

DETERMINATION OF THE EFFECT OF FREEZE DRYING PROCESS ON THE PRODUCTION OF PUMPKIN (*CUCURBITA MOSCHATA*) PUREE POWDER AND THE POWDER PROPERTIES

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Abstract

Pumpkin (*Cucurbita moschata*) is a good source of carotenoid, pectin, minerals, vitamins, phenolic compounds and terpenoids constituting its nutritional value. This study was intended to produce freeze dried pumpkin puree powder to be used as a functional ingredient in food products such as noodles, breads, cakes, soup and pasta products for improving their nutritional values and flavor. During the freeze drying process, total time was determined to be 9 hours by following the changes in the weight of the samples. According to the results, moisture content and water activity values of the pumpkin powders were in acceptable limits for safe storage of products. Freeze drying process decreased vitamin C content by 18%, total carotenoid content about 26% and the total phenolic compounds as 3%. Among the powder properties of the dried product, the bulk density was found to be 0.113 g/ml and average wettability and solubility times were determined as 710 and 16 s, respectively. Determination of flowability and cohesiveness in terms of Carr Index and Hausner ratio were evaluated as fair and intermediate levels respectively.

Keywords; Pumpkin, freeze drying, functional properties, powder properties

DONDURARAK KURUTMA İŞLEMİNİN BALKABAĞI (*CUCURBITA MOSCHATA*) PÜRESİ TOZU ÜRETİMİ VE ÜRÜNÜN TOZ ÖZELLİKLERİ ÜZERİNE ETKİSİNİN BELİRLENMESİ

Özet

Balkabağı (*Cucurbita moschata*) bileşimini oluşturan karotenoid, pektin, mineral, vitamin, fenolik bileşikler ve terpenoidlerin iyi bir kaynağıdır. Bu çalışmada makarna, ekmekek, kek, ve çorba gibi ürünlerin besin değerini ve lezzetini arttırmak amacıyla, bu ürünlerde katkı maddesi olarak kullanılabilecek dondurarak kurutulmuş balkabağı püresi tozu üretilmesi amaçlanmıştır. Dondurarak kurutma işlemi boyunca örneklerin ağırlığındaki değişim izlenerek toplam kuruma zamanı 9 saat olarak bulunmuştur. Balkabağı tozlarının nem ve su aktivitesi değerleri güvenli depolama için uygun sınırlar içinde bulunmuştur. Dondurarak kurutma işlemi C vitamini içeriğinde %18, toplam karotenoid içeriğinde %26 ve toplam fenolik madde içeriğinde %3 azalmaya neden olmuştur. Toz ürünün yığın yoğunluğu 0.113 g/ml ve ortalama ıslanabilirlik ve çözünürlük süreleri ise sırasıyla 710 ve 16 saniye olarak bulunmuştur. "Carr Index" ve "Hausner Ratio" olarak belirlenen akabilirlik ve yapışkanlık değerleri orta seviye olarak belirlenmiştir.

Anahtar kelimeler; Balkabağı, dondurarak kurutma, fonksiyonel özellikler, toz özellikleri

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INTRODUCTION

Pumpkin has a large range of uses as a valuable food source for human and animals. It is a good source of vitamins such as B₆, K, thiamine and riboflavin and minerals such as potassium, phosphorus, magnesium, iron and selenium. It also contains large amounts of carotenoid, pectin, phenolic compounds and terpenoids constituting its nutritional value (1, 2). Pumpkins are being processed to obtain juice, pomace, dried product, cake, filling material in pie, bread and soup. It is also used with the function of thickener, flavoring and coloring agent. Also, there exist several studies about the development of pumpkin based foods as its use in expanded snack and breakfast cereal products (3). The production of pumpkin is quite common in China, India, Ukraine, USA, Egypt, Mexico, Italy and Turkey. The pumpkins are quite stable after their harvest (e.g. 1-3 months). However, after peeling they are susceptible for microbial spoilage, moisture loss, color changes and softening. Due to large size of pumpkins and difficulties in removing their strong peels its sale in retail market in peeled and portioned way is much more suitable. For these reasons in order to prolong the shelf life of pumpkins, freezing and drying might be suitable techniques.

