} \frac{dX}{N} = \frac{S_S}{A} \int_{X_f}^{X_i} \frac{dX}{N}$
- $t = \frac{S_S}{A} \int_{X_f}^{X_c} \frac{dX}{N} + \frac{S_S}{A} \int_{X_c}^{X_i} \frac{dX}{N}$
- * Drying rate remains constant at N_C till X_C from X_i.
- 1. **Constant rate period:**
- $t_C = \frac{S_S}{A} \int_{X_c}^{X_i} \frac{dX}{N_c} = \frac{s_s(X_i - X_c)}{AN_C}$
- 2. **First falling rate period:**
- integral can be evaluated graphically ($\frac{1}{N}$ vs X)
- **Special case 1:**
- $N = mX_C + b$
- $N_C = mX_c + b$
- $N_f = mX_f + b$
- $m = \frac{N_C - N_f}{X_C - X_f}$
- $t_f = \frac{S_S}{A} \int_{X_f}^{X_c} \frac{dX}{N}$
- $- \frac{S_S}{A} \int_{X_f}^{X_c} \frac{dX}{mX+b} = \frac{S_S}{Am} ln(\frac{mX_C+b}{mX_f+b})$
- $= \frac{S_S}{A} \frac{X_C - X_f}{N_C - N_f} ln(\frac{N_C}{N_f})$
- Drying rate at $N = 0$ at equilibrium moisture, $X = X^*$
- $\therefore 0 = m X^* + b \rightarrow b = - m X^*$
- $\therefore \frac{N_C}{N_f} = \frac{mX_C - mX^*}{mX_f - mX^*} = \frac{X_C - X^*}{X_f - X^*}$
- $t_f = \frac{S_S}{A} \frac{X_C - X_f}{N_C - N_f} ln(\frac{X_C - X^*}{X_f - X^*})$

- $t_f = \frac{S_S}{A}\frac{X_C - X^*}{N_C} ln(\frac{X_C - X^*}{X_f - X^*})$

- $t = t_C + t_f = \frac{S_s(X_I - X_C)}{AN_C} + \frac{S_S}{A}\frac{(X_C - X^*)}{N_C} ln(\frac{X_C - X^*}{X_f - X^*})$

Problems for Practice:

1. During the batch drying test of a wet slab of material 0.35 m^2 and 7 mm thick, the falling rate 'N' was expressed as 0.95 (X – 0.01) where 'N' is the drying rate in kg/m^2.s and X is the moisture content in kg moisture/kg dry solid. The constant drying rate was 0.38 kg/m^2.s and slab was dried from one side only with edges sealed. Density of the dry material is 1200 kg/m^3. It is desired to reduce the moisture content from 35% and 5% on wet basis. What is the time needed for drying?
2. A wet solid is dried from 25% to 10% moisture under constant drying condition in 15000 sec. If the critical moisture content and equilibrium moisture content are 15% and 5% respectively, how long will it take to dry the solid from 32% to 8%?
3. A time of 5 hr was taken to dry a material from an initial moisture of 30% to a final moisture of 7%. Critical and equilibrium moisture are found to be 15% and 2% respectively. How much further time would be required to dry the material to a final moisture of 4%. All moisture contents are on wet basis.

References:

1. Mass Transfer Operations, Treybal R.E..3rd edition. Mcgraw Hill, 1981.
2. Principles of Mass Transfer and Separation Processes: B. K. Dutta, Prentice-Hall of India Private Ltd., 2007.
3. Separations in Chemical Engineering: Equilibrium Staged Separations: P. C. Wankat, Prentice Hall, NJ, US, 1988.

