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Ultrasound Pre-treated Osmotic Dehydration of Elephant Apple (*Dillenia indica*) Slices

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Osmotic dehydration of elephant apple fruit slices was optimized using a Box-Behnken design. Sugar concentration, immersion time and drying air temperature were chosen as the experimental input variables. These were optimized by estimating the desirable osmotic properties and by evaluating the phenolic compounds, antioxidant activity and rehydration ability. The regression coefficients of all the valid response models have been determined and the possible effect of variables at individual and interactive levels has been analyzed. Osmo-air dried elephant apple slices with acceptable quality could be produced by subjecting the raw slices to sugar syrup dipping at 60°Brix, for 4h followed by air drying at a temperature of 55°C.

Keywords: Ascorbic acid, Box-Behnken design, Optimization, Ultrasound

Introduction

Elephant apple (*Dillenia indica L.*) originates in the area of Indo-Malaysia, ranging from the Philippines to tropical Australia. It is based in the sub-Himalayan region of India, from Garhwal to Guwahati, West Bengal and Odisha.[1,2] Reviews of literature have shown that the plant has excellent therapeutic benefits along with phytochemicals, anti-oxidative properties and anti-diabetes activities.[1,3] The fleshy sepals of the fruit are high in ascorbic acid and tannin, including flavonoids.[3] Fruits are seasonal and principally perishable due to higher water activity. Reasonable preservation strategies should therefore be built to prolong the shelf life of this gift of nature and to produce value-added foodstuffs. Progress of innovative technologies developments is constantly taking place using different drying methods.[4] Osmo-dehydration is one of the preferred drying operations to obtain improved product quality.[5] A variety of study has been carried out on microwave-assisted drying[6], but very little literature examined the ultrasound—assisted osmotic dehydration of fruit slices critically. Ultrasound as a pretreatment to drying enhances the drying rate there by reducing the drying time and subsequent improvement in product quality.[1] Thus the goal of present research was to use ultrasound as a pretreatment to the drying of elephant apple slices.

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Materials and Methods

Materials

Properly matured, solid but unripe elephant apple fruits (10 kg) were collected from a farmer's field near OUAT, Bhubaneswar. The initial moisture content of fresh fruits was 88.5± 0.9 percent (wb). Damage-free fruits were selected, carefully washed and cleaned with cool water. The sepals were cut and separated from the central waste and stalk using a self-designed hand operated cutter.[7] Sepals were sliced length wise at a standard width of 10 mm, a typical size used by consumers. The slices were then peeled and were subjected to osmosis as per the experimental design. The chemicals from Qualigens Fine Chemicals (Mumbai, India) and HiMedia (Mumbai, India) were used in this research.

Response Surface Design of Osmo-Convective Drying

Osmo-air drying process of elephant apple slices was designed as per Box-Behnken concept. Response surface methodology (RSM) was used to produce response surface plots and to optimize process variables using a commercial statistical system MINITAB version 13 (Minitab Inc, USA, trial version).[8] Three input variables of experiment were sugar concentration, immersion time and drying air temperature and ranged from 40 to 60°Brix, 2 to 6h and 50 to 70°C. Total 15 sets of experiments were conducted and corresponding osmotic properties in terms of water loss and solids gain and quality characteristics such as rehydration ratio (RR), antioxidant activity (AOA), ascorbic acid (AA), and total phenolic (TP) content were quantified. The second order models generated for all the output variables were tested for significance and were used to optimize input variables to achieve best possible qualities in the finished product using multiple regression technique.[9]

$$Y=\alpha_0+ \alpha_1X_1+ \alpha_2X_2+ \alpha_3X_3+ \alpha_4X_1^2+ \alpha_5X_2^2+ \alpha_6X_3^2 +\alpha_7X_1X_2+ \alpha_8X_2X_3+ \alpha_9X_3X_1 \quad \dots (1)$$

Ultrasound Treatment

The ultrasonic vacuum assisted water bath (Kunshan Ultrasonic Instrument Co., Jiangsu, China) was connected with a vacuum pump, a digital timer and a temperature controller as presented in Fig. 1.[1] The elephant apple slices were imperiled to ultrasonic waves at a constant frequency of 35 kHz with intensity of 4870 W/m^3 at a sonication time of 20 minutes. The slice to solution ratio was maintained at 1:10 as per the findings from preliminary experiments.[1] The products were removed from the bath and weighed separately after wiping the surface. This pretreatment was given to all the 15 samples under osmo-convective drying experiment.

