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Osmotic assisted convective drying of pomegranate arils: Process optimisation, structural characterisation, and bioactive compound evaluation

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Abstract

Convective drying of pomegranate arils is time-consuming, energy-intensive, and affects the quality. Osmotic pre-treatment has been resorted prior to convective drying to improve drying performance and quality. In the present work, the osmosis factors such as temperature (30 - 60°C), time (50 - 250 min), and total soluble solids (TSS) (40 - 60°B) were studied using Box-Behnken design of response surface methodology. The quadratic models obtained adequately explained the process, and the optimum conditions were temperature (48.52°C), time (209.65 min), and TSS (51.31°B). The osmotic pre-treatment at optimum conditions followed by the convective tray drying reduced the drying time by nine hours, and the mean energy consumption by 0.172 MJ/g. Light microscopy revealed rupture and breakage of the honeycomb-like cellular structure of the pomegranate aril. Quality analysis of dried arils revealed that the texture (softness) and TSS improved by 11.75 N and 4.2°B, respectively. Likewise, sensorial quality parameters such as taste, mouth feel, and overall acceptability of the OATD (osmo-assisted tray-dried) arils significantly improved over the TD (tray-dried). However, there was a minor higher loss of 15.48, 12.52, and 15.88% in anthocyanin, phenols, and antioxidant capacity in OATD compared to TD. The OATD arils can be stored for up to six months in a modified atmosphere package (MAP).

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Introduction

Pomegranate has received attention in the recent past due to its myriad health benefits and disease-fighting abilities. The edible portion, pomegranate arils, has interesting amounts of flavonoids, anthocyanins, punicic acid, ellagitannins, alkaloids, fructose, sucrose, glucose, simple organic acids, vitamins, minerals, polyphenols, and other components (Zarfeshany *et al*., 2014). Pomegranate, being rich in bioactive compounds like polyphenols, has shown many health-related properties, such as antioxidant, anti-inflammatory, antihypertensive, anticancer, immune-modulatory, antimicrobial, and cardiovascular protective (Kandylis and Kokkinomagoulos 2020; Asgary *et al*., 2021). The pomegranate arils with a moisture content of around 80% (wb) or 400.10% (db) are highly susceptible to microbial contamination, and have very short shelf life. The availability of pomegranate fruits is also limited to certain season (Arendse *et al*. 2014), and the fruits are mostly consumed in fresh form.

Drying is an age-old technique for the preservation of food, wherein the water in the food is removed by vaporisation or sublimation. Therefore, water available for chemical, enzymatic, or microbial degradation reaction is reduced in the food material. Besides improvement in shelf life, drying reduces transportation and storage costs, and the need for cold storage. The important commercial drying techniques include convective tray drying, spray drying, freeze drying, ohmic heating, osmotic drying, *etc*. Freeze-drying is energy intensive and expensive due to its low-temperature and low-pressure operation (Suwelack and Kunke, 2002). Spray drying is suitable only for liquid food products like extracts, juices,

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syrups, concentrates, *etc*. (O'Sullivan *et al*., 2019). Mechanical convective tray drying is the most commonly used technique for perishables like fruits and vegetables, with 85% of dryers manufactured on this principle of drying (Kroehnke *et al*., 2021; El-Mesery *et al*., 2023). Convective hot air drying requires low capital investment but longer drying time, making the process costly (El-Mesery *et al*., 2023), and also affects quality due to hardening and losses in bioactive compounds.

Osmotic drying involves partial moisture removal by direct contact with the product with hypertonic medium. During osmotic drying, two major simultaneous counter-current transport processes are happening. One is the flow of moisture from the product to the osmotic solution, and the other is the flow of osmotic solute from the osmotic solution to the product through an interface. However, at the same time, the third transfer process is also in progress *i.e*. leaching of product solute (sugar, acids, minerals, and vitamins) into the solution. The basic indicators for the effectiveness of the drying process are the drying time required, the energy consumption during the drying process (Szadzinska *et al*., 2019), and the optimum retention of bioactive compounds. The combination of drying processes is being used by researchers to reduce drying time, energy consumption, and cost. Osmotic drying as pre-treatment has been studied in papaya (Chandra *et al*., 2021), kiwifruit (Roueita *et al*., 2020), and pomegranate (Bchir *et al*., 2012a). The main advantage of using osmotic pre-treatment is partial dewatering of the product due to the strong driving force of osmotic solution during osmotic pre-treatment.

There is scanty literature available on the combined effect of osmotic pre-treatment on the quality of pomegranate arils in terms of bioactive compounds and storability of dried arils. In the present work, the osmotic drying of pomegranate arils using a sugar solution was performed as pre-treatment before convective tray drying. The osmotic drying pre-treatment conditions were optimised for maximum moisture reduction, a solid gain, and minimum colour change and loss of bioactive compounds. Further, the convective tray drying followed by osmotic pre-treatment was studied for drying time required, retention of bio-actives, and sensorial quality, followed by storability of dried arils. The microscopic structure as affected by the osmotic pre-treatment was studied for the first time. The energy requirement in osmo-assisted tray drying was also studied which has not been reported hitherto.

Material and methods

The matured fresh pomegranate fruits (cv. Bhagawa) of 165 d from anthesis were harvested from a research farm, and stored in a cold room at 5°C until used for the experiments. Analytical grade chemicals (Hi-media, India) were used for biochemical analysis.

