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MODELLING OF OSMOTIC DEHYDRATION PROCESS OF PEAR (*PYRUS COMMUNIS* L.) IN TERNARY SOLUTIONS OF SUGAR AND CALCIUM SALT USING RESPONSE SURFACE METHODOLOGY

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ABSTRACT
The holistic effects of temperature (40-60°C), sugar concentration (40-60°B), calcium chloride (1-3%) and immersion time (120-180minutes) were studied during the Osmotic Dehydration (OD) of Pear Sp. in ternary solutions. The Box Behnken Design of Response Surface Methodology (R.S.M.) was used to optimize various process variables like Water Loss, Solids Gain, Weight Reduction and Overall Acceptability by sensory evaluation of the Osmosed Product. Thereby establishing these as the Response Variables during Osmotic Dehydration of Pear. Optimized conditions were found to be 59.57ºC temperature, 53.73ºB sugar concentration, 2.27% calcium lactate salt and 151.9 minutes immersion time; for minimizing solid gain and maximizing water loss, weight reduction and overall acceptability.

1. Introduction

Pear (Pyrus communis L.) originally belongs to temperate climate zone of Europe originated in Asiatic region (Sharma et al. 2003, Park et al. 2002). The pear fruit contains natural source of Vitamin C, B Complex Vitamins (folates and riboflavin), Vitamin K, organic and fatty acids, volatiles and minerals such as copper, iron, potassium, magnesium and fiber (Pavelkic et al., 2015, Amiripour et al., 2015). It is mostly liked by the consumers due to its low calorific value and high nutritive value (Salim et al., 2016) while volatile compounds present in pear contributes to its flavour. The colour of the pear depends on its pigments mainly chlorophyll (green) and carotenoid (yellow) (Pattee, 1985, Park et al., 2002). The chemical composition of pears influences its acceptable organoleptic characteristics (Pavelkic et al., 2015). According to FAO, 2014 data, pear is cultivated in 1574446 ha of area in the world while 42280 ha of area in India. World production of pear in 2014 was about 25798644 tonnes while that in India was 316700 tonnes (FAO, 2014). Mostly fresh pear is processed to make juices, alcoholic beverages, compotes, sweet courses, fruit yoghurt, marmalades and jams (Pavkov et al., 2010) while dried pears are used in bakery products and gravies (Pavelkic et al., 2015).The traditional preservation method is drying of matured pear fruit, which involves removal of moisture content of the fruit by hot air drying, utilizing various techniques. However, due to degradation in the nutritional quality of dried products, this process is not so effective. Removal of moisture from the fruit can also be done by method of osmotic dehydration. Osmotic dehydration (OD) is a technique which is based on the immersion of fruits in a hypertonic solution which creates a high osmotic pressure on the dipped fruits and vegetables and lowering its water activity (Raoult-Wack, 1994, Salim, 2016). However, the solute gain is less in comparision to the water loss from the fresh fruits and vegetables due to the semi-permeability of the cell membranes (Ahmed et al., 2016). OD depends on the properties of the biological material and complexity of each material varies from tissue to tissue and hence it is very challenging to optimize the processes and design the equipments for processing the biological materials (Tappi et al., 2017, Fernandez et al. 2004).