SCHOOL OF BIO AND CHEMICAL ENGINEERING

DEPARTMENT OF CHEMICAL ENGINEERING

UNIT – III – Mass Transfer - II – SCH1302

Introduction

Crystallization

It is a method of formation of solid particles within a homogeneous phase (vapor phase, liquid melt). Most of the crystalline solid substances are prepared either by solidification from liquid melt or by precipitation in chemical reaction. This unit operation involves knowledge of mass transfer mechanism, diffusion theory, heat transfer mechanism, hydrodynamics, and solubility curves, design of equipment and selection of equipments on the basis of principles of crystallization. Each manufacturing plant requires many unique features and these must be evaluated on an individual basis in order to achieve optimum result. It is true that mechanical design of the crystallizer has a significant influence on the nucleation rate due to contact nucleation with the parts of pump, impellor etc. when suspended in a supersaturated solution. This phenomenon yields varying rates of nucleation in scale up and differences in nucleation rate when the same piece of equipment is used to crystallize different materials. All these must be taken care of in order to achieve super saturation of liquid. Crystallization of a material from solution is important industrially because of the markets‟‟ demand due to some advantages of its crystalline solid forms. One of the important advantages of crystallization compared with other means of separation technique is that it produces highly „pure products with good appearance‟‟ from impure solutions. Energy input is low and products with high bulk density are obtained. Drying requirement is minimum due to low moisture content. Commercial crystallization processes manufacture crystalline products from the standpoint of special demands based on definite crystal shape, size and size range of particles (crystal size distribution, CSD). Generally, narrow size range is desired. Of course, requirements of scientific works are solely restricted to yield of crystal as high as possible and its purity. Crystalline products have high demand in the market due to some reasons and some advantages over liquid form products.

- Less degradation, less contamination, more stability than solution.
- More sales appeal than liquid.
- Purity of crystalline product is more than that of a solution.
- More economical than liquid form.

Crystallization process may be analyzed and judged from the standpoint of following aspects in order to produce good products and avoid problems encountered both within the industry and outside the industry.

1. Yield of a given product
2. Quality of product in terms of size, shape and purity of crystals
3. Uniformity in size and CSD
4. Rate of production of the desired crystals.
5. Energy requirements for cooling and evaporation.

High yield and rate of production of a crystalline product at acceptable cost leads to its economical viability. But, purity of a crystalline material as medicinal compound is mandatory requirement in Pharmaceutical industry. So, purity is the first and foremost demand of the purchaser in Pharmaceutical sector. Size, uniformity in size, crystal size distribution and crystal geometry are characteristics of particles that control many physicochemical factors in making of formulation drugs‟, stability and pharmacological factors.

Crystal geometry

A crystal is the most highly organized type of non-living matter. Molecules, atoms and ions are arranged in a crystal in orderly repetitive arrays called crystal lattices and it appears as polyhedrons having sharp corners and flat sides or faces.. The crystal systems are a grouping of crystal structures according to the axial systems used to describe their lattice. Each crystal system consists of a set of three axes in a particular geometric arrangement. There are seven unique crystal systems. The cubic / isometric system has the symmetry of a cube, that is the three axes are mutually perpendicular and of equal length. The other six systems in order to decreasing symmetry are as follows:

Hexagonal: Three equal axes are in one plane at 60^o to each other, one is at right angles to this plane but not necessarily of the same length as the others. Cross section is hexagonal.

Tetragonal: Three axes are at right angles, one is longer than the other two.

Orthorhombic: It is like tetragonal crystals except not square in cross–section (when viewing the crystal on end), forming rhombic prisms or dipyramids (two pyramids stuck together).

Trigonal: There is single 3–fold axis of rotation instead of the 6–fold axis of hexagonal division

Monoclinic: Two axes are at right angles in one plane and a third axis at some odd angle to this plane like skewed tetragonal crystals often forming prisms and double pyramids.

Triclinic: Three axes are at odd angles to each other; usually it is not symmetrical from one side to the other, which can lead to some fairly strange shapes. A crystalline substance may have the ability to form more than one form, called polymorphism.

Crystal size and shape factors

Shape factor or sphericity, φs is used to calculate the ratio of the total surface (SP) of a crystal to the crystal volume. For granular materials range of φs is from 0.6 to 0.95. The ratio Sp/Vp is very important parameter in any surface related phenomenon. Product containing smaller crystals is more soluble than that of bigger crystals since surface area per unit volume (Sp/Vp) is greater is smaller particles.

