Recent Advances in Drying Methods

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DRYING is an operation of removing a liquid; usually water, in the form of a vapour from a solid. The rate of drying, that is the rate of removal of water per unit mass of bone-dry material, is governed by the equation

$$dW/d\theta = \phi\,(p_s - p_a)\,A \ldots\ldots(1)$$

where, $dw/d\theta$ is the rate of drying per unit mass of bone-dry material, p_s, the vapour pressure of the evaporating liquid at the drying surface, p_a, the vapour pressure of the said liquid in the environmental atmosphere and A, the area of the drying surface.

The vapour pressure at the drying surface depends not only on the temperature but also on the total moisture content of the material, its distribution within the material and the manner in which it is held by the material. In a completely wet material, the moisture is usually distributed uniformly throughout the material except for some anisotropic material such as wood. The vapour pressure of water, if held by physical forces alone, is the same as its saturated vapour pressure at the same temperature. When the moisture is held by physico-chemical forces (i.e., when it is in combination), the vapour pressure is much lower, its value depending on the nature of the bond. The amount of combined water varies from practically zero for materials like sand, barytes, etc., 5-10% with certain pigments and dyes, to almost 100% in case of leather and tobacco.

Thus, in order to have satisfactory drying rates, it is necessary to have as large a vapour pressure difference as possible. This is achieved by keeping the temperature,—and hence the saturated vapour pressure, p_s,—at the drying surface as high as practicable. When, however, drying has to be carried out at room or even lower temperatures, the surroundings have to be kept at a very low pressure, in order to have a low value for p_a.

Apart from the driving force (p_s—p_a), the rate of evaporation at the drying surface is governed by two more factors. The first is the rate of diffusion of water vapour across the stagnant film of air at the drying surface and the second is the rate of movement of water from within the material to the drying surface.

The dependence of the rate of drying on diffusion is given by the equation

$$dW/d\theta = K_g\,(p_s - p_a)\,A \ldots\ldots(2)$$

where, K_g is the overall coefficient of diffusion. The thickness and hence the resistance of this film depends on the velocity of air across the drying surface, and is given by the equation

$$K_g = \phi\,G^{0.8} \ldots\ldots(3)$$

where, G is the mass velocity of air across the surface. Greater the velocity, thinner is the film and lesser its resistance to the diffusion of water power. Hence, for efficient drying, high rate of movement of air across the drying surface is necessary.

Initially, when the rate of movement of water from within the material is greater than the rate of diffusion of the vapour across the stagnant film, the evaporation takes place at the surface of the material, and since the rate of evaporation is governed by the rate of diffusion

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alone, the rate of drying under constant psychrometric conditions, is constant, when the above equations (2) and (3) are valid. But at some critical moisture content, the rate of movement of the liquid from within the drying solid decreases and the rate of drying progressively decreases until the equilibrium moisture content of the material under the operating conditions is reached. The rate of drying under these conditions is given by the equation

$$(dW/d\theta)_f = -K\,(W - W_e)\,A \ldots\ldots(4)$$

where, $W = \%$ moisture content (basic bone-dry material) at any moment,

$W_e = \%$ equilibrium moisture content expressed in the same units and

f = subscript denoting falling rate period.

The value of the constant K is a function of the constant rate period and is given by the equation

$$K = \frac{(dW/d\theta)_c}{A\,(W_c - W_e)} \ldots\ldots\ldots(5)$$

where, W_c = the critical moisture content and

$\left(\frac{dW}{d\theta}\right)_c$ = constant rate period.

Usually the drying surface is taken to be a geometrical plane, whose area, in general, is much less than that of the actual drying surface, which is far from being optically smooth. But the error involved is small and the rate of drying is usually calculated on this basis. While during the constant rate period, as stated previously, drying takes place at the surface of the material, during the falling rate period the drying surface recedes inwards, disappearing altogether at the equilibrium moisture content.

In order to keep the drying surface at the desired temperature and also to supply the necessary latent heat of evaporation, thermal energy is supplied from outside. The relationship between heat transfer and mass transfer is given by the equation

$$K_g\,(p_s - p_a) = \frac{h_t\,(t_a - t_s)}{\lambda} \ldots\ldots(6)$$

where, h_t = overall heat transfer coefficient,

t_a = temperature of the surrounding atmosphere,

t_s = temperature of the drying surface and

λ = latent heat of evaporation of water at the temperature of the drying surface.

