

**Original Research Paper**

## **Optimization of factors influencing osmotic dehydration of aonla (*Phyllanthus emblica* L.) segments in salt solution using response surface methodology**

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### **ABSTRACT**

**Optimization of process parameters is a critical requirement in food processing and food product industries for the development of highly acceptable product. Quantification of mass transfer kinetics under different processing conditions is essential step for optimizing the osmotic dehydration process. A Box-Behnken Design (BBD), adopted from response surface methodology (RSM) approach was used for evaluating and quantifying the moisture loss and solids gain kinetics of aonla segments in salt solution during the osmotic dehydration process. The independent variables were fixed at three levels (salt concentration- 2, 4, 6%; process temperature - 45, 50, 55 °C and process time - 60, 120, 180 minutes). The process responses were water loss percentage (WL%) and solids gain percentage (SG%). Validation experiments were conducted at optimum conditions to verify predictions and adequacy of the models. The optimum conditions predicted were 5.02% salt concentration, 54.8 °C temperature and 60.64 minutes process time to attain a desired effect of maximum water loss (6.42%) and minimum solid gain (1.09%) in osmotic dehydration of aonla in salt medium.**

**Keywords:** Aonla, Optimization, Osmotic dehydration, Response Surface Methodology

### **INTRODUCTION**

*Phyllanthus emblica* L. (aonla), which is indigenous to tropical India and Southeast Asia is one of the most indispensable crops in various traditional and folk systems of medicine. Being rich in ascorbic acid and bioactive constituents (ellagic acid, chebulinic acid, gallic acid, chebulagic acid, apigenin, quercetin, corilagin, leutolin, etc.), it has been recognized as an excellent source of anti-oxidants to consider as significant dietary source and explored for therapeutic potential against various diseases (Nashine *et al.*, 2019; Alsahli *et al.*, 2021). The area and production of aonla in India was estimated to be 92 (000 hectares) and 1039 (000 MT) respectively (NHB, 2019). To increase the shelf-life of aonla fruits many methods or combination of methods had been tried that undergo phases of high temperature and time combinations. These significant high temperature processing would impair fresh quality of the food and hence result in

the products without original flavor, color and textural attributes after rehydration (Alam *et al.*, 2010).

Being highly perishable in nature and highly astringent in taste, it demands processing and value addition for enhanced shelf life to extend its availability throughout the year (Liu *et al.*, 2012). Dehydration is found to be a preferred technique to preserve the aonla fruits in the context of producing the high-quality shelf-stable fruit products to enhance the product availability and extend the marketability (Li *et al.*, 2019). Since osmotic dehydration is considered as a pre-treatment to aonla dehydration that can improve nutritional, sensorial, and functional characteristics of fruit without altering its integrity, it is exploited effectively (Gantait *et al.*, 2021). The process optimization and product quality improvement should be focused well to achieve target specifications. In spite of the numerous researches that have been undertaken on this subject, it is still hard to establish



general rules about the variables that influence the osmotic dehydration. Rate of water loss and solid gain which are the intrinsic aspects of the mass transfer kinetics depends on both operating conditions and kind of cellular microstructure, as well as on the product form in which it was pretreated (Pandiselvam *et al.*, 2021). Mass transfer rate upsurges with product surface area and rise in temperatures. In contrast, the ratio of water loss to solid gain depends on the concentration of solute and its molecular weight (Pravitha *et al.*, 2022). Furthermore, the use of solutes of high molecular weight enables water loss at the expense of solid gain. The driving force for moisture diffusion is the high osmotic pressure employed by the osmotic medium. The moisture diffusion is accompanied by a concurrent counter-flow diffusion of solutes from the osmotic medium to the fruit matrix. Since the membrane for the osmotic process is not perfectly selective, native solids present in the cells can also be leached into the osmotic solution.

The optimization of the osmotic dehydration process is usually executed to guarantee the rapid processing conditions for development of a product with acceptable quality and achieve a high throughput capacity (Sharma *et al.*, 2020). Several studies have been carried out to evaluate the influence of process variables (concentration of the osmotic solution, process temperature, processing time, agitation, food geometry, medium to sample ratio, *etc.*) on the mass transfer kinetics of conventional osmotic dehydration processes (Tiroutchelvame *et al.*, 2015). It is important to note that these variables can only be altered over a restricted range, outside of which they adversely affect the quality even though the mass transfer rates may be enhanced (Souraki *et al.*, 2012; Herman-Lara *et al.*, 2013). Hence, there is a requirement to determine the optimum operating conditions that increases the mass transfer rates without affecting the quality (Garcia-Segovia *et al.*, 2010).

With this framework, response surface methodology (RSM) is an important tool in process and product improvement which enables the determination of the relationship between the response and the independent variables. It is characteristically utilized for mapping a response surface over a particular region of interest, optimizing the response, or for selecting operating conditions to achieve target specifications (Box and

Draper, 1965; Myers *et al.*, 1989; Myers *et al.*, 2016; Beegum *et al.*, 2018; Pandiselvam *et al.*, 2019; Pandiselvam *et al.*, 2022).

The objective of the present study was to quantify the effect of salt concentration, process temperature, and contact time on the moisture loss and solid gain during osmotic dehydration of aonla segments using a “Box Behnken Design”.

## MATERIALS AND METHODS

### Procurement of fruits

Freshly harvested aonla fruits of commercial variety “Krishna” of appropriate maturity (large-sized, sound fruits with adequate flavor and gloss, uniformity in color) procured from ICAR-Indian Institute of Horticultural Research, Bengaluru farms. Fresh mature fruits with uniform size, free from injuries, bruises, insect damages and diseases were used in this study.