Uniformity in size or crystals distribution

Particles of uniform size in a product are desirable for the convenience of selection of filter, washing, uniform time of dissolution and good appearance of product. Besides these, caking tendency of crystals during its storage period can be prevented since number of points of contact between crystalline particles is significantly less in uniform crystals. Actually a product batch contains various fractions of particles of different particle sizes. Wide range of particle sizes creates problem in many practical situations. So, crystal size range is made narrow by controlling some process parameters like

- adding seed crystals with desired size range.
- avoiding contact nucleation as far as possible

- removing a fraction of small size /finer particles
- dissolving smaller crystals, nuclei either by heating or adding some solvent
- increasing residence time for larger particles.

Yield

In crystallization processes, it is often needed to calculate the theoretical yield so that actual yield can be compared with it. Crystals are allowed to grow for sufficient period to reach targeted growth. Finally, mother liquor sheds its super saturation and attains equilibrium at final temperature. Now, solid crystals are separated from mother liquor and further processed. The yield can be predicted from the difference of concentration of feed solution and solubility of mother liquor (saturated). Predicted yield may not match actual yield due to slow growth longer time required to reach equilibrium, insufficient exposure of surface area when heavier particles settle at the bottom, less growth in the location with poor super saturation, viscous nature of solution etc. In such a situation, the final mother liquor may retain appreciable super saturation and actual yield is always less unless mode of nucleation is changed and above-mentioned causes are solved. The solubility is expressed commonly in terms of anhydrous salt per 100 mass of pure solvent or mass fraction. The calculation of yield of a nonhydrated salt (crystal without water of crystallization) from solution is simple since the amount of solvent in the liquid remains unchanged.

Purity of product

Purity of a crystalline product is judged by many ways, for example determination of melting point of crystalline substance. Generally it is thought that „crystal form‟ is the purest form of a substance, but it may retain impurities on its surface due to adherence of impure mother liquor on its surface while separating it from final magma. Smaller crystals will retain relativity more impurities than large crystals due to large specific surface area of the smaller crystals. “Washing of crystal’s surface” is done with the same solvent to remove impurities. Sometimes mother liquor is occluded within the empty space of an agglomerate / lump that leads to poor quality product. Proper washing of agglomerated crystal is difficult. Formation of agglomerated crystal during crystallization can be minimized by proper agitation.

Nucleation and Crystal growth

In the formation of crystal two-steps is required (1) generation of a new particle (2) its growth to macroscopic size. The first step is called nucleation. Neither formation of a new crystal nor its growth is possible in saturated or unsaturated solution. Rate of nucleation is very important for controlling crystal size distribution (CSD). The rate of nucleation is the number of new particles formed per unit time per unit volume of magma or solid free mother liquor.

The sequence of stages in the evolution of a crystal is as follows:

Cluster − embryo − nucleus − crystal

Nuclei are in a state of unstable equilibrium if it loses units, it dissolves, if it gains units, it grows and becomes a crystal.

Solubility of large crystal is less than that of smaller crystals in the micrometer range because of significant surface energy per unit mass on smaller crystals. Smaller crystals are in a state of unstable equilibrium in a supersaturated solution. As a result larger crystals grow until the small crystals disappear. This phenomenon is called Ostwald ripening.

The effect of particle size on solubility is a key factor in nucleation. At first, a loose unstable aggregate called „cluster‟ is formed when several molecules/atoms of solute come into contact due to random collision. Formation of embryo initiates lattice arrangement and formation of a new and separate phase.

Though embroys have short lives but it may grow to a size as that of nucleus, which is the smallest assemblage of particles.

Formation of stable nucleus depends on number of units assemble together. Nucleation may be of three types: Spurious nucleation, primary nucleation and secondary nucleation. Spurious nucleation produces crystals of poor quality with abnormal needle like or whisker like growths from the ends of the crystals. It occurs at large super saturation (ΔT >80 C). This is called needle breeding. Another growth related spurious nucleation called „Veiled growth‟ gives the crystal surface, milky appearance due to rapid crystal growth and occlusion of mother liquor into the crystal faces. This problem can be avoided by growing crystals at low super saturation and by using well-designed and operated pumps and agitators. The first, step of „nucleus‟ formation is called „nucleation‟ or primary nucleation. On an industrial super saturation driving force is necessary to initiate primary nucleation. The second chief mechanism in crystallization is called secondary nucleation. There, crystal growth is initiated with contact.