Osmotic Treatment and Convective Drying

In order to achieve the designed sugar concentration, the osmotic syrup was prepared by combining the various proportions of commercial sugar with distilled water.[10] The ultra-sonicated samples were weighed and immersed in the preheated osmotic solution to the designed temperature (40°C). This was maintained throughout the osmosis using a digital water bath (Wishwo instrument, Model-WBS-1000). To enable the proper soaking of the slices, the initial ratio of fruit slices to osmotic solution was maintained as 1:4.[11] The slices were withdrawn after each preset time period, drained quickly and rinsed with water for 30 seconds. After wiping the surface water, the slices were weighed and subjected to hot air drying until they attained a stabilized weight. The osmo-dehydrated slices, which had already lost some of its moisture, were subjected to hot air convective drying.[6] The fruit slices were dried in single layers in a convective dryer (IIC, Kolkata, India) fitted with an anemometer (Lutron AN-4201).

Determination of Osmotic Properties

During the osmotic dehydration process, the standard formulae from Behera *et al*. (2017)[(6)] were used to estimate the water loss and solid gain. Rehydration ratio is the ratio between the weights of the sample after rehydration to the weight of dried sample. The weight of the dried sample was taken as W_1. It was boiled with water for 10 minutes, and then was allowed to cool down. The sample was drained out from water and the surface was wiped with tissue paper. The final weight of the rehydrated sample was taken W_2.[12] The rehydration ratio was calculated as

$$RR = W_2 / W_1 \quad \dots (2)$$

Quality Characteristics of Dried Slices

Moisture content was determined by using standard AOAC official hot air oven method. Antioxidant potential and total phenolic content were determined by following the procedure of Lakshmi *et al*., (2019)[13]. Ascorbic acid was determined using 2-6 dichloroindophenol titration method and the formula used is as followed by Behera *et al*. (2019).[12]

Statistical Analysis

All the statistical procedure was carried out to test the validity of response models generated through regression coefficient and root mean square error. The significance of coefficients and variables were examined at 1 and 5% level of significance.

Results and Discussion

Ultrasound Pre-treated Osmo-Convective Drying of Fruit Slices

In Table 1, the values of different responses for all 15 experimental combinations are given. There is a wide range of variation in all responses, i.e. 36.2 to

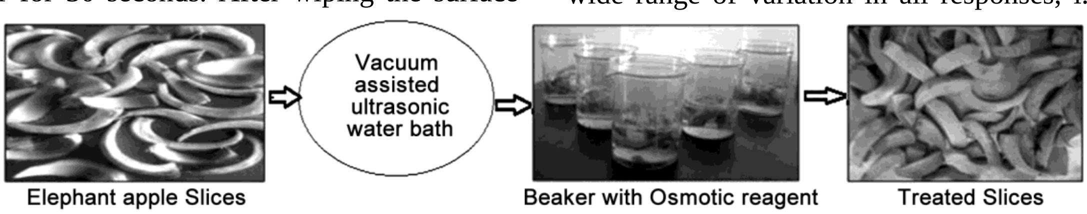

Fig. 1 — Schematic diagram for ultrasound pre-treated osmotic dehydration of elephant apple slices

Table 1— Experimental results for the Box-Behnken three factors osmo-convective drying