Osmotic drying pre-treatment

The fruits were washed in chlorine water (200 ppm), mopped, and the arils were extracted. The osmotic solution was prepared by adding sucrose to the distilled water for desired total soluble solids (TSS). A 100 g of pomegranate arils were used, and a solute to sugar solution ratio of (1:4) was maintained for osmotic dehydration (Bchir *et al*., 2012a). The arils and osmotic solution were placed in the glass beaker which in turn was placed in the water bath. The still water bath (model WBC012; Labqest, Borosil, India) was used for temperature control during osmosis. The uniformity of the temperature in the water bath was maintained by the stirrer operated at 440 rpm (Khoualdia *et al*. 2018). The temperature of the osmotic solution containing arils was raised to the desired temperature and duration. After the osmotic treatment, the arils were rinsed with distilled water to remove the excess solute adhering to the arils. The surface moisture was removed by placing the arils on blotting paper.

Experimental design

The Box-Behnken design of response surface methodology was used to study the effect of factors *viz*. pre-treatment temperature (A) (30 - 60°C), time (B) (50 - 250 min), and TSS (C) (40 - 60°B) of an osmotic solution on response variables such as water reduction (WR) (%), solid gain (SG) (%), total anthocyanin content (AC) (mg/100 g fresh matter (FM)), colour change (ΔE), total phenols (TP) (mg GAE/kg FM), and antioxidant capacity (AOC) (mg AAE/100 g FM). The selection of the range of factors such as osmosis pre-temperature, time, and TSS was based on the preliminary studies conducted. A total of 17 experimental runs were conducted separately, the results of which are tabulated (Table 1).

Table 1. Effect of independent parameters on response variables for osmotic drying pre-treatment of pomegranate arils.

Standard	Run	Factor 1	Factor 2	Factor 3	Response					
		A: Temp. (°C)	B: Time (min)	C: TSS (°B)	WR (%)	SG (%)	ΔE	AC (mg/100 g)	TP (mg GAE/kg)	AOC (mg AAE /100 ml)
6	1	60	150	40	45.02	22.35	15.14	11.16	770.43	14.60
9	2	45	50	40	12.69	0.25	3.31	24.79	1517.43	25.29
3	3	30	250	50	6.67	1.55	2.93	24.46	1458.10	24.10
13	4	45	150	50	21.46	1.21	1.6	23.01	1687.43	24.15
15	5	45	150	50	17.26	0.79	6.73	23.65	1659.10	23.80
10	6	45	250	40	27.45	1.04	3.96	21.41	1471.10	18.86
5	7	30	150	40	2.08	0.76	5.46	26.23	1495.10	25.04
11	8	45	50	60	14.26	0.31	14.32	24.37	1424.77	25.23
14	9	45	150	50	22.62	1.17	5.99	23.65	1622.43	23.77
17	10	45	150	50	24.12	0.78	1.64	23.11	1642.10	23.90
4	11	60	250	50	50.03	24.41	17.13	8.53	759.10	11.49
2	12	60	50	50	23.21	3.14	13.56	13.21	766.77	21.13
12	13	45	250	60	29.12	1.69	3.29	19.55	1254.77	18.51
7	14	30	150	60	3.67	0.69	1.44	25.12	1480.43	24.94
1	15	30	50	50	1.36	0.08	2.55	26.85	1568.10	27.87
16	16	45	150	50	20.43	1.17	3.16	23.72	1526.43	23.75
8	17	60	150	60	54.11	20.49	18.37	9.52	780.10	14.15

T: temperature; t: time; TSS: total soluble solids; WR: water reduction; SG: solid gain; ΔE: colour change; AC: anthocyanin content; TP: total phenols; and AOC: antioxidant capacity.

Response parameters

Water reduction (WR) and solid gain (SG)

The WR and SG were determined using Eqs. 1 and 2 (Bchir *et al*., 2012b):

$$WR = \frac{(w_0 - w_t)}{w_0} * 100 \quad \text{(Eq. 1)}$$

$$SG = \frac{(s_t - s_0)}{w_0} * 100 \quad \text{(Eq. 2)}$$

where, w = pomegranate arils' weight; s = pomegranate arils' dry matter; t = osmotic pre-treatment duration (min); and subscripts 0 and t = initial and osmotic treatment times, respectively.

Colour change (ΔE)

The colour parameters of the aril samples before and after osmotic pre-treatment were measured using CIE L*, a*, b* scale using a colorimeter, Labscan-XE (HunterLab, USA), defined as L*(lightness), a* (red-greenness), and b* (yellow-blueness) (Gaikwad *et al*., 2017). The colour change (ΔE) was measured using Eq. (3):

$$\Delta E = \sqrt{({L_2}^* - {L_1}^*)^2 + ({a_2}^* - {a_1}^*)^2 + ({b_2}^* - {b_1}^*)^2} \quad \text{(Eq. 3)}$$

Bioactive compounds

The osmotic pre-treated aril samples in different experimental runs were crushed with distilled water and filtered through Whatman filter paper No. 1, and the filtrate was used for further analysis. Total phenolic compounds (TP) were determined in terms of mg GAE /kg FM by the Folin-Ciocalteu (FC) colorimetric method, which is based on the chemical reduction of a mixture of tungsten and molybdenum oxides (Ainsworth and Gillespie, 2007). Total monomeric anthocyanin content (AC) was estimated using pH differential method (Darniadi *et al*., 2019). The FRAP method was used to determine antioxidant capacity (Benzie and Strain, 1996). The results were expressed as ascorbic acid equivalent antioxidant capacity (AEAC) per unit FM weight.

