Short communication

EFFICIENT FREEZE-DRYING OF MELON FRUITS: MINIMIZING TIME AND MOISTURE CONTENT WHILE MAINTAINING SUGARS AND PIGMENTS CONTENTS

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SUMMARY

Melon puree samples were tested to optimize freeze drying conditions in order to shorten the time of the freeze-drying process (within a 25 h period) and obtain powder material with low moisture content. Additionally, the determination of individual sugars composition and content, and total carotenoids (and chlorophylls) content were performed to test the quality of freeze-dried samples. Obtained results showed that analyzed samples had moisture content of 6.30%, pH of 6.47, refractive index (RI) of 1.346 and total soluble solids (°Brix) of 8.8. When compared to the fresh samples, the freeze-dried samples had 9.7 and 2.8 times higher amounts of sucrose and carotenoids, respectively. Presented testing showed the importance of sample preparation process, which can considerably reduce the lyophilisation time of this type of sample, especially for the underutilized agricultural plants in the production of value-added commodities.

KEYWORDS: Brix, carotenoids, *Cucumis*, fruits, melon, pH, puree, sugars

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INTRODUCTION

Melon (*Cucumis melo*) belongs to the Cucurbitaceae family and presents an important vegetable grown for its fruits. According to FAOSTAT data (FAOSTAT, 2013-2023), production of cantaloupes and other melons is in continuous growth, being the highest in 2023 (29,541,294.0 t from 1,092,354.0 ha). The majority of yield comes from China and other Asian countries (69.7% of the production), in general. Melons are most often consumed fresh. Due to their nutritional value, melons may be a promising candidate for application in functional food development.

One of the postharvest processing procedures of plant material, whether for further analyses or for formulation of different products (fruit and vegetable powders), includes freeze-drying. The method certainly extend shelf-life of foods and maintain microbiological safety and functional properties of vegetables and fruits. The process represents removal of water through sublimation of ice in the materials through high vacuum at low temperature, with the limitation of the oxidation process. Freeze-drying is generally recommended for drying of materials containing heat-sensitive antioxidant components such as tocopherols, ascorbic acid, carotenoids and plant phenolic compounds (Shofian et al., 2011).

Freeze drying of melon peel [cantaloupe melons (*Cucumis melo* L. var. *reticulatus*)], to provide value-added product were performed by Sroy et al. (2022). They freeze dried frozen samples (frozen at -80 °C for 6 to 8 h) at -50 °C, a pressure ranging from 1.5 to 2 bar, for 80 to 90 h. Additionally, Shofian et al. (2011) tested the quality of different fruits and vegetables [starfruit - *Averrhoa carambola* L., mango - *Mangifera indica* L., papaya - *Carica papaya* L.,

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muskmelon - *Cucumis melo* L., and watermelon - *Citrulus lanatus* (Thunb.)] and their antioxidant properties, by freeze-drying the frozen cubes of their fruits (frozen for 24 h, at -20 ± 1 °C) for three days until they were completely dried, concluding that samples retained the maximum amount of ascorbic acid after freeze-drying.

Nowak and Jakubczyk (2020) in their review reported that a general recommendation of dry matter content in freeze dried samples should be less than 95% for food, while for pharmaceutical products should be 98-99% or higher. Additionally they state that too high moisture content isn't favourable for longer storage and too low moisture content can damage the active material. However, the moisture content in some of the products presented in the paper ranged from 1.3 to 9.5% after freeze-drying.

Salehi (2023) reviewed freeze-drying methods on some fruits and vegetables and reported a range in final moisture content from 2 to 18% (for example, 22-26 h drying of tomato – 4.9%, carrot cylinders – 8.7%, carrot slices – 6.7, apple slices – 6.1-6.6%).

Since the reported time for freeze-drying in most cases ranges from two to four days, the purpose of this experiment was to test the possibility to shorten the time of freeze drying of melon puree and obtain powder material with low moisture content. Additionally, the testing includes determination of individual sugars composition and content, as well as total carotenoids content in fresh and freeze-dried fruits of melon with a view to compare their quality.

MATERIAL AND METHODS

Plant material (Picture 1), produced by Serbian farmers, was purchased from a supermarket located in Novi Sad (Vojvodina province, Serbia).

Raw material

Refractive index (RI) and total soluble solids (°Brix) values of fresh fruit juice were determined by Abbe refractometer (Carl Zeiss, Jena, Germany), and pH was determined by pH meter (WTW, Xylem Analytics Germany).

Melon fruit flesh was cut into cubes, homogenized (T10 basic, Ultra-Turrax, Ika, Germany) and frozen in leyers of 0.5 and 1.0 cm (-20 °C, minimum 24 h).