S N	X_1	X_2	X_3	SC, ºBrix	IT, h	DT, ºC	WL %	SG %	RR	AA mg/100g of DM	AOA μM of AAE/g of DM	TP mg of GAE/g of DM
1	−1	−1	0	40	2	60	36.2	5.49	1.98	55.750	1422	510
2	+1	−1	0	60	2	60	41.60	5.82	1.70	85.400	2663	483
3	−1	+1	0	40	6	60	42.70	7.34	1.92	67.230	3187	530
4	+1	+1	0	60	6	60	52.10	8.19	1.29	72.320	1869	450
5	−1	0	−1	40	4	50	39.00	7.36	1.82	83.350	2115	509
6	+1	0	−1	60	4	50	45.20	7.83	1.87	88.001	2286	517
7	−1	0	+1	40	4	70	38.60	7.10	3.14	54.210	2047	310
8	+1	0	+1	60	4	70	46.10	8.36	2.16	83.270	2293	223
9	0	−1	−1	50	2	50	39.10	6.13	1.70	82.400	1750	330
10	0	+1	−1	50	6	50	44.40	8.14	1.55	95.640	2289	430
11	0	−1	+1	50	2	70	39.20	5.06	2.40	82.740	1812	130
12	0	+1	+1	50	6	70	46.00	8.41	2.10	67.210	1516	114
13	0	0	0	50	4	60	41.50	7.61	1.94	97.620	1937	326
14	0	0	0	50	4	60	42.20	7.41	1.90	97.497	2273	321
15	0	0	0	50	4	60	41.80	7.23	1.89	98.230	2410	314

X_1: SC, X_2: IT, X_3: DT, SC: Sugar Concentration, IT: Immersion Time, DT: Dryer Temperature, WL: Water Loss , SG: Solid Gain, RR: Rehydration Ratio, AA: Ascorbic acid, AOA: Antioxidant Activity, TP: Total Phenolics, AAE: Ascorbic Acid Equivalent, GAE: Galic Acid Equivalent, DM: Dry Matter

Table 2 — Regression Coefficients of second order model for different responses

Predictors	Coefficients	WL	SG	RR	AA	AOA	TP
Intercept	α_0	41.83**	7.41**	1.91**	97.78**	2206**	326.3**
			Linear				
X_1	α_1	3.56**	0.36*	−0.23**	8.55**	42.5	−23.25**
X_2	α_2	3.63**	1.19**	−0.11**	−0.48	151.7	8.87
X_3	α_3	0.27	−0.06	0.35**	−7.74**	−96.50	−126.12**
			Quadratic				
X_1*X_1	α_4	0.68	0.01	0.06	−16.19**	211.0	155.83**
X_2*X_2	α_5	0.63	−0.71**	−0.24**	−11.40**	−132.45	17.08
X_3*X_3	α_6	−0.29	0.23	0.27**	−4.37**	−232.45	−86.41**
			Interaction				
X_1*X_2	α_7	1.00	0.13	−0.08*	−6.14**	−639.7**	−13.25
X_2*X_3	α_8	0.32	0.19	−0.25**	6.10**	18.75	−23.75*
X_3*X_1	α_9	0.37	0.33	−0.03	−7.19**	−208.75	−29.00*
R-sq		0.984	0.974	0.993	0.999	0.905	0.995

**Significant at 1%level, * Significant at 5% level

WL: Water Loss (%), SG: Solid Gain (%), RR: Rehydration Ratio, AA: Ascorbic acid (mg/100g of dry matter), AOA: Antioxidant Activity (μ M of AAE/g of dry matter), TP: Total Phenolics (mg of GAE/g of dry matter), AAE: Ascorbic Acid Equivalent, GAE: Galic Acid Equivalent

52.10% for WL; 5.06 to 8.41% for SG; 1.29 to 3.14 for RR, 55.75 to 98.23 mg/100g of dry matter for AA, 1422 to 3187 μM of AAE/g of dry matter and 114 to 517 mg/g of dry matter for TP. For a run order of 4, 1; 12, 11; 7, 4; 15, 12; 3, 1 and 3, 12 the highest and lowest values for WL, SG, RR, AA, AOA and TP, respectively, were achieved.

Regression Models and Effect of Variables on Various Responses

The significance of coefficients of second-order polynomial equations of all the output variables is displayed in Table 2. All models were checked for their adequacy using ANOVA methodology. There were non-significant values for lack of fit ($p > 0.05$) confirming the model validity (Table 2). A higher determination coefficient, R^2 (0.984, 0.974, 0.993, 0.998, 0.905 and 0.995 for WL, SG, RR, AA, AOA, & TP respectively) demonstrated the adequacy of the models for different responses.

