https://doi.org/10.15255/KUI.2022.045
KUI-14/2023
Original scientific paper
Received July 31, 2022
Accepted December 5, 2022

Polyphenols and Flavonoids Contents of Fresh and Dried Apricots Extracted by Cold Soaking and Ultrasound-assisted Extraction

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Abstract

This study was carried out to verify the influence of drying parameters on phenolic and flavonoid compounds of apricots (*Prunus armeniaca* L.) treated with sucrose, NaCl, and sodium bisulphite solutions dried by microwave at different powers (200, 400, and 800 W). We used two extraction methods, namely, cold soaking and ultrasound-assisted extraction (UAE). Total phenolics and flavonoids in fresh and dried apricots and apricot dough were estimated using the Folin-Ciocalteu reagent and the aluminium trichloride method, respectively. Fresh apricot contained considerable amounts of polyphenols and flavonoids by the cold soaking and UAE (285.43 and 165.49 mg GAE/100 g DM and 48.57 and 12.11 mg QE/100 g DM, respectively). Analysis of the data showed that the decrease in polyphenol and flavonoid contents of the dried treated apricots compared to the fresh material was significant. The greatest losses of these nutrients were recorded when applying the ultrasonic extraction method.

Keywords

Drying, polyphenols, flavonoids, cold soaking, ultrasonic extraction

1 Introduction

Apricot is the fruit of the common apricot tree, *Prunus armeniaca* L., of the *Rosaceae* family (*Pomoides* subfamily). In 2019, the Algerian production of apricots amounted to 256,890 t.[1] In general, *Prunus armeniaca* L. is used as a medicine in the treatment of skin diseases, parasitic diseases, and as a source of functional foods against cancer, heart disease, as well as for its ability to protect against chronic diseases.[2] Some studies have reported that apricot fruits are rich in phytochemicals, such as polyphenols, flavonoids, carotenoids, and vitamins C and E, which has been considered a good source of natural antioxidants for human nutrition.[2–6] Phenolic compounds have been associated with antioxidant activity and the prevention of degenerative diseases.[7] Recently, there has been a huge global demand for natural plant-based pigments, which are enriched with antioxidant potential and can replace artificial pigments, especially in the food, pharmaceutical, and cosmetic industries. The majority of petroleum-based or synthetic pigments are said to have adverse effects on human health, which can directly induce hyperactivity and allergenicity in children and other sensitive individuals. Modern health-conscious consumers demand natural plant-based pigments, especially in food applications, which has led researchers to mine plant waste, fruits, and vegetables to isolate natural bioactive pigments.[8] Many synthetic antioxidants, such as butylated hydroxytoluene (BHT) have been used for industrial processing, but their carcinogenic effect and involvement in liver damage are of great health concern. Therefore, the need for antioxidants from natural sources has received much attention, and efforts have been made to identify compounds that can act as antioxidants to replace synthetic antioxidants. Plants are a natural reservoir of substances with antioxidant potential. The use and development of natural antioxidants is highly valued because of their role in protecting human cells from free radical damage.[9] In this context, *Prunus armeniaca* L. displays high concentrations of phytochemicals and high antioxidant activity. It is therefore important to develop efficient methods for the extraction of phenolic compounds and flavonoids. Several extraction techniques can be used to extract bioactive compounds from fruits and vegetables, such as the use of chemical solvents, cold and hot soaking, microwave extraction, pulsed field extraction, and ultrasound-assisted extraction.[10] *Feng et al.*[11] stated that the long duration of conventional extraction methods resulted in damage to phenolic compounds. The authors also stated that the contact between the material

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and heat during extraction can also damage the bioactive compounds in the extracts. The UAE method has advantages such as time reduction.[12] *Huang et al.*[13] stated that UAE is a simple and effective extraction method. Ultrasonic waves damage the cell wall, resulting in the release of cell contents (including bioactive compounds) and heating of the liquid space. The use of ultrasonic waves for food extraction and preservation, because of their safety, environmental friendliness, and low instrumental requirements, is a powerful new processing technology.[14] The use of UAE reduces chemical consumption, extraction time, and degradation of target components. The improvement in extraction efficiency with UAE is attributed to mechanical effects and acoustic cavitation. Ultrasound produces an acoustic cavitation effect that facilitates the penetration of the extraction solvent. UAE has proven to be a technology that can be adapted at any time on a small or large scale.[15]

In our work, the drying efficiency of apricots treated with sucrose, NaCl, and sodium bisulphite solutions dried in the microwave were tested at different powers (200, 400, and 800 W) by the total polyphenol contents and flavonoids, and the same for apricot dough dried in an electric oven at 60 °C. The extraction was based on two methods: cold soaking and ultrasound-assisted extraction. To our knowledge, such work has never been done before.

