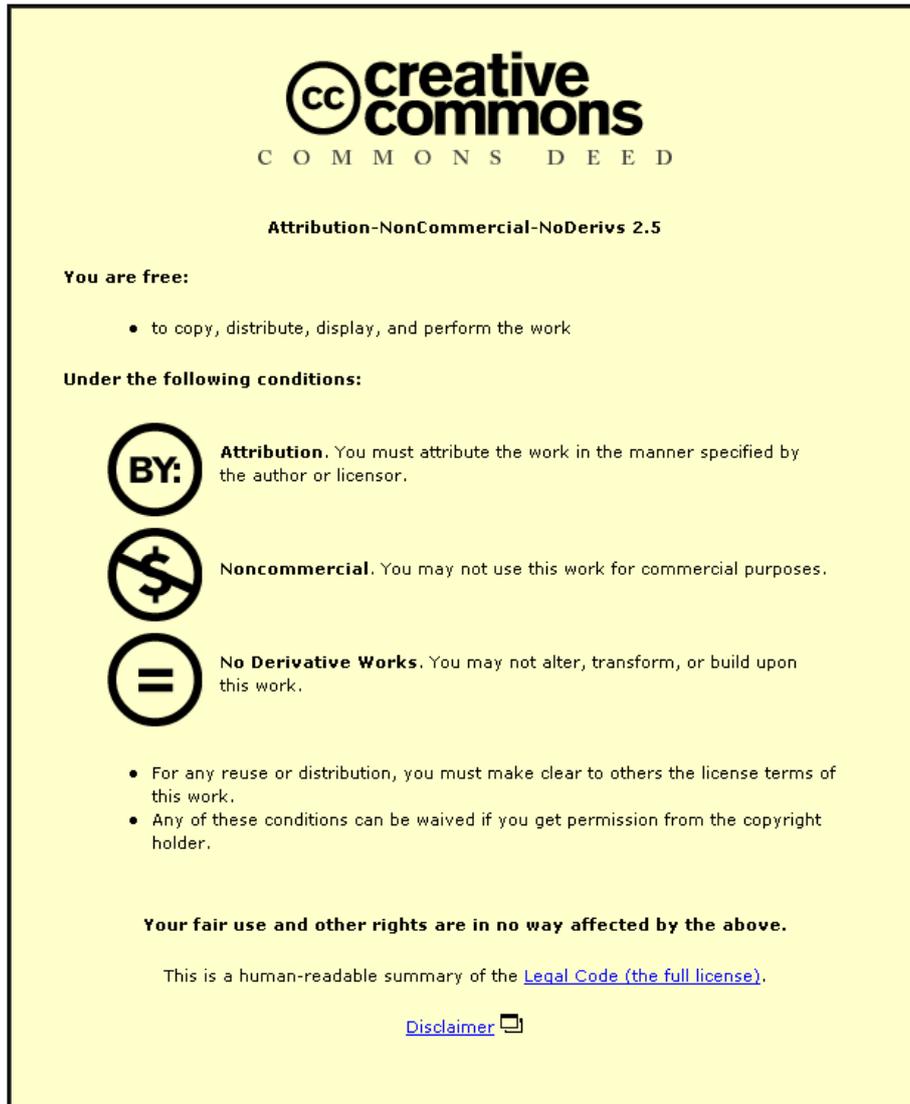


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Developments in fluidised bed freeze drying

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Freeze drying is a well-known, but relatively little used, method of food preservation. As its name implies, freeze drying involves first freezing the food to form ice crystals which then undergo direct sublimation from the solid to the vapour phase (primary drying), along with desorption of residual moisture from within the food matrix (secondary drying).

Freeze-dried foods can be found in any UK supermarket. By far the most common is freeze-dried coffee, but freeze dried herbs are also available and freeze-dried fruit is found in certain brands of breakfast cereal. These all have in common a convenience aspect when compared to fresh produce (avoiding, for example, the effort of preparing fresh filter coffee or maintaining a constant stock of fresh herbs or fruits), and a quality aspect when compared to spray-dried coffee or air dried-herbs and fruit.