Statistical analysis

Response surface methodology and Box-Behnken design of design expert software evaluation V.12 (Stat-Ease, Inc., Minnesota) was used for the regression and graphical analysis of the experimental data. The surface response of the fitted polynomial regression equations was generated to better understand the relationships between the factors and responses. Further, a numerical optimisation technique (Myers and Montgomery's desirability function) was employed for the simultaneous optimisation of the multiple responses. In order to search for a solution for multiple responses, the goals were combined into an overall composite function called the desirability function (Myers and Montgomery, 2002) All the independent factors, namely temperature, treatment time, and TSS of the osmotic solution, were set within the range. The response parameters WR, SG, AC, TP, and AOC were set for maximisation as we expected the highest WR, SG, and higher retention of AC, TP, and AOC. Whereas, ΔE was set for minimisation since we expected the lowest change in colour. Further, the osmotic pre-treatment was carried out at the optimised conditions (factors) in triplicate, and the average of the observed responses were compared vis-a-vis predicted responses using fitted polynomial regression equations for understanding the desirability of the prediction models.

The principal component analysis (PCA) was used for the response parameters AC, TP, AOC, ΔE, SG, and WR of the osmotic drying pre-treatment. The multivariate data analysis was performed using PAST 4.03 software. The results of the experiment on osmo-dried/pre-treated (OD), TD, and OATD were analysed for comparing means using one-way ANOVA in SPSS software.

Microscopic structure

The fresh and osmotic pre-treated arils at optimum conditions were used to study the tissues of its cut section. The microscopic images of the cellular structure of the tissues were viewed and recorded using a Nikon (Eclipse 90 i, Kawasaki, Japan) light microscope equipped with a Nikon camera (DS-Ri 1, Kawasaki, Japan).

Convective tray drying

The fresh and osmotic pre-treated arils were dried at 50°C with 1.25 kg/m^2 of tray loading. The drying of the arils was continued with aril samples (at the optimised condition) being dried in a convective tray dryer (Macro Scientific) up to an approximate moisture content of 10% (wb) or 11% (db), and drying curves were plotted for the control and osmotic pre-treated samples. The energy consumed during convective tray drying was measured using a

standard electric meter with a precision of 0.1 kwh, and recalculated in terms of MJ/g of moisture evaporated.

Quality analysis of fresh and dried arils

The fresh, OD, TD, and OATD arils were analysed for texture, together with ΔE, TSS, TP, AC, and AOC. The texture was measured by uniaxial compression of a single fresh or dried aril sample using a texture analyser (TA-XT Plus, Stable Micro Systems, UK), in strain mode at 80% strain. The 25 mm diameter cylindrical probe was used at a test speed of 0.5 mm/s, pre-test speed of 1 mm/s, and post-test speed of 10 mm/s (Bchir *et al*., 2012b). The TSS of the samples was measured using a digital pocket refractometer, with values corrected to 20°C. The ΔE, TSS, TP, AC, and AOC were determined as described earlier.

Sensory evaluation

The TD and OATD aril samples were evaluated by a panel of judges on a nine-point hedonic scale (Ranganna, 2000). The parameters used for evaluation were colour, taste, texture, mouth feel, and overall acceptability.

Storage of dried pomegranate arils

The OATD pomegranate arils dried at the optimised condition with an approximate final moisture content of 10% (wb) or 11% (db) were used for the storage studies. The dried arils were packaged in a packet of laminated aluminium foil. The packets were filled with air (control) and modified atmosphere (30% CO_2 + 70% N_2), and stored at 30°C for six months. The samples were drawn at intervals of one month for microbial quality analysis. The total aerobic plate count and total yeast and mould count were determined and expressed as log CFU/g (Gaikwad *et al*., 2017).

Results and discussion

Water reduction (WR) and solute gain (SG)

The WR and SG are the most important parameters, and varied from 1.36 - 54.11% and 0.08 - 24.41%, respectively (Table 1). It was observed that there was no water reduction for a pre-treatment time of 50 min at 42°C, and even up to 250 min at 36°C (Figure 1a). Higher WR has been observed at 48 ± 3°C and beyond. The little WR up to 42°C or meagre SG up to 54°C (Figure 1b) can be explained by the fact that the higher temperature decreased the viscosity of the sugar solution, reducing the external resistance to the mass transfer of the moisture from the aril and solute transfer to the aril through the membrane. Similar results were reported in the osmotic drying of pomegranate arils (Mundada *et al*., 2010).

It is interesting to note that at constant TSS of 50°B and temperature of 50°C and above, there was rapid WR up to 180 min of pre-treatment time (Figure 1a), and then it became constant. Similarly, it was observed that SG was rapid up to 180 min, then became constant, and then decreased for temperatures above 54°C (Figure 1b). The initial rapid WR and SG were followed by a constant phase, and then decreased later on, and can be explained by the higher osmotic driving force between the hypertonic solution and the inside of the pomegranate aril at the start of osmotic drying, which decreased over time, and governed both counter-current mass transfer processes (Gaoula and Harris, 2012; Lopez *et al*., 2020).

The formation of a solute layer on the cell membrane surface was also the reason for the decreased rates of WR and SG after a certain time (Gaoula and Harris, 2012). Equilibrium was reached in the present case after 180 min, which varied in different cases studied in the past depending on the commodity.

It was also observed that WR and SG increased with an increase in TSS (Figures 1c and 1d). The contour lines (Figures 1c and 1d) show that, for the same amount of WR or SG, lower osmosis time was required at higher TSS. The WR was more pronounced at higher TSS compared to SG. The increase in SG was quite low compared to WR. The increased WR with an increase in TSS can be explained by the higher osmotic pressure of the osmotic solution leading to a higher driving force (Alam *et al*., 2019).

