PRACTICAL EXPERIENCES OF AN OPERATIONAL SIZE FREEZE DRYING EQUIPMENT

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ABSTRACT*: In Hungary there is a revolutionary new method, a joint application of an air-turbo refrigerator and sublimation drier. The complex equipment is suitable for production of an excellent quality dried food. The study shows the construction of the system and demonstrates some examination of the typical parameters, for example the drying curve, chemical components, rehydration and surface hardness.*
KEYWORDS*: refrigerator, parameters, freeze drying*

1 INTRODUCTION

Nowadays, in the 21st century, there are some special quality requirements against the dried vegetables and fruits, for example stable microbiological, physical and chemical parameters, moreover production of excellent storing, packaging and transporting features (Verghese, 2010). Besides these characters, the product should have the best chemical components for making functional foods and supplements. Fulfilling the above mentioned preservation demands, only a few drying methods is suitable to produce the necessary quality. The most gently and healthy way is the vacuum freeze drying (Antal, 2017). Freeze drying is the ideal method due to minimal shrinkage resulting in a porous product with excellent rehydration capacity, soft texture, bright color, superior taste (Ratti, 2001). In Hungary an extended research work has been going on to select different dehydrated fruit and vegetable powders since 2004, which are suitable for production of functional preventive and curative nutrition and supplements.

The main goal is to prevent or cure some serious illnesses, caused by negative hormonal change. The large scale drying equipment, applied in our experimental work, is a new method, which follows the dehydration process during lyophilisation (freezing, sublimation, post-drying), but the freezing happens not by the conventional compressor cooling unit, but with the help of an air-turbo refrigerator (Antal, 2009). The present paper details the large capacity sublimation drying machine, examination of mass-transport, moreover introduce some special quality parameters, as the chemical components, rehydration capacity and product hardness.

2 MATERIAL AND METHODS

2.1 Raw materials

Production of supplements happened by the following dried vegetables and fruits: broccoli, tomato, pumpkin, green pepper, colewort, carrot, cauliflower, orange, pineapple, apple, grape. In our study we should like to present the results of pumpkin (Cucurbita maxima L.) drying. The production area of pumpkin is about 350 hectares, mainly in Szabolcs County (Nagydobos) and Békés-Csongrád County.

The physiological effects of the pumpkin are very comprehensive. The pumpkin has a high level of carbohydrate from starch, fruit-sugar and glucose. The one part of starch changes to sugar under roasting. We can find some protein with trace fat in it. The pumpkin contains a small amount of fibre, but it is reach of vitamins and minerals. It is a considerable β-carotene source, the potassium content assures the optimal acid-base balance. This vegetable has a blood-pressure control and diuretic effect (Géczi, 2003).

The antioxidants and pre vitamins (lutein, zeaxantin) restrict the free radicals, boost the immune system, and decrease confirmation of circulation and tumour diseases. The pumpkin seed contains 30-38 % oil, the curative power is due to its omega-3 fatty acid. The measurements were executed by a regional (Nagydobos) variety, grown in Szabolcs County.

The pumpkin samples were washed with tap water, hand-peeled and cored with a knife, and then cut into cubes of 10 mm thickness using a hand-operated slicer. The samples were divided into ten groups, each group of samples weighed 200 g.

2.2 Vacuum-freeze drier

For running the sublimation drier two technical equipments are necessary. One of them is the air-turbo refrigerator. This unit can produce minus 50-130°C range cooling demand by atmospheric air. The other unit is the sublimation equipment, which promotes the dehydration in vacuum.

In the coding process there are not any chemicals compare with the present methods, so it is an environmental protectionist and more economical technology. The main functional theory of the installation is that the cooling procedure eliminates the evaporation and compression phase. The conventional supercharger refrigerator can only produce minus 50-55°C, but we can achieve about 85-110°C by the developed method. The 100-120°C hot air, leaving the equipment under running, can utilize for useful energy (for heating the drying unit), so we can realize a closed system.