2 Experimental

2.1 Preparation of apricot extracts

The apricot variety selected in our study is widespread (local variety; Rosé de Manaa from the wilaya of Batna, Algeria). It has a spherical shape, more or less flattened on both poles. Sampling was carried out in two to three homogeneous plots. Fruits were randomly selected from several clusters at different heights and orientations, harvested at full maturity (July), and stored in cold storage at 4 °C. The average moisture content of apricots on a wet basis was approximately 85.93 %.

In this work, the efficiency of the drying process of apricot by domestic microwave was tested by extraction of polyphenols and flavonoids, and compared to the raw material (apricot without drying). For this purpose, three treatments were performed with the following solutions, sucrose (60° Brix), NaCl (6 %), and sodium bisulphite (6 %) before the drying operation. The drying process started at powers of 200, 400, and 800 W; and stopped at a residual moisture content of 5 %. It should be noted that the present work is the continuation of a previous work already published in 2021.[1] The power was varied because the temperature is not exploitable with the microwave (especially in a domestic microwave as used in this work). In

Table 1 – Types of products made from apricot (*Prunus armeniaca* L.) by microwave

Treatment	Product		
sucrose (60° Brix)	 dried at 200 W	 dried at 400 W	 dried at 800 W
NaCl (6 %)	 dried at 200 W	 dried at 400 W	 dried at 800 W
sodium bisulphite (6 %)	 dried at 200 W	 dried at 400 W	 dried at 800 W

addition, the microwave technology is a rapid and efficient technique for drying fruits and vegetables, in which electromagnetic radiation plays an important role in heating the inside and outside parts of the considered products. Technologies related to microwave drying are more advantageous separately or in combination because of a short drying period, better product quality, and flexibility in drying a wide variety of dried products with added value compared to other methods such as the electric oven.[16] On the other hand, the method of drying the dough after preliminary tests by microwave showed the appearance of pores on the texture of the dough due to the rotation of the plate of the microwave, which gave a texture unacceptable to tasters. It was therefore replaced by an electric oven at 60 °C which gave an acceptable texture of the dough. The temperature of 60 °C was considered as the best temperature for drying the fruits; since the reduction of the water content was more evident, the time was shorter compared to the other temperatures (40 and 50 °C), allowing to avoid the enzymatic browning reactions.[17] Finally, the extraction of polyphenols and flavonoids was performed for each treatment and each power according to two techniques: cold maceration and ultrasound-assisted extraction (UAE). In addition, the drying time was set according to the time prediction model obtained by artificial neural network (ANN) (200 W, 400 W, and 800 W for 540, 440, and 320 s, respectively), according to a previous study.[1] For this purpose, microwave power (W), total apricot weight (g), moisture content (%), dry matter content (%), and moisture ratio (MR) were calculated and fed into the ANN model to predict the drying time. The products made from apricots (*Prunus armeniaca* L.) treated with sucrose, NaCl, and sodium bisulphite dried by microwave at different powers (200, 400, and 800 W), and apricot dough dried by electronic oven at 60 °C are presented in Table 1 and Fig. 1.

Fig. 1 – Apricot dough

2.1.1 Cold soaking

An amount of 4 g of fresh apricot and dried products were ground and homogenised, then macerated in 400 ml of methanol for 24 h under continuous stirring in a shaker. After filtration through 0.45 µm filter paper, the filtrates were concentrated in rotary vacuum evaporator at a rotation of 80 rpm and a temperature of 35 °C. After concentrating, the residue was recovered with 10 ml of pure methanol and stored at −18 °C.

2.1.2 Ultrasound-assisted extraction (UAE)

UAE was made using an ultrasonic bath (Soniclean 220 V, 250 W, and 50 kHz, Soniclean Pty. Ltd., Thebarton, Australia). An amount of 4 g of fresh apricot were considered, and the processed products were crushed and homogenised, followed by maceration in 400 ml of methanol for 30 min in an ultrasonic bath at 45 °C. Filtration was then performed before concentrated in a rotary vacuum evaporator. The recovery of the residue was performed with 10 ml of pure methanol; it was then stored at −18 °C.