### Optimization process

Based on the literature reviews independent variables for this study were selected as concentration of osmotic solution, process temperature and treatment duration which were found to influence the quality of the final product appreciably (Eren and Kaymak-Ertekin, 2007; Alam *et al.*, 2010). The independent osmotic process variables and their levels in the form of coded variables for three-factor three level response surface analyses are given in Table 1. A three-factor three level Box-Behnken design model with three replicates at the central point, which gives 15 experiments was selected to study the influence of processing parameters on mass transfer attributes (Table 2). The levels of processing parameters were chosen as independent variables: Osmotic solution concentration, Treatment temperature, and Process duration; whereby each of these variables was tested at three different coded levels: low (“-1”), medium (0), and high (+1). Outcomes of preliminary trials were aided in setting the range of these independent variables. The responses considered attaining optimum condition were water loss (WL) and solid gain (SG) as these are the combination of significant processes or fluxes co-occurring, determining the attainment of equilibrium in osmotic dehydration process as a

unit operation and also important for both quantitative modeling and knowledge of the kinetics of mass transfer in the system.

### Osmotic dehydration experimental setup

The desired concentration of osmotic solution of salt (2-6%) was prepared and 150g of aonla segments were immersed in beaker containing osmotic solutions (600 ml) of varied temperatures (45-55 °C), fruit to solution ratio (1:4 w/v) in order to ensure that the concentration of osmotic solution did not change significantly during the experiment and contact time of 60-180 min. From the preliminary experimental results obtained from experiments conducted in the laboratory, the process variables and their ranges were selected. The osmotic dehydration process was carried out in a temperature-controlled chamber (Digital Multi Chamber Water Bath (Model-WMB 306, Daihan Scientific Co., Ltd). The experiments were carried out in randomized replicated order to minimize the variability in the observed responses due to extraneous factors. All the experiments were performed in triplicate and the mean value was used for the determination of mass transfer parameters.

### Osmotic dehydration process

For each experiment, known weight of aonla segments were put in the glass beakers having calculated volume (as per STFR-Solution to fruit ratio of 1:4) of osmotic solutions of different concentrations pre-set at the desired temperature by water bath at atmospheric pressure. This dehydration process was carried out in a water bath with temperature ranges from 45-55 °C to make the process effective. At the specified times the aonla segments were removed from the osmotic solutions and rinsed with water to remove surplus solvent adhering to the surfaces. These osmotically dehydrated aonla segments were then spread on the absorbent paper to remove free water present on the surface. A proportion of pre-treated aonla segments (5–8 g) were used for determination of dry matter by oven method (AOAC, 2000). The remaining part of each sample was dried to final moisture content of 10 % (wet basis) using hot air drier (Tray dryer, TD-2A, CM Envirosystems Pvt. Ltd) pre-set at 60°C air temperature.

### Determination of mass transfer properties

In osmotic dehydration, both water loss and solid gain take place concurrently. The reduction in weight is

attributed to the loss of water from the sample and increase in the weight of the sample due to solute gain from the osmotic solution. The evaluation of mass transfer between the solution and samples during osmotic dehydration process were estimated by using the parameters such as water loss % (WL) and solid gain % (SG) and the parameters were calculated by using the following equations (Sridevi and Genitha, 2012)

$$WL(\%) = \frac{(W_o - W_t)}{M_o} \times 100$$

$$SG(\%) = \frac{S_t - S_o}{M_o} \times 100$$

where,  $M_o$  = initial mass of the sample (g);  $W_o$  = initial water mass of the sample (g);  $W_t$  = water mass of the sample (g) after dehydration;  $S_o$  = initial dry mass of the sample (g) and  $S_t$  = dry mass of the sample (g) after dehydration

### Microstructure analysis

The fresh, and osmotically treated aonla segments were examined using scanning electron microscopy (SEM) in order to determine the effect of osmotic dehydration process on the microstructural changes of the tissue. Samples were cut into cubes with a sharp blade and mounted on aluminium SEM stubs and fixed it. The microstructure of the tissue was examined by a scanning electron microscope (Hitachi, TM3030 plus) and the images were recorded at the magnification of 250X (Mayor *et al.*, 2008).

### Experimental design

An experimental plan and further statistical analysis of data with regression model fitting for each response were carried out by using response surface analysis with Box-Behnken Design (BBD), SAS Statistical Software package version 9.3 (SAS Institute, Cary NC). Response values were analysed by fitting the data in a second-order polynomial model. The generalized second-order polynomial model proposed for predicting response variables is given as:  $Y = B_0 + B_1X_1 + B_2X_2 + B_3X_3 + B_{12}X_1X_2 + B_{13}X_1X_3 + B_{23}X_2X_3 + B_1^2 X_1^2 + B_2^2 X_2^2 + B_3^2 X_3^2$

In this equation, Y represents the dependent variable (the estimated response) and  $X_i$  ( $i = 1-3$ , 1-C (concentration), 2-T (Temperature), 3-t (time)) represent the independent variables. Coefficients of the

polynomial were represented by  $B_0$  (constant term),  $B_1$ ,  $B_2$  and  $B_3$  (linear coefficients for C, T and t respectively),  $B_1^2$ ,  $B_2^2$  and  $B_3^2$  (quadratic coefficients), and  $B_{12}$ ,  $B_{13}$  and  $B_{23}$  (interactive coefficients). Model adequacy by ANOVA. The fitness of the models was further affirmed based on statistical parameters such as coefficient of determination ( $R^2$ ), F test value and lack of fit. Interaction effects of independent variables were pictorially represented by 3-D response surface graphs for better depiction of results. The stepwise procedure adopted for response surface methodology analysis is given in Fig.1.

