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Osmotic Dehydration of Pomegranate (*Punica granatum* L.) Using Response Surface Methodology

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Abstract. For studying osmotic dehydration of pomegranate arils, a mathematical model was developed to quantify the responses of water loss, weight reduction and solute gain using response surface methodology. Under the experimental conditions, 15-32% water was lost, whereas 6-13% solids were gained. The high value (> 0.98) for determination coefficient (R^2) and adequate precision (> 38) and a low value for coefficient of variance (< 2.5) was achieved for the developed model. Optimisation of the model with the goal of maximum water loss and minimum solute gain resulted in 24.5% and 9.6% values, respectively, whereas, with the goal of minimum water loss and maximum solute gain resulted in 15.6% water loss and 13.8% solute gain.

Keywords: pomegranate, osmotic dehydration, mathematical modeling

Introduction

Pomegranate (*Punica granatum* L.) is a fruit of tropical and subtropical regions. It is widely cultivated in Iran, Spain, Egypt, Afghanistan and India (Adsul and Patil, 1995). The edible fruit is a berry with a rounded hexagonal shape, and has thick reddish skin and around 400-600 seeds (Al-Said *et al*., 2009). The pulp bearing seeds are called "arils". Dehydrated arils are known as "Anardana" in local language in India and Pakistan and are used in culinary and traditional medicines. The arils are either consumed as fresh or their juice is extracted. The juice may also be used in processed products like jams and jellies.

Drying conditions of pomegranate arils, significantly affect essential functional properties. Pomegranate is usually dried in open environment (sun drying) due to which the resulting product contains dust, insects and other contaminants. Moreover, open environment (sun drying) does not result in consistent product due to varying humidity and temperature conditions (Doymaz and Pala, 2002). Industrial dryers have been proposed (Doymaz, 2004) to avoid these problems. However, industrial dryers are not only expensive but result in low quality product as well due to the use of hot air for drying the product. An alternate drying method is osmotic dehydration.

Osmotic dehydration (OD) is widely used to remove water from fruits and vegetables by dipping them in aqueous solutions of low molecular weight compounds e.g., sucrose at high concentration. During OD, water is lost from the product, whereas solids are transferred from the dipping medium to the product simultaneously (Madamba, 2003). OD thus results in energy saving and improved product quality (Raoult-Wack, 1994).

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Rate of OD depends on several variables including temperature, immersion time and solute concentration. Successful application of osmotic dehydration requires mathematical modelling of process variables. Mathematical modelling helps in dealing with multiple factors to optimise the desired outcome by simulating the process variables and allowing the quantification under various conditions (Jalali *et al*., 2008).

Using response surface methodology (RSM), the aim of this work had been to study the effects of temperature, immersion time and concentration on the weight reduction (WR), water loss (WL) and solute gain (SG) during the OD of pomegranate arils. The OD parameters were simulated and optimised using mathematical model.

Meterials and Methods

Sample preparation. Pomegranate fruits of approximately same size, weight and maturity level were purchased from the local market. The arils were manually separated from the fruits and the peel was discarded. The arils were then subjected to osmotic treatment.

Osmotic dehydration. Osmotic solutions were prepared with commercial sucrose. For every experiment, 150 g sample (arils) was dipped in 1 L osmotic solution for different time intervals, temperatures and concentrations (Table 1). During this treatment the solution was continuously stirred, on shacking water bath. After the treatment, arils were superficially dried with an absorbent paper, manually. The samples were weighed after the process to calculate the percentage of weight reduction. (WR), water loss (WL) and solute gain (SG), according to the following formulas:

$$SG = \frac{W_{st} - W_{so}}{W_o} \times 100 \quad (1)$$

$$WR = \frac{W_o - W_t}{W_o} \times 100 \quad (2)$$

$$WL = SG + WR \quad (3)$$

Where:

W_{so} = weight of solids present in pomegranate arils before treatment,

W_{st} = weight of solids present in pomegranate arils after treatment,

W_o = weight of pomegranate arils before treatment and

W_t = weight of pomegranate arils after treatment.

All the experiments were conducted in triplicate and average values were taken for calculation.