This heat is transferred from the source to the material either by conduction through the walls of the container or by convection from the air or by radiation from the surroundings. But while the rate of heat transfer, either by conduction or by convection, is directly proportional to the temperature difference between the source and the stock, it is directly proportional to the difference in the fourth power of the temperature of the source and the temperature of the stock, in the case of radiation. This makes radiation by far the most efficient means of heat transfer as heat transfer by radiation is independent of the surrounding atmosphere. Therefore, it is possible to have the source at a very high temperature (because higher the temperature of the source, higher the rate of heat transfer) and yet heat the stock to the desired temperature and supply the requisite heat energy without the risk of overheating. Supply of thermal energy by radiation for drying operation has, in recent years, found increasing application.

Infra-red dryers :

Radiations, most effective from the point of heat transfer, occupy in the electro-magnetic spectrum a region called the infra-red; hence dryers employing radiant thermal energy are called infra-red dryers. These are essentially funnel type of dryers, eminently suitable for continuous operation. The material to be dried passes at a predetermined rate through the tunnel where it is exposed to the radiation, the commonest source being infra-red lamps. These lamps have tungsten filaments and have a colour temperature of about 2200°C which is lower by 300°C than that of ordinary incandescent lamps used for lighting purposes. Lamps with carbon filaments have a very high initial efficiency but their life is short. The radiant energy is evenly distributed over the stock by means of reflectors, which, usually parabolic in shape and plated with aluminium and though less efficient than those plated with gold, are less costly.

Wherever fuel is cheap, the radiant energy may be supplied by refractories heated to incandescence, usually by means of surface combustion. Though the colour temperature of these refractories is lower than that obtained with an infra-red lamp, the flue gases produced by the fuel burnt may be made to pass over the stock, thus augmenting the supply of thermal energy and hence the capacity of the dryer, making its operation more economic.

In infra-red drying, the temperature attained by the stock is usually higher than any wet-bulb temperature and depends, among other factors, upon the intensity of radiation, the heat capacity and absorptivity of the material and the heat losses to the surroundings. The rate of drying per unit area of drying surface is given by the equation, based on the assumption that the area receiving the radiation equals the area losing heat by convection :

$$\left(\frac{dW}{A/d\theta}\right)_c = \frac{3{\cdot}41\,\alpha\,I - h_c\,(t - t_a)}{\lambda} \quad \ldots(7)$$

where, t = temperature of the stock, °F,
t_a = temperature of surrounding, °F,
α = absorptivity, that is the ratio of heat absorbed to heat received, of the stock,
I = intensity of radiation, watts/sq. ft.,
h_c = convection heat transfer coefficient, B.T.U./(hr.) (sq. ft.) (°F) and
λ = latent heat of evaporation of the liquid at temp. t°F., B.T.U./lb.

This method of drying is suitable for thin layers and sheets which can be heated rapidly and uniformly by radiant energy, provided its absorptivity is high. Its application for drying of materials such as paper, photographic films, enamel frit and plastic granules, glue on carton flaps, latex on wire gauze, textiles, abrasive powder and foodstuffs is on the increase. In short, any material which can be produced in sheets can be dried by this method.

A bank of infra-red lamps can be operated at 40-80% efficiency ; with the absorptivity of the materials ranging from 0.5 to 1.0, the overall efficiency of a properly designed dryer is of the order of 30-60%.

Infra-red dryers have the advantages of low cost of installation, quick drying times and flexibility of operation. These, however, are more than offset by the heavy replacement charges of the lamps and high power consumption. The overall cost of infra-red dryers is about ten times (0.2 units/lb. of water evaporated) higher than that of convection type of dryers. There is also an added disadvantage that it cannot be employed to dry bulky materials. But these dryers have been successfully used to boost up existing capacities because of their flexibility and where high rate of turnover is more important than the cost of evaporation (as infra-red dryers will dry in minutes where convection drying will take hours).