### Model optimization and validation

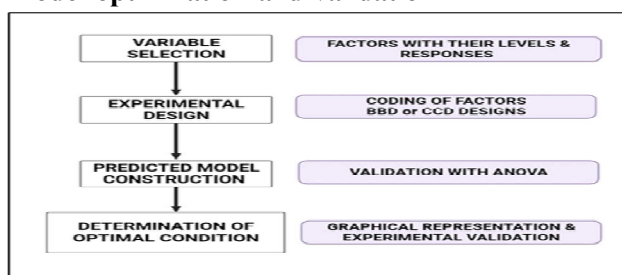


Fig. 1 : Step-wise procedure adopted for response surface methodology analysis

Optimization of independent variables was performed by multi-criteria methodology with the analysis of Derringer function or desirability function (Bezerra *et al.*, 2008). Further validation of the optimization was performed by comparing predicted and experimental value. The deviation between these two can be assessed by calculating absolute error by using equation as below:

$$\text{Absolute error (\%)} = \frac{\text{Experimental value} - \text{Predicted value}}{\text{Predicted value}} \times 100$$

## RESULTS AND DISCUSSION

The observed values of response variables are given in Table 2. The generalized second-order polynomial model proposed for predicting response variables were generated. The data was analyzed employing multiple

regression technique to develop a response surface model. A second order model with and without interaction terms were tested for their adequacies to describe the response surface and  $R^2$  values were calculated. The models were compared based on their coefficient of determination ( $R^2$ ), adjusted coefficient of determination ( $R^2$  -adj), predicted coefficient of determination ( $R^2$  -pred), and the probability (p) of lack of fit. All two models (WL and SG) were tested for their adequacy using ANOVA technique. F-values for the lack of fit were nonsignificant ( $p < 0.01$ ) thereby confirming the validity of the models. The significant terms of the response variable were determined by ANOVA and given in Table 3.

### Effect of process variables on responses

The mass transfer parameters i.e., water loss (WL) and solid gain (SG) reflecting as one of the quality attributes of aonla during osmosis. The rate of water loss during osmotic dehydration depends upon the solution temperature, solution concentration, and immersion time (Alam and Amarjit ,2010; Salimi *et al.*, 2020; Pinto *et al.*, 2021; Zeghib *et al.*, 2022).

### Effect of osmotic dehydration process parameters on the water loss (WL %)

WL is a major parameter of mass transfer that shows the efficiency of the osmotic dehydration process. A wide variation in all two responses was observed for different experimental combinations from 1.76 to 6.08 % in WL. The maximum value (6.25%) of water loss was observed for experimental combination of salt concentration of 6%, treatment temperature of 55! and treatment duration of 120 minutes (Table 2). Among the process variables studied, the concentration witnessed maximum effect on water loss. It can be seen that water loss was found to be significantly affected by concentration individually, ( $p < 0.01$ ) and in the quadratic and interaction terms on water loss, whereas other factors such as temperature, time and

**Table 1 : Coded and uncoded values of process variables and their levels during osmotic dehydration of aonla segments in salt solution.**

Independent Variables	Coded Levels	-1	0	+1
Solution concentration, %	$X_1$	2	4	6
Temperature, °C	$X_2$	45	50	55
Time, minutes	$X_3$	60	120	180

$X_1$ ,  $X_2$ ,  $X_3$ : Coded independent variables

**Table 2 : Outline of the experimental design matrix and observed values of response variables**

Treatments runs	Independent variables			Responses	
	Concentration (%)	Temperature (!)	Time (minutes)	WL %	SG %
1	2	45	120	1.85	0.22
2	4	55	180	2.64	0.76
3	2	50	60	1.75	0.25
4	4	45	180	2.08	0.66
5	4	50	120	2.21	0.85
6	6	50	60	6.03	1.00
7	6	55	120	6.25	1.07
8	2	50	180	1.80	0.33
9	4	55	60	6.05	1.22
10	6	50	180	6.08	1.24
11	4	50	120	2.21	0.85
12	4	45	60	2.06	0.74
13	4	50	120	2.21	0.85
14	2	55	120	1.76	0.35
15	6	45	120	5.11	1.56

X<sub>1</sub>, X<sub>2</sub>, X<sub>3</sub>: Coded independent variables; WL: Waterloos; SG: Solid gain

**Table 3 : ANOVA of the second order polynomial models of the various responses.**

ANOVA	WL %	SG %
Mean of response	3.33	0.79
RMSE	0.79	0.21
R <sup>2</sup>	0.94	0.90
Adj- R <sup>2</sup>	0.82	0.71
PRESS	50.17	3.56
Model F value	8.45	4.81
P value	0.01 <sup>S</sup>	0.04 <sup>S</sup>
Lack of fit	0.93 <sup>NS</sup>	0.96 <sup>NS</sup>

S - Significant at 1-5%; NS - Non-significant

its interactions found non-significant effect on water loss (Table 4).

Second order response surface model resulted in significant ( $P < 0.06$ ) estimates for the factor concentration and its interaction as tested by their corresponding student t-test value being significant ( $P < 0.05$ ) (Table 4). The RSM solution is minimum

for the response WL at 1.16 corresponding to the critical values of 2.38, 46.15 and 107.26 for concentration, temperature and time, respectively. All three linear terms, interaction and quadratic terms of solution concentration (C) showed a significant ( $p < 0.05$ ) influence in the model prediction of WL ( $R^2 = 0.94$ ). WL was significantly influenced ( $P < 0.05$ ) by the variation in salt concentration only with insignificant linear, quadratic and interaction terms of temperature and time. Presence of interaction of process variable WL with concentration, temperature and time variables as seen in the plots justified for the choice of RSM with interaction effect as constructed (Fig. 3). The sign and magnitude of the coefficients indicate the effect of the variable on the response. Negative sign of the coefficient means decrease in response when the level of the variable is increased while positive sign indicated increase in the response. Significant interaction suggests that the level of one of the interactive variables can be increased that of other decreased for constant value of the response (Draper and Hunter, 1966; Montgomery, 2004). From Table 4 and Fig. 3, levels of salt concentration were in positive correlation with WL in both linear and

interaction terms. The increasing trend of WL with solute concentration could be due to the enhanced osmotic pressure gradients. Comparable results showing the enhancement of the solution concentration resulting in an increase of the osmotic pressure gradients and, hence, higher WL in solutions (Cichowska *et al.*, 2018).