A unique feature of freeze dried foods is their porosity, which originates from the formation of ice crystals in the freezing step. When these sublime they leave behind voids to create a porous structure. However, to maintain this structure the food must be held below its so-called “collapse temperature” (closely related to the glass transition temperature) so that the material remains in a rigid (glassy) state as the ice sublimates. For many foods, particularly those with a large proportion of low molecular weight components (such as sugars), the collapse temperature can be below -30°C and this restricts the temperatures used at the start of freeze drying. However, as the material dries out from secondary drying the glass transition temperature and hence the collapse temperature rises. The goal of freeze drying is to dry the product to the point that it can still maintain the porous structure at room temperature (and preferably higher). An advantage of the porosity is the ability of a food to rehydrate quickly via capillary action, and the rehydration properties of freeze-dried foods are usually superior to those dried by other methods. This porosity also aids the freeze drying process by providing pathways through the material for vapour diffusion.

Freeze dried foods are generally regarded as being of higher quality compared to foods dried at higher temperatures, retaining better colour, texture and flavour. However, considering the total amount of food sold in supermarkets, the amount of freeze-dried produce sold is very low. In the 1960s there was huge enthusiasm for the technology as a potential panacea for the mass production of high quality shelf stable food items, but this has never been fully realised.

The major problem is the cost of production. This arises from the long drying times and the large energy duties required. Most commercial freeze driers use either radiative and conductive heating of trays to provide the latent heat of sublimation required to convert ice to water vapour. However, an equivalent amount of cooling duty is required to operate condensers in the freeze dryer. These provide very low temperature surfaces which remove water vapour from the freeze dryer and keep the very low vapour pressures of water needed for freeze drying to occur. Condenser temperatures of -60°C are typical, so the energy requirements for this refrigeration duty are large.

Conventional freeze dryers are operated under vacuum, but contrary to popular belief the main purpose of vacuum pumps is the removal of non-condensable gases. The amount of water removed by the vacuum pumps is tiny compared to that taken out by the

condensers, but by operating under vacuum the water molecules can reach the condensers relatively unhindered by collisions with air molecules.

Atmospheric freeze drying

However, there is no absolute requirement for freeze drying to take place in a vacuum. All that is needed is that the vapour pressure of water is below the saturation vapour pressure of ice at the temperature of the food being dried. This means that freeze drying can be performed with air at atmospheric pressure just as long as it is very dry (i.e. the dew point is below the temperature of the food). Indeed this was achieved many centuries ago by the Incas who preserved food by freeze drying on the mountain slopes of the Andes. The first published demonstration of atmospheric freeze drying however was not presented until the 1950s (Meryman, 1959). Atmospheric freeze drying is nevertheless a very common occurrence as it occurs in freezer cabinets from frozen food that has not been properly sealed in its packaging – it is better known as “freezer burn”.

Fluidisation

The ability to freeze dry using air opens the door to other potential methods of freeze drying, including fluidisation. Fluidised beds are well known to provide very efficient convective heat and mass transfer, and it is possible to freeze dry foods by contacting them with cold, dry gas in a fluidised bed.

There is currently a commercial atmospheric freeze drying plant in Hungary operated by Aroma Dry Kft. which uses exactly this principal to freeze dry peas and sweet corn produced from local farms. The process recycles the air exiting the fluidised bed by passing through a condenser to reduce the dew point and then warming to reduce the relative humidity. Such a system offers scope for significant heat integration, and thus energy savings, by using the heat from the condenser to supply heat to the reheater via a heat pump (as the heat needs to be pumped up a temperature gradient). Figure 1 shows a possible arrangement.