Ready to use intermediate moisture (IM) food products produced by Osmotic Dehydration (OD) are gaining importance now a days owning to its high nutritive content in comparison to be produced by any other drying methods (Pavelkic et al., 2015). This is due to the reason that OD has little effect on the flavour of the final product and its nutrients is preserved during the process. OD also provides benefits in decreasing the energy cost and retains the colour of the fruits and vegetables by inhibiting the browning of enzymes present in the food product (Salim et al., 2016, Pavelkic et al., 2015). OD modifies food composition in addition to water removal from the plant tissues. So, if the product is impregnated with desirable solutes then, it can improve the nutritional and sensorial characteristics of the final product (Tappi et al., 2017; Akbarian et al., 2014; Silva et al. 2014; Barrera et al., 2004). Selection of solute as an osmotic agent in the osmotic solution is a fundamental issue as it alongwith affects the dehydration kinetics and process cost, it also has an effect on the organoleptic and nutritional properties of the osmosed product. Several authors have considered Sucrose (Suc) as an optimal osmotic agent as it has higher efficiency than glucose (Tappi et al., 2017, Saputra, 2001) whereby reducing the enzymatic browning and aroma losses (Tappi et al., 2017; Cortellino et al., 2011; Qi et al., 1998; Lenart, 1996). Various investigators have performed OD with calcium salt in osmotic solutionmwhich has been used to increase the firmness of plant tissue while at the same time increasing the process efficiency, restricting sugar gain as sugar has higher molecular weight than the calcium salt it remains at the surface of the plant tissue and selectively allowing calcium to enter the tissue and here by at the same time increasing the water loss (Tappi et al., 2017; Mavroudis et al., 2012; Ferrari et al., 2010; Pereira et al., 2006). Pereira et al., (2006) stated that calcium salt disintegrates to form pectic polymers which by cross linking reinforces cell walls of the plant tissue and hence is able to reduce the damage from dehydration. When the conc. increases, damage to cell membrane may occur as reported by Anino et al., (2006). Also calcium being the desired solute, its salt have been used in osmotic solutions as a method for getting a fortified product high in nutrition, hence increasing the consumer intake due to being a calcium fortified product (Tappi et al., 2017; Silva et al. 2014; Barrera et al., 2004). It has also been stated that due to the addition of calcium salt the metabolic activity and respiration rates of different fruit species gets decreased (Tappi et al., 2017; Castello et al., 2010, Luna-Guzman et al., 1999; Lester, 1996) and therefore the stability of the product during storage gets potentially enhanced, especially considering that a lower respiration rate may lead to a longer shelf life. In addition, calcium present in the fruit affects the membrane and cell wall structure and functioning (Tappi et al., 2017; Maurel, 2007; Peiter et al., 2005).

Response surface methodology (RSM) is a statistical technique used to optimize the process variables in an experiment design and model development of a process. It has been widely used as an effective method for process and product improvement (Maran et al., 2013a, Maran et al., 2013b, Maran et al., 2013c). It helps in mapping a response surface of different variables to optimize the responses or for the selection of operating conditions or consumer requirements (Maran et al., 2013 c). It reduces the number of experiments, helps in the study of the interaction of variables, modeling and analysis of the responses with statistically valid results (Saxena et al., 2015). This approach helps an investigator to make efficient exploration of a process system (Amiripour, et al., 2015). Several studies have been conducted using RSM for OD (Amiripour, et al., 2015, Saxena, et al., 2015, Maciel, et al., 2015, Gupta, et al., 2014, Patil, et al., 2014, Ganjloo, et al., 2014, Saxena, et al., 2012, Mercali, et al., 2011, Saxena, et al., 2009, Ozdemir, et al., 2008, El-Aouar, et al., 2006, Uddin et al., 2004)

This research aimed to assess the influence of solution temperature (40-60 oC), sugar concentration (40-60 oB), calcium lactate salt concentration (1-3%) and immersion time (120-180 min) of osmotic dehydration (OD) of pear, establish process conditions which are able to provide maximum water loss, weight reduction and Overall acceptability score (OAA) and minimum solid gain and water activity in the product and optimizing using response surface methodology. In addition, the osmotic dehydration kinetics at optimized conditions was also studied and fitted to different empirical models viz. Magee's model, Page's model and Azuara's model so as to generate the data to design the OD systems.

2. Materials and methods

2.1. Preparation of sample

Fresh pears of uniform size were obtained from Agricultural Produce Marketing Committee (APMC), Azadpur market, New Delhi, India and were stored in the refrigerator before the use for the experiments. The fruits were washed using potable water, hand peeled with knife, de-seeded and vertically cut into four pieces. The average moisture content of pears was found to be 85.285% wet basis by oven drying method subjecting the uniform mash of fresh pears to 105 ^{o}C for 5 hours (AOAC, 2000). The osmotic solutions in the range of 40-60 ^{o}B were prepared by mixing food grade sucrose (amorphous refined sugar) and food grade calcium lactate salt in powder form (Universal laboratories, New Delhi, India) in the range of 1-3% w/w with calculated quantity of distilled water according to the experimental design. The conc. of sugar solutions was determined using a portable hand held refractometer (Omega RFH 101 & 201 of measuring range of 28-62% and 58-92%. In each of the experiments, fresh osmotic solution was used. All the experiments were done in triplicate and the average value was taken for calculations. The sample to solution ratio of 1:5 by weight was chosen to avoid significant dilution during OD (Sangamithra*et al.*, 2014, Uddin et al., 2004; Le Marguer, 1988).