The formation of nuclei is attributable to fluid shear and to collisions between existing crystals with each other or with the walls of the crystallizer and rotary impellers or agitator blades. On impact with these moving parts of crystallizer, soft or weak crystals can break into fragments and so give new crystals. Attrition is the only source of new crystals that is independent of super saturation. Crystal growth is a diffusion process; solute molecules reach the growing surface by diffusion through the liquid phase and are organized into space lattice. Growth rate of most crystals is linear with super saturation. The rate of deposition is proportional to driving force between the bulk of the liquor and that wetting the surface of the crystal that is approximately saturated with respect to crystals of that size. The driving force will vary because of the increasing solubility for crystals with lower size range. Crystal growth takes place in metastable zone that lies between saturation and nucleation limits. In this region the solution is supersaturated and no nucleation occurs when crystals are growing.

Miers super saturation theory

In the year 1927, Miers, SIR, H.A. postulated a theory on super saturation. Miers theory explains growth of nuclei with respect to super solubility and solubility curve under some limitations. Let us know first about solubility curve. When equilibrium is attained at final temperature, mother liquor becomes saturated in crystallization process and rate of formation of nucleus is balanced by the rate of dissolution of nucleus. The equilibrium relationship is the solubility curve. Solubility data for different solids had been expressed as function of temperature. Solubility chart displays various types of profiles like curves with positive and high slope (KNO3), positive and very low slope (NaCl), negative slope (MnSO4−H2O). For most of the materials, solubility curves follow firstly i.e. high and positive slope. Solubility curve represents the maximum concentration of solutions that can be obtained by bringing solute into equilibrium with solvent. This curve represents final concentration of mother liquor toward which supersaturated solution approaches. Super saturation is attained by decreasing temperature of highly concentrated solution or decreasing amount of solvent by evaporation or by both.

Miers super saturation theory is explained with the help of following figure.

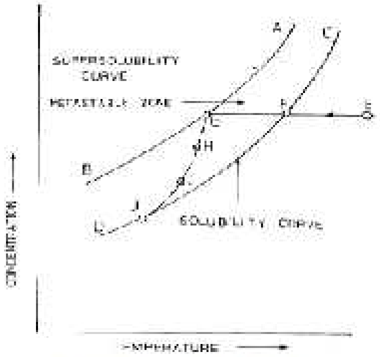


Fig. 1: Miers solubility curve

AB – super solubility curve, CD – solubility curve, E – Feed location, under saturation, F–solution cools to saturation, G – Metastable zone, nucleation begins. H– Concentration of mother liquor decreases with crystal growth, I – Crystal growth during cooling and decrease of concentration of solution, J – Exit location, saturated solution

Figure 3.1 Mier's Solubility Curve

Super saturation method

1. Super saturation by cooling
2. Super saturation by evaporation of solvent.
3. Super saturation by adiabatic evaporation (cooling plus evaporation)
4. Salting out by adding a substance that reduces the solubility of the substance of interest.

In case of solubility with strong temperature dependencies the cooling method is more attractive whereas with low dependencies on temperature the evaporation method is adopted. When solubility curve decreases appreciably with temperature i.e., rate of decrease of solubility with the decrease of temperature is high, so curve is steeper. KCl, $AgNO_3$, $CuSO_4$, $5H_2O$ and $Na_2HPO_4 - H_2O$ are the example of crystals that follow Method

1.Substances like NaCl, Na_2SO_4, and $CaCl_2$ follow method

2. Solubility curves of these substances are flat type i.e. solubility does not decrease appreciably with the decrease of temperature. In this case, yield will be very poor if method 1 is followed. The third method, cooling adiabatically under a vacuum is the most important method for large-scale production. If a hot solution is evaporated under a vacuum, the solvent flashes because total pressure is less than the vapor pressure of the solvent at the temperature at which it is introduced. Now, the solution becomes supersaturated due to evaporation of solvent and adiabatic cooling. The last method is not very much used. In this method, a foreign substance is deliberately added to reduce the solubility to such an extent that the desired solute crystallizes.

