

UNIT: III

(DRYING)

CLASS:22

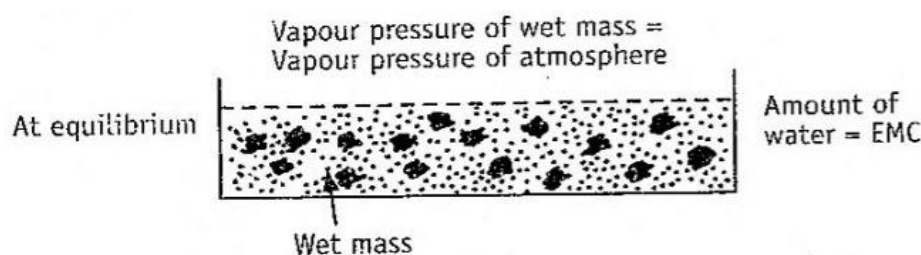
**TOPIC: MEASUREMENT & APPLICATIONS OF EQUILIBRIUM MOISTURE
CONTENT, RATE OF DRYING CURVE**

Equilibrium Relationships

Air of constant temperature and humidity is passed over the wet solid. After a long exposure, equilibrium is reached. On attaining equilibrium, further exposure will not alter moisture content in the solid. At this stage, Vapour pressure of the wet solids is equal to that of the surrounding atmosphere. Hence there is no driving force for mass transfer.

Equilibrium moisture content (EMC): It is the amount of water present in the solid which exerts a vapour pressure equal to the vapour pressure of the atmosphere surrounding it.

The moisture (content) which is more difficult to remove in practice is the equilibrium moisture content. This moisture content is retained by the solid under steady state conditions. This water (or liquid) does not vaporize and does not produce vapour pressure. From that point, full vapour pressure cannot be developed. Equilibrium moisture content in a wet mass is shown below.

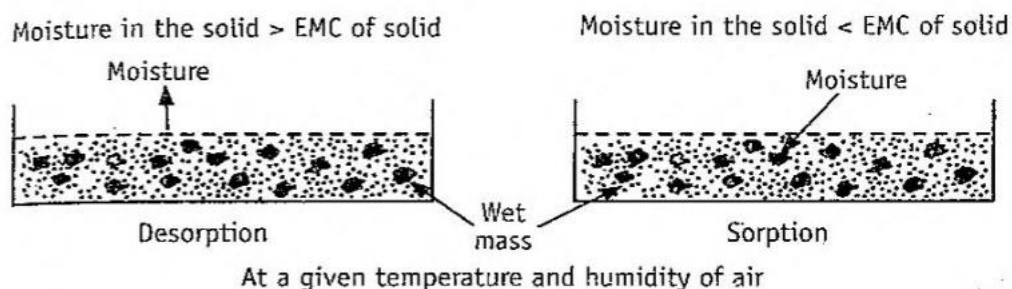


The characteristics of air such as temperature and humidity are maintained constant. Depending on these conditions, the solids may absorb or lose moisture.

When air (of constant temperature and humidity) is continuously passed over the solid containing moisture more than EMC, then solid loses water continuously till EMC is reached. This phenomenon is known as *desorption*.

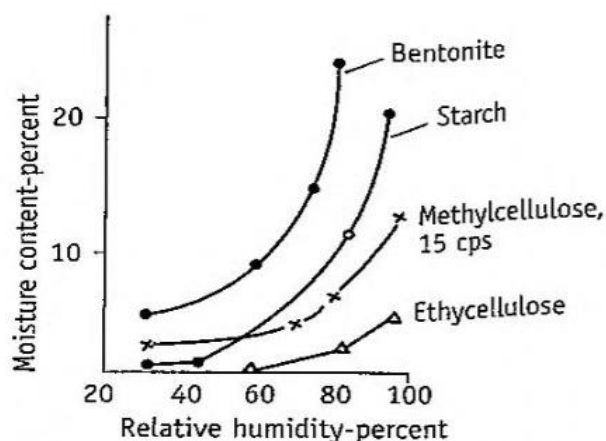
When air (of constant temperature and humidity) is continuously passed over the solid containing moisture less than EMC, then solid adsorbs water continuously till EMC is reached. This phenomenon is known as *sorption*.

The behaviour of desorption and sorption is shown below. From the above observations it is clear that, material can be dried up to EMC but not below it.



Measurement of EMC: The EMC of a material can be determined as follows. The solid samples are placed in a series of closed chambers such as desiccators. Each chamber consists of solution (desiccant), which maintains a fixed relative humidity in the enclosed air spaces. In other words, the solid samples are exposed to several humidity conditions. The exposure is continued until the material attains a constant weight (equilibrium conditions). The difference in the final and initial weights gives the moisture content.