**Effect of osmotic dehydration process parameters on solid gain (SG %)**

A wide variation in all two responses was observed for different experimental combinations from 0.22 to 1.56 % in SG. The maximum value (1.56%) of solid gain was observed for experimental combination of salt concentration of 6%, treatment temperature of 45°C and treatment duration of 120 minutes. It can be seen from Table 4 that solute gain was found to be significantly affected by concentration individually ( $p < 0.01$ ) whereas in the quadratic and interaction terms showed non-significant effect on solid gain. Studies elucidated similar findings that in osmotic dehydration, the concentration gradient between the intracellular fluid and osmotic solution create a difference of osmotic pressure, which leads to diffusion of water and solid molecules through the semi-permeable membrane of this fruit to achieve osmotic equilibrium, thus increase in solute concentration led to increases in SG (Wiktor *et al.*, 2022).

**Summary of fit and actual vs predicted plot analysis**

RSM is able to capture 93.8% and 89.6% of model response using the experimental runs considered for three response factors each at three level as discussed earlier, which is also supported graphically for WL and SG respectively. In the Actual-by-Predicted Plot (Fig. 2), the mean line falls outside the bounds of the 95% confidence curves (red-dotted lines), which tells you the model is significant. Results of ANOVA table revealed that the model can adequately express the response variable (Prob(F) being  $< 0.05$ ) which is also evident with the results of lack of fit (Prob(F) being  $> 0.05$ ). PRESS (Prediction Residual Error Sum of Squares) statistic value along with low PRESS root mean square error (RMSE) of 1.82 too indicated that least prediction error at this solution and supported that the model fits well for the runs considered (Table 3). As the F-values for the lack of fit were non-significant ( $P < 0.05$ ), the model adequacy is well attained.

The prediction formula as elucidated below, were used to construct surface plots individually for WL and SG by considering two independent variables at a time. cursory look into the various surface plots too revealed that the maximum water loss to the extent of 6.42 % and maximum solute gain of 1.08 %, which corresponds to optimum level of 5.02 %, 54.8°C and 60.64 minutes for concentration, temperature and time, respectively.

**Prediction formula for Water loss (WL %):**

$$Y_2 = 2.21 + 2.03875 * ((C - 4) / 2) + 0.7 * ((T - 50) / 5) + -0.41125 * ((TIME - 120) / 60) + (C - 4) / 2 * (T - 50) / 5 * 0.3075 + (C - 4) / 2 * (TIME - 120) / 60 * -1.11022302462516e-16 + (T - 50) / 5 * (TIME - 120) / 60 * -0.8575 + (C - 4) / 2 * (C - 4) / 2 * 1.12 + (T - 50) / 5 * (T - 50) / 5 * 0.4125 + (TIME - 120) / 60 * (TIME - 120) / 60 * 0.585$$

**Prediction formula for Solid gain (SG %):**

$$Y_1 = 0.85 + 0.465 * ((C - 4) / 2) + 0.0275 * ((T - 50) / 5) + -0.0275 * ((TIME - 120) / 60) + (C - 4) / 2 * (T - 50) / 5 * -0.155 + (C - 4) / 2 * (TIME - 120) / 60 * 0.04 + (T - 50) / 5 * (TIME - 120) / 60 * -0.095 + (C - 4) / 2 * (C - 4) / 2 * -0.095 + (T - 50) / 5 * (T - 50) / 5 * 0.045 + (TIME - 120) / 60 * (TIME - 120) / 60 * -0.05$$

Where, **Y1**: Solid gain (%); **Y2**: Water loss (%); **C**: Concentration of the salt solution (%); **T**: Temperature of the process (°C)

**Optimization of osmo-dehydration process**

Only a significant and precise model can supply reliable and essential information for optimizing the results (Ade Omowaye *et al.*, 2002; Romero *et al.*, 2022). The predictive models are used to generate response surfaces within the experimental range. The response surface plot is the theoretical three-dimensional plot (3D surface) showing the relationship between the response and the independent variables (Bezerra *et al.*, 2008; Yuan *et al.*, 2018). It is a two-dimensional screen of the surface plot, in which, ranges of constant dependent variables are drawn in the plane of the independent variables (Alam and Singh, 2010; Shameena *et al.*, 2019). Fig.4 shows the response surface plot of aonla WL rate under the effects of input parameters of osmotic temperature, time, and solute concentration, considering the interactive effect of variables. Some profiles for the

