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Freeze-drying and its application to some Finnish agricultural products

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Abstract. This study deals with the freeze-drying of berries, vegetables, roots and pork produced in Finland. The products were freeze dried whole, in slices or cubes. The pressure in the drying chamber was 10—20 Pa and the maximum surface temperature +23— +40°C. The products were packed after freeze-drying into glass jars under air and in polyester-aluminium-polyethene foil under vacuum or nitrogen atmosphere. The drying temperatures and drying times were measured. During storage for 4 months, the differences in quality were evaluated.

The drying times ranged from 8 to 25 hours. The moisture content of the dry products was 1—3 %. The sublimation rate at the beginning of the drying process was very rapid, being thereafter constant. Bilberries, carrots and radishes collapsed during drying. Most of the products dried moistened and underwent aroma changes during storage in glass jars under air. The products packed under vacuum or nitrogen atmosphere proved stable.

Introduction

Freeze-drying is a dehydration method which has not yet been commercially applied by the Finnish food industry. The basic principle of freeze-drying involves sublimation of water at low temperatures and pressures which are determined by the physical properties of water. The temperature and pressure of the system have to be below those of the triple point of water. The pressure is usually between 10 and 100 Pa and the highest surface temperatures between +20 and +60°C. Freeze-drying as a preservation method has been carefully reviewed by King(1970), Karel (1975) and Mellor (1978).

Flink and Knudsen (1983) have defined freeze-drying as a three-step process, these steps being freezing, drying and dehydration of the material. In freezing, the temperature of the material is lowered and the water is solidified, in drying the water is sublimated and in dehydration the water bound is removed. During freeze-drying, the material to be dried involves two regions, the dry layer and the ice layer (Karel and Flink 1973). Heat is usually supplied by radiation to the surface of the material and is conducted through the drying region to the interface where the frozen water is sublimated. This heat is used to sublimate the water bound in the drying region, to superheat the vapor passing through the drying region and to increase the internal energy of the material in the drying region (MELLOR 1978).

The most common configuration, usually the most desirable one, of materials to be freeze-dried is the slab form. In freeze-drying the surface temperature of the material is rapidly brought to the maximum permissible level and maintained at this level by adjusting the radiator temperature according to the specific program. In practice the changes are done by feedback devices. The maximum surface temperature is usually dictated by quality considerations, especially those related to flavor and colour. The temperature of the frozen layer must be maintained below a critical level which depends on the nature of the product and its thermal history (KAREL 1973).

Compared with other food dehydration methods, freeze-drying is superior in retaining the shape, size, colour, aroma and nutrition value of food material. The retention of these properties is a function of preparing and freezing as also processing conditions. FANG et al. (1971) have studied the effects of pretreatment and freezing on the quality of freeze-dried mushrooms. KAREL and FLINK (1973) studied freeze-drying and the effects of freezing on the quality of freeze-dried food. MÄLKKI and HEINONEN (1978) have studied freeze-drying of onions. FLINK (1982) has reviewed the effect of processing on the nutritive value of freeze-dried food. Freeze-drying has been widely used in the manufacture of instant coffee and tea. It has also been applied to industrial meat and vegetable processing.

Finnish agricultural products are generally considered of high quality. Freeze-drying is a gentle dehydration method which can be used to retain this quality also in dry products. The purpose of this study was to establish the suitability of some Finnish food products to freeze-drying.

Materials and methods

In this study, a number of berries, vegetables, roots and pork were freeze-dried. The products were purchased fresh from local dealers in 1984. The materials and their pretreatment conditions are given in table 1.

Table 1. Materials and their pretreatment.

Material	Form	Pretreatment
bilberry	whole berries	no pretreatment
strawberry	whole berries	no pretreatment
carrot	cubes, 1 cm^3	washed, scaled, cut
celery	cubes, 1 cm^3	washed, scaled, cut
horseradish	cubes, 1 cm^3	washed, scaled, cut
parsnip	cubes, 1 cm^3	washed, scaled, cut
potato	cubes, 1 cm^3	washed, scaled, cut
radish	cubes, 1 cm^3	washed, scaled, cut
rutabaga	cubes, 1 cm^3	washed, scaled, cut
turnip	cubes, 1 cm^3	washed, scaled, cut
broccoli	slices	washed, cut
brussels sprouts	halves	washed, cut
cabbage	slices	washed, cut
cauliflower	slices	washed, cut
pork	cubes, 1 cm^3	cut

Freezing conditions

Freezing conditions are given in table 2; thereby freezing was done in air tunnel, flow freezer and in a freeze-drier.

Table 2. Freezing conditions in various experiments.