The machine contains the following main parts: a seven degree axial compressor, turbo-expansion axial refrigerator, two regenerating units, three-valve hydraulic chamber, multiplicity unit and an electric motor.

The main favorable properties of the air-turbo refrigerator are:

the unit uses atmospheric air as refrigerant and carrier agent, so the application of the machine is simple and safe;

it does not need water for chilling the cold agent;

the refrigeration process is generated by the direct connection with the air (cold agent) in the different objects;

the planned nominal running parameters can be reached quickly;

preliminary drying of the air is not necessary.

The other important part of the system is the sublimation device. This unit has two chambers for treating 160 kilograms material one by one. In this equipment you can find the turbo refrigerator, control system, refrigerant moving system and vacuum chamber system, on the branch of sublimation unit and condenser.

The working medium of the turbo refrigerator is the atmospheric air. Heating of the working space of the sublimation device and condenser can be happened by utilization of the pressed hot air in the compressor, without any plus energy.

The Figure 1 illustrates the main parts of the industrial lyophilisation equipment (sublimation drier – left picture, air-turbo refrigerator – right picture).

The sublimation process is realized by two phases. At the beginning the raw material goes through a quick freezing at a low temperature. During this phase the water content becomes to ice crystals. The second phase is the dehydration in vacuum: the crystal water eliminates due to sublimation (it changes to vapour on a high temperature). In this step the mass of product decreases by 80-90%, and there is not significant change in appearance and cell texture. The Table 1 contains the main parameters of the industrial drier.

Figure 1. Industrial lyophilising device with the additional parts

For comparing the collected data, the experimental work was also completed in our laboratory, by the ARMFIELD FT33 type vacuum freeze drying equipment (Kerekes, 2008).

The water content of the raw material and dried product was determined by the PRECISA HA60 type moisture control instrument. These data were very important for evaluating of the whole experimental work.

2.3 The electronic penetrometer

The research work included to measure some mechanical parameters of the examined biological materials, for example the hardness of the fruits. The basic theory is to press a special load head to the raw or dried material, until a determined deformation, while measuring the force and determining the maximum value of it. In this way

Table 1. Technical parameters of sublimation drier

Description	Data
Sublimation drier	
External dimension	11×8×3 m
Equipment weight	12 t
Number of working chamber	2 pieces
Surface of working shelve	11 m^2 ×2
Dimension of working shelf	1,05×0,83 m
Number of working shelve	12×2 pieces
Productive capacity of equipment	160 kg×2/cycle
Minimal temperature of shelve	-60 °C
Maximal temperature of shelve	+75 °C
Minimal temperature of condenser	-100 °C
Limit values of sublimation pressure	3000 Pa – 6 Pa
Air-turbo refrigerator	
External dimension	4,955×2,58×2,445 m
The equipment weight	4,8 t
Required power	110 kW
Speed of electric motor	3000 1/min
Cold-air consumption	3400 kg/h
Created air temperature	+120 – -130 °C

we can measure only one point at the beginning part of the force-deformation curve. This measurement can be repeated many times at a given material, so the average hardness can be calculated at the end of the trials.

The instrument includes different size control heads, with 4, 6 or 8 mm diameters, and the deformation can be adjusted by different spacer rings (0.15 mm, 0.30 mm or 0.60 mm). Applying this method a gentle measurement (without any destruction) can be carried out at the soft and flexible materials. The successful measurement requires applying suitable load head and spacer ring.

The portable hardness control unit includes the following parts: electronic penetration unit, measuring interface, power unit, computer, and software. The electronic penetration device is a special construction, a spherical form, using by hand. This metal head unit contains the force measurement cell, the load head and the spacer ring (Géczi, 2003). Evaluating the results, the elasticity coefficient can be calculated by the rate of the load-tension and deformation, with the following equation (1):

$$c_e = \frac{\sigma}{z} \qquad (1)$$

In the equation ce is a elasticity coefficient [kPa/mm] and σ is a load tension [kPa] and z is a deformation [mm].