Drying constitutes an alternative way to increase the consumption of pumpkin and allows its use during the off-season. Besides having long shelf life, dried products have some storage and transportation advantages. Classical method of hot air drying has the disadvantages of long drying times, significant quality losses, etc. Freeze drying is a dehydration process in which water is removed by sublimation of ice from frozen material. Freeze drying is the best method for drying foods with high quality which compensates the high operating costs. Color, flavor, texture, nutrients, taste, appearance, chemical composition, shape and biological activity of the fresh samples undergo only slight changes (4). So, it is generally recommended for drying of foods containing heat-sensitive components and mainly for antioxidant components such as tocopherols, ascorbic acid, carotenoids and phenolics (5). Numerous studies were carried out with freeze drying of on the foods that contain these heat sensitive materials such as star fruit,

muskmelon, watermelon, papaya, mango (5), acerola (6), marionberries, strawberries, corn (7), pumpkin slices (1, 4) with minimum quality loss.

Several drying techniques such as convective drying (1, 4, 8, 9 - 17), freeze drying (1, 4), microwave drying (2, 14), vacuum drying (4, 18) and osmotic dehydration (15, 16) were studied for drying of pumpkin. In some other studies, the effects of combined methods such as microwave-air (14) and vacuum-microwave (4) on drying kinetics of pumpkin slices were also investigated.

The properties of food powders such as bulk density, hygroscopicity, degree of caking, dispersibility, wettability, solubility, particle size and distribution are useful for design and control of processing, handling, storage operations and product quality control (19). In the literature, there exist some studies dedicated to the effect of feed properties and drying conditions on the physical properties of freeze dried powders such as bulk density (1, 4, 20, 21), true density (1), wettability (21), solubility (20-22), porosity (1) and particle density (20).

The objective of this study is to produce freeze dried pumpkin puree powders as a functional ingredient in food products such as soups, noodles, breads, cakes and pasta products for improving their nutritional values and flavor.

MATERIAL AND METHODS

The fresh pumpkins were obtained from a local supermarket in Izmir, Turkey. They were peeled; seeds removed and were grinded in a home type blender.

Freeze Drying

Experiments were performed in a pilot scale freeze dryer (Armfield, FT 33 Vacuum Freeze Drier, England). The pumpkin puree were frozen in a layer of 3 mm in the petri dishes at - 40 °C in an air blast freezer for two hours, then freeze dried under vacuum (13.33 Pa absolute pressure), at - 48 °C condenser temperature for nine hours. The temperature of the heating plate was set to +30 °C which accelerated the sublimation process and it was constant during the drying process.

For this process, each experiment for increasing time periods was carried out with new samples of equal mass and moisture loss was determined by weighing the petri dishes using a digital balance with 0.01 precision (Ohaus AR2140, USA). The powder was obtained by grinding the dried material in a blender (Tefal Smart, MB450141, Turkey) and stored in glass jars in the dark at $20 \pm 1^\circ\text{C}$ until further tests were carried out.

Physical and Chemical Analyses

Moisture, ash and vitamin C contents of the fresh pumpkin puree (FPP) were determined by using AOAC methods (23). Water activity was measured using a Testo-AG 400 (Germany) relative humidity and temperature measurement device. The Ph values of the samples were measured by using a ppt meter Inolab WTW pH 720 (Germany). The experiments and measurements were replicated three times.

The color values of FPP (L^* , a^* and b^* values) were measured with a Minolta CR-400 Colorimeter (USA) and results were expressed in accordance with the CIE Lab. System. The L^* value, is a measure of lightness which ranges between 0 and 100. Increases in a^* value in positive and negative scales correspond to increases in red or green color, respectively. The b^* value represents color ranging from yellow (+) to blue (-).

To determine the total phenolic content of FPP (1:1 kg/kg) was prepared. 1 ml of juice (fresh grounded pumpkin or reconstituted puree powder) was taken into a test tube and Folin-Ciocalteu's (Sigma-Aldrich Corp.) reagent (5 ml) that diluted 10- fold before use and 2% Na_2CO_3 (15 ml) were added. After reaction for 2 hours, absorbance was measured at 760 nm. A calibration curve of gallic acid (3, 4, 5-trihydroxybenzoic acid) (Sigma-Aldrich Corp.) was prepared. The results were given as gallic acid equivalents per grams of sample (24).

The total carotenoid content was determined according to the method of Lee and Castle (25) that was modified for pumpkin. A sample of 2 ml FPP was mixed with 38-ml extraction solvent (hexane-acetone-ethanol (50:25:25 v/v/v)) with the homogenizer at speed 4 (Ultra-Turrax Ika-Werke, Staufen, Germany). After centrifugation (4000 rpm, 10 min, 5°C) (CFC free Universal

Hettich Zentrifugen), the absorption was measured with a Varian Cary 50 Scan (Australia) spectrophotometer at 450 nm. Total carotenoid content was determined as milligrams of β -carotene per gram of sample (db) as Eq. 1:

$$A = \frac{(\varepsilon bxc)}{1000} \quad (\text{Eq. 1})$$

where, A = absorbance value

ε = molar absorbance coefficient $\varepsilon(1\text{cm}) = 2505$

b = extent of unit light way, 1 cm

c = milligram per gram total carotenoid content.