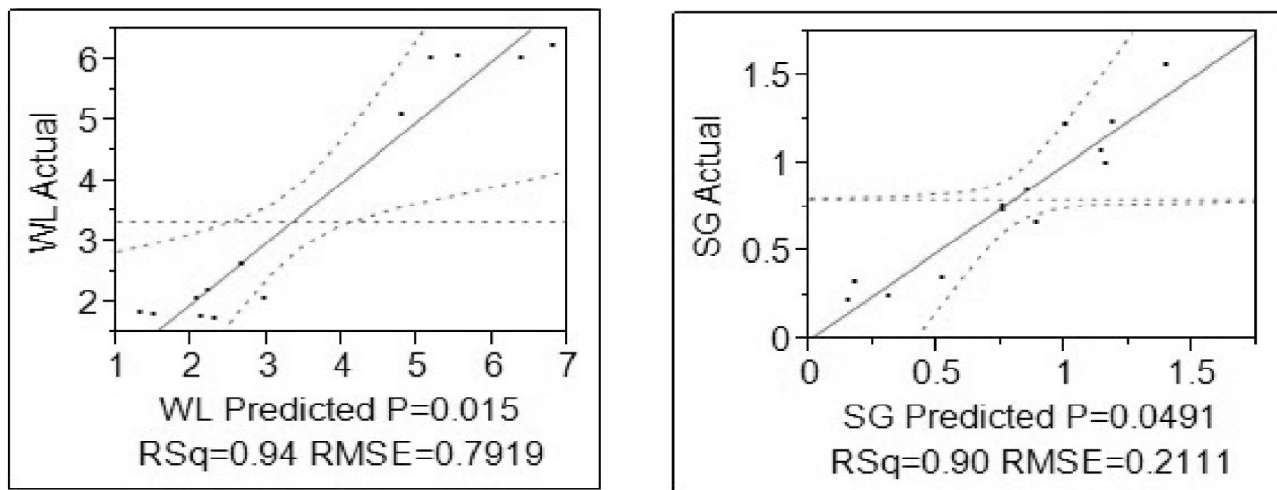


Fig. 2 : Actual vs Predicted plot for WL % and SG %

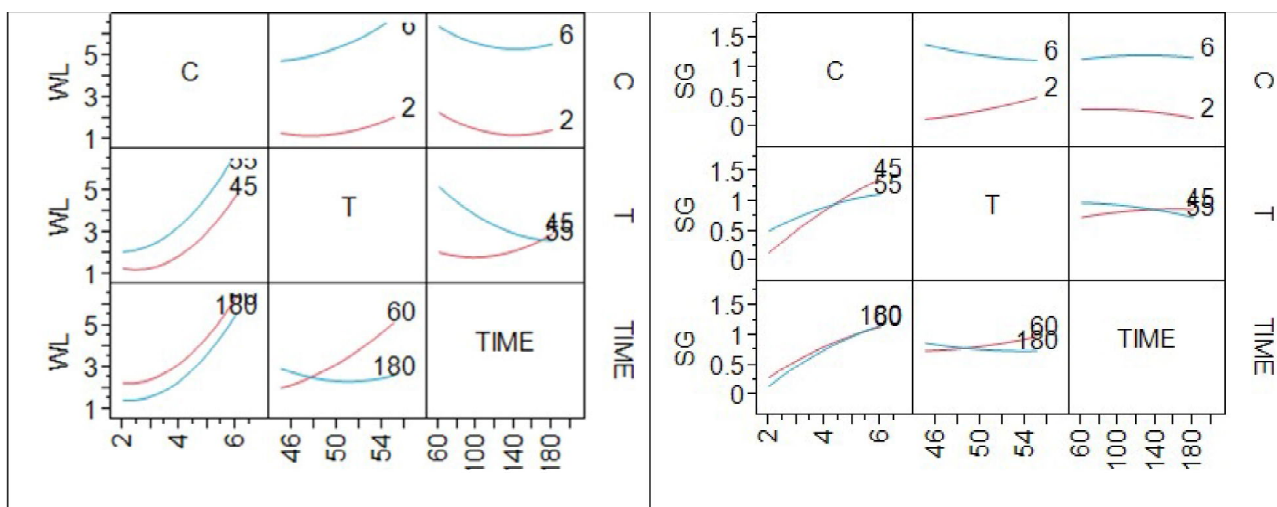


Fig. 3 : Interaction profiles of WL and SG with process variables

**Table 4 : Regression coefficients of the quadratic polynomial for salt during optimization**

Coefficients	WL %	SG %
Intercept	2.21 <sup>S</sup>	0.85 <sup>S</sup>
C (2,4,6)	2.03875 <sup>S</sup>	0.465 <sup>S</sup>
T (45,50, 55)	0.7 <sup>NS</sup>	0.0275 <sup>NS</sup>
Time (60,120, 180)	-0.41125 <sup>NS</sup>	-0.0275 <sup>NS</sup>
C*T	0.3075 <sup>NS</sup>	-0.155 <sup>NS</sup>
C*Time	-1.11e-16 <sup>NS</sup>	0.04 <sup>NS</sup>
T* Time	-0.8575 <sup>NS</sup>	-0.095 <sup>NS</sup>
C*C	1.12 <sup>S</sup>	-0.095 <sup>NS</sup>
T*T	0.4125 <sup>NS</sup>	0.045 <sup>NS</sup>
Time*Time	0.585 <sup>NS</sup>	-0.05 <sup>NS</sup>

\* Significant at 1 and 5% by t-test; S-Significant; NS-Non-significant



quadratic response surface plot in the optimization of the two parameters were obtained by keeping the other parameter at zero levels for WL rate in order to visualize the interaction effect of the two factors on the response. The osmotic stress may consequence in disruption of cell membrane and thereby enhanced the cell permeability, enabling the mass transfer between fruit matrix and osmotic medium. Thus, by increasing salt concentration, solute diffusivity can attain a maximum level, with succeeding drop thereafter. In addition, at higher concentrations, the solids could collect faster on surface of product, causing an extra hindrance for mass transfer causing least rate of solid gain (Eren *et al.*, 2007; Herman-Lara *et al.*, 2013).