2.2 Determination of polyphenols

Total polyphenols were determined according to the method described by *Lekbir et al.*[18] and *Ciric et al.*[19] An amount of 0.5 ml of various extracts, namely fresh, dried apricot and apricot dough were diluted with 5 ml of distilled water; then 0.5 ml of Folin-Ciocalteu reagent was added. After 3 min, 0.5 ml of sodium carbonate (7.5 %) was added, and the mixture was incubated in the dark for one hour at room temperature. The absorbance was measured at 760 nm using the VWR UV-1600PC Vis spectrophotometer. Total polyphenols were quantified by calibration curve obtained from measuring the absorbance of a known concentration of gallic acid standard. The results were expressed as in mg of gallic acid equivalent (GAE) *per* 100 g of dry matter (DM) for fresh apricot and dried products.

2.3 Determination of flavonoids

The aluminium trichloride method was used to quantify the flavonoids in apricot extracts. For this purpose, 1 ml of sample (prepared in methanol) was added to 1 ml of $AlCl_3$ solution (2 % in methanol). After 10 min of reaction, the absorbance was read at 430 nm. Flavonoids were quantified by calibration curve obtained from measuring the absorbance of a known concentration of quercetin standard. The results were expressed as mg of quercetin equivalent (QE) *per* 100 g DM for fresh apricot and dried products.[18]

2.4 Statistical analysis

Experimental data were expressed as means ± standard deviations. All determinations were performed in triplicate. A statistical analysis of the results was made using the XLStat 2014 software. An equal mean hypothesis was tested by analysis of variance (ANOVA). The mean was significantly different in comparison to Duncan's test ($p \leq 0.05$).

3 Results and discussion

3.1 Total polyphenols content (TPC)

The results of the TPC of extracts: fresh apricots and apricots treated with sucrose, NaCl, and sodium bisulphite solutions dried by microwave at different powers (200, 400, and 800 W), and apricot dough dried in electric oven at 60 °C extracted by cold soaking and UAE are presented in Table 2 and Fig. 2.

Table 2 – TPC of fresh apricots, pretreated apricots dried by microwave at different powers (200, 400, and 800 W), and apricot dough dried in electric oven at 60 °C extracted by cold soaking method and UAE

Product extract	TPC/ mg GAE/100 g DM	
	Cold soaking	UAE
Apricot	285.43 ± 0.00^{a}	165.49 ± 0.01^{a}
1a	245.88 ± 0.01^{ab}	16.94 ± 0.02^{b}
1b	87.66 ± 0.13^{c}	16.15 ± 0.01^{b}
1c	127.058 ± 0.01^{b}	13.68 ± 0.05^{c}
2a	76.84 ± 0.01^{cd}	12.73 ± 0.01^{c}
2b	75.23 ± 0.00^{cd}	15.02 ± 0.07^{bc}
2c	41.65 ± 0.02^{d}	16.13 ± 0.06^{b}
3a	264.12 ± 0.04^{ab}	12.78 ± 0.04^{c}
3b	261.54 ± 0.13^{ab}	14.34 ± 0.04^{bc}
3c	251.53 ± 0.00^{ab}	17.28 ± 0.05^{b}
Apricot dough	100.90 ± 0.11^{b}	9.81 ± 0.03^{d}

1: Apricot treated with dried sucrose at: 1a. 800 W, 1b. 400 W, 1c. 200 W; 2: Apricot treated with NaCl dried at: 2a. 800 W, 2b. 400 W, 2c. 200 W; 3: Apricot treated with sodium bisulphite dried at: 3a. 800 W, 3b. 400 W, 3c. 200 W.

a, ab, b, bc, c, cd, d, de, e: in each column, the means followed by a different letter are significantly different at $p < 5$ % threshold (Duncan's test).

For the UAE technique, the TPC was about 165.49 mg of GAE/100 g DM, while the cold soaking method allowed reaching the highest level of TPC (285.43 mg GAE/100 g DM), showing that this latter method led to a higher TPC extraction compared to the UAE method.

These fresh apricot TPC values were lower than those obtained by *Akin et al.*,[20] *Ali et al.*,[21] and *Vega-Gálvez et al.*,[2] who reported the following ranges of TPC amounts, 4233.70 to 8180.49 mg of GAE/100 g DM for the variety Bursa and Tokalogleu from the Malatya region of Turkey, 4900 to 7310 mg of GAE/100 g DM for the variety Shai and Habi from the northern region of Pakistan, and 4452.7 mg of GAE/100 g DM, for the variety Tilton, Salamanca province, Coquimbo region, Chile, respectively. This decrease can be attributed to a difference in the variety of apricots used or the complexity of the group of phenolic compounds, as well as to the extraction and analysis methods.