All the mentioned analyses were also applied to freeze dried pumpkin puree powders (FDPPP) by rehydrating them the initial moisture content to compare the changes of the properties. Total color change (ΔE) of freeze dried pumpkin puree with respect to fresh pumpkin puree was calculated by Eq. 2;

$$\Delta E = \sqrt{\Delta L^2 + \Delta a^2 + \Delta b^2} \quad (\text{Eq. 2})$$

Analysis of Powder Properties

For the determination of bulk density, 20 g of powder was gently loaded into a 100 ml graduated cylinder. The measured volume was used to calculate the bulk density (ρ_{bulk}) according to the relationship: mass/volume. For the determination of the tapped density (ρ_{tapped}); the loaded cylinder dropped 70 times onto a rubber mat form a height of 15 cm and the final volume was recorded to be used in the calculations (26, 27). The wettability of the powders was determined by measuring the time for completely wetting 10 g of sample placed around a beaker of 250 ml containing 100 ml of distilled water (at 25°C) (28). The average solubility times of FDPPP were determined by adding 2 g of the powder to 50 ml of distilled water at room temperature. The samples were stirred vigorously with a magnetic stirrer and the time required for the powders to dissolve completely was recorded (29). Flowability and cohesiveness of the powder were evaluated in terms of Carr index (CI) and Hausner ratio (HR) (26), respectively. Both CI and HR were calculated from the bulk (ρ_{bulk}) and tapped (ρ_{tapped}) densities of the powder as shown below (Eq. 3 and 4) and classified according to Table 1.

Table 1. Classification of the flowability and cohesiveness of the powder based on the CI and HR values (23).

CI (%)	Flowability	HR	Cohesiveness
<15	Very Good	<1.2	Low
15-20	Good	1.2-1.4	Intermediate
20-35	Fair	>1.4	High
35-45	Bad		
45>	Very Bad		

$$CI = \frac{(\rho_{tapped} - \rho_{bulk})}{\rho_{tapped}} \times 100 \quad (\text{Eq. 3})$$

$$HR = \frac{\rho_{tapped}}{\rho_{bulk}} \quad (\text{Eq. 4})$$

Experimental results were the means of (\pm standard deviation, SD) three parallel measurements.

RESULTS AND DISCUSSION

Drying Curves

The drying behaviour for the freeze drying process of FPP was determined from the mass loss in samples of known initial moisture content. The drying times required for freeze drying are substantially long when compared to the conventional drying methods which may change depending on temperature, air velocity and humidity. During drying process, the total time was determined to be 9 hours by following the change in weight of the samples which can be compared with a drying time of 8 hours for convective drying around 30 °C (8). The change of the moisture content (dry basis) of samples versus time was shown in Figure 1. As expected, the higher percentage of weight loss of moisture content occurs in the early stages of drying and the moisture content decreased considerably with increasing the drying time.

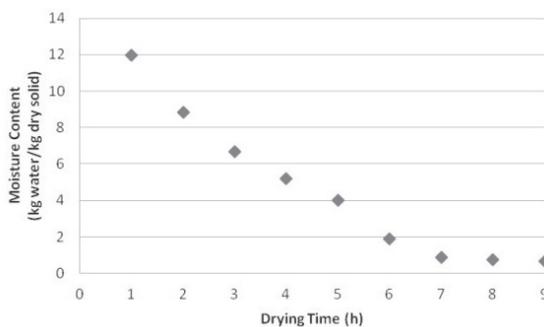


Figure 1. The variation of moisture content (db) as a function of drying time for pumpkin puree

Drying rate is described as the amount of water removed per time and per drying area. The drying rate values were calculated for different drying times and plotted against the moisture content for drying of FPP as shown in Figure 2. Two falling rate periods were observed in drying of FPP at freeze dryer. The first falling rate period occurred at the moisture contents up to 4.03% (d.b), while the second falling rate period occurred at the moisture contents below 1.88% (d.b). Between the moisture contents of 4.03% (d.b) and 1.88% (d.b) a constant rate period was observed. The observed drying behaviour of FPP consistent with drying of banana (30) and carrot (31).