The analysis of variance (ANOVA) of the quadratic model for WR showed a significant model (R^2 = 0.9833) ($p < 0.05$) and a non-significant lack of fit. Similarly, an ANOVA of the quadratic model for SG also depicted a significant model (R^2 = 0.9807) ($p < 0.05$) and a non-significant lack of fit.

The results of regression analysis for WR and SG can be expressed as second-order polynomial in Eqs. 4 and 5. The mathematical models in Eqs. 4 and 5 were derived from Figures 1a, 1c, and 1b, 1d, respectively:

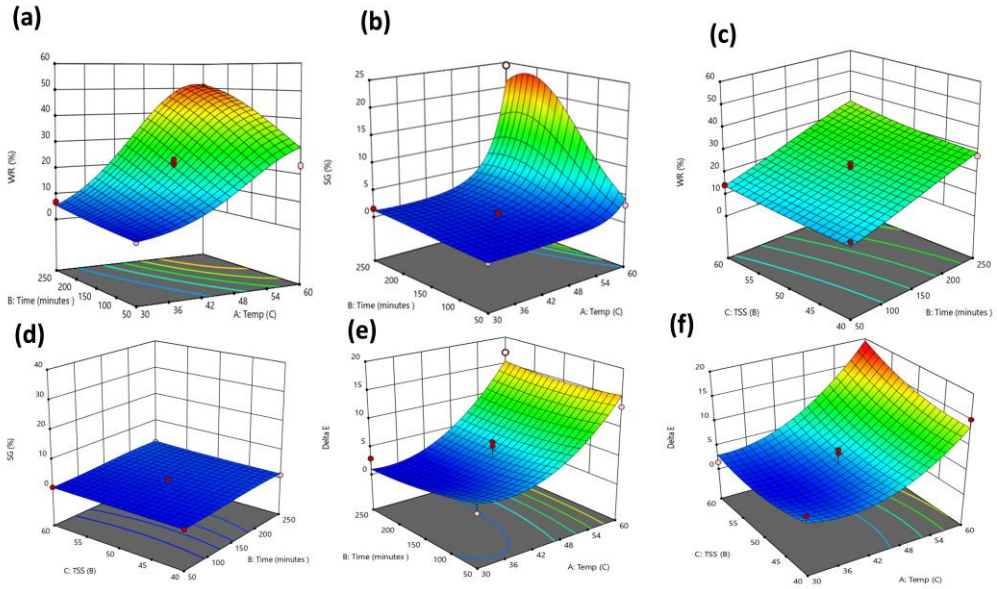

Figure 1. Effect of temperature, time on **(a)** the water reduction and **(b)** solid gain, and **(e)** colour change (ΔE); TSS and time on **(c)** water reduction and **(d)** the solid gain, and **(f)** colour change (ΔE).

$$\text{Log10(WR)} = -1.32 + 0.5767 * \text{A} + 0.2088 * \text{B} + 0.0504 * \text{C} - 0.0895 * \text{AB} - 0.0417 * \text{AC} - 0.0063 * \text{BC} - 0.2704 * A^2 - 0.0475 * \text{B}^2 + 0.0144C^2 \quad \text{(Eq. 4)}$$

$$Log10(SG) = +0.0005 + 0.7156 * A + 0.4406 * B + 0.0270 * C - 0.0972 * AB + 0.0000 * AC + 0.0317 * BC + 0.5290 * A^2 - 0.2833 * B^2 + 0.0660 * C^2 \quad \text{(Eq. 5)}$$

where, A = temperature, B = time, and C = total soluble solids.

The coefficients of factors A, B, and C (Eqs. 4 and 5) show that temperature has the most pronounced effect on WR and SG, followed by time and TSS.

Change in colour (ΔE)

During the osmotic drying process, the L* value increased while the a* and b* values decreased (Table 2), causing ΔE to vary from 1.44 to 18.37 (Table 1). The increase in osmosis temperature beyond 50°C has shown significant increase in ΔE thereby showing a colour change (Figure 1e). At a temperature of 50°C and beyond, the time of osmosis had significant effect on ΔE. The change in colour at the higher temperature of osmosis can be attributed to the leaching of anthocyanin induced by reduced viscosity and higher osmotic driving force (Bchir *et al*., 2012a). The TSS had significant effect on ΔE only beyond 54°C temperature for TSS of 50°B and beyond (Figure 1f). It was observed that L* values increased, whereas a* and b* values decreased with an increase in temperature, time, and TSS (Table 2). The higher L* values after osmotic pre-treatment were due to the protective effects of sugars on colour, and the presence of sugar over osmo-dehydrated samples (Zhao *et al*., 2017). The decrease in a* and b* values can be attributed to the decrease in anthocyanin pigment and yellowness, respectively.

ANOVA of the quadratic model for ΔE showed significant model (R^2 = 0.8874) (p < 0.05), with a non-significant lack of fit. Results of regression analysis revealed the linear Eq. 6 for ΔE. The mathematical model in Eq. 6 have been derived from Figures 1e and 1f, which depict the relationship between the response ΔE and factors temperature, time, and TSS:

$$\Delta\text{E} = +3.82 + 6.48 * A - 0.8037 * B + 1.19 * C + 0.7975 * AB + 1.81 * AC - 2.92 * BC + 4.55 * A^2 + 0.6680 * B^2 + 1.73 * C^2 \quad \text{(Eq. 6)}$$

Bioactive compounds

Anthocyanin content (AC)

Anthocyanins are the pigments that impart a red colour to the arils, and they varied from 8.53 to 26.58 mg/100 g FM (Table 1). The temperature and time of pre-treatment had significant effect on AC of arils during osmotic pre-treatment, with a prominent effect of temperature compared to time. The increase in temperature beyond 45°C showed a sharp decrease

Table 2. Quality of fresh pomegranate arils in comparison with tray-dried and osmotic-assisted tray-dried arils.