The list of complete second order models developed are as follows (Eqs 3 to 8):

$$WL = 41.83 + 3.50*SC + 3.63*IT \quad \ldots (3)$$

$$SG= 7.41 + 0.36*SC +1.19*IT – 0.71*IT*IT \quad \ldots (4)$$

$$RR = 1.91−0.23*SC– 0.11*IT + 0.35*DT – 0.24*IT*IT + 0.27*DT*DT – 0.08* SC*IT– 0.25* IT*DT \quad \ldots (5)$$

$$AA = 97.78 + 8.55*SC – 7.74*DT– 16.19*SC*SC – 11.40*IT*IT – 4.37*DT*DT – 6.14*SC*IT + 6.10*IT*DT + 7.19*DT*SC \quad \ldots (6)$$

$$AOA = 22.06 -639.7*SC*IT \quad \ldots (7)$$

$$TP= 326.3 – 23.25*SC – 126.12*DT + 155.83*SC^2– 86.41*DT^2– 23.75*IT*DT -29.00*IT*SC \quad \ldots (8)$$

It was seen from the Eqs (3–8) that all the variables had a major influence on the therapeutic qualities of the slices of elephant apple fruit. However, the sugar concentration (SC) and immersion time (IT) had the most prominent effect on all responses, and the drying air temperature did not have any effect on osmotic properties.[10]

a) ***Effect of Variables on WL and SG*****:** WL was influenced by SC and IT greatly (P<0.01) and was linearly related with both of these within the experimental range. Similar trend was observed for solid gain (SG) with additional significant effect of immersion time at quadratic level (p<0.01). The higher water loss may be due to improvement of mass transfer by use of ultrasound during the treatments.[10]

b) ***Effect of Variables on RR*****:** IT and DT were highly significant (P<0.01) at all the levels. Of course the quadratic effect of SC and its interactive effect with DT were not significant. IT was negatively related to RR at all levels.[6]

c) ***Effect of Variables on AA*****:** AA was significantly affected by all the input variables at all levels except a linear IT. This could be due to the phenomena that AA is susceptible to high drying temperature and soluble in water up to critical point.[10]

d) ***Effect of Variables on AOA*****:** It was found that AOA was not significantly impacted by the process variables. The cross-product effect of SC and IT, however was strongly (P<0.01) associated (negative).

e) ***Effect of Variables on TP*****:** It was observed that SC and DT had a profound impact on TP, although IT had no effect. At linear and all forms of interaction, both SC and DT were negatively correlated to TP. SC's quadratic relationship, however was positive and that of DT was negative. Due to the presence of certain volatile compounds and soluble compounds which may be leached during the soaking process, the reduction of TP with SC and DT is probable.

Optimization of Process Variables

The desirability function for the parameters to optimize all but SG at a target value was established in the current analysis. The responses obtained for WL, SG, RR, AA, AOA, and TP in the case of individual response optimization were 52.03 percent, 8 percent, 3.09, 102.14 mg/100g of dry matter, 2834.19 μM of AAE/g of dry matter, 588.5 mg/g of dry matter, respectively. Since the product inquiry seeks to provide an acceptable procedure with optimum preservation of quality parts, it was necessary to work out and check the outcomes of the multiple response optimizations. Similar findings were reported by Barman *et al*. (2017).[10] Here, compromised response parameters were obtained to match the output characteristics to an acceptable range. The optimum conditions thus derived for SC, IT and DT were 1.00,−0.0101 and −0.4545 (coded values) and 60ºBrix, 3.98 h≈ 4 h, 55.5ºC ≈ 55ºC (uncoded values), respectively. With these optimum input variables, the value of WL, SG, RR, AA, AOA, and TP were estimated as 45.70 %, 7.76 %, 1.754, 90.015 mg/100g of dry matter, 2451.4 μM of AAE/g of dry matter, 503.09 mg/g of dry matter. The overall desirability was 0.984. The results obtained by actual experiment were very close to these values and had an acceptable deviation of within ± 0.5%. Another set of experiment was carried out under these set of optimum input parameters without ultrasound treatment. The output properties WL, SG, RR, AA, AOA, and TP were measured to be 32.70 %, 6.76 %, 1.354, 80.015 mg/100g of dry matter, 2415.6 μM of AAE/g of dry matter, 480.10 mg/g of dry matter respectively. On comparison, it was observed that the ultrasound treated sample had higher water loss for which it could be dried faster and could retain better quality components like AA and TP with a higher RR.