Crystallization Equipments

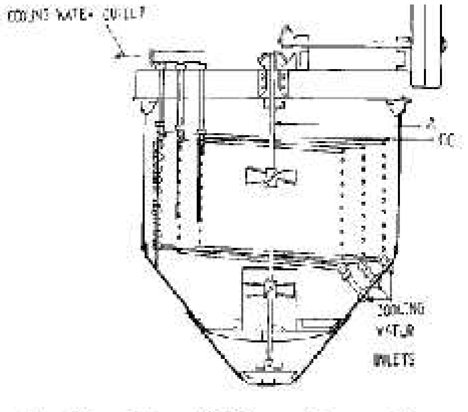
There are different types of equipments used for making crystals. The design of these equipments is based on the method of super saturation.

Names of equipments:

1. Super saturation by cooling alone − Tank crystallizer, Agitated batch crystallizer. These two are used in batch process, though tank crystallizer is obsolete one. Swenson Walker crystallizer falls under this category and it is used in continuous process.
2. Super saturation by adiabatic cooling: Different kinds of vacuum crystallizers are used to make crystals by this method.
3. Super saturation by evaporation: Krystal evaporator and salting evaporator are two examples under this type of super saturation. All these equipments are made of stainless steel.

Description of some crystallizers:

(i)Agitated batch crystallizer: The crystallizer body is equipped with a centrally located agitator, cooling pipes. The upper part of the vessel is cylindrical and closed at the top. The lower part of the vessel is conical and its bottommost part is used to drain out final magma. Magma is defined as the slurry containing product crystal and saturated mother liquor. Hot concentrated solution of a substance is induced in the crystallizer and it is agitated with the impeller. Cold water flows though the cooling coils to transfer heat from the hot solution. Cold water transfers heat due to heat of crystallization too. As temperature drops, super saturation is achieved that initiates crystallization. Agitator facilities heat transfer operation throughout the solution and maintains uniformity of temperature in the solution and keeps the growing crystals in suspension so that these can grow uniformly. Thus agitator prevents formation of aggregates.



A – Impellor, CC- cooling coil
Fig. 3: Agitated batch crystallizer

Figure 3.2 Agitated Batch Crystallizer

Rate of production of crystals is low due to batch process. Since temperature at the surface of cooling coil is least so rapidly formed crystals get deposited on the surface of cooling coil and cause hindrance in heat transfer rate. These are the disadvantages of agitated batch crystallizer. After certain period of growth, magma is transferred to centrifuge where crystals are separated from mother liquor.

The Swenson−Walker crystallizer: It consists of an open semi cylindrical trough, bottom part of which is welded with a jacket to run cold water through the jacket in counter current direction to that of solution. It is equipped with a slow speed long pitched spiral agitator moving at 7 r.p.m., that is fitted as close to the bottom of the trough as possible i.e. a narrow clearance is maintained. A single unit (trough) is 24 inch wide and 10 feet long. Length of crystallizer may be increased up to 40 feet by joining 4 units. If more units are required, these can be arranged one above another and solution fall from the upper row to the next lower one.

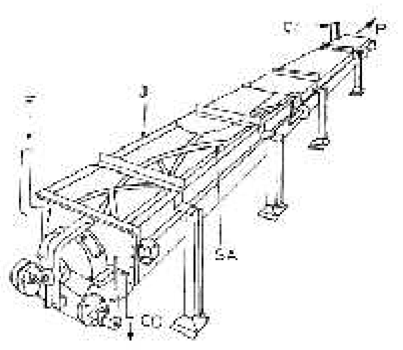


Fig. 4: Swenson Walker Crystallizer: F-feed, J- Jacket, CI-coldwater inlet, CO-coldwater outlet, SA-spiral agitator, P- product.

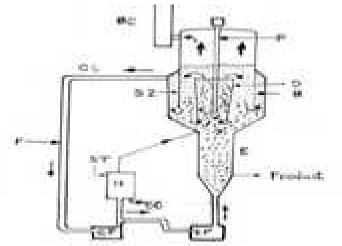
Figure 3.3 Swenson Walker Crystallizer

The hot concentrated solution is fed at one end of the trough and flows to the other end slowly. When solution is cooled to desired degree of super saturation, crystals start forming. At the end of the crystallizer, crystals along with mother liquor overflow to a draining table that separates mother liquor from crystals. Mother liquor is returned to the process and wet crystals are conveyed to a centrifuge. The spiral agitator functions by conveying crystals in the forward direction and it keeps particles in suspension and prevents accumulation of crystals at the bottom of trough. This agitator lifts the deposited crystals from the bottom and spreads throughout the solution and thus keeps these crystals in suspension facilitating uniform growth of crystals.