2.4 Rehydration Ratio

The measurement of the water rehydration ratio was based on the following procedure. 100 ml of distilled water was brought to a temperature of 35 and 75°C in a constant temperature water bath. Then a precisely weighed 0.5 g sample of the dried material was placed in a plastic vessel and immersed for 60 and 90 min. Afterwards the samples were taken out (when the time reached 0.5, 5, 10, 15, 30, 60 and 90 min) and blotted with tissue paper to eliminate excess water on the surface. The weights of dried and rehydrated specimens were measured with an electronic digital balance (model JKH-500, Jadever, Taiwan) having a sensitivity of 0.1 g.

Rehydration ratio (RR) of dehydrated samples was estimated using the equation given below (2) [3].

$$RR = \frac{W_r}{W_d} \qquad (2)$$

where Wr is the drained weight of the rehydrated sample [g], and Wd is the weight of the dry sample used for rehydration [g]. All experiments were performed in triplicate and the average values were reported.

3 RESULTS AND DISCUSSIONS

3.1 Heat and mass transport processes

During the drying process one of the most important task to determine the drying diagrams (change of the water content in function of the time).

The Figure 2 demonstrate the change of core temperature, temperature of the condenser chamber and the press (1-0,5 mbar) in the working unit, in function of the time.It was found that the temperature for pumpkin samples decreased in the first three hour from +20 to -40^{O}C.The freezing step followed by the first drying step, in which mainly the frozen water is removed by sublimation (from -40 to 0°C). A second drying step follows, in which bound water (unfrozen) is removed by desorption (from 0 to 35°C). When the product temperature is close to the temperature of the shelf (working chamber), the drying process is complete.

The Figure 3 shows the change of moisture level in pumpkin samples, during the laboratory freeze drying. The sublimation of moisture happened at 0,06-0,04 mbar vacuum. The shape of the drying curve during lyophilisation is a third degree polynomial (brown line).Obviously, a good agreement exists between the experimental data and the mathematical model, which is confirmed by the high values of the R2 (0.99).

The shape of drying curve indicates that the drying period of the vacuum-freeze drying process is longer than the convective dehydration, because of the minor drying rate. The drying rate is determined by the heat transfer, due to the required slight heat-flux. The speed average of dehydration was 3,34 %/h in the laboratory and 2,6 %/h (percent per hour) in the plant. Because of this, the drying times were 32 and 24 hours at plant and laboratory freeze driers.

3.2 Analysis of the chemical contents

The most important chemical components of the dried pumpkin samples were determined in the Agricultural and Molecular Research Institute of the University of Nyíregyháza. The results can be studied in Table 2.

Summarizing the completed examinations and experiences, it can be quoted, that the chemical components of the vegetable lyophilized in the plant drier are better than that of the pumpkin samples sublimated in laboratory.

The main reason of it, that the vegetable was frozen very quickly on a lower temperature by the air-turbo refrigerator. In the plant freeze drier the cell walls suffered a smaller degradation by the ice crystals. Moreover the smaller drying rate was favorable to assure a relative stable structure.

3.3 Rehydration and hardness tests

The food industry has an increasing demand for dried greens and fruits to extend the selection for the market. The rehydration directs to restore the original features of the materials.