Dielectric drying :

The unsuitability of infra-red heating, and for that matter even heating by convection and conduction, is due to the fact that as the drying progresses, there are set within the material, temperature gradients which are necessary for transference of heat. These gradients are very steep if the material happens to be bulky and a poor conductor of heat. This leads to poor drying rates and overheating, sometimes ending in deterioration of the material being dried. These disadvantages will be eliminated if, by some means or the other, thermal energy can be generated *in situ* within the material.

A poor conductor of heat is usually a poor conductor of electricity, technically called a "dielectric." The most characteristic property of a dielectric is that, when placed in an electric field, a definite potential gradient is established within it. This stress produces an electrical strain or displacement, the material thereby acquiring energy which is distributed uniformly throughout its volume and which is given by the equation

$q/v = xF^2$.........(8)

where, q = electrical energy,
v = volume of the material,
x = dielectric constant and
F = field strength.

Now, a displacement or strain of elastic types gives rise to no net absorption of energy in any number of completed cycles. But a strain retarded by any force of frictional or viscous nature, however, will lag in phase with respect to the driving force, giving rise to a definite loss of energy which is dissipated in the form of heat. This process is analogous to the generation of heat by friction.

This lag, denoted by the symbol δ, is characteristic of every dielectric material. Concentration of power in any element of volume of the dielectric is given by the equation

$q/v = 5/9E^2.\ f.\ \varepsilon'' \times 10^{-12}$......(9)

where, q = electrical energy, watts,
v = volume, of the dielectric, cc,
E = potential difference across the dielectric, volts,
ε'' = loss factor = x x tan δ and
f = frequency in kilocycles per second.

Hence, it will be seen that when a homogeneous material is placed in a uniform field oscillating at a high frequency, the heat generated will be distributed *uniformly throughout the material*. By this process, thick layers of a material, which may be poor conductors of heat, can be uniformly heated, an operation that cannot be performed by any other method. If anything, the temperature at the centre of the material may be higher than the temperature at the surface, a reversal of the usual conditions of heating.

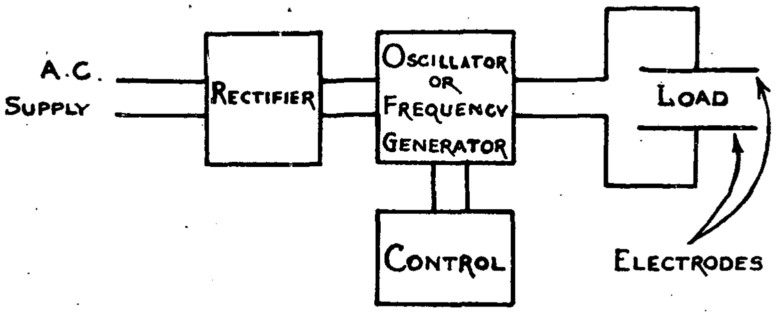

Fig 1

The material to be dried by this method is made the dielectric of a huge condenser through which passes an electrical field, alternating at a high frequency. The heat generated supplies the latent heat for drying the material. It is possible by this method to take advantage of different loss factors of components of a heterogeneous material and heat one component preferentially to the other.

In order to obtain reasonable rates of drying and yet to maintain reasonable potential differences, it is necessary to operate at very high frequencies, which range from 2 to 100 megacycles/sec. This method of drying has been applied to dry materials such as ceramic wares exceeding thicknesses of 3″, rayon cakes, textiles, sponge rubber and for the initial concentration of heat sensitive solutions such as penicillin.

The installation capacities of dielectric drying units vary from 2 KW to 100 KW, a capacity of 100 KW corresponding roughly with an evaporation rate of 5 lb. of water per minute. The cost of drying varies from 0.025 to 0.014 units per pound of water evaporated, and compares favourably with that of infra-red drying and is of the same order as that existing in other methods of drying. But the high installation cost and the necessity of having skilled labour for operation, however, are great obstacles in the universal adoption of this method.

Freeze drying:

Many a time there arises in industry the need of drying labile or heat sensitive materials such as drugs and biological preparations. Low temperatures of the stock mean low value of p_s, and consequently, to obtain a positive driving force, p_a must be lower still. This is ordinary vacuum drying. Freeze drying is a special form of vacuum drying with certain characteristics of its own.