Analysis of the results revealed losses in the TPC of the extracts of apricots dried with microwaves or the extracts of the apricot dough dried in the electric oven at 60 °C, and extracted by cold soaking or UAE, of which the most important loss was recorded for the dried product extracted by UAE.

Regarding cold soaking, the analysis of the results revealed losses in TPC of the extracts of dried apricots compared to fresh apricots, the most important of which were recorded for apricots treated with NaCl solution dried by microwave at different powers of 200, 400, and 800 W with loss values of: 243.78, 210.20, and 208.59 mg of GAE/100 g DM with a significant difference, respectively, followed by the apricot dough dried in electric oven at 60 °C with a loss value of 185.43 mg of GAE/100 g DM with a significant difference, followed by the apricots treated with sucrose solution dried by microwave at different powers 400, 200, and 800 W with loss values of 197.58, 158.37, and 39,55 mg of GAE/100 g DM with a significant difference, respectively, and finally the apricots treated with sodium bisulphite dried by microwave at different powers (800, 200, and 400 W) registered lower losses (39.31, 3.90, and 23.89 mg of GAE/100 g DM with a significant difference, respectively) compared to the other treatments.

The UAE extraction showed very important losses of TPC for the apricot dough dried at 60 °C (155.68 mg of GAE/100 g DM of the losses of the TPC of the dough of apricot by contribution to the fresh apricot). These results were lower than those found by *Khairuddin et al.*,[22] who gave a value of 354.61 (mg GAE/100 g) of dried apricot from the Malaysian region.

Comparing the results obtained from the TPC of dried apricot extracts and apricot dough extracted by cold soaking and UAE to fresh apricot, we can conclude that a decrease in TPC of dried products during drying can also be attributed to the binding of polyphenols with other compounds such as proteins. Interactions between polyphenols and proteins can also lead to the formation of adducts through covalent (irreversible) bonds,[23,24] or to changes in the chemical structure of polyphenols that cannot be extracted or determined by the available methods.[25] Some authors have also reported a decrease in phenolic content in dried apricots, also attributed to enzymatic activity of polyphenoloxidase (PPO); this reflects the ability of PPO to use different phenolic compounds as substrates at temperatures between 55–60 °C.[26] It was found that, during the dehydration process, PPO activity remained high for longer periods when the drying temperature was around 55–60 °C, while shorter exposure periods were required to inactivate the enzyme at temperatures of 75–80 °C.[27,28] On the other hand, the different fluctuations on the TPC can be due to the variation of the microwave power used in relation to the time needed for drying. Indeed, the increase in the drying power allows to reduce the time of exposure to heat because of the fast drying of the whole apricots (case of cold soaking); while on the other hand, it also promotes the degradation of bioactive compounds (case of UAE).

By comparing the two methods of extraction used in our work, it was found that cold soaking was the most efficient

for the extraction of TPC from fresh and dried apricots and apricot dough, compared to UAE on the one hand, and the pretreatment of apricots with sodium bisulphite was the most efficient for preserving TPC during drying, compared to sucrose and NaCl treatments on the other hand.

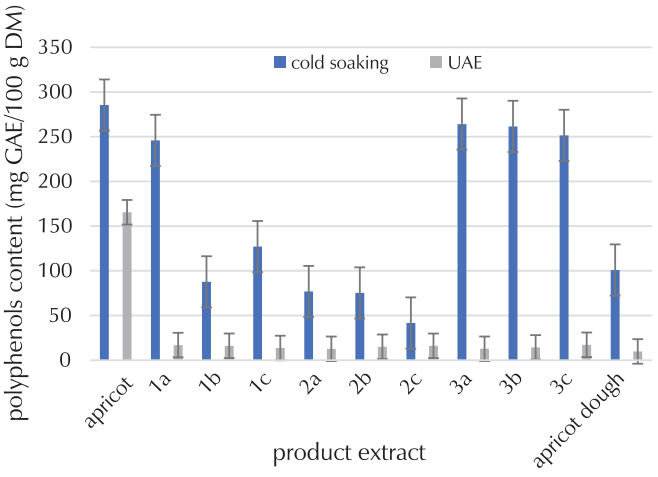

Fig. 2 – TPC of fresh apricots, pretreated apricots dried by microwave at different powers (200, 400, and 800 W), and apricot dough dried in electric oven at 60 °C extracted by cold soaking method and UAE

3.2 Flavonoids content

The results of the flavonoids of fresh apricots and treated products dried by microwave at different powers (200, 400, and 800 W), and the apricot dough dried in electric oven at 60 °C extracted by maceration soaking and UAE are given in Table 3 and Fig. 3.