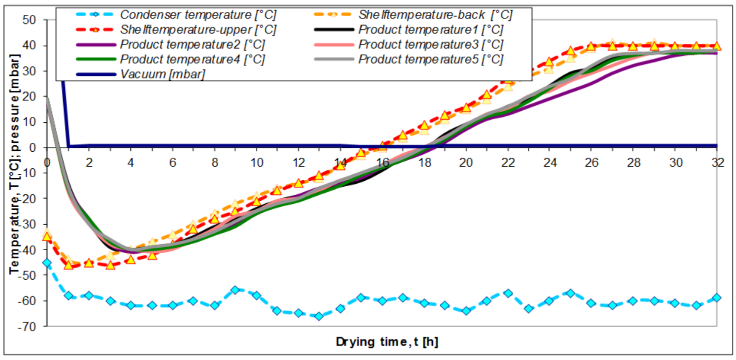

Figure 2. Curve of freeze-drying of the pumpkin in sublimation drier

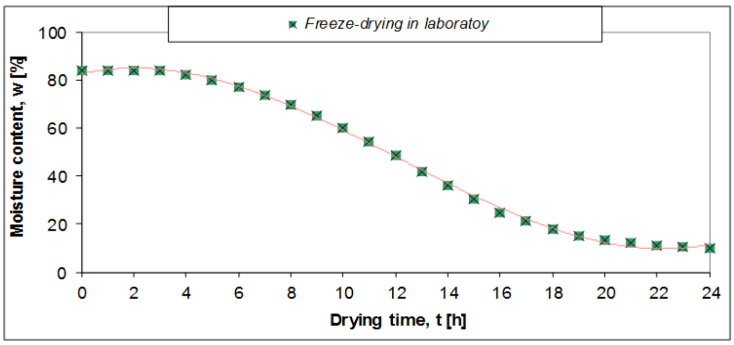

Figure 3. Drying curve of the pumpkin samples in laboratory drier

Table 2. Chemical composition of lyophilized pumpkin

Description	Lyophilized in plant	Lyophilized in laboratory
General data		
Water [%]	1,2	3,8
Protein [%]	8,8	8,2
Fat [%]	2,8	2,71
Carbohydrate [%]	70,3	62,9
Fiber [%]	8,4	7,3
Ash [%]	3	2,6
Mineral materials		
Na [mg/100g]	40,2	33,2
K [mg/100g]	1123,6	1071,3
Ca [mg/100g]	127,4	123,5
Fe [mg/100g]	2,9	2,8
P [mg/100g]	330,5	314,3
Chemical items		
Total-carotene [mg/100g]	30,1	25,4
β-carotene [mg/100g]	26,2	20,3
Cryptoxanthin [mg/100g]	2,1	1,3
Nicotinamide [mg/100g]	8,5	6,2
Ascorbic acid [mg/100g]	77,6	52,1
Carbohydrates		
Glucose [g/100g]	22,2	20,7
Fructose [g/100g]	23,3	21,9
Saccharose [g/100g]	25,4	20,8

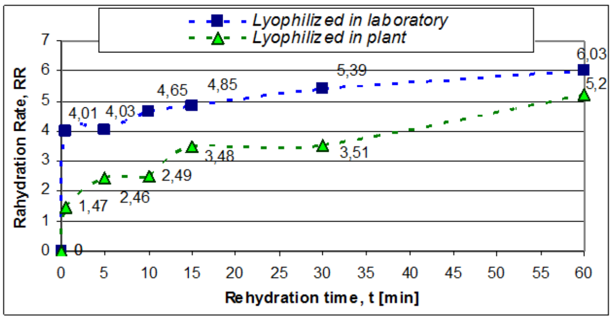

Figure 4. Rehydration curves of the dried pumpkin at 35°C

The Figure 4 and 5 illustrate the change of rehydration rate of the dried pumpkin. On the figures you can notice that the samples lyophilized in laboratory have a higher rehydration rate than the pumpkin dried in the special large capacity freeze drier. This might be due to a smaller pore size left by
faster freezing, which is filled up slowly with distilled water.