Table 1. Variables and experimental design levels of osmotic dehydration

Variable	Coded symbol	Levels				
Coded value		-2	-1	0	1	2
Time (min)	A	25.5	49.0	72.5	96	119.5
Temperature (°C)	B	28.5	35	41.5	48	54.5
Concentration (°Bx)	C	25	34	43	52	61

Experimental method and statistical analysis. A central composite experimental design was used to study the effects of time, temperature and concentration on the OD parameters. This experiment design allows the modelling of a second-order polynomial that describes the responses. Data were analysed by multiple regressions through the least square method to fit in the following equation.

$$Y = b_o + b_1A + b_2B + b_{12}AB + b_{11}A^2 + b_{22}B^2 + b_3C + b_{13}AC + b_{23}BC + b_{33}C^2 \quad (4)$$

The analysis of variance (ANOVA), response surfaces and other statistics were executed using design expert software (1996, V.5.0.3).

Results and Discussion

The results of water loss, solute gain and weight reduction for the 20 trials generated by the central composite design are shown in Table 2.

Relative extent of solute uptake is usually expressed in terms of dehydration efficiency index (DEI) which is the ratio of water loss/solid gain (WL/SG). The value of WL/SG in this study was found to be approx. 2 (2.15-2.57). Thus the water removed was almost double the solute gained under the process conditions studied. Value of DEI may be desired either high or low depending on the end use. In case of Anardana, a higher DEI is required as the end use is in culinary. On the other hand, low DEI value is preferred in case of "candying". Structure of the raw material had a significant effect on SG and WL (Lazarides *et al*., 1997; Lazarides and Mavroudis, 1996). Since there is no reported study on pomegranate, values are not available for comparison. Mujica-Paz *et al*. (2003), working on osmotic dehydration of melons, got the WL/SG values ranging from

Table 2. Effect of osmotic treatment on water loss (WL), solid gain (SG), weight reduction (WR) and WL/SG

Treatment No.	Time (min)	Temperature (°C)	Concentration (°Bx)	WL (%)	SG (%)	WR (%)	WL/SG
1	49.0	35.0	34.0	15.66	6.44	9.22	2.43
2	96.0	35.0	34.0	17.85	7.86	9.98	2.27
3	49.0	48.0	34.0	23.55	9.52	14.03	2.47
4	96.0	48.0	34.0	25.90	10.65	15.25	2.43
5	49.0	35.0	52.0	20.89	8.51	12.38	2.45
6	96.0	35.0	52.0	22.83	9.80	13.03	2.33
7	49.0	48.0	52.0	28.56	11.46	17.11	2.49
8	96.0	48.0	52.0	31.61	13.22	18.39	2.39
9	25.5	41.5	43.0	19.46	8.44	11.02	2.31
10	119.5	41.5	43.0	24.30	11.30	13.00	2.15
11	72.5	28.5	43.0	16.45	6.81	9.64	2.42
12	72.5	54.5	43.0	32.77	13.01	19.75	2.52
13	72.5	41.5	25.0	18.08	7.02	11.06	2.57
14	72.5	41.5	61.0	29.03	11.66	17.37	2.49
15	72.5	41.5	43.0	22.56	8.97	13.59	2.52
16	72.5	41.5	43.0	22.50	8.92	13.58	2.52
17	72.5	41.5	43.0	22.98	9.30	13.68	2.47
18	72.5	41.5	43.0	22.92	9.25	13.67	2.48
19	72.5	41.5	43.0	23.45	9.67	13.78	2.42
20	72.5	41.5	43.0	23.19	9.47	13.72	2.45

1.3 to 2.2 depending on the concentration of the immersion solution.