2.2. Osmotic dehydration

Sample of 50 g of pear slices were weighed and totally submerged into the osmotic solutions as per the experimental design. At each time of sampling (120-180 min), the pear slices were drawn out and quickly rinsed under a fast flowing stream of cold water, then gently blotted with adsorbent paper and weighed with an analytical balance (Schimadzu make) with an accuracy of $\pm$ 0.001g and the residual moisture and solid content after the osmosis was determined using the oven drying method (AOAC, 2000). Experiments were performed in triplicates and the average values were reported in order to average out any possible in accuracies. OD kinetics were analysed using the gravimetric equation (1-3) for the mass transfer parameters, water loss (WL), solid gain (SG) and weight reduction (WR) and were expressed in percentage of initial composition (Ozen*et al.,* 2002; Singh *et al.,* 2007; Fernandes and Rodrigues, 2008). The response surface methodology with Box-Behnken design using the software Design Expert Trial 7.0.0 version, Statease Inc., Minneapolis, USA was used to optimize the processing conditions process variables (Table 1) during OD of pears.

Table 1. Experimental variables and their levels used for Response Surface Methodology (Box Behnken Design) osmotic dehydration of Pear fruits

	Symbols	-1	0	1
Temperature (°C)	A	40	50	60
Sugar Concentration (°B)	B	40	50	60
Calcium salt (%)	C	1	2	3
Treatment time (min)	D	120	150	180

These response variables were calculated using the data of weight and moisture content of each sample.

$$\% WL = \frac{(W_0 - W_t) + (S_t - S_0)}{W_0} \times 100 \quad (1)$$

$$\% SG = \frac{S_t - S_0}{W_0} \times 100 \quad (2)$$

$$\% WR = WL - SG \quad (3)$$

where, W_0 is the initial weight of sample taken before OD, Wt is the weight of the sample after OD at the time t, S_0 is the initial weight of solids of the sample and S_t is the weight of solids (dry matter) in the sample after OD at the time t.

2.3. Water activity

The equilibrium amount of water which is available for the hydration of materials is referred to aswater activity. If there is a value of unity, it indicates pure water, whereas zero indicates the total absence of any water molecule (bone-dry matter). a_w of a sample only depends upon its water content and corresponding temp. Dew point equipment (Aqua Laboratory, Decagon CX-2, Decagon Devices Inc., Pullman, Washington, USA) was used for the determination of a_w at 25 °C.

2.4. Color measurements

The color of the pear slices was measured using Labscan XE colorimeter (HunterLab, Inc., Reston, VA, USA) under D65 illuminating lamp conditions keeping the observer angle of 10°, after calibrating it with a white ceramic tile. The color values were expressed as *L*- value (lightness/ darkness), *a*- value (redness/greenness) and *b*- value (yellowness/ blueness) on the Hunter scale.

2.5. Sensory evaluation

Osmosed product was evaluated for color, taste, texture and its overall acceptability (OAA). Ten trained panelists conductedthe sensory evaluation using a nine point hedonic scale (9: excellent; 7: good; 5: acceptable (limit of marketability); 3: poor and 1: extremely poor) (Larmond, 1977). The samples were coded and were drawn from each level of the experimental design, and presented to the panelists randomly in a whitelight illuminated room and maintained at 25°C.

2.6. Optimizationand statistical analysis

Second order polynomial equation 4 was used to fit the experimental data which further described the combined effect of all the variables on the response Y and determined the interrelationship among the variables.

$$Y_i = ß_0 + ß_1X_1 + ß_2X_2 + ß_3X_3 + ß_4X_4 + ß_{11}X_1^2 + ß_{22}X_2^2 + ß_{33}X_3^2 + ß_{44}X_4^2 + ß_{12}X_1X_2 + ß_{13}X_1X_3 + ß_{14}X_1X_4 + ß_{23}X_2X_3 + ß_{24}X_2X_4 \quad (4)$$

where Y_i is the predicted response for WL, SG, WR, *L*- value, *a*- value, *b*- value, a_w, and OAA score.β_0 is the estimated coefficient of the fitted response at the center point of the design, β_1, β_2, β_3, β_4 are linear, β_{11}, β_{22}, β_{33}, β_{44} quadratic and β_{12}, β_{13},β_{14}, β_{23}, β_{24} are interaction coefficients, respectively. Non linear statistical optimization procedure was used to fit the polynomials for various responses studied. The surface plots were used to show the fitted polynomial equations so that the relationship between the response and experimental levels of each factor can be visualized and used to interpret the optimum conditions. Graphical optimization technique was used to find workable optimum conditions by fixing two of the variables at predetermined optimum level. Therefore, osmotic dehydration process was optimized on conditions giving maximum WL, WR and OAA score, minimum SG and a_w and determines the levels of temp., sugar solution conc., Ca salt and immersion time in osmotically dried product.