Quality parameter	Treatment			
	FA	TD	OD	OATD
ΔE	0.00 ± 0[d]	5.36 ± 0.23[c]	6.98 ± 0.43[a]	6.17 ± 0.13[b]
Texture (N)	32.06 ± 1.41[c]	89.65 ± 4.12[a]	29.25 ± 4.07[d]	77.83 ± 4.36[b]
TSS (°B)	16.23 ± 0.06[d]	20.83 ± 0.29[c]	22.83 ± 0.29[b]	25.03 ± 0.06[a]
AC (mg/100 g FM)	27.61 ± 1.54[a]	22.09 ± 3.09[b]	19.77 ± 1.07[bc]	17.81 ± 0.31[c] (35.48%)
TP (mg GAE/kg FM)	1717 ± 10.00[a]	1504 ± 15.28[b]	1405 ± 7.64[c]	1289 ± 10.41[d] (24.94%)
AOC (mg AAE/100 g FM)	28.28 ± 0.35[a]	22.97 ± 0.00[b]	20.32 ± 0.35[c]	18.48 ± 0.35[d] (34.65%)

FA: fresh arils; OD: osmotic dried/pre-treated; TD: tray dried; OATD: osmotic-assisted tray-dried; ΔE: colour change; AC: anthocyanin content; TP: total phenols; and AOC: antioxidant capacity. Values are mean ± standard deviation. Means followed by different lowercase superscripts within the same row are significantly different ($p < 0.05$). Values in parenthesis in OATD column are percentage reduction in bioactive compounds as compared to fresh arils.

in AC, which can be attributed to the migration of anthocyanin pigment from aril pulp to the osmotic solution. Similar observations were reported by other researchers (Mundada *et al*., 2010; Bchir *et al*., 2012a; Sharif *et al*., 2018). The magnitude and duration of heating also had significant effect on the stability of AC, which also might be the reason for the lower AC in osmotic pre-treated arils. Further, it was also observed that the TSS did not have significant effect on AC of the pre-treated aril samples during the osmosis process. Similar results were observed in the case of the osmotic drying of wild blueberries (Sharif *et al*., 2018).

ANOVA of the quadratic model for AC showed significant model ($R^2 = 0.9987$) ($p < 0.05$), with a non-significant lack of fit. The results of regression analysis revealed a second-order polynomial (Eq. 7) for anthocyanin content:

$$\mathrm{AC} = +23.43 - 7.53 * A - 1.91 * B - 0.6287 * C - 0.5730 * AB - 0.1317 * AC - 0.3604 * BC - 4.84 * A^2 + 0.3186 * B^2 - 0.5767 * C^2 \quad \text{(Eq. 7)}$$

Total phenols (TP)

The osmotic pre-treatment affected TP in pomegranate arils, which ranged between 759.10 and 1687.43 mg GAE per kg FM (Table 1). Total phenols decreased as temperature and pre-treatment time increased. The temperature had significant effect on the total phenols, particularly beyond 48°C. Further, it was seen that the effect of temperature was more prominent than time. The effect of time of pre-treatment on TP was greater at lower temperatures. The decrease in TP was due to the migration of phenolic compounds from the aril pulp to the solution caused by a concentration gradient between the solution and pomegranate pulp inside the arils. The migration process is hastened by the temperature (Bchir *et al*., 2012a; Sharif *et al*., 2018). The thermal degradation of TP with temperature and time was also reported by Vardin and Yilmaz (2018), and can be attributed to the activation of some oxidative and hydrolytic enzymes, the release of bound phenols, the partial degradation of lignin leading to the release of phenolic acid derivatives, and the start of thermal degradation of the phenolic compounds (Maillard and Berset, 1995). The TSS of the osmotic solution did not have significant effect on TP. The result was supported by the findings in the case of the osmotic drying of wild blueberries (Sharif *et al*., 2018).

ANOVA of the quadratic model for TP showed significant model ($R^2 = 0.9850$) ($p < 0.05$), with a non-significant lack of fit. The results of regression analysis revealed a second-order polynomial (Eq. 8) for total phenols:

$$\mathrm{TP} = +1627.50 - 365.67 * A - 41.75 * B - 39.25 * C + 25.58 * AB + 6.08 * AC - 30.92 * BC - 387.49 * A^2 - 101.99 * B^2 - 108.49 * C^2 \quad \text{(Eq. 8)}$$

Antioxidant capacity (AOC)

The AOC is one of the important quality parameters, and mainly contributed by TP, AC, and ascorbic acid present in the arils. During osmotic drying, the temperature of the hypertonic solution and the osmosis time both significantly affected AOC of pomegranate arils. Interestingly AOC was observed to decrease sharply beyond 42°C and 50 min of

osmosis time. This threshold temperature for a sharp decrease in AOC increased with the increase in osmosis time. At higher osmosis temperatures, AOC decreased sharply. The loss of AOC with temperature can be associated with a decrease in the bioactive compounds, such as TP and AC, due to thermal degradation and leaching into an osmotic solution during osmotic drying pre-treatment. The loss of antioxidant activity during drying with an increase in temperature and drying time was reported by other researchers (Bchir *et al*., 2012a; Vardin and Yilmaz, 2018; Rahaman *et al*., 2019). The total soluble solids or concentration of the osmotic solution had little effect on AOC during osmotic drying of pomegranate arils.