Material	Freezer	Temp. °C	Time h
bilberry	freeze-drier	—40	4
strawberry	freeze-drier	—40	4
carrot	freeze-drier	—40	4
celery	air tunnel, flow freezer	—45	2
horseradish	freeze-drier	—40	4
parsnip	air tunnel, flow freezer	—45	2
potato	freeze-drier	—40	4
radish	freeze-drier	—40	4
rutabaga	air tunnel, flow freezer	—45	2
turnip	freeze-drier	—40	4
broccoli	air tunnel, flow freezer	—45	2
brussels sprouts	air tunnel, flow freezer	—45	2
cabbage	air tunnel, flow freezer	—45	2
cauliflower	air tunnel, flow freezer	—45	2

Freeze-drying

An Edwards model EF 10/10 freeze-drier, shelf area 2 m^2 and condenser capacity 15—20 kg, was used for drying of the products. Refrigereant R502 was used for cooling of the shelves and condenser. The shelves can be electrically heated to a preset value. Vacuum was maintained with an Edwards model ED660 rotary vacuum pump with a displacement effect of 39.6 m^2h^{-1}.

In all experiments, the products were transferred onto prefrozen (t < —40°C) shelves of the freeze-drier. The products were loaded to the drying chamber on aluminium trays. All products except pork were loaded immediately after freezing. Pork was stored frozen at —20°C for four weeks before freeze-drying. The pressure in the drying chamber was lowered to about 10 Pa before heat was applied to the product. The condenser was cooled to about —50°C, and the pressure in the drying chamber was 10—20 Pa. The drying parameters are given in table 3. The pressure in the drying chamber was measured with an Edwards pirani 11 gauge. The temperature of the shelves, product surface and ice layer were measured with Cu-CuNi-thermocouples which were connected to a 12 point Honeywell recorder with a measurement range of —50°C to +100°C. The end point of the drying process was determined by the preset temperature whereafter the vacuum was broken with air. The final moisture content was measured by placing 2—3 g of the materials in an air oven at +105°C for 18 h and determining the weight loss.

Packing of the products

The products were packed immediately after freeze-drying into glass jars under air and in polyester-aluminium-polyethene foil under vacuum or nitrogen and stored at room temperature for 4 months.

Additional experiments

Turnip was freeze-dried as raw and blanched cubes (1 cm^3) to examine the drying behaviour of turnip at different temperatures. Pork was freeze-dried at a surface temperature of +40°C in five experiments to examine differences in various batches, each batch weighing 2.5 kgs.

Results

Freeze-drying

The temperatures of the products during freeze-drying are given in figures 1—3. The drying times were 18 to 25 hours. Figure 4 shows the temperatures of turnip during freeze-drying at different surface temperatures. The drying details of pork are given in table 4 and figure 5.

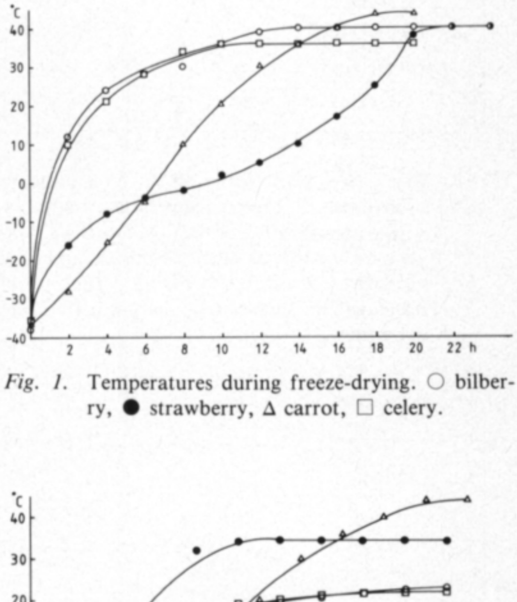

Fig. 1. Temperatures during freeze-drying. ○ bilberry, ● strawberry, Δ carrot, □ celery.

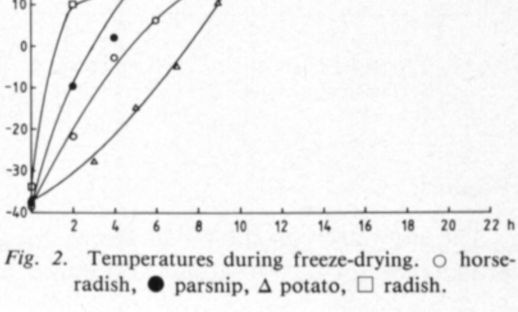

Fig. 2. Temperatures during freeze-drying. ○ horseradish, ● parsnip, Δ potato, □ radish.