Equilibrium moisture curve is drawn by taking relative humidity (%) on x-axis and moisture content on y-axis (Figure).



EMC curves for tabletting material.

Application of EMC: The EMC curve permits the selection of the experimental conditions to be used for drying the product. Drying should be stopped when the moisture content reaches the level of the EMC under the exposed conditions. Over drying can be avoided, because over-dried solid quickly regain the moisture from the ambient conditions.

If the moisture content is to be reduced, the relative humidity of the ambient air must be reduced as a first step. This can be done mechanically on a large scale using an air-conditioning system. On a small scale, desiccators are employed. Some materials, such as tablet granules, have superior compaction properties with a small amount (1-2%) of residual moisture content.

Factors affecting EMC: EMC values vary depending on a number of factors.

Nature of material:

1. Nonporous insoluble solids have an EMC of practically zero. Example is talc.
2. For fibrous or colloidal organic substances, the EMC values high and variable.

- For porous solids, the EMC values are much higher and variable. The water may be held in fine capillaries that have no access to the surface. The dissolved solid may reduce the vapour pressure and water may be molecularly bound.

Nature of air:

- For air of zero humidity, EMC of all materials is zero.
- As the temperature of air increases, the EMC of solid decreases.

EMC of solids is a constant for a given temperature and humidity of air. If the equilibrium curves (Figure) are continued up to 100% RH, the moisture content so obtained (EMC with saturated air) is the least moisture at which the material can exert a vapour pressure as high as that exerted by liquid water at the same temperature.

Free moisture content (FMC): *Free moisture content* (FMC) is the amount of water that is free (easy) to evaporate from the solid surface.

Under the conditions of saturation humidity (100% RH), the EMC is the minimum moisture content. Under these conditions the water must be bound water which is minimum at that temperature. The remaining is unbound for which the FMC for a given condition can be written as:

Free moisture content (FMC) = total water content - equilibrium moisture content (EMC)

The distinction between free and equilibrium moisture depends on the drying conditions.

Rate Relationships

Rate relationships can be studied by considering a simple model, which mimic the conditions of a dryer. In this model, wet slab of material of sufficiently high moisture content to be dried is placed in a tray whose bottom and sides are insulated. The air is blown over the solid under constant drying conditions (air velocity, temperature, humidity and pressure are maintained constant). The superficial water diffuses through the surrounding stationary air film and is carried away rapidly by the moving air stream.

During drying, the surface temperature falls to the wet-bulb temperature that corresponds to the temperature and humidity of air.

The rate of transfer of water vapour from the surface into air is:

$$N = \frac{k_g}{RT} (p_{wi} - p_{wa})$$

where p_{wi} = partial pressure of water vapour at the surface, mPa

p_{ws} = partial pressure of water vapour in the air, m Pa

N = moles of vapour transferred per unit area in unit time

R = Ideal gas constant, kJ/kmol.K

T = absolute temperature, K

K_g = mass transfer coefficient, mol per unit area

The K_g mass transfer coefficient is a function of:

- temperature f

- air velocity
- angle of air incident

The mass of liquid per unit time (w) is expressed by rewriting equation (I) as:

$$W = \frac{M_w \times A}{RT} K_g (P_{wi} - P_{wa})$$

where M_w = molecular weight of water vapour (18 g/mol)

High velocity or angle of incidence diminishes the thickness of the stationary air layer in contact with the liquid surface, thereby lowering the diffusional resistance.

The rate of evaporation may also be expressed in terms of heat transfer across the laminar film from the drying gases as:

$$Q = hA(T_a - T_s)$$

where Q = rate of heat transfer, J/s

h = heat transfer coefficient, J/s. m².K

A = area of surface, m

T_a = temperature of drying air, K

T_s = temperature of the surface, K.

Equation refers across the laminar flow from drying gases to the surface. The heat transfer coefficient is also a function of air velocity and the angle of impingement.

Actually, mass transfer rate (w) occurred due to the latent heat of vaporization (λ) is:

$$w = \frac{hA}{\lambda} (T_a - T_s)$$

When these conditions pertaining to drying, the surface temperature, T_s (though consider as equal to the wet-bulb temperature), is normally much lower than the temperature of drying gases. The observation is applicable to drying of thermolabile substances. Its solids are present in the surface, the rate of evaporation is modified, then the overall effect depends on the structure of the solids and the moisture content.