Optimization of independent variables was performed by multi-criteria methodology with the analysis of Derringer function or desirability function showed that the prediction profiler constructed with high desirability of 0.82 corresponding to maximum water loss to the extent of 6.42% and minimum solid gain of 1.08% which corresponds to optimum level of 5.02%, 54.8! and 60.64 min. for concentration, temperature and time, respectively (Fig.5). The prediction profiler is a way to interactively change variables and look at the effects on the predicted response.

### Validation studies

Osmotic dehydration experiments were conducted at the optimum process conditions (at concentration of 5.02, temperature of 54.58! and process time of 60.64 min.) for testing the adequacy of model equations for predicting the response values. The observed experimental values (mean of 3 experiments) and values predicted by the equations of the model are presented in Table 5. Therefore, it could be concluded from above discussion that model is quite adequate to assess the behavior of the osmotic dehydration of aonla. The fitted values predicted by the models were compared with the experimental data. Under these optimal conditions, the experimental value of WL and SG rate is consistent with the predicted value with 1.09% and 3.66% difference respectively (Liu and Peng, 2017; Pravitha *et al.*, 2021; Behir *et al.*, 2020). Perusal of results presented in Table 5 showed minimal deviation between observed and predicted values for both the responses, which further strengthen the conclusion drawn about the suitability of the model developed.

### Adequacy of the models

#### Residual analysis

Even though, the F-values for the lack of fit were non-significant ( $p < 0.05$ ) which clearly shows the model adequacy, residual analysis is done to confirm the developed model to make sure that it gives a sufficient approximation to the actual values residual is the difference among the observed and fitted values (Table 6). Model generated residuals were subjected to detailed residual analysis as sole reliance of  $R^2$  and adjusted  $R^2$  is not enough to ensure the repeatability of the RSM results. Run test statistic Z value in both the cases being less than 1.96, showed that the residuals are randomly distributed. Also, Shapiro-Wilk test statistic value of near to unity for both the response variables too ensured that the residuals are normally distributed. These two conclusions further strengthen the adequacy of RSM models constructed in this study.

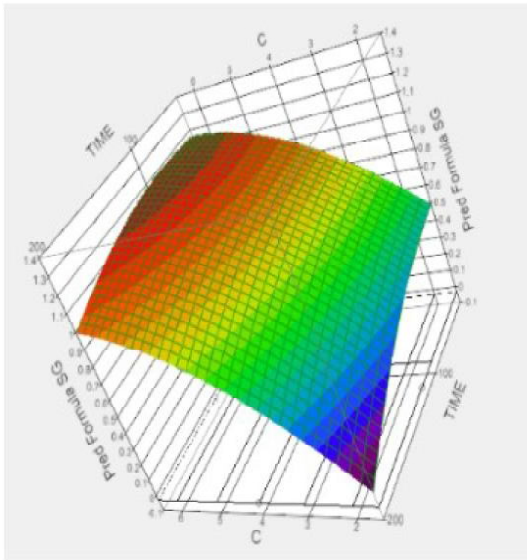
#### Microstructure analysis

The image analysis enabled to show how far the solid penetrates inside the fruit and to estimate the shrinkage factor of the fruit during the osmotic dehydration (Rodrigues and Fernandes, 2007).

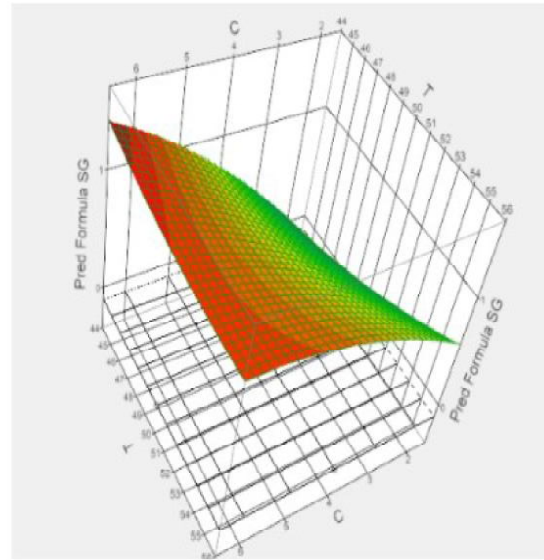
**Fresh sample:** It appeared as a compact structure comprising of swollen cells intimately bonded each other through extensive cell-to-cell attachments (Fig. 6a) with a greater degree of cell compartmentalization and small intracellular spaces of fresh tissue were noticed (Nunes *et al.*, 2008).

**Osmotically treated sample:** It is observed that osmotic dehydration process (at optimal condition) altered the tissue structure compared to the untreated samples (Fig. 6b). The cells were observed shrunk and distorted and their contour seemed irregular and wrinkling probably due to the solubilization of polysaccharides that constitutes the cells walls (Brochier *et al.*, 2015). Also, the water loss and the pre-concentration of salt on the surface of the tissue during the process caused shrinkage and tissue collapse. Furthermore, water loss induces the plasmolysis of cells and solid gain gives consistency to the tissues. There are several experimental findings in the literature that are consistent with our claims regarding the occurrence of cell structure modification during osmotic processing (Delgado *et al.*, 2005).