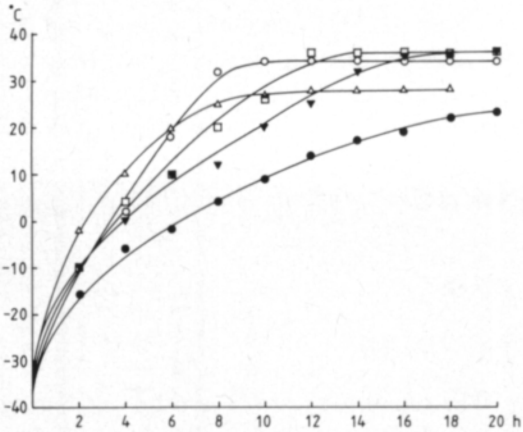

Fig. 3. Temperatures during freeze-drying. ○ rutabaga, ● turnip, Δ broccoli, □ cabbage, ▼ cauliflower.

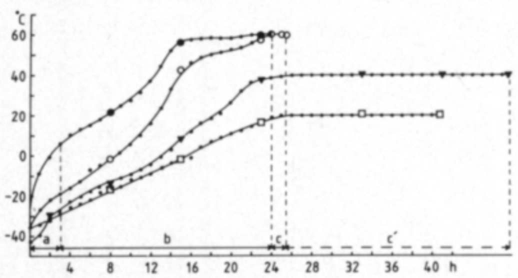

Fig. 4. Freeze-drying of turnip. ● shelf temperature in first case, ⊙ surface temperature +60°C, ▼ surface temperature +40°C, □ surface temperature +20°C, a) rapid sublimation, pressure increases, b) constant sublimation period, c) and c') maximum surface temperature period, dehydration.

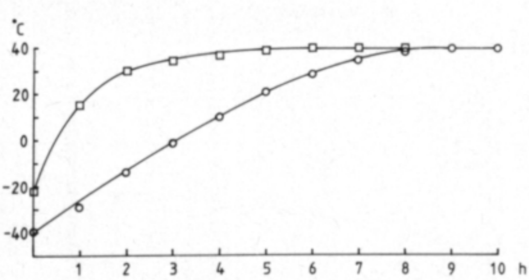

Fig. 5. Freeze-drying of pork. □ shelf temperature, ○ surface temperature.

Products

The moisture content of all freeze-dried products was 1—3 %. Observed changes in freeze-drying or in freeze-dried products are given in table 5. The products packed under nitrogen atmosphere or under vacuum proved more stable than the products packed under air.

Table 3. Temperatures in freeze-drying.

Product	Initial temp. °C	End temp. °C
bilberry	—38	40
strawberry	—38	40
carrot	—37	43
celery	—36	34
horseradish	—36	23
parsnip	—38	34
potato	—37	43
radish	—36	23
rutabaga	—38	34
turnip	—36	23
broccoli	—37	28
brussels sprouts	—37	28
cabbage	—36	34
cauliflower	—36	34

Table 4. Temperatures in different experiments during freeze-drying of pork.

Time h	Experiment Temperature °C				
	I	II	III	IV	V
0	—40	—39	—40	—38	—40
1	—35	—25	—34	—24	—29
2	—10	—10	—24	—14	—13
3	0	2	—18	8	0
4	16	9	2	17	5
5	24	18	12	24	28
6	30	24	21	31	36
7	38	30	29	35	40
8	40	36	35	39	40
9	42	38	41	40	40
10	42	40	41	40	40

Discussion

The purpose of the study was to establish the suitability of some Finnish agricultural products to freeze-drying. Most of the products tested proved suitable for freeze-drying. However, commercial freeze-drying may be applied only after specific processing conditions have been determined for each product.

Table 5. Changes in products during freeze-drying and storage.

Product	Freeze-drying	After freeze-drying	Storage in glass jars
bilberry	collapsed	moist, rapid moisture uptake	aroma changes
strawberry		moisture uptake	aroma changes
carrot	collapsed	moisture uptake	aroma changes
celery		moisture uptake	aroma changes
horseradish			colour turns red
parsnip		moisture uptake	aroma changes
potato		moisture uptake	aroma changes
radish	collapsed	moisture uptake	aroma changes
rutabaga		moisture uptake	aroma changes
turnip		moisture uptake	aroma changes
broccoli		moisture uptake	aroma changes
brussels sprouts		moisture uptake	aroma changes
cabbage		moisture uptake	aroma changes
cauliflower		moisture uptake	aroma changes

In this study, the drying times for various products were rather long, ranging from 8 to 25 hours, the longest time being applied to drying of strawberries. Strawberries were dried as whole berries up to 3 cm in diameter.