A part of the plant material was analyzed as fresh sample and another part was crushed, divided in four pieces and freeze dried (Alpha 2-4 LSC basic, Martin Christ, Germany) under vacuum, for 25 h (main drying), at -80 °C and chamber pressure 0.1 mbar. Before choosing the final conditions, we tested the samples under conditions reported in the literature (Lopez-Quiroga et al., 2020; Nowak & Jakubczyk, 2020), Table 1. Accordingly, the most effective treatment included 24 h process duration and 0.5 cm thick layer.

Picture 1. Melon fruits (A), puree of melon fruits (B), freeze-dried puree of melon fruits (C), stored samples (D)

Moisture content

Fresh and freeze-dried melon puree (3 g approximately each) were weighed using analytical balance prior to drying the samples in the oven at 105 °C for 3 h. Samples were then left to cool in the desiccator and were reweighed until it reached a consistent weight. Moisture content (%) was calculated based on wet weight basis (Ph Jug V, 2000).

Analyzed samples were frozen in 0.5 cm thickness and after 25 h of freeze-drying, moisture content was 6.30 ± 0.11%. Lyophilised samples were kept in a desiccator and protected from light with aluminium foil.

Individual sugars composition and content were analyzed in fresh, freeze-dried, and samples obtained after the rehydration (1 h, 25 °C) of previously freeze-dried samples. Parameters for rehydration were taken from Lopez-Quiroga et al. (2020).

Total chlorophyll a, b and carotenoids contents were determined in fresh and freeze-dried samples, according to method described by Wellburn (1994). The fresh or freeze-dried plant material (0.1 g) was ground with cooled mortar and pestle and then homogenized with 10 ml of 80% acetone. After extraction, extracts were centrifuged for 10 min at

5000 rpm. Absorbance was recorded at 470, 646 and 663 nm (Perkin Elmer Lambda 25 UV/Visible spectrophotometer). All pigments contents were calculated from equations described in applied method and given as mg/kg dry/fresh weight.

For aqueous extracts, 10.0 and 0.5 g of fresh and freeze-dried plant material were homogenized with 30 and 10 ml of HPLC pure water, respectively. After 30 min of shaking on a laboratory shaker, extracts were centrifuged for 10 min at 5000 rpm and supernatants were filtered through 0.22 μm regenerated cellulose syringe filters into vials.

Table 1. Testing of freeze-drying conditions for melon puree samples

Freeze-drying conditions		Moisture (%)
Process duration (h)	Layer thickness (cm)	
24	0.5 (A)	8.41 ± 0.80
	1.0 (B)	47.52 ± 13.70
30	0.5 (A)	9.90 ± 0.36
	1.0 (B)	39.13 ± 13.62
Fresh sample		92.07 ± 0.60

Individual sugars were determined using HPLC system (LC-40D XS Nexera, Shimadzu, Japan) coupled with Refractive Index Detector (RID-20A) by isocratic method recommended by the producer (Shimadzu, Application news, No. SCA_190_028) and a previously described protocol (Abbas, 2020). Samples (10 μl injection volume) were separated at 40 °C by using Shim-pack GIST NH2 column (4.6 mm, × 250 mm, 5 μm). HPLC pure acetonitrile/water, 8:2 (v/v), was used as mobile phase at flow rate of 2 ml/min, run time 8 min. Quantification of sugars was performed by comparing retention times and integrating peaks and converting them to concentrations by comparison against standards of glucose (Fluka), fructose and sucrose (Thermo Scientific).

Values of the biochemical parameters were expressed as means ± standard error of determinations made in triplicates and tested by ANOVA followed by comparisons of means by the Duncan's test and t-test ($p<0.05$). All statistical analyses were performed using XLSTAT software.

RESULTS AND DISCUSSION

Obtained results showed that analyzed samples had pH 6.47, refractive index (RI) 1.346±0.001, and total soluble solids (°Brix) 8.8±0.7. Results from the literature ranged from 4.03 to 6.52 for pH values and from 3.00 to 12.53, and even 15.00-18.00 for °Brix (Mendlinger & Pasternak, 1992; Stepansky et al., 1999; Albuquerque et al., 2006; Gur et al., 2016; Chikh-Rouhou et al., 2021).

According to Stepansky et al. (1999), who studied 56 *Cucumis melo* genotypes from 23 countries, almost all the sucrose-accumulating sweet varieties (cantalupensis and inodorus types) have near-to-neutral pH values (6.00-6.75). In the study, sucrose content were from 0.8 to 66.3 mg/g, while glucose and fructose were in ranges 1.6-29.5 and 1.1-30.2 mg/g, respectively.