ANOVA of the quadratic model for AOC showed significant model (R^2 = 0.9984) ($p < 0.05$), with a non-significant lack of fit. The results of the regression analysis revealed a second-order polynomial (Eq. 9) for AOC:

$$\text{AOC} = +23.87 - 5.07 * A - 3.32 * B - 0.1190 * C - 1.47 * AB - 0.0895 * AC - 0.0725 * BC - 2.51 * A^2 - 0.2182 * B^2 - 1.68 * C^2 \quad \text{(Eq. 9)}$$

Optimisation of osmotic drying process

The final optimised process conditions obtained were temperature (48.52°C), time (209.65 min), and TSS (51.31°B) with predicted values of responses WR (34.98%), SG (2.25%), ΔE (5.48%), AC (19.93 mg/100 g), TP (1453.42 mg GAE/kg), and AOC (20.23 mg AAE/100 g). This solution which has the highest combined desirability (0.622) was nearest to the response goals. At optimum factors, the average of three replications for the observed responses was WR (29%), SG (3.11%), ΔE (6.98), AC (19.77 mg/100 g FM), TP (1405.43 mg GAE/kg FM), and AOC (20.32 mg AAE/100 g FM). Therefore, the use of optimum conditions derived has provided higher WR, SG, AC, TP, AOC, and lower ΔE. It has been seen that the results of the observed responses and predicted values of the responses are quite close. Thus, it can be said that the fitted polynomial regression equations for response variables were suitable for the prediction.

Principal component analysis (PCA)

PCA is one of the most popular multivariate techniques because it reduces the dimensionality, compresses the noise, and correlates measurements in a simple informational sub-space of the dataset. The dimensional analysis of the response parameters, *viz*. WR, SG, ΔE, AC, TP, and AOC, has generated the bi-plot (Figure 2). The variability within the dataset was clearly explained by only two principal components (PC1 and PC2). PC1 accounted for 86.64% of the variability in the dataset, while PC2 accounted for 6.99% of the total 93.63% variability (Figure 2). AOC, AC, and TP fell in the same quadrant of the PC1, whereas ΔE, SG, and WR fell in the opposite quadrant of the PC1. This indicated that the bioactive compounds such as AC, TP, and consequent AOC were lower and affected by the higher osmosis temperature and time, while WR, SG, and ΔE were higher. The experimental runs R1, R11, and R13 fell in the opposite quadrant to the experimental runs R2, R3, R7, R8, R14, and R15, and followed the opposite trend, as the former represented runs with higher osmotic temperatures and times, and the latter represented runs at lower temperatures and times.

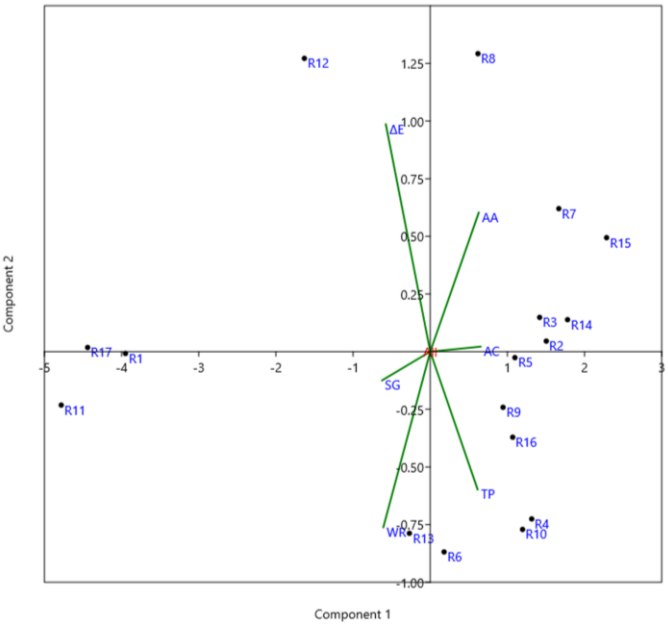

Figure 2. Bi-plot between two principal components (PC1 and PC2) in rotated space generated from PCA of response parameters for different osmotic drying pre-treatment conditions.

Microscopic structure

The tissue of the pomegranate aril is composed of a several cells and intracellular spaces. The microscopic image of the cut section of the fresh aril (Figure 3a) shows the tissue with cells and intercellular spaces. It is interesting to note that the cells are arranged in grid like a hexagonal honeycomb structure (Figure 3a). The moisture removal and

solute incorporation during osmotic pre-treatment certainly affected the cellular structure of the arils (Figure 3b). It can be observed that there is a loss of shape, disruption, and breakdown of the cell wall of the osmo-treated samples compared to the control arils. Several studies, including in apple (Muniz-Becera *et al*., 2020) and strawberry (Prinzivalli *et al*., 2006) have reported the disintegration, disconnection, and breakdown of the cell wall, and the subsequent destruction of tissue continuity during osmotic dewatering.