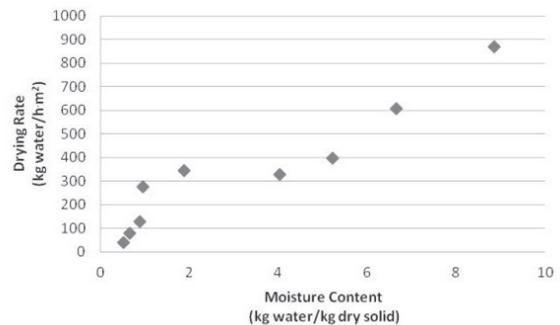


Figure 2. The variation of drying rate as a function of moisture content of pumpkin puree.

Results of Physical and Chemical Analyses

The average values of the experimental results of the FPP and FDPPP were given at Table 2. The average initial moisture content of FPP was found as 92.34 % (wet basis, wb) and drying process removed 95.75% of water leading to a final moisture content of 3.93% (wb). This result is similar to the value of moisture content for freeze dried mango as 4% (wb) (32), however, lower than that of strawberry slices (9% wb), pineapple (7%) and guava (5% wb) which were also freeze dried in the studies by and Marques et. al. (32) and Kirmaci et. al. (33) respectively. The moisture content of FDPPP was found to be higher than freeze dried guava concentrate

Table 2. Results of the analyses applied on fresh pumpkin puree and freeze dried pumpkin puree powders (db).

	Moisture content (%) (wb)	Ash content (%) (db)	Water Activity	pH	Vitamin C Content (mg/100g) (db)	Total Carotenoid Contents (ppm) (db)	Total Phenolic Contents (mg GAE/g) (db)
Fresh Pumpkin	92.34±0.26	0.69±0.07	0.988±0.006	7.38±0.02	20.20±0.07	10.09±0.003	225.22±1.1
Freeze Dried Pumpkin Puree Powders	3.93±0.55	4.11±0.08	0.197±0.001	7.47±0.03	16.56±0.06	7.47±0.008	218.47±3.85

(2.36% wb) (20), marionberries and strawberries cultivars (0.1-0.2%), corn cultivars (0.7-1.1 %) (7) and papaya (2% wb) that were freeze dried in the study of Marques et. al. (32).

Ash contents (%) of FPP and FDPPP were found as 0.69% and 4.11% (db), respectively. The amount for FPP was lower than those reported by Fennema et. al. (34) as 0.8-1.4% and Guine et. al. (8) as 1.1%. This may be due to variations in cultivar selection.

Water activity has long been considered as one of the most important quality factors for dried products especially for long term storage. Water activity is related with moisture content and responsible for biochemical reactions (35). The values of water activity among 0.20 and 0.40 ensure the stability of the product against browning and hydrolytical reactions, lipid oxidation, auto-oxidation and enzymatic activity. Water activity value of FDPPP was found as 0.197 similarly to the range of water activity of freeze dried acerola as about 0.2 obtained by Marques et. al. (6) after 8 hours of freeze drying process.

The values in Table 2 were expressed in dry basis for a better comparison of the FPP with dried powder. From these values, it is possible to see that the chemical compositions such as vitamin C and total phenolic contents of both fresh and dried pumpkin were very similar. This means that the freeze drying process did not influence the nutritional value of the final product significantly. The vitamin C loss of pumpkin puree (18%) was found to be similar to the vitamin C loss for guava concentrate (18.8 %) (20). The loss of vitamin C was also found to be less than freeze dried guava (37.47%) and pineapple (27.31%) (32), and it was more than freeze dried acerola (13%) (6), mango (3.05%) and papaya (6.91%) (32). Shofian et. al.,

(5) did not observe any significant change between the ascorbic acid content of the fresh and freeze-dried star fruit, mango, papaya muskmelon and watermelon. The vitamin C losses can be due to not only the freeze-drying, but also by the operations before drying such as cutting, slicing and freezing. Therefore, grinding process may cause more vitamin C losses for the pumpkin puree. The vitamin C losses for freeze-dried fruits are considerable smaller when compared the vitamin C losses caused to other drying methods due to the low temperatures and to the use of vacuum in the process (32).

The total phenolic contents (TPC) of water extracts of FPP and FDPPP were determined by Folin Ciocalteu method. The results obtained by using the calibration curve ($A = 0.1041GAE - 0.0469$, $R^2 = 0.9977$) were determined as gallic acid equivalents per 100 ml of water extract of freeze dried pumpkin puree powder (mg GAE/g pumpkin puree powder). TPC of FPP and FDPPP were found as 225.22 mg GAE/g fresh pumpkins and 218.47 mg GAE/g pumpkin puree powders. The determined values are much higher than 0.39 mg GAE/ g for freeze dried pumpkin flour given by Que et. al., (1). Different extraction processes may be the reasons for the differences between the determined values of TPC. Although freeze drying process only reduced the TPC as 3% in this study, Shofian et al.(5) expressed significant differences for the amounts of TPC between the fresh and freeze-dried fruit samples (starfruit, mango, papaya, watermelon). In their study, freeze drying process caused TPC losses of 24%, 23%, 40% and 48% for star fruit, mango, papaya and watermelon, respectively.