The results of WR, WL and SG were fitted on a quadratic model and analyzed statistically (Table 3A, 3B and 3C). High F-value for model (>132) and low F-value for lack of fit (<0.4) implies that the model is significant. This is strengthened by low Fisher F-test value ("Pmodel > F" < 0.0001). The determination coefficient (R^2) – a measure of how well the responses are likely to be predicted by the model – was found to be >0.98 which reveals the good fitness of the model (Table 4). Adjusted R^2 (Adj. R^2) is the value of R^2 adjusted for the number of explanatory terms and sample size in a model. The value of the Adj. R^2 was also found to be high (>0.97), showing that the high value of R^2 is not just due to added terms. The difference between the Predicted R^2 and Adj. R^2 was also found to be low (<0.01). Low values of coefficient of variance (<2.5) that was found in this study indicate that the deviation between the experimental and the predicted values is low. Adequate precision measures the signal to noise ratio. A ratio greater than 4 is desirable. In this work, the ratio was found to be >38, which indicates an adequate signal. Petchi and Manivasagan (2009), working on osmotic dehydration of radish, obtained similar statistical parameters.

Table 3A. ANOVA for response surface quadratic model: water loss

Source	Sum of squares	df	Mean square	F value	p-value prob > F
Model	410.408	9	45.601	507.304	< 0.0001
A-Time	23.062	1	23.062	256.566	< 0.0001
B-Temperature	264.235	1	264.235	2939.580	< 0.0001
C-Concentration	114.600	1	114.600	1274.910	< 0.0001
AB*	0.203	1	0.203	2.263	0.1634
AC*	0.026	1	0.026	0.286	0.6045
BC*	0.033	1	0.033	0.363	0.5603
A^2	1.606	1	1.606	17.865	0.0018
B^2	4.628	1	4.628	51.488	< 0.0001
C^2	0.687	1	0.687	7.645	0.0200
Residual	0.899	10	0.090	-	-
Lack of fit*	0.229	5	0.046	0.341	0.8685
Pure error	0.670	5	0.134	-	-
Cor total	411.307	19	-	-	-

* = not significant.

Table 3B. ANOVA for response surface quadratic model: solid gain

Source	Sum of squares	df	Mean square	F value	p-value prob > F
Model	66.930	9	7.437	132.311	< 0.0001
A-Time	8.019	1	8.019	142.664	< 0.0001
B-Temperature	37.940	1	37.940	675.015	< 0.0001
C-Concentration	19.761	1	19.761	351.588	< 0.0001
AB*	0.004	1	0.004	0.074	0.7913
AC*	0.030	1	0.030	0.541	0.4791
BC*	0.031	1	0.031	0.552	0.4747
A^2	0.636	1	0.636	11.321	0.0072
B^2	0.722	1	0.722	12.851	0.0050
C^2*	0.019	1	0.019	0.337	0.5744
Residual	0.562	10	0.056	-	-
Lack of fit*	0.146	5	0.029	0.350	0.8630
Pure error	0.416	5	0.083	-	-
Cor total	67.492	19	-	-	-

* = not significant.

Table 3C. ANOVA for response surface quadratic model: water reduction

Source	Sum of squares	df	Mean square	F value	p-value prob > F
Model	153.3327	9	17.0370	4309.2478	< 0.0001
A-Time	3.8834	1	3.8834	982.2432	< 0.0001
B-Temperature	101.9250	1	101.9250	25780.4213	< 0.0001
C-Concentration	39.1849	1	39.1849	9911.2313	< 0.0001
AB	0.1494	1	0.1494	37.7931	0.0001
AC*	0.0002	1	0.0002	0.0496	0.8283
BC*	0.0000	1	0.0000	0.0051	0.9444
A^2	4.2640	1	4.2640	1078.5051	< 0.0001
B^2	1.6937	1	1.6937	428.3952	< 0.0001
C^2	0.4780	1	0.4780	120.8938	< 0.0001
Residual	0.0395	10	0.0040	-	-
Lack of fit*	0.0095	5	0.0019	0.3141	0.8852
Pure error	0.0301	5	0.0060	-	-
Cor total	153.3722	19	-	-	-

* = not significant.

Table 4. Statistical parameters for quadratic model

Parameters	WL	SG	WR
SD	0.2998	0.2371	0.0629
Mean	23.2271	9.5641	13.6629
C.V. %	1.2908	2.4788	0.4602
Press	2.6741	1.6848	0.1150
R-squared	0.9873	0.9917	0.9997
Adj R-squared	0.9758	0.9842	0.9995
Pred R-squared	0.9622	0.9750	0.9993
Adeq precision	38.4352	40.0759	237.2569

The diagnostic curves (normal probability, predicted *vs* actual and residual *vs* run) help to find out any abnormality in the data points. Normal probability curve should not show any pattern; it should be a straight line with few scattered points (Fig. 1A). Predicted *vs* actual and residual *vs* run (Fig. 1B and 1C) help to detect any value or group of values giving large deviation in the model. In our case, these curves show that all the data points are adequately explaining the model.