However, drying times are still long for this process (of the order of 20 hours for peas). For freeze drying to be more attractive it makes sense to examine ways in which drying times can be shortened

Speeding up the process – spray freeze drying

As drying times vary approximately with the square of the sample thickness, one way to increase drying rates is to reduce the dimensions of the material. Whilst this may not be desirable for peas, it is perfectly feasible for liquid based products such as coffee. A liquid feed stream can be atomised into small droplets in a manner similar to spray drying, and then rapidly frozen to form an icy powder which is subsequently freeze dried. This is called the spray-freeze-drying technique

It is common for this freeze drying step to be performed using a conventional shelf based freeze dryer in pharmaceutical applications to produce powders with the very porous morphology. However this does not take advantage of the small particle size as heat is only transmitted by conduction through the powder bulk.

The most recent work in this field has been by Mumenthaler and Leuenberger (1991) who used cold desiccated gas to first freeze and then dry the spray in an integrated fluidised bed at atmospheric pressure. Due to the small particle sizes produced, drying times of two hours were achieved, i.e. an order of magnitude faster than conventional freeze drying.

A problem with the atmospheric spray-freeze-drying process, however, is the very large quantities of cold dry gas that need to be circulated through the bed. As freeze-drying must be performed below the collapse temperature of the material (which can be as low as -30°C) the ice phase exerts an extremely low vapour pressure, e.g. ~ 40 Pa at -30°C .

Even in a best case scenario whereby the exit gas of the fluidised bed is fully saturated, according to the Ideal Gas Law approximately 4000 kg of bone dry gas is required to pass through the bed in order to sublime 1 kg of ice.

Although this can be partly offset by recirculating (and drying) the gas, this is a huge quantity of gas. The refrigeration and drying requirements for such a mass of gas to meet the required specification are considerable and severely damage the economics of the process.

A further consideration is that to pass this mass of gas through the bed in a reasonable time – a few hours perhaps - requires a very large gas volume flow rate and hence velocity. This leads to particles being swept out of the fluidised bed where they must be caught by a gas-filter (as observed by Mumenthaler and Leuenberger, 1991). Although this can still dry the particles, it does render the fluidised bed, with its high heat and mass transfer capability, redundant.

Using less air - sub-atmospheric spray-freeze-drying

One means of alleviating the problem of circulating large mass flows of gas is to apply a partial vacuum to the fluidised bed process. The gas is, after all, merely an inert heat transfer medium and plays no part in determining the driving force for mass transfer.

As an example, if the process is operated at -30°C and at 0.1 bara then the mass of gas required would be approximately 400 kg (one tenth of the amount at 1 bara). Although a vacuum capability increases the capital cost and operational difficulty of the plant, it does significantly reduce the mass of dry gas required. The lower density at lower pressures also reduces the inertial (non-viscous) drag forces on the particles making them less likely to be swept from the bed.

To test this out a spray-freeze-drying rig has been constructed at Loughborough, in which the fluidised bed freeze-drying section is capable of operating at reduced pressures (see Figure 2). This was made possible by the award of an EPSRC (UK research council) fast stream grant (grant ref: GR/N16662). The equipment is in two parts: a spray freezing chamber and a separate sub-atmospheric fluidised bed freeze drying unit. Spray frozen particles are first formed by spraying the concentrate through a heated hydraulic nozzle into a spray chamber cooled by nitrogen gas from a 500 litre liquid nitrogen dewar. The frozen particles are collected from the outlet of the chamber in cooled, polystyrene containers.

The spray frozen particles are then loaded into the fluidised bed freeze dryer, which consists of a polycarbonate cylinder inside a stainless steel vacuum vessel. The depth of the bed is typically 1 cm, which yields a product mass of around 5 g. The fluidisation gas is provided from the liquid nitrogen dewar via a trim gas heater and evacuated via a vacuum pump ($100\text{ m}^3\text{h}^{-1}$).

The temperatures of the inlet and outlet gas are measured using thermocouples. The dewpoint (humidity) of the outlet gas is also monitored to provide an indication of the drying rate. The inlet temperature must crucially be maintained below the collapse temperature of the material, otherwise the porous structure of the product will be lost.