At the beginning of freeze-drying, sublimation is very rapid, and the pressure in the system increases. Thereafter the sublimation rate becomes almost constant, and the rise of temperature in the material is slower until maximum surface temperature is reached, and the rest of the moisture is evaporated. This is shown clearly in the case of turnip and pork (Figures 4 and 5).

As pointed out by KING (1975), collapse of solid foods manifests as excessive shrinkage and poor rehydration. In this study, excessive shrinkage occured only in the radish. The water content of radish is relatively high, about 93 % (TURPEINEN 1975), being the highest of the products studied. This may explain the more intensive collapse in radish than in other products. On the other hand, the membranes and cell walls of radish may be weaker than in other products studied. However, the sublimation temperature of radish was much higher than that of other products except bilberries. The behaviour of bilberries is similar to that of radish according to their freeze-drying curves (Figures 1 and 2). It is possible that both of these materials tend to melt during freeze-drying. The carbohydrate content of all the products studied was high. Usually carbohydrates tend to promote collapse (KING 1975). TSOUROUFLIS et al. (1976) observed that the higher the concentration of the initial solution, the higher the collapse temperature. In this respect the bilberries and radishes differ very much from each other, and collapse of bilberries might be due to increased pressure inside the bilberries during freeze-drying. This can also be observed in the very rapid rise of temperature at the beginning of freeze-drying. It is evident that also other berries with skin behave similarly.

The amount of uptake of water by the dried product is important in determining the quality of the product and, if there has been any significant biochemical change, e.g. denaturation of protein and hardening of the surface, rehydration will be poor (HOLDSWORTH 1971). HOLDSWORTH (1971) has also pointed out that the histological changes which take place during freeze-drying revealed that whereas total collapse of the cell structure was common to all air-dried vegetable tissue, the freeze-dried products showed no cell collapse, only some wall rupture. The main observation in this study was that most of the freeze-dried products did not undergo structural changes.

In this study, most freeze-dried products had a very rapid moisture uptake from the air, and if stored unpacked, stickiness, softening and caking of the products were observed. It is possible that this phenomenon is a function of carbohydrate content of the products. The carbohydrate structure and structure transitions are very important in the agglomeration, caking and flow of powder materials (Flink 1983). In sugar products, slight caking is observed at a_w between 0.25 and 0.30 (Quast and Teixeira neto 1976).

This study revealed clearly that the freeze-dried products must be packed under vacuum or nitrogen immediately after freeze-drying to avoid moisture uptake and oxidation, and thereby prevent excess aroma and colour losses during storage. The findings of Berlin et al. (1966) and Bishov et al. (1971) also support these results. This study showed the benefits of freeze-drying to maintain the original freshness of raw materials after processing and rehydration. Bilberry was the only product which proved unsuitable for freeze-drying, because its skin did not allow proper sublimation of ice, and this resulted in a poor quality and a moist final product. Collapse and aroma retention in freeze-drying are dependent on the moisture and temperature gradients during freeze-drying. Most of the work was done with model systems (Flink and Karel 1970, Flink et al. 1974, To and Flink 1978 a,b, Tsourouflis et al. 1976), and little attention was paid to solid foods. Further studies will be conducted to determine the optimal conditions for freeze-drying of solid foods and to overcome the problems related to packing and storage.

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SELOSTUS

Joidenkin suomalaisten maataloustuotteiden soveltuvuudesta pakkaskuivaukseen

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Tutkimuksessa pakkaskuivattiin kotimaisia marjoja, vihanneksia, juureksia ja sianlihaa. Materiaalit pakkaskuivattiin käsittelemättöminä, viipaloituina ja kuutioituina. Kuivauskammion paine kuivauksen aikana oli 10—20 Pa ja suurin pintalämpötila +23°C—+40°C. Kuivatut tuotteet pakattiin pakkaskuivauksen jälkeen lasitölkkeihin sekä polyesteri-alumiini-polyeteeni pusseihin tyhjiöön tai typpikaasuun. Pakkaskuivauksen aikana mitattiin kuivauslämpötilat ja kokonaiskuivausaika. Tuotteiden laatua seurattiin neljä kuukautta.

Pakkaskuivausajat olivat 8—25 h. Lopputuotteiden kosteuspitoisuus oli 1—3 %. Sublimoitumisnopeuden muutos pakkaskuivauksen alussa oli suuri, minkä jälkeen se oli vakio. Mustikka, porkkana ja retiisi kärsivät pakkaskuivauksessa rakennemuutoksia. Useimmat suojaamattomat tuotteet kostuivat pakkaskuivauksen jälkeen. Lasipakattujen tuotteiden aromi muuttui voimakkaasti varastoinnin aikana. Tyhjiö- ja typpipakatut tuotteet osoittautuivat stabiileiksi.