Response Surface Analysis

The surface profiles for different output variables were drawn as a function of two input variables with the third one at optimum value which are presented in Fig. 2 (a–f). At given DT, WL and SG increased with increase in SC and IT. The influence of IT is higher than that of SC in both cases. But SG increased rapidly with increase in IT but at a reduced rate towards the higher value (Fig. 2 a, b). At constant DT for any given IT, with increase in SC, RR increased up to center point and then again decreased (Fig. 2c). Change in AA took quadratic shape with all the parameters with a variation in magnitude. AA

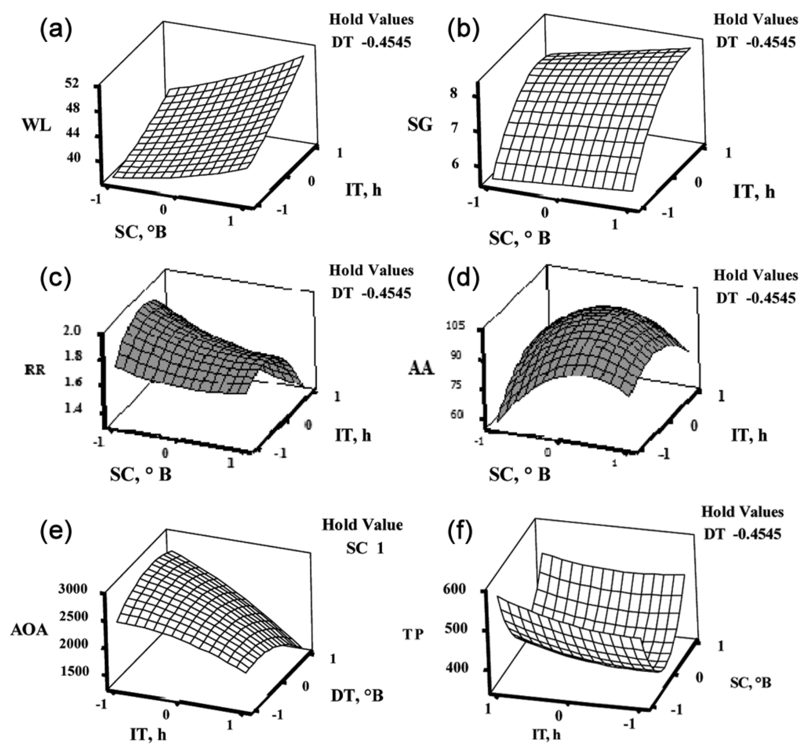

Fig. 2 — Surface plot of a) WL vs SC, IT; b) SG vs SC, IT; c) RR vs SC, IT; d) TP vs SC, IT; e) AOA vs IT, DT; f) TP vs SC, IT

increased up to center point and then reduced (Fig. 2d). No definite pattern could be observed with AOA. At given SC, AOA decreased with increase in IT. AOA decreased up to center point with increase in SC and thereafter started increasing. The change was in reverse trend with DT (Fig. 2e). At constant DT, the value of TP was minimum with SC at 50ºBrix. For a given value of IT, TP followed the similar trend with increase in SC (Fig. 2f).

Conclusions

A very effective alternative for preserving elephant apple slices is osmotic dehydration with ultrasound as a pre-treatment. A number of compromised process parameters for osmo-convective drying of slices were calculated as sugar concentration of 60°Brix, immersion time of 4h and drying air temperature of 55°C to fulfill all the response functions within the appropriate range. The values of WL, SG, RR, AA, AOA, and TP were 45.70 %, 7.76 %, 1.754, 90.015 mg/100g of dry matter, 2451.4 µM of AAE/g of dry matter and 503.09 mg/g of dry matter, respectively with an overall desirability of 0.985. Multiple regression analysis of observed values of osmo-convective drying using the Box-Behnken RSM design showed that the proposed second-order models for WL, SG, RR, AA, AOA and TP were acceptable and sufficient with coefficient of determination more than 0.9 in each case. The regression coefficients and the probability of variables affecting the response at all the levels have been extensively studied. Among all the variables, sugar concentration and immersion time had the most critical effect on all the responses. The findings of the present investigation are useful in selecting the most suitable processing technology to obtain high-quality processed elephant apple slices for direct consumption or for inclusion into a composite food with enhanced shelf life.

Conflict of Interest

The authors declare that there are no conflicts of interest.

Acknowledgement

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