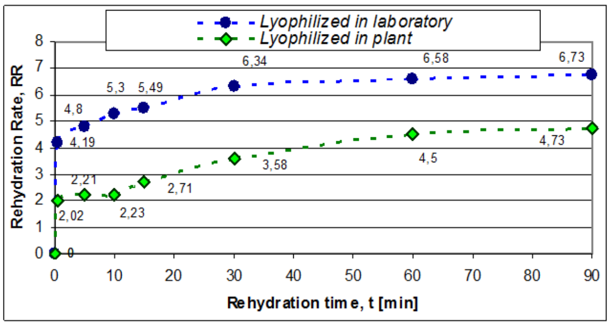

Figure 5. Rehydration curves of the dried pumpkin at 75°C

The RR of laboratory dried samples doubled than the dried in plant when the rehydration process was reached from 0 to 0.5 min. In addition, when the soaking time was increased from 30 min to 60 and 90 min, the RR of samples was increased slightly at lyophilzed in laboratory.

Moreover it is true, that the value of RR is increasing at a higher temperature of the water. We experienced that the rehydration rate did not change significantly after 60 minutes.

The Figure 6 summarizes the value of hardness of pumpkin, in raw state, after drying and rehydration.

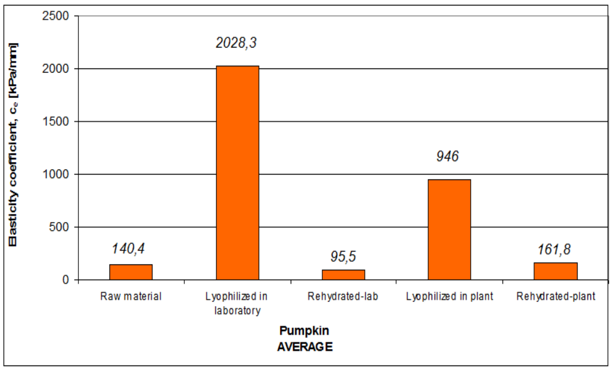

Figure 6. Change of surface hardness of pumpkin

It was found that, the hardness values of plant and laboratory freeze dried samples were 946 and 2028 kPa/mm. The fresh pumpkin presented a firmness of 140 kPa/mm, which is low compared with the hardness values of lyophilized ones. The hardness tests of the product also supported our experiences that the surface of the material dried in the large capacity freeze drier was more flexible and softer comparing to the surface of sample dried in laboratory. But the rehydration tendency of the experimental material was the opposite, the laboratory samples could be rehydrated better than that of the vegetable dried in plant.

4 CONCLUSIONS

The freeze drying can produce the best quality for conserving of horticultural yields, but the production cost is the highest, because of the large investment and running costs. This conclusion was certified by our measurements.

Summarizing the completed examinations and experiences, it can be quoted, that the chemical components of the vegetable lyophilized in the plant drier are better than that of the pumpkin samples sublimated in laboratory.

The main reason of it, that the vegetable was frozen very quickly on a lower temperature by the air-turbo refrigerator. In the plant freeze drier the cell walls suffered a smaller degradation by the ice crystals. Moreover the smaller drying rate was favourable to assure a relative stable structure.

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The vacuum freeze-dried material has a porous structure, but other biological materials dried by conventional method degrade by shrinkage. The considerable porosity of the product assures a fast recovering of the original properties during rehydration. Naturally the large surface, causing a quick swelling, increases oxidizing danger, so it is necessary a special packing procedure in a neutral gas-room, at the event of some product, which contain decomposed color and flavor materials.

Finally, it can be concluded, that the practical industrial trials verified, that the sublimation drier is suitable for production of basic materials for food supplements. But it is important to decrease the drying period of the sensitive and special quality greens, because the dehydration can be finished shorter way, according to the laboratory measurements.

Further consideration that the running cost of the industrial drier can moderate if the air-turbo refrigerator is installed in a temperature-controlled room, so we can avoid preheating of refrigerant (oil) for vacuum pump. Additional opportunity to reduce the processing time of the freeze-drying process, the utilization of combined (hot-air + freeze-drying, vacuum drying + freeze-drying or infrared + freeze drying) processes are proposed.

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