The mature fruits of melon have disaccharide sucrose, as the major sugar, and two of its hydrolysis products, glucose and fructose (Gur et al., 2016). Another studies (Singh et al., 2022; 2025), that explored phytochemical compositions and the sensory and functional quality attributes of muskmelons, revealed sucrose contents were from 0.9 to 55.7 mg/g fresh weight (f.w.), while glucose and fructose were in ranges 0.7-17.3 and 2.8-30.4 mg/g f.w., respectively. Other studies (Albuquerque et al., 2006; Ercan et al., 2023) reported sucrose, glucose and fructose contents in fresh fruits in range: 22.9-78.6; 10.2-34.0; 11.7-23.0 mg/g of fresh fruit weight.

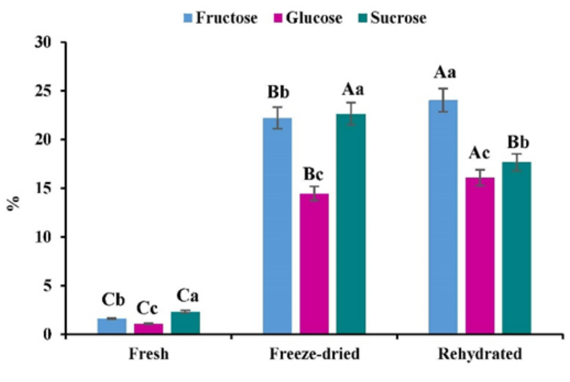

Figure 1. Fructose, glucose and sucrose contents in fresh, freeze-dried and rehydrated melon pulp. Values marked with the different upper case and low case letters differ significantly among and within the processing treatment at $p<0.05$ (Duncan's test), respectively

Our study confirmed the published results on sucrose, glucose and fructose contents in fresh fruits, since our results were 23.4, 10.8, and 16.3 mg/g f.w., respectively. Content of sucrose in freeze dried samples was 22.6% (226.5 mg/g freeze-dried weight), and it was 9.7-fold the amount of sucrose in the fresh puree (Figure 1). Sucrose contents were lower, and glucose and fructose contents were higher in rehydrated samples by 1.1-fold change when compared to freeze-dried samples (Figure 1).

Table 2. Pigments contents (mg/kg) in fruits of the tested melon samples

Sample	**Chlorophyll a**	**Chlorophyll b**	**Carotenoids**
Fresh	1.49±0.02	2.39±0.04	27.74±2.16
Freeze-dried	3.23±0.49*	5.19±0.79*	78.64±3.34*

Results marked with asterisk differ statistically at $p<0.05$ (t-test)

White, green and orange colour of melon fruit is determined by a combination of chlorophyll and carotenoid pigments (Gur et al., 2016). Tested pigments contents were 2.2-2.8 times higher in freeze dried samples when compared to fresh ones (Table 2). According to published data, chlorophyll a, b and carotenoids (β-carotene) were: 0.4-3.5, 0.4-2.9, 0.0-6.6 mg/kg fresh weight, respectively (Chikh-Rouhou et al., 2021; Singh et al., 2025). Some studies presented levels of β-carotene from 13.5 to 37.6 μg/g (Singh et al., 2022; 2025).

CONCLUSIONS

As it was expected, freeze-dried samples had higher contents of sugars and total carotenoids (due to higher amount of dry weight), however, this research emphasized the importance of the thickness of the layer in which the samples are frozen. Presented testing showed the importance of sample preparation process, which can considerably reduce the lyophilisation time of this type of sample.

REFERENCES

Abbas, H.M.K., Huang, H.X., Wang, A.J., Wu, T.Q., Xue, S.D., Ahmad, A., Xie, D.S., Li, J.X., & Zhong, Y.J. (2020). Metabolic and transcriptomic analysis of two *Cucurbita moschata* germplasms throughout fruit development. *BMC genomics*, *21*, 1-13. https://doi.org/10.1186/s12864-020-6774-y

Albuquerque, B., Lidon, F.C., & Barreiro, M.G. (2006). A case study on the flavor properties of melon (*Cucumis melo* L.) cultivars. *Fruits*, *61*(5), 333-339. https://doi.org/10.1051/fruits:2006032

Chikh-Rouhou, H., Tlili, I., Ilahy, R., R'him, T., & Sta-Baba, R. (2021). Fruit quality assessment and characterization of melon genotypes. *International Journal of Vegetable Science*, *27*(1), 3-19. https://doi.org/10.1080/19315260.2019.1692268

Ercan, M., Çoklar, H., Akbulut, M., Yavuz, D., Seymen, M., & Yavuz, N. (2023). Effect of irrigation regime on chemical, physico-chemical, and functional properties of melon fruits and seeds. *Gesunde Pflanzen*, *75*(6), 2835-2845. https://doi.org/10.1007/s10343-023-00900-w

FAOSTAT (2013-2023).https://www.fao.org/faostat/en/#data (accessed on 28 April 2025).