According to the results obtained by cold soaking (Table 3 and Fig. 3), the fresh apricot contained 48.57 mg EQ/100 g DM of flavonoids, namely, lower than that obtained by *Vega-Gálvez et al.*[2] who reported a flavonoid content of 2104.7 mg EQ/100 g DM for the variety of Tilton, province of Salamanca, region of Coquimbo, Chile. This difference may be due to a difference in the variety of apricot or the method of extraction and analysis used.[8]

Analysis of the results revealed losses in flavonoids for the microwave dried products extracted by cold soaking, the most important of which were recorded for apricots treated with sodium bisulphite and dried at 400 and 200 W (respectively 3.89 and 3.98 mg EQ/100 g DM) and apricots treated with NaCl and dried by microwave at 800 W, with a value of 3.84 mg EQ/100 g DM, with a significant difference compared to fresh apricot. The highest flavonoid content was recorded for apricots treated with NaCl solution dried at 400 W and apricot dough (respectively 11.89 and 6.30 mg EQ/100 g DM with a significant difference). Comparing our results with the fresh product, it can be concluded that there was a decrease in the flavonoids content of the elaborated dried products, showing a negative effect of drying on these nutrients, in agreement with

Table 3 – Flavonoids contents of fresh apricots, pretreated apricots dried by microwave at different powers (200, 400, and 800 W), and apricot dough dried in electric oven at 60 °C extracted by cold soaking method and UAE

Product extract	Flavonoids content / mg QE/100 g DM	
	Cold soaking	UAE
Apricot	48.57 ± 0.03^{a}	12.11 ± 0.01^{a}
1a	4.28 ± 0.04^{de}	2.16 ± 0.04^{d}
1b	5.36 ± 0.11^{d}	3.69 ± 0.08^{bc}
1c	5.65 ± 0.02^{d}	2.63 ± 0.03^{d}
2a	3.84 ± 0.02^{e}	10.74 ± 0.05^{ab}
2b	11.89 ± 0.01^{b}	3.69 ± 0.04^{bc}
2c	7.87 ± 0.26^{c}	2.72 ± 0.04^{d}
3a	5.36± 0.04^{d}	3.50 ± 0.04^{bc}
3b	3.89 ± 0.01^{e}	3.64 ± 0.05^{bc}
3c	3.98 ± 0.03^{e}	4.54 ± 0.06^{b}
Apricot dough	6.30 ± 0.07^{cd}	3.55 ± 0.04^{bc}

1: Apricot treated with dried sucrose at: 1a. 800 W, 1b. 400 W, 1c. 200 W; 2: Apricot treated with NaCl dried at: 2a. 800 W, 2b. 400 W, 2c. 200 W; 3: Apricot treated with sodium bisulphite dried at: 3a. 800 W, 3b. 400 W, 3c. 200 W.

A, ab,b, bc, c, cd, d, de, e: in each column, the means followed by a different letter are significantly different at the p significantly different at $p < 5$ % threshold (Duncan's test).

Ghellam et al.[29] who observed similar drying effect on the leaves of *Corchorus olitorius* dried by microwave.

In the present work, 16.42 g of whole pitted apricots were dried (14.11 g wet weight and 2.31 g dry weight) to a weight of 7.31 g; this variation in the final dry weight should be related to residual moisture. Despite this, the effect of methods and treatments and drying conditions were the main factors affecting the characteristics of dried apricots (polyphenols, flavonoids and extraction, *etc.*), in agreement with the findings of *Chong et al.*[30] and *Joardder et al.*[31] for final leaf drying of herbs in food.

According to the results obtained by the UAE method (Table 3 and Fig. 3), the apricot contains 12.11 mg QE/100 g DM of flavonoids. This value is lower than that obtained by the cold soaking method (48.57 mg EQ/100 g DM of flavonoid) for the same variety of Rosé from the Bouzina region of the wilaya of Batna in Algeria; this difference is due to the extraction method used. By comparing the two methods of extraction used in our work, an important loss of flavonoid content of the dried products extracted by the UAE method can be noticed, and hence, the cold soaking method is more efficient than UAE.