In this process, the water is removed from the stock at pressures and temperatures low enough to maintain it in a frozen state, so that *drying occurs by sublimation.* The material to be dried is frozen at an appropriately low temperature, usually in its containers, and placed on shelves in the drying chamber which is maintained at a suitably low pressure, usually of the order of 1 mm of mercury. The heat of sublimation of water is more than enough to keep the material in a frozen state ; in fact, some heat must be supplied to the material to maintain its temperature.

At the temperatures maintained during freeze drying, the frozen stock tends to maintain its original shape. As drying progresses, since ice is immobile, immediate recession of the ice surface inside the material takes place, and yet, since no resistance is offered to the escaping vapours by the rigid material, the rate of drying remains constant almost throughout, in contrast to ordinary vacuum drying, irrespective of the position of the ice layer, till all the free moisture is removed. Freeze drying is not suitable for removing chemically bound moisture. In the case of penicillin, 99.5% of total moisture content is removed at a constant rate with the material at—20°C. The temperature then rises rapidly and may even reach that of external surroundings, when the remaining moisture is removed, the final moisture content being hardly 0.05%, this being by far a lower moisture content than that can be obtained by any other method of drying. Care must be taken that no melting occurs during this process ; otherwise, deterioration of the material and decrease in the drying rate will occur. The characteristics of the freeze drying operation can be readily ascertained from Fig. 2.

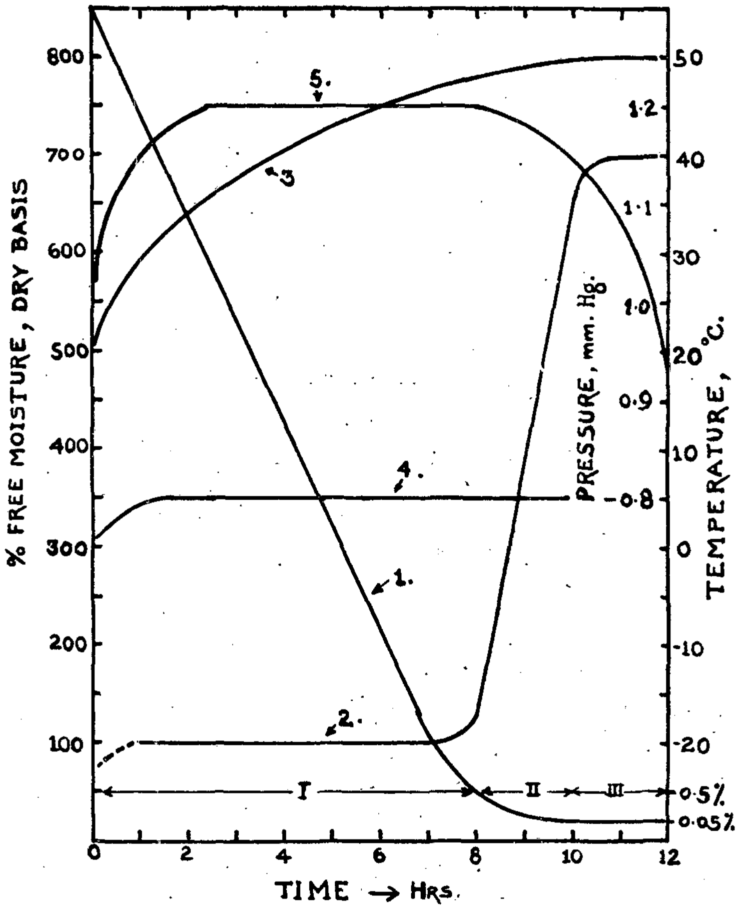

FIG. 2.

The rate of drying during the constant rate period is given by the equation

$$dW/A/d\theta = \frac{U\,\Delta\,t}{\lambda} \;......(10)$$

where, Δt = temperature difference between the ice and the heating surface,

λ = latent heat of sublimation and

U = overall heat transfer co-efficient, which has a value of the order of 1 B.T.U./(Hr.) (sq. ft.) (°F).