Periodically the slab is weighed. The difference in the weights of two successive periods gives the loss of moisture content, i.e., amount dried. The moisture present in the solid can be expressed on a wet weight or dry weight basis. Then the following calculations are made:

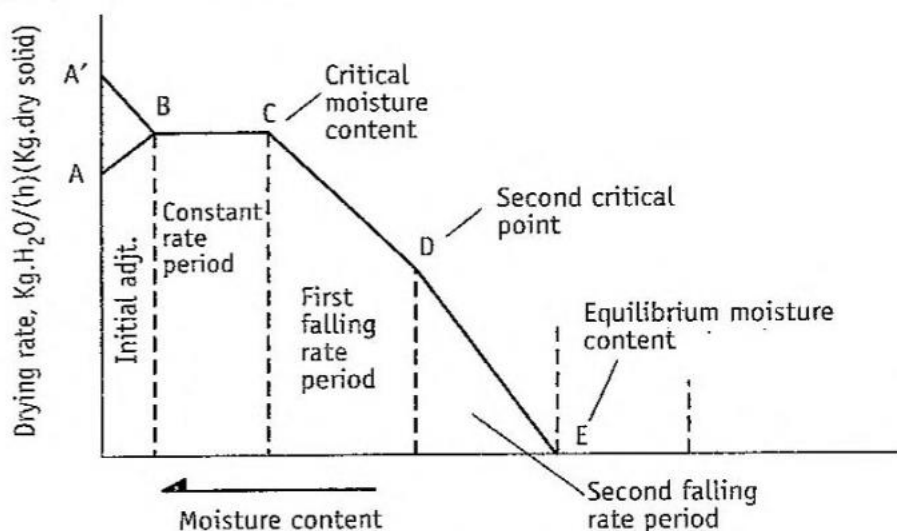
$$\% \text{ Loss on drying (LOD)} = \frac{\text{mass of water in sample (kg)}}{\text{total mass of wet sample (kg)}} \times 100$$

$$\% \text{ Moisture content (MC)} = \frac{\text{mass of water in sample (kg)}}{\text{mass of the dry sample (kg)}} \times 100$$

$$\text{Drying rate} = \frac{\text{weight of water in sample (kg)}}{\text{Time (h)} \times \text{weight of the dry solid (kg)}}$$

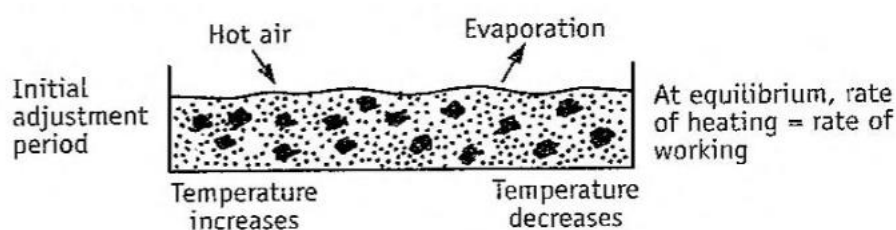
Drying rate is plotted against the midpoints of the time period. Similarly, midpoints of the moisture content values can be plotted.

From the data obtained by the above experiment, a graph is plotted by taking FMC on x-axis and drying rate on y-axis. The curve so obtained (Figure) is called drying rate curve. It represents different changes during drying as explained below.

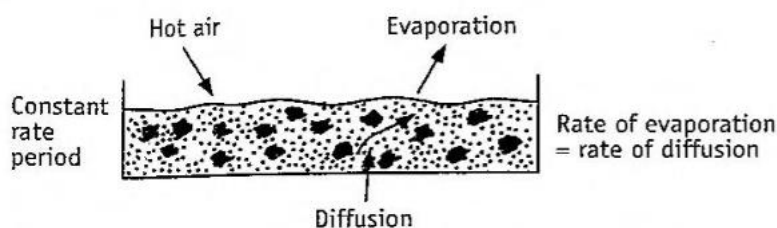


FMC curve.

(1) The time corresponding to AB represents the initial *adjustment period*. During this period, the solids absorb heat and the temperature increases. At the same time, the moisture begins to evaporate and thus tends to cool the drying solid. After some time, the temperature stabilises (heating and cooling rates become equal). This temperature is equal to the wet bulb temperature of the drying air and is referred by the point B.

