CHAPTER 9

DRYING

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Abstract

Drying is a crucial unit operation in pharmaceutical manufacturing, involving the removal of moisture or other solvents from solid, semi-solid, or liquid materials. This process is essential for enhancing the stability, handling properties, and shelf life of pharmaceutical products. Various drying mechanisms, including evaporation, vaporization, and sublimation, are employed depending on the nature of the material and desired end-product characteristics. Different types of dryers, such as tray dryers, fluidized bed dryers, spray dryers, and freeze dryers, are used based on the specific requirements of the formulation. Factors affecting drying efficiency include heat and mass transfer rates, material properties, and equipment design. The concept of drying kinetics, including constant rate and falling rate periods, is explored to understand the drying process dynamics. Critical process parameters, such as inlet air temperature, airflow rate, and product bed depth, are discussed in relation to their impact on product quality. Special considerations for heat-sensitive materials and the drying of hygroscopic substances are addressed. Quality control measures, including moisture content determination and uniformity testing, are essential to ensure consistent product quality

Keywords: *Moisture removal, Solvent evaporation, Drying kinetics, Stability enhancement, Lyophilization, Process optimization*

Learning Objectives

After completion of the chapter, the student should be able to:

- Define drying and its importance in pharmaceutical manufacturing.
- Explain the principles and mechanisms of various drying processes.
- Describe different types of drying equipment used in pharmaceuticals.
- Discuss the factors affecting drying efficiency and product quality.
- Explain the concept of moisture content and its measurement techniques.
- Analyze the impact of drying on drug stability and properties.
- Evaluate methods for optimizing drying processes in pharmaceutical production.

Drying is defined as the removal of liquid from a solid by thermal method. When large amount of liquid is evaporated from a solution /suspension / slurry the process is called 'evaporation'. The final product is a concentrated liquid / slurry.

When very small amount of liquid is evaporated from solids the process is called 'drying'. The final product is a 'dried solid'.

PURPOSE OF DRYING

1. Drying is most commonly used in pharmaceutical industries in the preparation of granules, which can be packed in bulk or compressed into tablets or filled in capsules.
2. Drying is required for processing of materials like, drying of aluminium hydroxide, spray drying of lactose

and preparation of powdered extracts.

3. Drying is used to reduce the bulk weight that lowers the transportation and storage costs of that material.
4. Drugs obtained from plant and animal sources, when dried, becomes more friable. Thus, drying helps in size reduction of natural drugs.
5. Animal and vegetable drugs are preserved against mold growth in dried condition.
6. Dried products often are less more stable than moist ones as in the case of effervescent salts, aspirin, hygroscopic powders, ascorbic acids and penicillin.

SOME DEFINITIONS

Moisture content (MC)

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

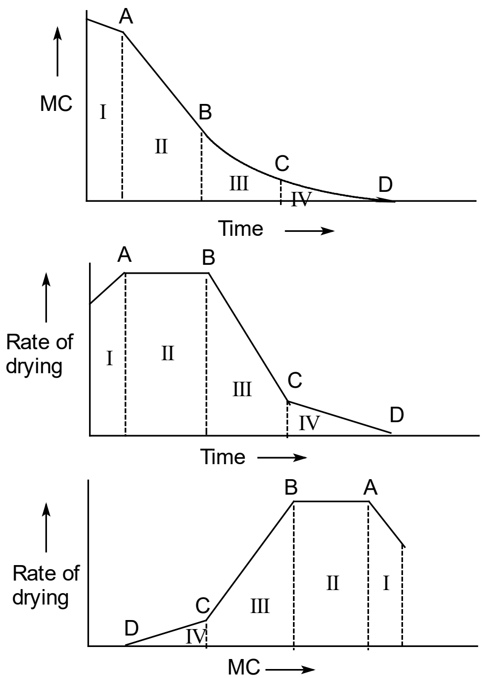
Loss on drying (LOD)

$$\% \text{ LOD} = \frac{\text{weight of water in sample}}{\text{total weight of wet sample}} \times 100$$

THEORY OF DRYING

A typical drying cycle shows the following phases:

Phase-I: Initial adjustment period
Phase-II: Constant rate period
Phase-III: First falling rate period
Phase-IV: Second falling rate period



B: Critical Moisture Content (CMC)
D: Equilibrium Moisture Content (EMC)

The rate of drying of a sample can be determined by suspending the wet material on a scale or balance in a drying cabinet and measuring the weight of the sample as it dries as a function of time.

Phase-I: Initial adjustment period

A wet material when kept for drying it begins to absorb the heat and the temperature of the material increases. At the same time, the moisture begins to evaporate and thus cools the drying solid. After a period of initial adjustment, the rates of heating and cooling become equal and the temperature of the drying material reaches the *wet-bulb* temperature of the *drying air*.