The efficiency of freeze or sublimation drying depends upon two factors: firstly, on the rate of removal of the vapour generated and secondly, on the rate of supply of heat, which must be high enough to maintain efficient sublimation rate without allowing the temperature of the material to drop, but not high enough to melt the material.

The commercial success of sublimation drying depends largely on the efficiency of the evacuation system which consists of adequate ducts for the passage of a large volume of vapours generated, glands and traps to prevent inleak of air and a pumping system. For small capacities, oil-sealed rotary vacuum pumps can be used with nearly 100% efficiency at 100-200μ pressure. These efficiencies are obtained by the removal of all condensible vapours on the high pressure side of the pump, thus reducing back pressures to a minimum. This means a virtual addition of another stage.

The vapours may be removed by a variety of methods. A chemical dessicant such as P_2O_5 may be used. This method, however, is used only on laboratory scale. The vapours may also be removed by chilling them by refrigerants. The cold trap may be replaced by a rotary condenser where the ice formed is continuously scraped off. Or alternatively, they may be absorbed in a low vapour pressure brine such as chilled LiCl or physically adsorbed on materials such as silica gel. The last two methods are ideally suitable for continuous operation. For large capacities, however, steam ejectors, usually in 4-5 stages, are preferred because of their economic operation.

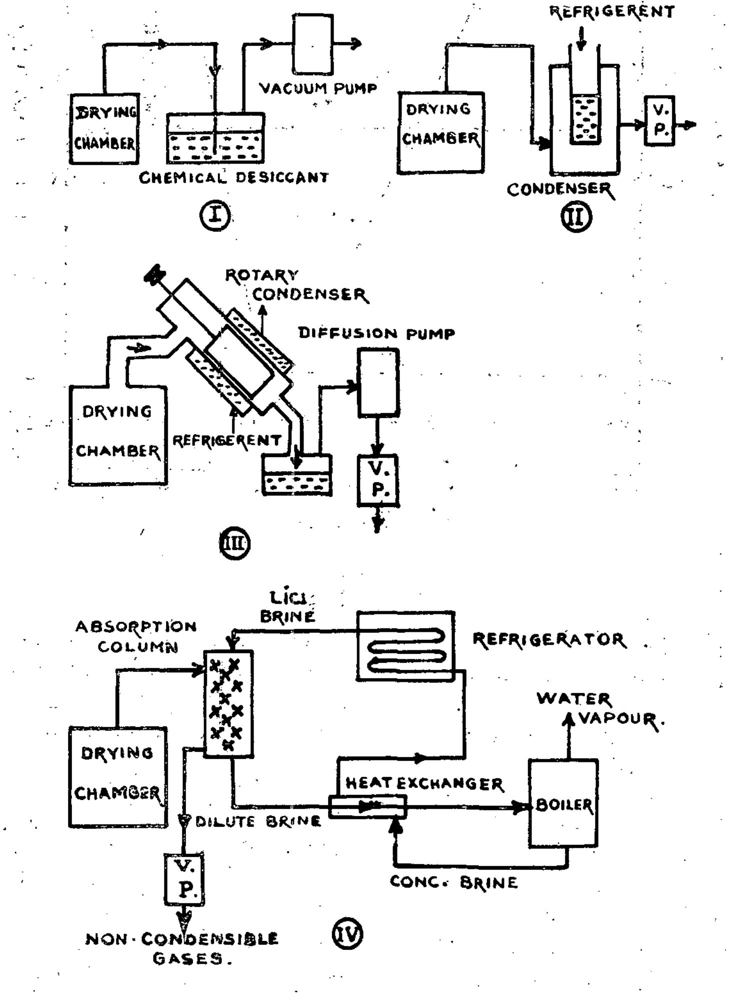

Fig. 3.

Heat is supplied to the frozen material either by conduction through coils in which water at temperatures up to 200°F circulates or by radiation; the latter, in addition to being the more efficient system, offers less danger of overheating and spoiling the work.