Gur, A., Gonda, I., Portnoy, V., Tzuri, G., Chayut, N., Cohen, S., Yeselson, Y., Meir, A., Bar. E., Davidovitz-Rikanati, R., & Saar, U. (2016). Genomic aspects of melon fruit quality. In R. Grumet, N. Katzir, J. Garcia-Mas (Eds.), Genetics and genomics of Cucurbitaceae (pp. 377-408). Cham: Springer International Publishing. https://doi.org/10.1007/7397_2016_29

Igual, M., Flores-León, A., Picó, B., Martínez-Monzó, J., & García-Segovia, P. (2023). Physicochemical, structural, and functional properties of snake melon (*Cucumis melo* subsp. *melo* var. *flexuosus*) microencapsulated with pea protein and pea fibre by freeze-drying. *Foods*, *12*(14), 2679. https://doi.org/10.3390/foods12142679

Lopez-Quiroga, E., Prosapio, V., Fryer, P.J., Norton, I.T., & Bakalis, S. (2020). Model discrimination for drying and rehydration kinetics of freeze-dried tomatoes. *Journal of Food Process Engineering*, *43*(5), p.e13192. https://doi.org/10.1111/jfpe.13192

Mendlinger, S., & Pasternak, D. (1992). Effect of time of salinization on flowering, yield and fruit quality factors in melon *Cucumis melo* L. *Journal of Horticultural Science*, *67*(4), 529-534. https://doi.org/10.1080/00221589.1992.11516279

Nowak, D., & Jakubczyk, E. (2020). The freeze-drying of foods - The characteristic of the process course and the effect of its parameters on the physical properties of food materials. *Foods*, *9*(10), 1488. https://doi.org/10.3390/foods9101488

Salehi, F. (2023). Recent progress and application of freeze dryers for agricultural product drying. *ChemBioEng Reviews*, *10*(5), pp.618-627. https://doi.org/10.1002/cben.202300003

Shofian, N.M., Hamid, A.A., Osman, A., Saari, N., Anwar, F., Dek, M.S.P., & Hairuddin, M.R. (2011). Effect of freeze-drying on the antioxidant compounds and antioxidant activity of selected tropical fruits. *International Journal of Molecular Sciences*, *12*(7), 4678-4692. https://doi.org/10.3390/ijms12074678

Singh, J., Metrani, R., Jayaprakasha, G.K., Crosby, K.M., Jifon, J.L., Ravishankar, S., Brierley, P., Leskovar, D.L., Turini, T.A., Schultheis, J., & Coolong, T. (2022). Profiling carotenoid and sugar contents in unique *Cucumis melo* L. cultigens harvested from different climatic regions of the United States. *Journal of Food Composition and Analysis*, *106*, 104306. https://doi.org/10.1016/j.jfca.2021.104306

SAŽETAK

EFIKASNA LIOFILIZACIJA PLODA DINJE: UMANJENJE VREMENA I SADRŽAJA VLAGE UZ OČUVANJE SADRŽAJA ŠEĆERA I PIGMENATA

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Uzorci pirea dinje testirani su radi optimizacije uslova liofilizacije, kako bi se skratilo vreme samog procesa liofilizacije (do 25 h) i dobio sprašeni biljni materijal sa niskim sadržajem vlage. Pored toga, izvršeno je određivanje sastava i sadržaja pojedinačnih šećera, kao i sadržaja ukupnih karotenoida (i hlorofila) kako bi se testirao kvalitet liofiliziranih uzoraka. Dobijeni rezultati su pokazali da su analizirani uzorci imali sadržaj vlage od 6,30%, pH od 6,47, indeks prelamanja (RI) od 1,346 i ukupne rastvorljive čvrste materije (°Brix) od 8,8. U poređenju sa svežim uzorcima, liofilizirani uzorci su imali 9,7 i 2,8 puta veće količine saharoze i karotenoida, respektivno. Data ispitivanja pokazala su važnost procesa pripreme uzoraka, koji može značajno smanjiti vreme liofilizacije ove vrste uzorka, posebno za nedovoljno iskorišćene, gajene biljke u proizvodnji hrane sa dodatom vrednošću.
KLJUČNE REČI: Brix, karotenoidi, *Cucumis*, plodovi, dinja, pH, pire, šećeri

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