3. Results and discussions

Preservation of pearby osmotic dehydration instead of conventional drying was done by development of high moisture stabilized pear pieces through an optimized process. OD over conventional drying is highly desirable and advantageous for a high quality product with improved shelf life.

3.1. Osmotic dehydration process

Experiments were conducted for different combinations of the process variables viz. temp. of ternary solution , sugar conc., calcium salt conc. and immersion time and their responses WL, SG, WR, *L*- value, *a*- value, *b*-value, $a_w$and OAA score were obtained (Table 2). Optimization of the process was carried out as per the Box-benhken design of the response surface methodology. ANOVA was used to evaluate the significance of linear, quadratic and interaction effect of each variable on the responses. The sum of squares of all the responses were found to be significant ($p<0.01$). A second order polynomial model (Eq 8) explains the variability in responses with high coefficient of determination ($R^2> 0.90$) representing good fit (Table 3). The statistical lack of fit test for all responses was non-significant ($p>0.05$) and therefore, it conforms to the statistical methodology used and is applicable in the case of osmotic dehydration of pear slices (Khuri and Cornell, 1996; Saxena *et al.*, 2009). The combined effect of the two variables on any response for each fitted model was studied as the function of two independent variables. So, the software was used to develop three-dimensional plots of the response surface of variables while keeping the other two variables at centre value. Response surface plots were generated for each combination of variables keeping the other two variables at centre points as constant factor. Effects of variables on responses were explained by the plotted response surface figures.

3.2. Influence of variables on water loss

The range of water loss varied from 14.86 to 28.33% (Fig 1a). Experiment no. 17, resulted in highest WL which corresponded to 50 ºC temp., 50 ºB sugar conc., 3% calcium salt conc. and 180 min immersion time while experiment no. 25 resulted in minimum WL which corresponded to 50 ºC temp., 40 ºB sugar conc., 2% calcium salt conc. and 120 min immersion time (Table 2).

Table 2. Experimental design and values of response variables for osmotic dehydration of Pear

Experimental run	A:Temperature	B:Sugar Conc.	C: CaCl2	D: Time	WL	SG	WR	*L*- value	*a* - value	*b* -value	aw	OAA
	(ºC)	(ºB)	(%)	(min	(%)	(%)	(%)					
1	40	50	2	120	17.46	5.7	11.81	69.55	-3	17.506	0.736	6.83
2	50	50	3	120	19.55	6.89	12.69	60.81	-3.12	17.546	0.652	8.01

3	50	60	3	150	23.27	7.5	15.71	48.18	-3.21	17.371	0.691	7.94
4	40	40	2	150	17.35	6.53	10.87	68.11	-3.22	17.085	0.671	7.35
5	50	50	1	120	18.37	6.45	11.97	64.59	-2.94	17.371	0.823	8.22
6	40	50	2	180	25.47	8.27	17.26	52.78	-2.31	17.452	0.799	7.19
7	50	60	2	120	17.69	5.91	11.75	54.23	-3.26	17.597	0.723	8.26
8	50	50	2	150	25.71	8.11	17.55	44.34	-3.52	17.25	0.843	8.4
9	60	50	2	180	27.97	8.68	19.33	46.56	-1.99	17.536	0.688	6
10	50	40	2	180	23.01	8.19	14.78	47.08	-2.8	17.435	0.7	8.07
11	50	60	1	150	21.53	8.03	13.45	51.01	-3	17.196	0.724	7.95
12	60	50	1	150	22.92	7.69	15.17	58.46	-2.53	17.114	0.605	5.76
13	50	50	2	150	25.73	8.13	17.57	44.14	-3.5	17.27	0.753	8.42
14	60	50	3	150	24.87	8.11	16.71	54.98	-2.71	17.254	0.789	6.9
15	60	60	2	150	22.54	7.23	15.36	52.18	-2.67	17.276	0.849	6.72
16	50	50	1	180	26.42	9.16	17.31	48.48	-2.22	17.345	0.788	7.23
17	50	50	3	180	28.33	9.55	18.85	43.09	-2.45	17.6	0.692	8.42
18	50	50	2	150	25.81	8.21	17.65	45.74	-3.42	17.35	0.575	8.5
19	40	50	1	150	21.2	7.38	13.78	65.73	-2.86	17.023	0.675	6.91
20	50	50	2	150	25.8	8.2	17.64	45.74	-3.43	17.34	0.688	8.49
21	60	40	2	150	20.22	6.71	13.56	61.09	-2.8	17.176	0.821	6.53
22	50	40	3	150	19.99	8.05	11.88	56	-3.42	17.236	0.827	8.54
23	60	50	2	120	19.16	5.9	13.29	63.43	-2.66	17.455	0.808	6.95
24	50	40	1	150	18.66	6.72	11.89	62.53	-3.2	16.981	0.778	7.57
25	50	40	2	120	14.86	5.22	9.6	65.2	-3.37	17.404	0.842	8.21
26	40	50	3	150	22.34	7.77	14.51	59.75	-3.09	17.313	0.849	6.76
27	50	50	2	150	25.73	8.13	17.57	44.14	-3.5	17.27	0.84	8.42
28	40	60	2	150	21.16	6.76	14.45	57.29	-2.95	17.335	0.85	6.91
29	50	60	2	180	26.32	8.26	18.01	38.71	-2.5	17.593	0.842	7.78