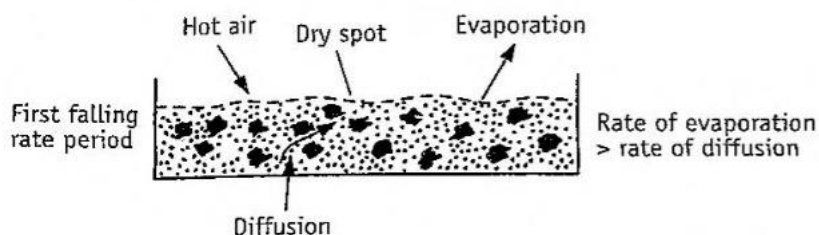


(2) The time corresponding to BC represents the constant rate period. The temperature remains constant and rate of drying is constant. The moisture evaporating from the surface is replaced by the water diffusing from the interior of the solid. The rate of diffusion is equal to the rate of evaporation. The moisture content at the end of constant rate (point C) is referred to as the critical *moisture content* (CMC).



(3) The time corresponding to CD represents the first falling rate period (or unsaturated surface drying). During this period, the surface water is no longer replaced at a rate fast enough to maintain a continuous film on the surface. Dry spots begin to appear and the rate of drying begins to fall off. The

point D is referred to as the second critical point. At this point, the film of surface water is completely evaporated.



(4) The time corresponding to DE represents the second falling rate period. During this period, the rate of drying falls even more rapidly than the first falling rate. During this period, the rate of drying is dependent on the rate of diffusion of vapour of moisture to the surface of the solid. Point E is referred to as the *equilibrium moisture content*.



(5) Beyond E, the drying rate is equal to zero. Therefore, temperature and moisture content remain constant. Beyond, E, continued drying is waste of time and energy

The curves may have different shapes for different levels of moisture. If the drying is carried above the level of CMC, only constant period occurs. If the drying is started for the material whose initial moisture content is less than CMC, then falling rate period occurs.

The drying process is related to wet-bulb and dry-bulb temperatures. Drying (migration of liquid) largely occurs during the constant-rate period and first falling-rate period. As drying continues, crystallization also occurs to a certain extent, though it is not the objective. If the ingredients are water soluble, then segregation of the soluble ingredients occur during drying. Therefore, blending is essential after drying (dry blending). The curves may have different shapes for different levels of moisture. If the drying is carried above the level of CMC, only constant rate period occurs. If the drying is started for the material whose initial moisture content is less than CMC, then falling rate period occurs.

The falling rate periods put together gives nearly a linear plot and hence possible to model the drying process. A simplified equation is:

$$\ln \frac{M_t}{M_0} = -kt - 0.2$$

Where k = first order rate constant, h^{-1}

M_0 = moisture content at initial period, kg (g)

M_t = moisture content (mass) at any time t , kg (g)

A plot is drawn by taking $\ln$ on y axis and time on x axis. A straight line is obtained with a negative slope, $-k$. This model permits the evaluation of drying process in a tray dryer and impact of process parameters (bed thickness and drying temperature). Drying can be attempted at different

temperatures and a relationship can be drawn between $\ln k$ vs. $1/T$ (a pattern similar to Arrhenius plot for drug loss against temperature), i.e. moisture loss against temperature

MCQ Each carry 1 mark

1. Talc has EMC of practically equal to
 - a. High
 - b. One
 - c. Variable
 - d. Zero**
2. Which one of the following types of products is having an EMC of practically zero?
 - a. Non-porous & insoluble**
 - b. Non-porous & soluble
 - c. Porous & insoluble
 - d. In drying curve,
3. initial adjustment period involves one of the following processes.
 - A. Surface water is no longer replaced at a rate fast enough from the bulk.
 - B. Temperature and rate of drying remains constant.
 - C. Temperature increases and subsequently decreases.**
 - D. Temperature remain constant.
4. In a drying process, once the material reaches equilibrium moisture content, the rate of drying proceeds one of the following.
 - A. Constant
 - B. High
 - C. Low
 - D. Zero

Each carry 2 marks

1. Define critical moisture content and equilibrium moisture constant.
2. Define bound water and free moisture content.
3. How do you obtain the rate of drying curve for a given drying operation? Give its applications.

Each carry 5 marks

1. Describe the drying rate curve for a nonporous granular solid
2. Give the measurement and application of Equilibrium moisture content (EMC)
3. Describe the drying rate curve. Explain its applications.
4. Describe the rate of drying curve for a crude fibrous drug.

Each carry 10 marks

1. Give detail note on rate relationship
2. Explain the theory behind drying and rate of drying with suitable graphs
3. How do you classify dryers? Describe in detail the constant rate and falling rate periods. Add a note on critical moisture content.