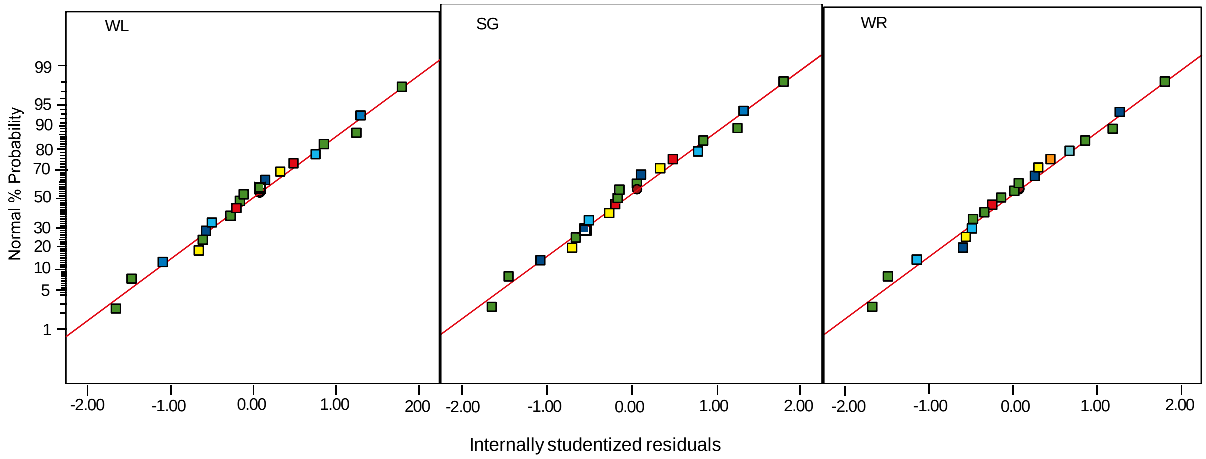

Fig. 1A. Diagnostic curves, normal plot of residuals (WL = water loss, SG = solid gain, WR = weight reduction).

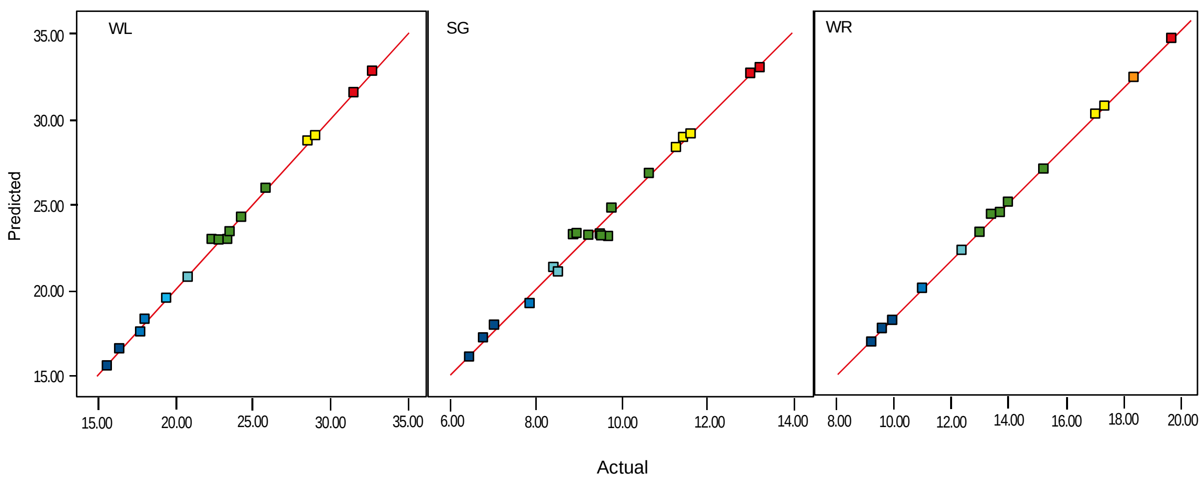

Fig. 1B. Diagnostic curves, predicted *vs* actual curves.