The process variables which were expressed as the linear and quadratic terms had significant ($p<0.05$) effect on WL as shown from the analysis of variance.The values of the regression coefficient of the fitted second order polynomial was used for the interpretation of relationship between responses and variables. Itcan be seen that the effect on WL in descending order of the process variables on the basis of the values of regression coefficient are immersion time (a_4=4.20), sugar conc. of ternary solution (a_2=1.54), temp. of ternary solution (a1=1.06) and calcium salt conc. (a3=0.77). These results indicated that time of immersion and sugar conc. are more important variables influencing the water loss of pear slices in comparison to the temp. and calcium salt conc. The interaction of process variables, showed a significant effect on WL during OD ($p<0.05$)(Table3).

Table 3. Regression coefficients of the fitted second-order polynomials representing the relationship between

Coefficients	WL (%)	SG (%)	WR (%)	L- value	a value	b value	a_w	OAA
a_0	25.76	8.16	17.60	44.82	-3.474	17.30	0.8446	8.45
a_1	1.06*	0.16*	0.90*	-3.04*	0.173*	0.01*	-0.0263*	-0.26*
a_2	1.54*	0.19*	1.35*	-4.87*	0.102*	0.09*	-0.0063*	-0.06*
a_3	0.77*	0.20*	0.57*	-2.33*	-0.104*	0.11*	0.0239*	0.24*

a_4	4.20*	1.34*	2.87*	-8.43*	0.340*	0.01*	-0.0148*	-0.15*
a_{12}	-0.37*	0.07*	-0.45*	0.48	-0.035	-0.04*	0.0160*	0.16*
a_{13}	0.20*	0.01	0.20*	0.63	0.013	-0.04*	0.0325*	0.32*
a_{14}	0.20*	0.05	0.15*	-0.03	-0.005	0.03*	-0.0328*	-0.33*
a_{23}	0.10*	-0.47*	0.57*	0.93	0.002	-0.02	-0.0243*	-0.25*
a_{24}	0.12*	-0.16*	0.27*	0.65	0.048	-0.01	-0.0090*	-0.09*
a_{34}	0.18*	-0.01	0.21*	-0.40	-0.013	0.02	0.0360*	0.35*
a_{11}	-1.74*	-0.60*	-1.12*	10.09*	0.483*	-0.05*	-0.1476*	-1.50*
a_{22}	-3.75*	-0.80*	-2.97*	4.05*	0.032*	-0.03*	-0.0143*	-0.12*
a_{33}	-1.14*	0.22*	-1.39*	5.55*	0.238*	-0.07*	-0.0351*	-0.32*
a_{44}	-1.50*	-0.42*	-1.05*	3.16*	0.505*	0.24*	-0.0173*	-0.20*
R^2	0.9999	0.9986	0.9998	0.9947	0.9909	0.9893	0.9976	0.9976
*Significant at 5% level								