Vacuum Crystallizer: This falls in the category of modern type crystallizers. It is closed vessel, at its top vacuum is maintained by a condenser with the help of a steam jet vacuum pump or booster. Large quantity of material can be processed in small floor space and labor cost is also saved in this type of crystallizer.

Vacuum crystallizer consists of a cone-bottomed vessel, which is fitted with a barometric condenser at the top and propellers in the conical part, elutriation leg at the bottom of the vessel. Hot saturated solution (feed) well above the boiling point at the pressure in the crystallizer is introduced into the main crystallizer body via a slow speed low head circulating pump and a heater, through a tangentially cut inlet pipe that causes a swirling motion within solution. Liquid level in the crystallizer body is maintained up to certain mark. At the top of liquid, vacuum is maintained that corresponds to be boiling point of the solution lower than the feed temperature. The solution so introduced into a vessel in which a vacuum is maintained, the solution flashes and solvent, evaporates due to adiabatic evaporation. As a result solution gets cooled spontaneously to equilibrium temperature; since it is adiabatically cooled i.e. both the enthalpy of cooling and the enthalpy of crystallization provide enthalpy of vaporization. Super saturation is generated due to both cooling and evaporation.Temperature gradient and concentration gradient thus formed due cooling and evaporation are two driving potential to create nucleation. Vapors are formed in the empty space above the solution and discharged through the top outlet. It is evident that the vapor from the crystallizer cannot be condensed at low pressure with the usual cooling water temperatures available.This vapor is compressed and then condensed by ordinary cooling water. The crystals so formed in the body due to super saturation, fall through the conical bottom of vessel into elutriation leg that helps to classify and separate bigger size particles.

Now, product magma (crystal along with mother liquor) is discharged through outlet and fed to centrifuge, part of mother liquor is re-circulated. Function of tangential inlet of the pipe is that it creates swirling motion and thus facilitates flashing of solvent and equilibrates magma within the vapor. The simple vacuum crystallizers are not equipped with propellers. So, the effect of static head due to solution creates problem in generating adequate nucleation.



Vacuum Crystallizer: B- barometric condenser, D- Downpipe, F- feed ST-steam inlet, SC- steam condensate, H-heater, PM- Pump, C-centrifuge, M-mother liquor, P-product crystal.

Figure 3.4 Vacuum Crystallizer

Draft tube baffle (DTB) crystallizer:

Draft tube baffle crystallizer is based on the same principle as that of vacuum crystallizer but it is more versatile and effective equipment. The main crystallizer body is equipped with a condenser at the top, elutriation leg at the conical bottom, a recycling loop at the side of the body, a centrally located propeller, a draft tube around the propeller and an annular space within the crystallizer body.

A pump assembly is used to maintain circulation of fluid. Hot solution enters the main crystallizer body. The solution is super saturated adiabatically under the vacuum maintained at the top, due to evaporation of solvent and decrease of temperature of solution.

Draft tube acts as battle. It controls circulation by promoting axial flow of fluid thus facilitating maintenance of temperature and concentration gradient throughout the solution and allowing fine particles to flow in the upstream. In this crystallizer, all the fine particles are not grown simultaneously. Some fraction of fine particles is removed in the upstream.

When fluid enters the annular zone, bigger particles are settled here due to baffling effect and very finer particles float in the upstream and thus unwanted nuclei are removed through the recycle loop. In this way magma density is sharply increased within the body. A part of this recycled liquid is mixed with the fresh feed and is pumped to the main crystallizer body via a heater and another part of liquid is introduced into the elutriation leg via an elutriation pump.

Elutriation leg facilitates in classification of crystals according to size while flowing in the upward direction through it. Upward stream carries away some smaller particles into the main crystallizer body for further growth. Product is discharged through the outlet and fed to the centrifuge to separate out crystals from mother liquor.