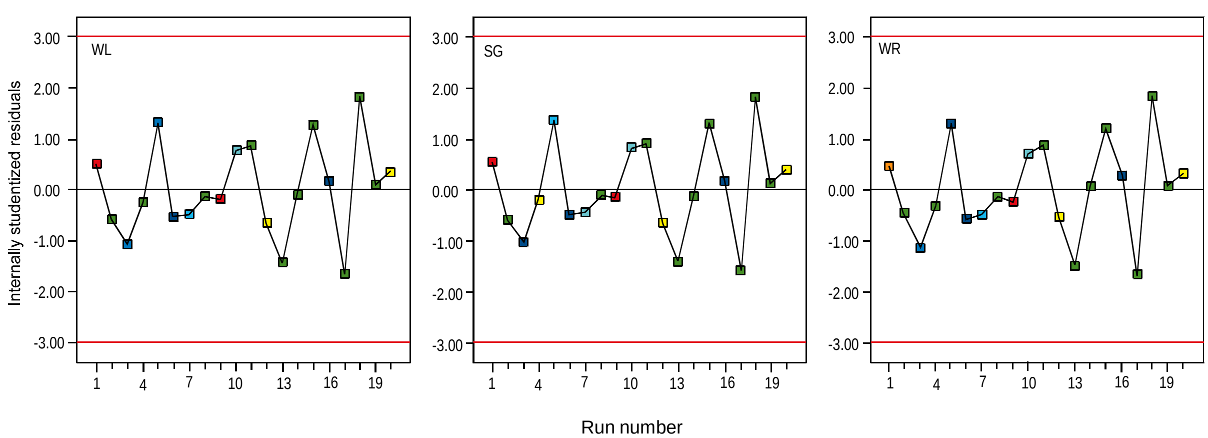

Fig. 1C. Diagnostic curves, residual *vs* run curves.

However, the generated model was not significant with respect to all the factors studied (Table 5). In all cases, interaction effects were not found to be significant except for WR for which time, temperature interaction was significant. This fact can also be observed in Fig. 2(A-F). These surfaces are rectangular planes without any elliptical curvature.

For WL, linear time factor was found to be positive whereas quadratic time factor was negative. This can be observed in Fig. 2A. This response surface curved with a plateau by increase in time. Alam and Singh (2010) working on osmotic dehydration of aonla fruit also found positive sign for linear time term and negative sign for quadratic time term. This indicates that initially water is lost quickly, while this loss gets lower and lower with time. In comparison, for SG, linear time term was found to be negative whereas quadratic term was positive. This type of behaviour is the result of continuous uptake of solute (Raoult-Wack, 1994). The linear temperature term was found to be negative but quadratic term was positive for both WL and SG. This implies that at low temperature, the mass transfer phenomenon is low whereas it increases at higher temperature not only due to the higher kinetic energy of molecules but also due to the change in the structure of the fruit membrane at higher temperature (Torreggiani, 1993). Both the linear and quadratic concentration terms were found to be positive for WL and SG; however, in case of SG the quadratic term was

Table 5. Coefficients of model equations

Factor	WL	SG	WR
Intercept	5.29859	4.26041	1.03818
Time	0.06260	-0.03037	0.09297
Temperature	-0.34025	-0.15265	-0.18761
Concentration	0.05711	0.02904	0.02806
Time* Temperature	0.00104*	0.00015*	0.00089
Time* Concentration	0.00027*	0.00029*	-0.00002*
Temperature* Concentration	0.00109*	0.00106*	0.00003*
Time^2	-0.00046	0.00029	-0.00075
Temperature^2	0.01015	0.00401	0.00614
Concentration^2	0.00204	0.00034*	0.00170

* = not significant.