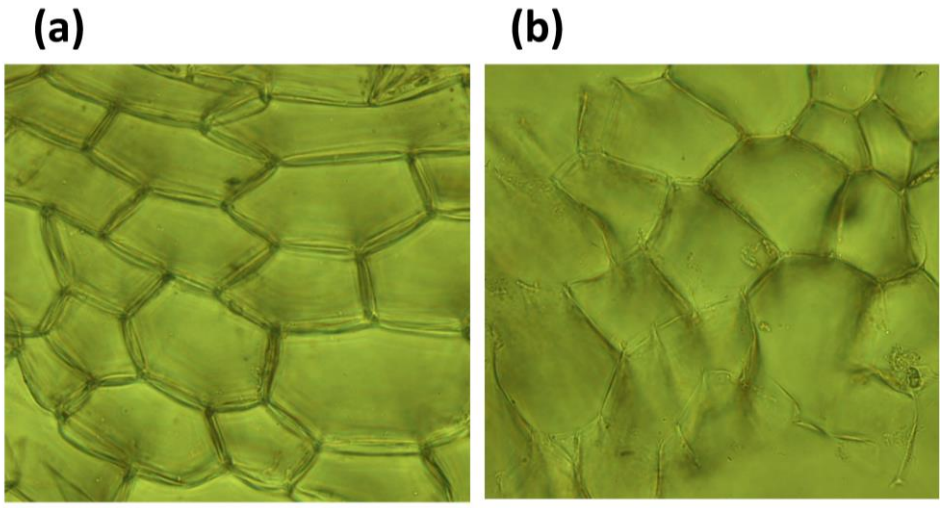

Figure 3. Microscopic view of **(a)** control aril and **(b)** osmo pre-treated arils.

Convective drying

Moisture kinetics and energy consumption

The drying curves for TD and OATD of arils were plotted (Figure 4). During osmotic pre-treatment, the moisture content of arils decreased to 67.42% (wb) from 80% (wb) or 207.10% (db) from 400.10% (db). The drying curve revealed that OATD required a drying time of 5 h, whereas TD required 14 h of drying time to reach an approximate moisture content of 10% (wb) or 11% (db), saving 9 h of drying time. The drying rate during the OATD was higher compared to TD. This could have been due to the cell wall breakage during the osmotic drying pre-treatment of pomegranate arils. The TD at 60°C of pomegranate arils from the moisture content of 271.80% db to 8.70% db required 6 h (Kingsly and Singh, 2007), which was higher than OATD and lower than the TD in the present work. This might be explained by the initial moisture content difference between the pomegranate arils used for drying. The drying times for pomegranate arils dried at 55°C using convective hot air, vacuum, ultrasound-assisted vacuum drying, and freeze-drying at -55°C were 15.83, 10.83, 9.50, and 57 h, respectively (Ozay-Arancioglu *et al*., 2021).

The mean energy consumption per unit weight of arils during TD was 0.23 MJ/g, whereas that for OATD was 0.058 MJ/g, which was 25.21% lower

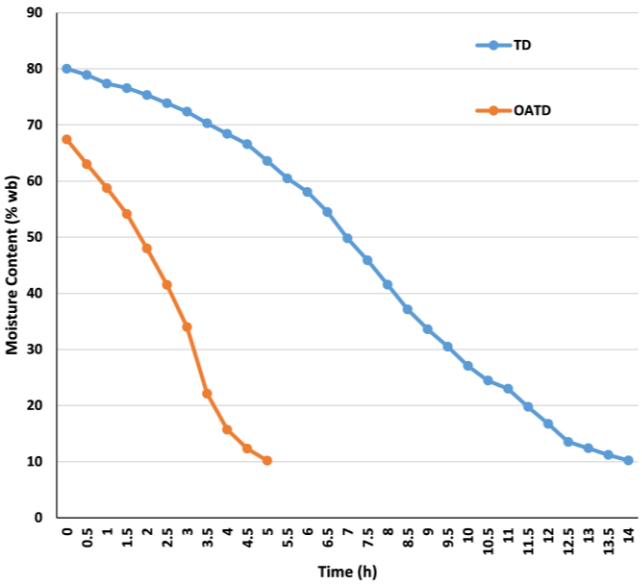

Figure 4. Moisture kinetics during convective drying of control and osmo pre-treated pomegranate arils.

than TD. This led to savings on electric energy consumption during the drying process. Based on the results obtained, OATD showed 2.8 folds decrease in drying time, and 4 folds decrease in energy consumption than TD, making it an energy-efficient and cost-effective alternative drying method for the industrial drying of pomegranate arils. The OATD is a process essentially easy to adopt on a commercial scale as it is easy to upscale, and requires low capital

investment. Further, since the process is energy efficient, it requires low recurring costs in commercial enterprise.

Quality of dried pomegranate arils

The fresh arils (FA), TD, OD, and OATD arils were studied for quality characterisation (Table 2). The texture of the OD arils was softer. However, among the final dried products, OATD arils were softer and had lower texture (77.83 N) compared to TD arils (89.65 N), which agreed with the observations of previous researchers (Correa *et al*., 2011; Rahaman *et al*., 2019). The change in colour (ΔE) in fresh aril was significantly different for all the treatments, with the highest colour change being noticed in OD, followed by OATD and TD. The higher colour change in OD can be attributed to the loss of the anthocyanin colour pigments. The higher colour changes in OD than in OATD suggested that the losses of AC due to leaching were greater during OD. Further, TD followed by OD during OATD reduced more moisture, and the concentration effect of AC due to the reduction in moisture showed lower colour change. The change in colour (ΔE) in the case of microwave drying at 350 w and hot air drying at 60°C of pomegranate arils was 23.72 and 8.38, respectively (Horuz and Maskan, 2015), which was quite higher (6.17) than in OATD observed in the present work.