This type of crystallizer produces crystals with narrow crystal size distribution. Draft Tube Baffle Crystallizer is an elaborated Mixed Suspension Mixed Product Removal (MSMPR) design, which has proven to be well suited for vacuum cooling and for processes exhibiting a moderate evaporation rate. MSMPR is an idealized crystallizer model that follows some stringent requirements. The DTB crystallizer has proven to be suitable for many products such as boric acid, Glauber salt, citric acid

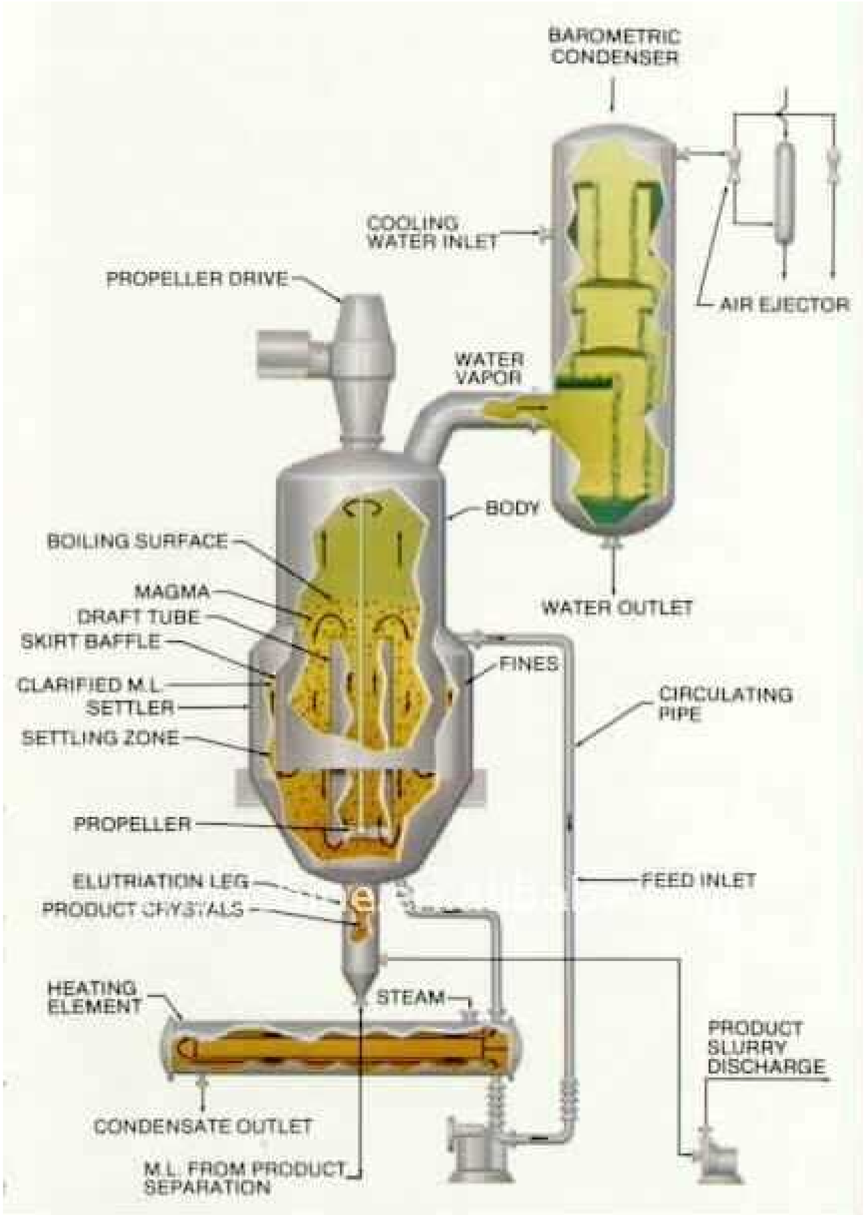
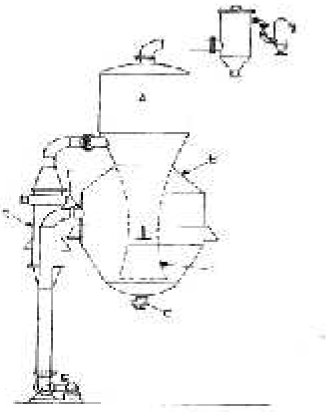


Figure 3.5 DTB Crystallizer

The Krystal Crystallizer: The Krystal or Oslo crystallizer is extensively used where large quantities of crystals of controlled size are required. The basic principle is to flow a supersaturated solution to bed of crystals which are maintained in a fluidised state. The Krystal crystallizer consists of a vapor head (A) that is attached with a condenser at the top and connected with a long specially designed discharge tube (E) extended well to the bottom of crystal growth chamber B.