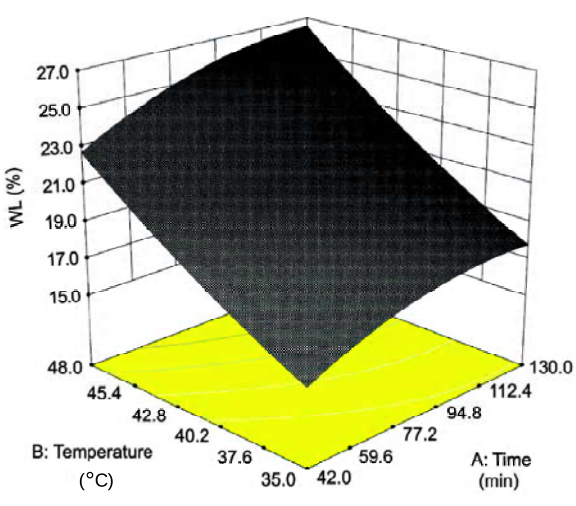

Fig. 2A. Response surface for water loss (WL) at 34° Brx.

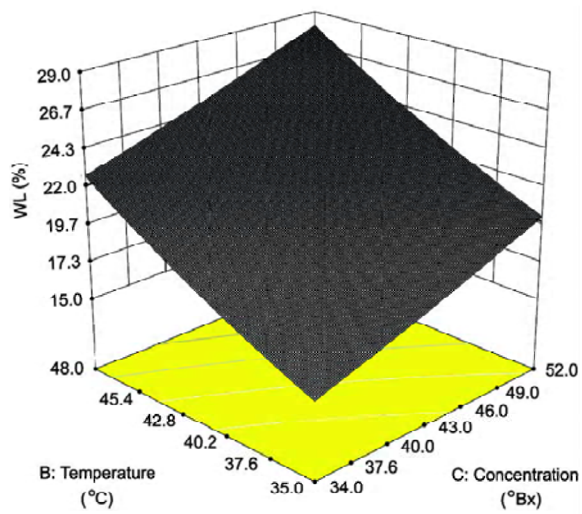

Fig. 2B. Response surface for WL at 42 min.

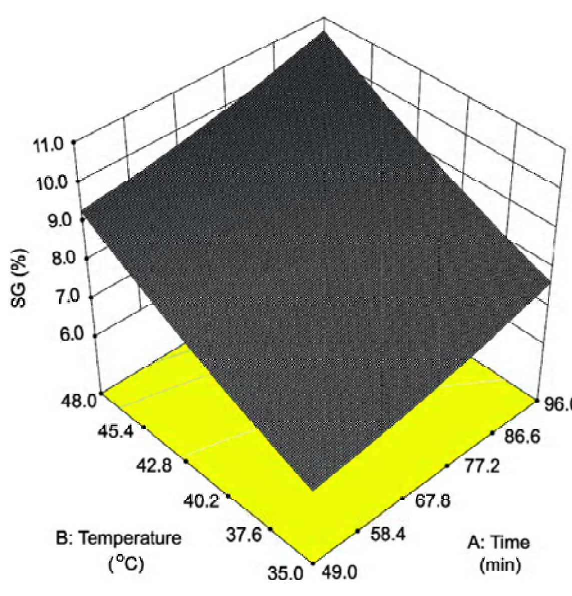

Fig. 2C. Response surface for solid gain (SG) at 34° Brix.

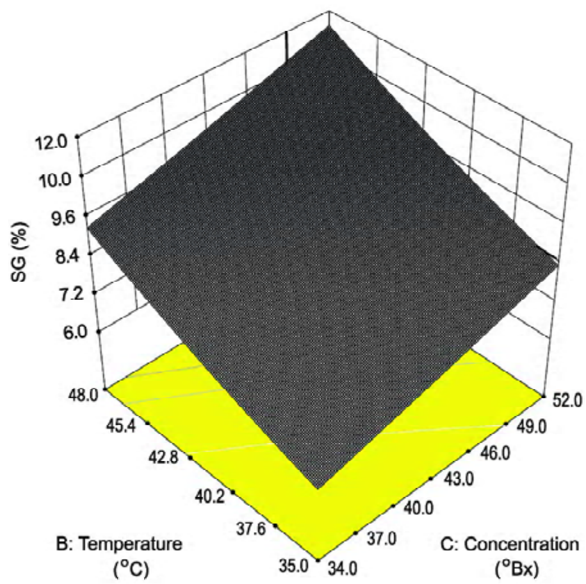

Fig. 2D. Response surface for SG at 42 min.