Manufacturers of pharmaceuticals and biological products prefer the drying chamber method of freeze drying because thereby the material is dried in its container and hence can be sealed aseptically *in situ*, which is a major consideration. The manufacturers of dried foodstuffs, on the other hand, prefer to work in continuous operation. Two methods are employed. The material to be dried is previously concentrated and sprayed on the wall of the drying chamber where it is frozen and when dried, the product is scraped off and collected in bins. Otherwise, the material is previously frozen and spread on endless belts made of an infra-red transparent material (such as "tefloglass"), where it dries. Thermal energy is supplied by infra-red lamps.

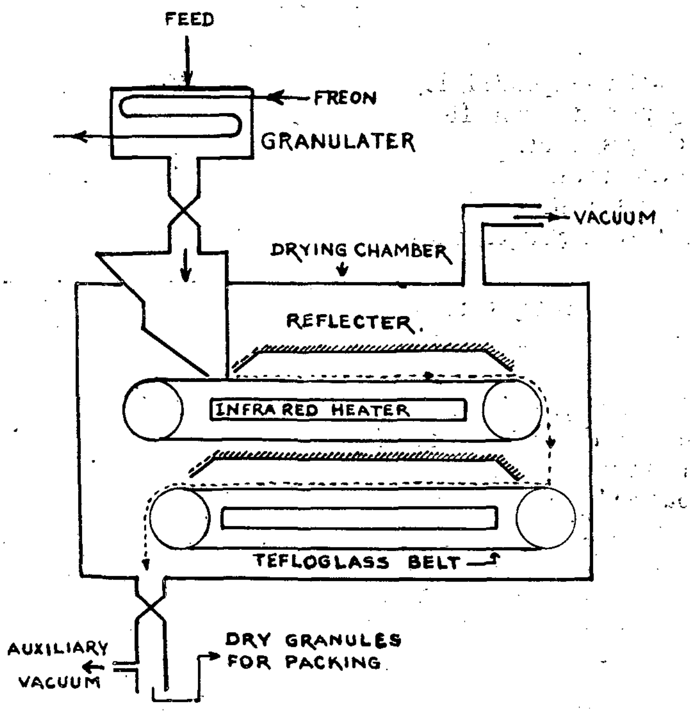

FIG 4.

Freeze drying offers a number of advantages over conventional drying methods. Since the temperature of the material to be dried is well below that at which many substances undergo a chemical change it is possible to dry labile substances such as blood plasma, viruses and pharmaceuticals without the danger of their decomposition. For the same reason no bacterial growth or enzymic change can occur. The final moisture content is reduced to a minimum (about 0.05%), hence the final product stores well, resisting attacks by bacteria and enzymes even at room temperatures. There is a minimum loss of volatile components, hence all the aroma and taste of the original product is retained in the dried powder, which makes the process highly sought after by manufacturers of food products such as orange juice and soluble coffee powder. If properly carried out, no bubbling or foaming of the material can occur in this process, therefore no denaturation of products such as gelatine and protein products can take place. Because the solute remains evenly distributed without undergoing contraction as the solvent sublimes, the final product assumes a sponge-like structure in which form very efficient dispersion and/or solution of even lyophobic sols can take place, a highly desirable property in injectibles and dyes. Absence of oxidation of the products during drying is an additional advantage.

There is one drawback, however, namely the high cost of installation and fabrication, which is ultimately reflected in the cost of operation, as will be seen from table I. The cost is about five to ten times as high as that incurred by conventional drying methods, and in the long run, it will be the deciding factor.

Table 1

Basis 1 hour.

1. Capacity water evaporated, lb.	10	50	200
2. Operating pressure, mm. Hg.	1	1	1
3. Labour charges, units.	1.25	1.25	1.25
4. Depreciation, units.	0.26	0.92	1.92
5. Steam charges, units.	0.06	0.3	1.6
6. Power and water charges, units.	0.3	0.6	1.7
Total charges, units.	1.87	3.07	6.47
7. Cost of water evaporated, units/lb.	0.187	0.062	0.032

In short, by freezing, it is possible to dry and maintain the potency of biologicals, solubilities of pharmaceuticals and the flavour and aroma of food products. But because of the high costs of operation and installation, the process is used on industrial scale only when the quality of the product and not the cost is the deciding factor.

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