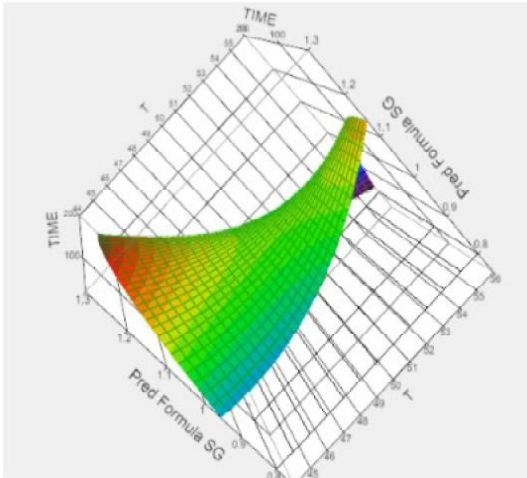




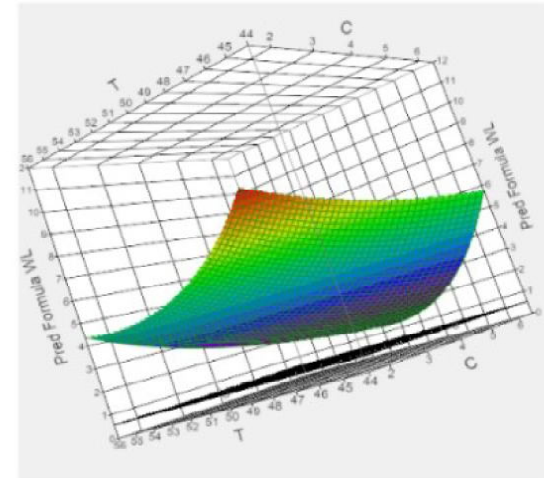
**a**



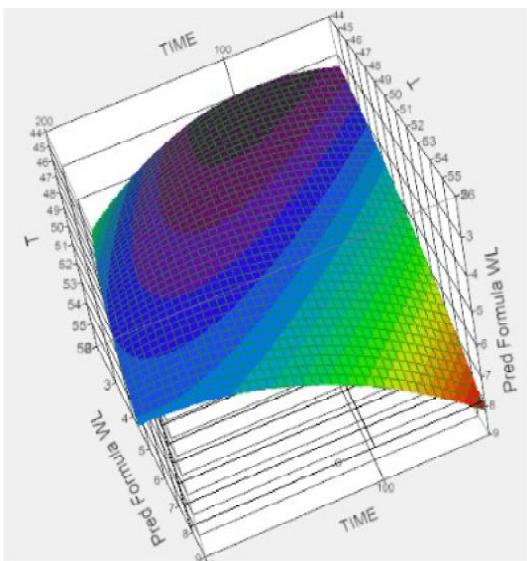
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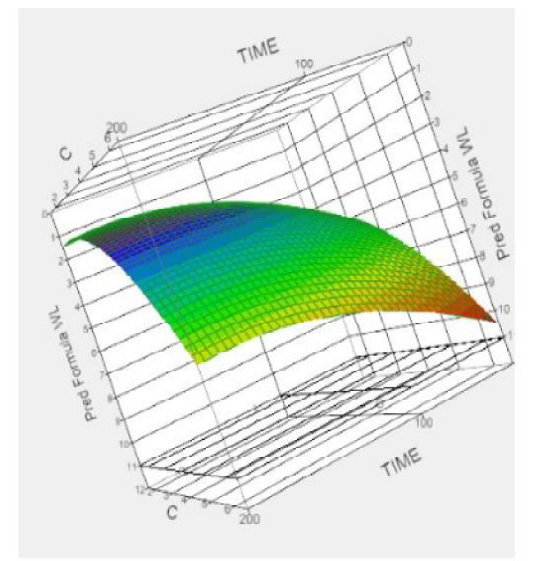
**c**



**d**



**e**



**f**

Fig. 4 : Illustration of profile for the quadratic response surface plot in the optimization of two variables for WL and SG

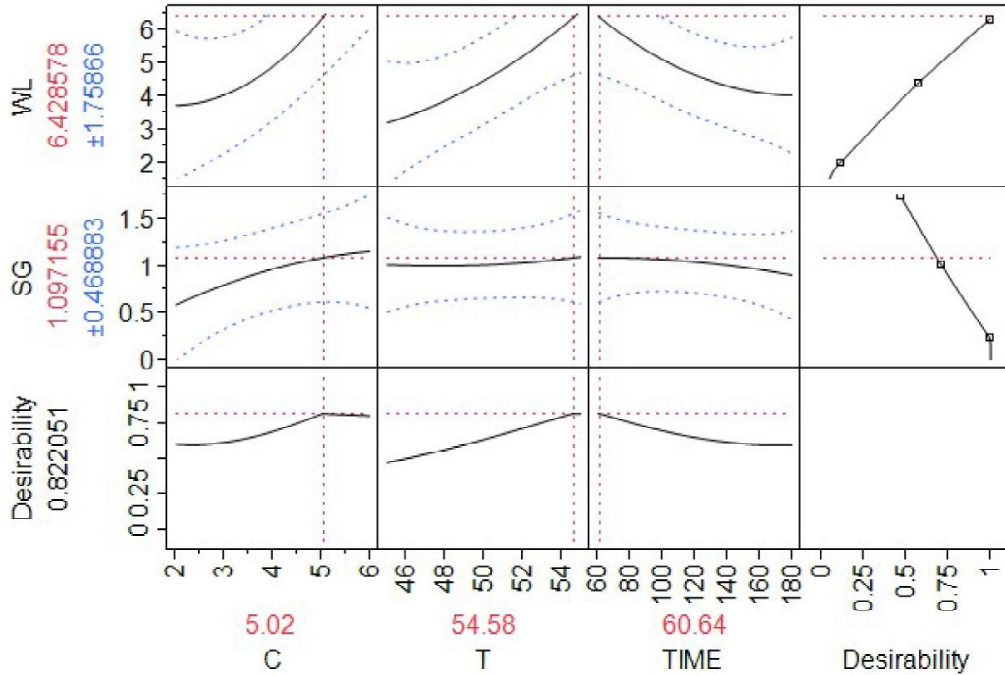


Fig. 5 : Prediction profiler for optimized conditions

**Table 5 : Predicted and experimental values of response at optimum process conditions for osmotic dehydration of aonla.**

	Predicted value	*Experimental value	Absolute error (%)
<b>Water loss (WL)</b>	6.42	6.35	1.09
<b>Solid gain (SG)</b>	1.09	1.13	3.66

Experimental values were expressed by average value in triplicate for eliminating the experimental errors.