Phase-II: Constant rate period

During this period there is continuous liquid film over the surface of the solid. Evaporation from the film (at wet bulb temperature) proceeds at a constant rate and the film is continuously replaced by the underlying moisture. As long as the delivery of water from the interior of the material is sufficient to keep the surface completely wet, the drying rate remains constant.

Drying rate at this phase is given by:

$$\frac{dW}{d\theta} = \frac{Ah_c\,(t_d - t_w)}{\rho L \lambda}$$

where, $\frac{dW}{d\theta}$ = Rate of drying in $\left(\frac{\text{kg of water}}{\text{kg of dry solid}}\right)$

A = Area exposed to drying

t_d, t_w = Dry bulb and wet bulb temperature of the drying air respectively

ρ = Bulk density of solid

L = Thickness of solid bed

λ = Latent heat of vaporization of water

h_c = Convection heat transfer coefficient

Phase-III: First falling rate period

Due to dry spots on the surface, the area of constant mass transfer decreases and the heat transferred to the dry spots will be utilized to raise the temperature of the material to the dry bulb temperature. Thus as the number of dry spots increase, heat transfer and mass transfer rates fall which is called the first falling rate period. The point from which Phase-II starts is called **critical point** (B). Moisture content at which this point appears is called **critical moisture content (CMC)**.

Phase-IV: Second falling rate period

In this period the capillaries are empty, no film is present on the surface. The moisture movement takes place only by diffusion. In this phase the rate of drying falls and it is lesser than first falling rate. The starting of second falling rate period is called **second critical point** (C).

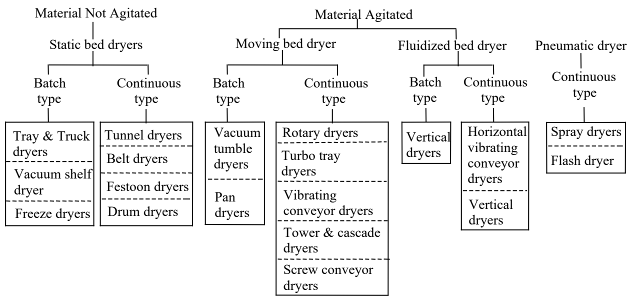
At the end the drying rate becomes zero. The moisture content at this point is called **Equilibrium Moisture Content** (EMC). EMC may be defined as the *moisture content of the solid when drying limit has attained by use of air at a given temperature (dry bulb) and humidity of air.* EMC depends on the *nature of the material, temperature* and *humidity*.

<u>Utility of drying curve</u>

1. From the drying curve the *time for drying* a batch of material in a certain dryer can be estimated.
2. The size of a particular dryer can be determined for drying a substance from one moisture level to the desired moisture content.

CLASSIFICATION OF DRYERS

Classification based on solid handling



Classification of dryers, based on methods of solids handling

Classification based on heat transfer mechanism

A. Convection dryers

(a) Tray or shelf dryers (b) Tunnel dryers

(c) Rotary dryers (d) Fluidized bed dryers

B. Conduction dryers

(a) Vacuum oven (b) Freeze dryers

C. Radiant heat dryers

(a) Infra-red dryers

TRAY DRYER / TRUCK DRYER / SHELF DRYER / CABINET DRYER / COMPARTMENT DRYER

Construction

It consists of a small cabinet or a large compartment in which trays containing wet materials are placed. The compartment wall is insulated to reduce heat loss.

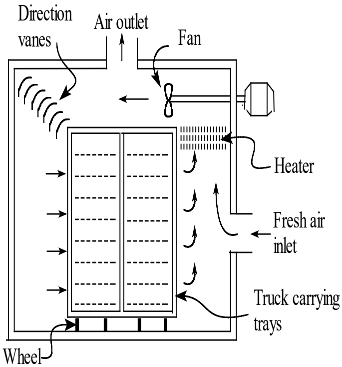


Fig. Truck dryer

- In tray dryers the trays are directly placed inside the cabinet.
- The truck dryer the trays are loaded on to the trucks (shelves on wheel) and then the trucks are introduced inside the heating cabinet.

The bottom of the trays are either perforated or having wire-mesh bottom.

- The material is heated by hot air circulated by means of fans that removes the humid air from the cabinet.

The trays containing the load remain in the dryer until drying is complete, after which they are withdrawn, emptied and recharged for drying the next batch.

Energy sources: Dry air can be heated either by electricity or steam.

Applications

- Drying of crude drugs, chemicals, powders, tablet granules etc.
- It is a batch process and materials can be handled separately.