Carotenoid content is an important parameter for the determination of the final quality of dehydrated vegetables such as carrots and pumpkin as it is a

determining factor in color and nutritional quality. Carotenoids are sensitive to heat, oxygen, light, and enzymes (36). The total carotenoid content (TCC) of FPP was found as 10.09 ± 0.003 ppm (db) which was similar to the value determined by Dutta et. al. (37) as 10.9426 ppm and quite higher than 4 ppm which is expressed by Badr et. al. (38). The composition of carotenoids may be influenced by some factors such as cultivar, maturation stage, geography, climate, harvesting and analysis method (37). Freeze drying process decreased the TCC of pumpkin puree about 26%. The result for freeze dried pumpkin puree powder was found to be higher than the determined values by Nawirska et. al. (4). In their study it was observed that the TCC of freeze dried slices of 12 pumpkin cultivars were ranged between 5 mg/100 g and 160 mg/100 g and the highest average TCC was obtained from freeze dried samples, then vacuum microwave and the lowest value was observed by convective drying.

The color of dried products is an important quality factor which reflects the sensory attractiveness and the quality of the powders (35). Even though a functional food could provide several health benefits to consumers, without visual attraction to the consumers it cannot be marketable. Thus, the color of the supplemented products should ideally remain unchanged after production. The color values (L^* , a^* and b^*) of FPP were found as 47.75, 16.66 and 31.31 respectively. The L^* , a^* and b^* values increased throughout the drying period to the final values of 76.57, 22.84 and 47.18 respectively. The color values are comparable with the measurements obtained by Que, et.al., (1) for L, a and b value of 80.15, 13.43 and 48.63 respectively. The main difference is in a value which is the indication of redness and the main difference taking place between the varieties. Nawirska et. al. (4) observed that the lightness (L^*), redness (a^*) and yellowness (b^*) values of freeze dried slices of 12 pumpkin cultivars were ranged between 70-82; 2-22 and 32-49, respectively. The color values of FDPPP were also consistent with their study. To determine the total color change (ΔE) of FDPPP with respect to FPP, the FDPPP were rehydrated the initial moisture content and ΔE was calculated as 31.94 ± 0.74 .

Analysis of Powder Properties

The powder properties such as bulk density, wettability and solubility are related to ease of reconstitution. These properties are influenced by the nature of the feed (solid content, viscosity and temperature) and operational conditions (20). The moisture content of powders also has important effect on powder properties such as bulk density, solubility, wettability, flow behaviour etc. (29). The bulk density of FDPPP was found as 0.113 ± 0.0006 g/ml which was lower than 0.33g/ml for freeze dried pumpkin puree (1), 0.65g/m^3 for guava concentrate (20) and 0.402g/ml for kiwi puree powder (21). Solubility problems occur when foods are subjected to higher temperatures, especially in products with high concentration of solids (29). The freeze dried powder could be reconstituted instantly with water at room temperature. FDPPP were found easy soluble as 16 ± 0.58 seconds of average soluble time that was less than 257 s for freeze dried gilaboru powder (22) and 26 s for freeze dried kiwi powder (21). In a study by Mahendran (20) guava concentrate was dried with spray, tunnel and freeze driers and the freeze dried guava powder was found highly soluble (96%) compared with the other drying methods. The average wettability time of FDPPP was found to be 710 ± 33.04 seconds which was higher than freeze dried kiwi puree with 1.34s (21). The flowability and cohesiveness properties in terms of Carr Index and Hausner ratio were evaluated as fair (23.14 ± 0.79) and intermediate (1.30 ± 0.01) levels respectively. Flowability of a particle is affected by several factors as particle size, particle size distribution, particle morphology, moisture content and the hygroscopic and electrostatic nature of the material. In a free flowing powder, particles should, ideally, be spherical with a relatively large particle size and be moisture free. Since the freeze dried puree in form of pellets was ground in this study the morphological properties are not considerable. Therefore, flowability is directly related with more dried powders which also lead to lower cohesiveness between particles.

CONCLUSION

The present work describes possibility of producing pumpkin puree powder by freeze drying and the changes in some physicochemical and powder properties of powders. The results showed that freeze drying can satisfactorily be applied for drying of pumpkin puree to obtain powders that can be used as an ingredient for flavoring and improving nutritional value purposes. The possible uses of this dried product in food systems and storage potential might be studied in future projects.

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