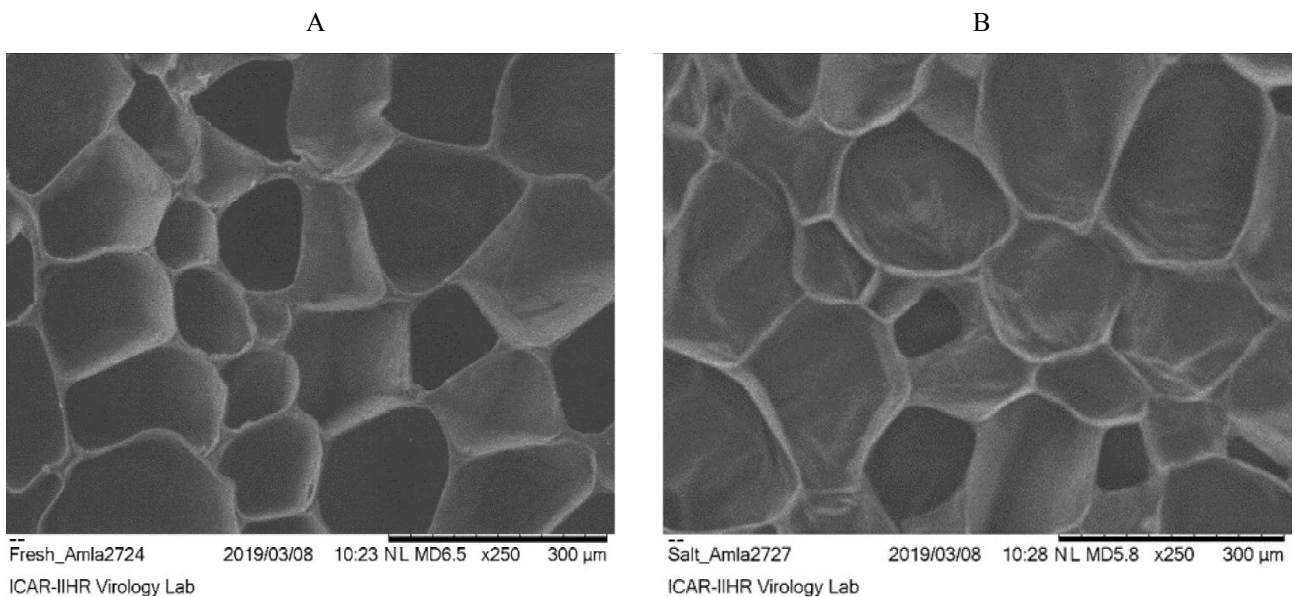


Fig. 6 : a) Fresh aonla b) Osmotically treated (salt) aonla

**Table 6 : Predicted and experimental values of responses at optimum conditions for salt medium.**

Concentration (%)	Temperature (°C)	Time (Minutes)	WL %	SG %	Predicted SG	RES SG	Predicted WL	RES WL
2	45	120	1.85	0.22	0.15	0.07	1.31	0.54
4	55	180	2.64	0.76	0.75	0.01	2.64	0.00
2	50	60	1.75	0.25	0.31	-0.06	2.29	-0.54
4	45	180	2.08	0.66	0.89	-0.23	2.95	-0.87
4	50	120	2.21	0.85	0.85	0.00	2.21	0.00
6	50	60	6.03	1.00	1.16	-0.16	6.37	-0.34
6	55	120	6.25	1.07	1.14	-0.07	6.79	-0.54
2	50	180	1.80	0.33	0.17	0.16	1.47	0.34
4	55	60	6.05	1.22	1.00	0.23	5.18	0.87
6	50	180	6.08	1.24	1.18	0.06	5.54	0.54
4	50	120	2.21	0.85	0.85	0.00	2.21	0.00
4	45	60	2.06	0.74	0.75	-0.01	2.06	0.00
4	50	120	2.21	0.85	0.85	0.00	2.21	0.00
2	55	120	1.76	0.35	0.52	-0.17	2.10	-0.34
6	45	120	5.11	1.56	1.39	0.17	4.77	0.34

WL: Water loss; SG: Solid gain; RES WL: Residual water loss; RES SG: Residual solid gain

## CONCLUSION

Response surface methodology was found effective for process optimization of parameter using Box-Behnken response surface design was successfully employed in this investigation to estimate and identify the optimal osmotic condition in order to prepare osmotically dehydrated aonla segments using salt solution as an osmotic agent. From the experimental results, second order polynomial models were developed for the responses (water loss and solid gain). The regression equations obtained can be used for optimum conditions for desired responses within the range of conditions applied in this study. The F-values for the lack of fit were non-significant ( $p < 0.05$ ) which clearly shows the model adequacy. Graphical techniques, in connection with RSM, aided in locating optimum operating conditions, which were experimentally verified and proven to be adequately reproducible. The results exhibited that osmotic solution concentration, process temperature and process time have significant effects on the osmotic dehydration process of amla. Response surface methodology was employed to model the effects of the process parameters on the quality attributes of

the soaked product and an optimal combination of 5.02% salt concentration, 54.8! temperature and 60.64 min. process time to attain a desired effect of maximum water loss (6.42%) and minimum solid gain (1.09%) in osmotic dehydration of aonla in salt medium.

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