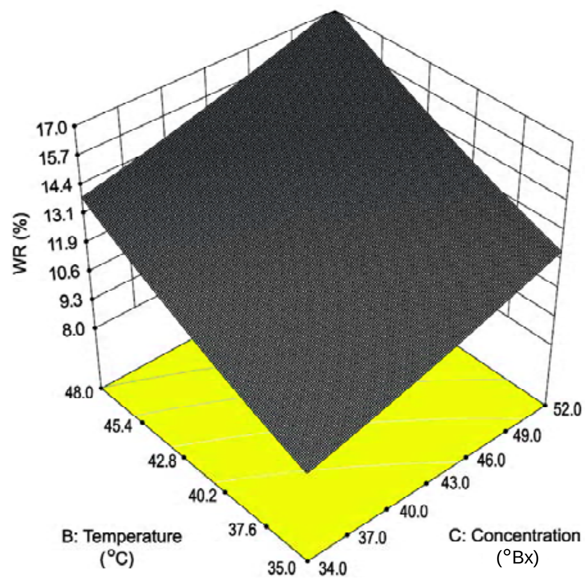

Fig. 2F. Response surface for WR at 42 min.

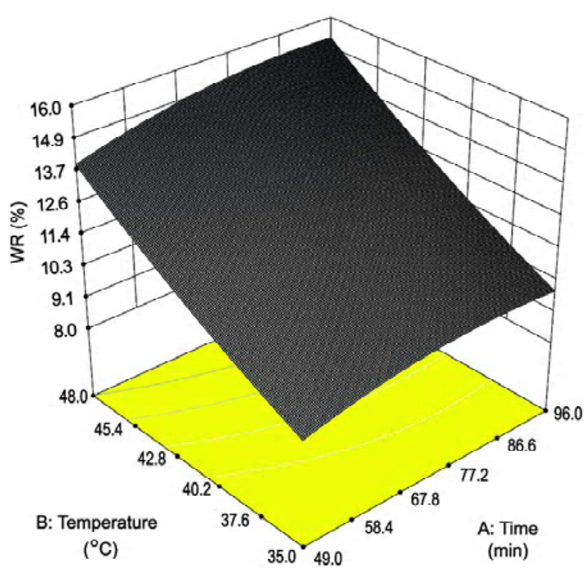

Fig. 2E. Response surface for weight reduction (WR) at 34° Brix.

not significant. Higher concentration results in greater difference of osmotic pressure, therefore, greater removal of water but increase in concentration results in steady increase in solid gain. This might be due to increase in external resistance. Borsato *et al*. (2009) working on osmotic dehydration of pineapple pieces showed that external resistant is also important along with the major resistance caused by the semi-permeable membrane of plant material.

Table 6 shows the optimum conditions under two different scenarios. This optimisation was performed on Design Expert software which uses the desirability function as described by Myers *et al*. (2009).

Table 6. Optimisation of model under different conditions

Factor	Goal	Solution	Goal	Solution
A: Time	is in range	65.96	is in range	188.41
B: Temperature	is in range	48.00	is in range	35.05
C: Concentration	is in range	34.00	is in range	34.04
WL	maximize	24.48	minimize	15.62
SG	minimize	9.66	maximize	13.84

Two different goals were chosen on the basis of two different possible end uses. The first goal was to maximize the WL, keeping the SG lowest. It is to be used when osmotic dehydration will be used as pre-treatment of conventional drying for production of anardana. Whereas the second goal was chosen for possible application of candied pomegranate arils. For this purpose, minimum WL was used as it results in shrinkage, keeping SG the maximum. From Table 6 it is obvious that this goal can be achieved when the temperature and concentration are low and time is high. This result is in agreement with the traditional candied fruit (*Murabba*) making practices.

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