Feed solution is usually introduced into the suction of pump C and then it is sent to vapor head through heater. Vapor formed at the top is released and discharged into condenser and vacuum pump. The hot solutions in the chamber A become supersaturated due to evaporation but crystals are not formed in this chamber.

Super saturation produced in chamber A is discharged on the crystals suspended in an upward flowing stream of liquid at the bottom of Krystal growth chamber. When the liquid becomes saturated at equilibrium, it leaves the chamber through F for recirculation. Coarser particles remain at the bottom and finer particles remain at the top of crystal bed and re-circulated liquid carries away finest particles and enters the vapour chamber via the heater and pump.



Krystal crystallizer: A- vapor head. B-crystal growth chamber, C- circulating pump, D- heater or cooler, E- discharge tube, F- overflow to circulating pump, G- product outlet.

Figure 3.6 Krystal Crystallizer

Material Balance in Crystalliser

$$F = C + M + E$$

$$FX_f = CX_c + MX_m + EX_e$$

Example Problem

In a process producing KNO_3 salt, 1000 kg/h of a feed solution containing 20 wt % KNO_3 is fed to an evaporator, which evaporates some water at 422 K to produce a 50 wt % KNO_3 solution. This is then fed to a crystallizer at 311 K, where crystals containing 96 wt % KNO_3 is removed. The saturated solution containing 37.5 wt % KNO3 is recycled to the evaporator. Calculate the amount of the recycle stream R in kg/h and the product stream of crystals P in kg/h.

F = 1000 kg/hr

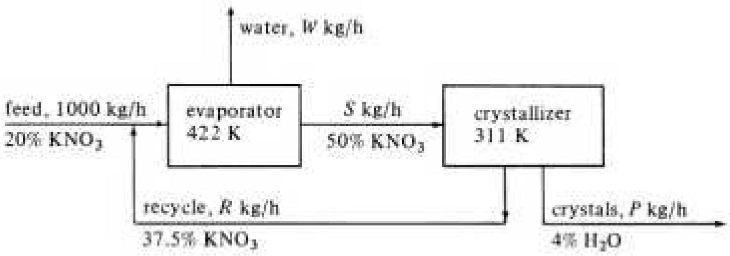
$X_e = 0$

$X_f = 20\% = 0.2$

$X_c = 96\% = 0.96$

$$1000(0.20) = W(0) + P(0.96)$$

$$P = 208.3 \text{ kg crystals/h}$$



$S=R+208.3$

For a KNO_3 balance on the crystallizer,

$$S(0.50) = R(0.375) + 208.3(0.96)$$

Solving the above equations

***R* = 766.6 kg recycle/h and *S* = 974.9 kg/h.**

Problem for Practice:

1. 5000Kg of KCl solution at a temperature of 80°C is cooled to 20°C in an open tank. The solubility's of KCl at 80°C and 20°C are 55 parts per 100 parts and 35 parts per 100 parts of water. Estimate the yield of KCl crystals by,
   (a) Assuming 5% water is lost by evaporation.
   (b) Assuming no loss of water by evaporation.
2. A 35% solution of Na_2CO_3 weighing 6000 kg is cooled to 20°C to yield crystals of $Na_2CO_3 . 10H_2O$. During cooling 4% by weight of original solution is lost due to vaporization. Find the weight of crystal formed. Data: Solubility at 20°C = 21.5 g of Na_2CO_3/100 g of water.

References:

Mass Transfer Operations, R. E. Treybal's, McGraw Hill International Edition, 1981

W.L. McCabe, J. Smith and P.Harriot, Unit operations of Chemical Engineering, McGraw Hill International Edition 2001.

B. K. Dutta, Principal of Mass Transfer and Separation Processes, 1st edition, 2012