Experiments to date have used maltodextrin, tea, coffee and whey, some of which have very low collapse temperatures (-30°C). Even at these temperatures drying times of the order of one to two hours have been achieved. Scanning electron microscopy images of the porous products are shown in Figure 3. This provides a low powder bulk density of the order of 200 – 300 kg m⁻³.

Future developments

Our two current aims are to find areas of application in which spray freeze-dried products confer a quality and functional advantage over those from other manufacturing methods and to investigate areas for process optimisation and cost reduction.

In the current Loughborough rig the nitrogen gas only passes once through the fluidised bed before being evacuated to the environment. In the commercial atmospheric freeze drying process described earlier, the fluidising gas is recycled using a heat pump to integrate the energy flows and produce significant energy savings. We are therefore wishing to test the technical feasibility and economic effectiveness of recycling the drying gas via a heat pump in a sub-atmospheric spray freeze drying process.

Paradoxically for a process that has been traditionally viewed as a high energy, there may be significant environmental benefits from a successful low energy freeze drying process, as dried food products are (once dried) very environmentally friendly. They do not require refrigeration (which requires energy), and they do not spoil even if stored over long periods of time which eliminates food wastage. The removal of large amounts of water also means that transport and storage is also much easier. Such considerations are likely to become ever more important in the future.

To take this further we are currently leading a consortium of universities and companies in a DEFRA (Department for Environment Food and Rural Affairs) funded FeasibilityLINK project. Our academic partners are Dr Judith Evans of the Food Refrigeration and Processing Engineering Research Centre (FRPERC) at the University of Bristol, and Dr Bettina Wolf of the Division of Food Sciences at the University of Nottingham. Our industrial partners are Air Products, Kerry Ingredients and CCC Consultants.

The objective of the FeasibilityLINK scheme is to identify suitable materials that may have commercial potential to be freeze dried using this method. We are currently testing dairy powders, tea and coffee powders, encapsulated flavours and extracts, and tomato and meat based powders. This is envisaged to pave the way for a full LINK scheme which would involve constructing a hygienic demonstrator rig using heat pump technology to spray-freeze-dry kilogramme quantities of foods. The products from the demonstrator rig will be directly tested in food formulations by industrial partners. We will be wishing to add one or two more partners for the full LINK scheme, so enquiries from interested parties are very welcome!

We hope to demonstrate that sub-atmospheric spray-freeze-drying has the potential to produce food products more quickly and cheaply and using much less energy than is currently possible by conventional vacuum freeze-drying processes.

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Acknowledgements

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Figures:

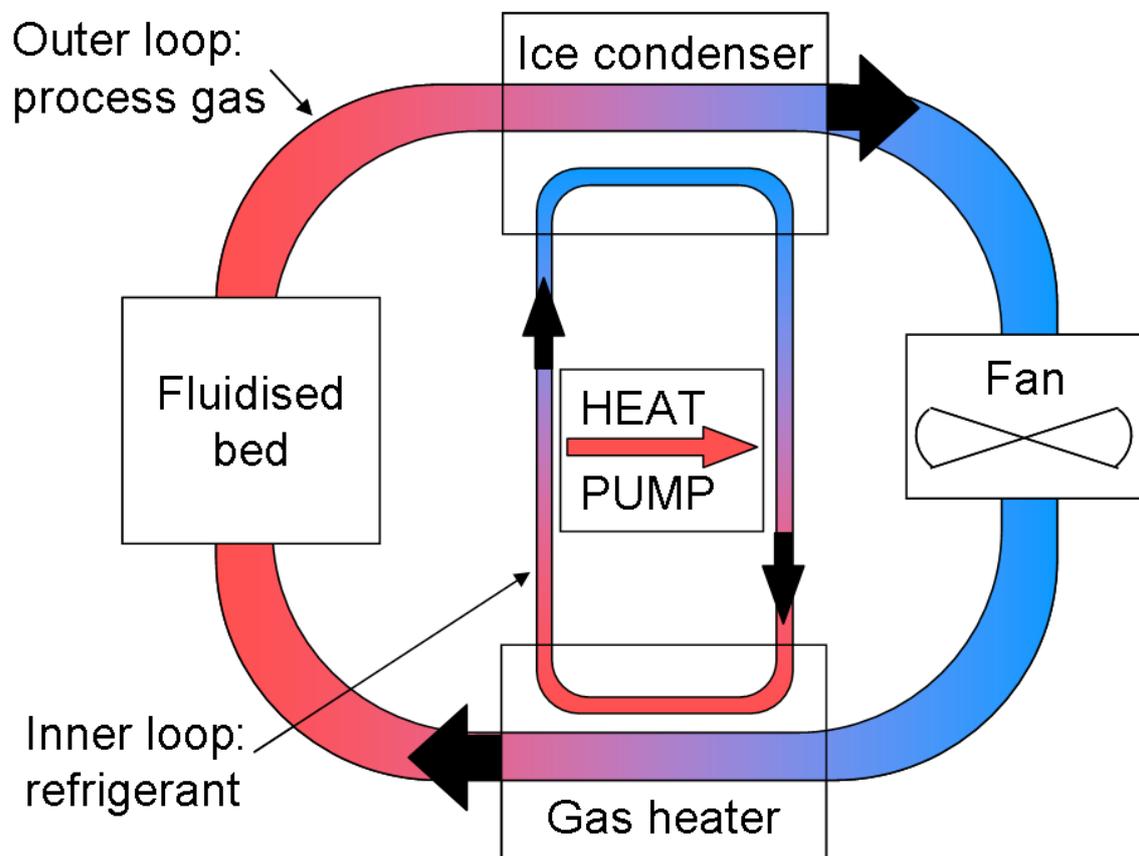


Figure 1. Schematic of a method of integrating heat flows using a heat pump in the fluidised bed freeze drying process.



Figure 2. The spray-freeze-drying apparatus used at Loughborough. Spray-freezing initially occurs in the large (black) chamber located on the right of the photo. The spray frozen particles are collected from the base of this chamber and transferred to the sub-atmospheric fluidized bed freeze-drying apparatus on the left of the photo.

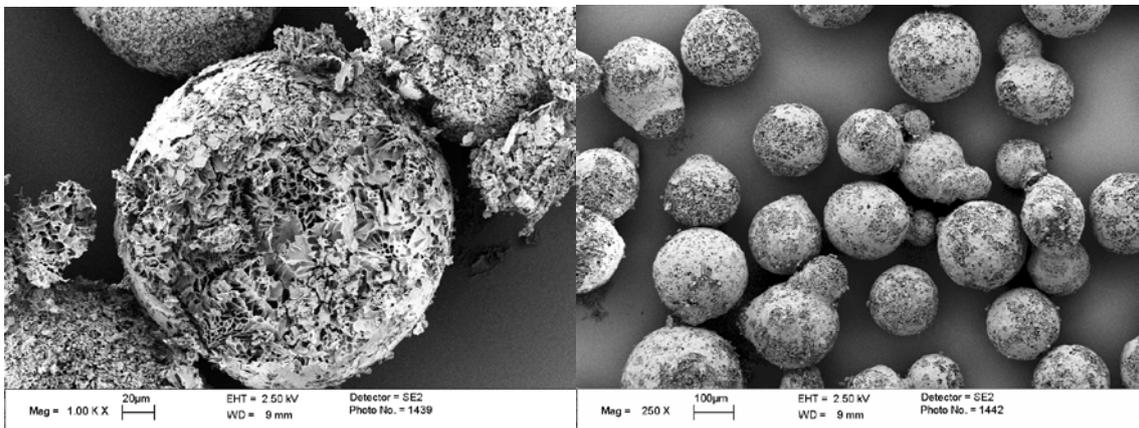


Figure 3. SEM images of spray-freeze-dried (a) maltodextrin and (b) tea. Fields of view are 350 µm and 1.5 mm respectively.

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