The comparative evaluation of the final dehydrated product showed that TSS of OATD samples was significantly higher (25.03°B) compared to that for TD (20.83°B), which can be attributed to the loss of moisture and solid gain (Xiao *et al*., 2020). Hence, the highest TSS was observed in OATD samples where there was solid gain during osmotic treatment, and moisture loss during both osmosis and tray drying. The higher TSS to the tune of 25°B positively affected the acceptability of the dried arils. The percent increase in TSS of OATD arils over TD was found to be 4.2°B, and texture (softness) improved by 11.75 N; likewise, sensorial quality such as taste, mouth feel, and overall acceptability of the OATD arils significantly improved over TD.

Lower AC (17.81 mg/100 g FM), TP (1289 mg GAE/kg FM), and AOC (18.48 mg AAE/100 g FM) were observed during OATD compared to AC (22.09 mg/100 g FM), TP (1504 mg GAE/kg FM), and AOC (22.97 mg AAE/100 g FM) during TD (Table 2). However, there was a minor higher loss of 15.48, 12.52, and 15.88% in AC, TP, and AOC in OATD compared to TD. The loss of AC and TP during OATD was due to the leaching of bioactive compounds during the osmosis process, which was discussed in depth in earlier sections. In the present work, the percent reduction in bioactive compounds as compared to fresh arils was (35.48%) AC, (24.94%) TP, and (34.65%) AOC, respectively, for OATD. Ozay-Arancioglu *et al*. (2021) observed that the percent loss in AC and TP compared to fresh arils during convective hot air, vacuum, and ultrasound-assisted vacuum drying was 55.34, 36.14, 23.25%, and 25.43, 22.26 and 16.30% respectively. The percent loss of AC and TP was higher in convection hot air drying, at par with vacuum, and lower than ultrasound-assisted vacuum drying in comparison to OATD observed in the present work.

The photographs of the fresh, tray-dried, and osmo-assisted tray-dried aril samples show the quality of dried arils. The radar plot shows the mean sensory score for the TD and OATD pomegranate aril samples. The sensory scores for taste, texture, and mouth feel, as well as overall acceptability, were higher for the osmo-assisted tray-dried arils, whereas the sensory score for colour was higher for the tray-dried arils. The better taste in the case of the OATD arils might be due to the solid gain in terms of sugar contributing to enhanced taste. The sensory data for the texture are in line with the texture analysis using a texture analyser, which might be due to the hydrothermal effect during osmotic pre-treatment leading to the softness of the seeds. The overall acceptability, which is the culmination of the other sensory parameters, was higher for OATD arils than for TD arils. TD arils had higher sensory score for colour than OATD arils.

Storage

The results of the storage studies revealed that there was no microbial growth for up to three months in OATD arils stored at 30°C, and packaged either in a laminated aluminium foil pouch filled with air (control) or modified atmosphere (30% CO_2 + 70% N_2). However, the control samples had shown a total aerobic plate count of 5.43 log CFU/g, and a total yeast and mould count of 0.17 log CFU/g at the end of the fourth month. Here the total aerobic plate count has exceeded the acceptable limits (5 log CFU/g), and the total yeast and mould count was within the acceptable limits (4 log CFU/g) for dried products as per FSSAI guidelines. The increase in the total aerobic plate count in control samples might be due

to the presence of oxygen in the pouch. Meanwhile, the MAP (30% CO_2 + 70% N_2) samples had no microbial growth of either type even at the end of a six-month storage period. The higher storability for MAP can be attributed to the antimicrobial effect of CO_2 during storage.

Conclusion

The studies on osmotic pre-treatment of the pomegranate arils revealed that the temperature, time, and TSS of osmotic pre-treatment had significant effect on WR, SG, and ΔE, and quadratic model explained the relationship of the variables. The time and temperature had significant effect on the bioactive compounds of the pomegranate arils namely AC, TP, and AOC during osmotic pre-treatment. The quadratic model adequately explained the relationship between these factors with the variables. The optimum osmosis pre-treatment conditions were temperature (48.52°C), time (209.65 min), and TSS (51.31°B). The application of the optimised pre-treatment conditions reduced the moisture content of the arils to 67.42% wb or 207.10% db from the initial moisture content (80% wb or 400.10% db). The osmotic pre-treatment followed by the convective tray drying resulted in a decrease in drying time by 9 h, and a corresponding decrease in the mean energy consumption per unit weight of the arils, 0.172 MJ/g. The light microscopy revealed the honeycomb-like structure of the pomegranate aril cell which was ruptured and broken due to osmotic pre-treatment. The quality analysis revealed a texture reduction of 11.75 N in OATD arils, and corresponding increase in softness over tray-dried arils. The TSS was increased by 4.2°B in OATD arils over the TD arils. However, there was a loss of 15.48, 12.52, and 15.88%, respectively in AC, TP, and AOC, respectively, compared to TD. The sensory evaluation concluded the superiority of the OATD arils over TD arils in terms of the texture, taste, mouth feel, and overall acceptability, but not in colour. The OATD arils can be stored for up to six months in MAP (30% CO_2 + 70% N_2) in laminated aluminium foil at room temperature. The nutritious and convective dried arils with higher shelf life can either be consumed directly or used as an ingredient in snacks, energy bars, ice cream, yogurts, milkshakes, salad dressings, *etc*. The future direction of the research in osmo-assisted drying of pomegranate arils may be focused on profiling of phenols and anthocyanin, as affected by the drying process, *in vitro* bioaccessibility of bioactive compounds for OATD arils, and accelerated storage studies for long-term storability of the dried arils.

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