Analysis of the results revealed losses of the flavonoid content of the elaborated products, and the most significant losses were recorded for apricots treated with sucrose solution and dried by microwave at 800 and 200 W (2.16 and

2.63 mg EQ/100 g DM with a non-significant difference, respectively). The highest flavonoids content was recorded for apricots treated with NaCl and sodium bisulphite solutions dried at 800 and 200 W (10.74 and 4.54 mg QE/100 g DM with a significant difference, respectively). Compared to the fresh product, there was a decrease in the flavonoids content after drying. *Casquete et al.*[32] show that the duration of the treatment causes the disruption, disorganisation of tissues, and especially cellular decomposition, leading to an important enzyme-substrate interaction during the treatment and an increase in enzymatic activity, which causes a reduction in the flavonoids content of the dried products.

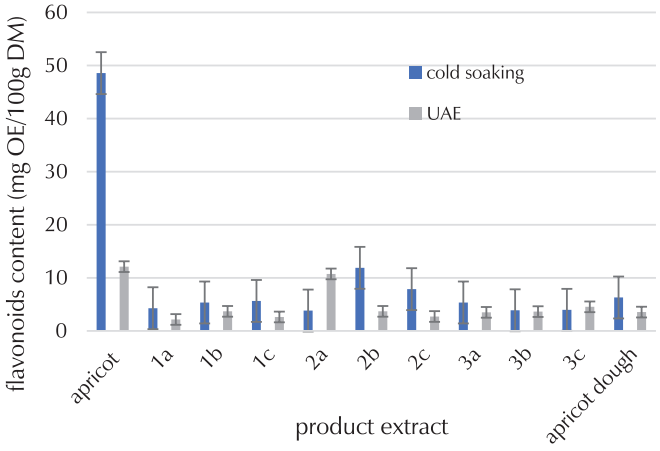

Fig. 3 – Flavonoids contents of fresh apricots, pretreated apricots dried by microwave at different powers (200, 400, and 800 W), and apricot dough dried in electric oven at 60 °C extracted by cold soaking method and UAE

4 Conclusion

The main results show that fresh apricot fruits contain high amounts of polyphenols and flavonoids. The treatment of apricots with sucrose, NaCl, and sodium bisulphite dried by microwave at different powers (200, 400, and 800 W), and apricot dough dried in electric oven at 60 °C showed a negative influence on TPC and flavonoid contents compared to fresh apricots, extracted by cold soaking and UAE. Moreover, cold soaking showed to be the most efficient method for the extraction of nutrients (TPC and flavonoids) from *Prunus armeniaca* L.

ACKNOWLEDGEMENTS

This study was supported by the research laboratory LETPPÉM (Laboratory for the Improvement of Phytosanitary Protection Techniques in Mountain Ecosystems) and LAPAPEZA (Laboratory for the Improvement of Agricultural Productions and Protection of Ecosystems in Dry Areas) of the University Batna 1, Algeria.

List of abbreviations

UAE – ultrasound-assisted extraction
GAE – gallic acid equivalent
DM – dry matter
OE – quercetin equivalent
TPC – total polyphenols content

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SAŽETAK

Sadržaj polifenola i flavonoida u svježim i sušenim marelicama ekstrahiranim hladnim namakanjem i ekstrakcijom potpomognutom ultrazvukom

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U ovom radu ispitan je utjecaj parametara sušenja na fenolne i flavonoidne spojeve marelica (*Prunus armeniaca* L.) tretiranih otopinama saharoze, natrijeva klorida i natrijeva bisulfita te sušene u mikrovalovoj pećnici pri različitim snagama (200, 400 i 800 W). Primijenjene su dvije metode ekstrakcije, hladno namakanje i ekstrakcija potpomognuta ultrazvukom (UAE). Ukupni fenoli u svježim i suhim marelicama te tijestu marelica određeni su primjenom Folin-Ciocalteu reagensa, dok su flavanoidi određeni metodom s aluminijevim(III) kloridom. Obje metode ekstrakcije pokazale su da je svježa marelica sadržavala znatne količine polifenola i flavonoida. U suženim marelicama zabilježene su znatno manje količine polifenola i flavanoida. Najveći gubici tih nutrijenata zabilježeni su prilikom primjene ekstrakcije potpomognute ultrazvukom.

Ključne riječi
Sušenje, polifenoli, flavonoidi, hladno namakanje, ekstrakcija potpomognuta ultrazvukom

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Izvorni znanstveni rad
Prispjelo 31. srpnja 2022.
Prihvaćeno 5. prosinca 2022.