WL increased with the increase in immersion time, temp., solution conc., in osmotic solution, while loss of water was also affected by calcium salt conc. It reveals that water loss increased more with the increased sugar conc. than in temp. of ternary solution. During the investigation, it was found that at the higher sugar conc. and process temp., the water loss was faster and also reduced the immersion time to reach the equilibrium.As temperature increases the membrane permeability increases, leading to swelling and plasticization of the cell membranes therefore favoring mass transfer from the tissue (Mercali*et al.*, 2012; Lazarides*et al.*, 1995).With the rise in temp., the viscosity of the solution decreases and the external resistance to mass transfer reduces allowing water and solute transport easier (Mercali*et al*,. 2012; Tonon*et al.*, 2007).Sugar having higher molecular weight than calcium lactate might have remained on the surface of the pear slices and may have allowed higher impregnation of calcium salt into the pear slices resulting in enhanced water loss. The sugar conc. and calcium salt have a synergistic effect on WL and increased the firmness of plant tissues as well (Pereira *et al.*, 2006, Ferrari *et al.*, 2010 Mavroudis*et al.*, 2012; Mercalli *et al.*, 2012 and Amiripour*et al.*, 2015). Similar results have also been reported for osmotic dehydration of cherry tomatoes by Derossi*et al.*, (2015), aloe vera gel cubes by Pisalkar*et al.*, (2011) and pumpkin by Mayor *et al.*, (2007). The possible damage caused to the cell membranes of the pear slices by the osmotic dehydration process may have been compensated by the calcium ions in the solution by cross linking the pectic polymers and a calcium fortified product has been obtained (Barrera *et al.*, 2004, Anino*et al.*, 2006 and Silva *et al.*, 2014).Three dimensional response surface was plotted for the WL. Fig. 1a shows the most significant interaction plot between process variables. It is selected on the basis of high correlation coefficient among all interaction of the variables. It shows that as temp. increases, WL increases. In the same way as the calcium salt conc. increases, WL increases.

The multiple regression equation at 5% level of significance and neglecting the non-significant terms provided a good fit for describing the relationship between the process variables and response. The uncoded process variable form of the developed model, is as follows:

$$WL = -185.34 + 1.89 * Temp. + 4.01 * Sugar\ Conc. + 2.89 * Calcium\ salt + 0.57 * Time - 0.004 * Temp. * Sugar\ Conc. + 0.02 * Temp. * Calcium\ salt + 0.0007 * Temp. * Time + 0.01 * Sugar\ Conc. * Calcium\ salt + 0.0004 * Sugar\ Conc. * Time + 0.0061 * Calcium\ salt * Time - 0.0174 * Temp^2 - 0.04 * Sugar\ Conc.^2 - 1.14 * Calcium\ salt^2 - 0.0017 * Time^2 \quad (1)$$

3.3. Influence of variables on solid gain

The range of SG varied from 5.22 to 9.55% (Fig 1b). Experiment no. 17, resulted in highest SG which corresponded to 50 °C temp., 50 °B sugar conc., 3% calcium salt conc. and 180 min immersion time while experiment no. 25 resulted in minimum SG which corresponded to 50 °C temp., 40 °B sugar conc., 2% calcium salt conc. and 120 min immersion time (Table 2). The process variables which were expressed as the linear and quadratic terms had significant ($p<0.05$) effect on SG as shown from the analysis of variance. The values of the regression coefficient of the fitted second order polynomial was used for the interpretation of relationship between responses and variables. Itcan be seen that the effect on SG in descending order of the process variables on the basis of the values of regression coefficient are immersion time (a_4=1.34), calcium salt conc. (a_3=0.20), sugar conc. of ternary solution (a_2=0.19), temp. of ternary solution (a1=0.16) and. These results indicated that time of immersion and calcium salt conc. are more important variables influencing the SG of pear slices in comparison to the temp. and sugar conc. (Table 3). SG increases more with the increase in sugar concentration than increase in temperature as a_2 is higher than a_1 while SG also increases with the increase in the osmosis time. The interaction of process variables, between temp. and sugar conc., sugar conc. and calcium salt conc., sugar conc. and immersion time showed a significant effect on SG during OD ($p<0.05$) (Table 3).

Three dimensional response surfaces were plotted for the SG. Fig. 1b shows the most significant interaction plot between process variables. It is selected on the basis of high correlation coefficient among all interaction of the variables. SG increased with increase in immersion time and sugar conc. as well as with increase in calcium salt and temp. (data not reported). Permeability of cell membrane is the reason for the solid gain. Permeability is lost due to the effect of the increase in temperature of the syrup and allows the solute to enter by losing its selectivity. The viscosity of the solution decreases at high temperatures and influences solid gain, because of the decrease in viscosity ultimately decreases the resistance to diffusion of solute into the sample tissue. Similar results have been reported for OD of cherry tomatoes by Derossi*et al.*, (2015), aloe vera gel cubes by Pisalkar*et al.*, (2011) and pumpkin by Mayor *et al.*, (2007). Due to high conc. difference between the pear slices and osmotic solution, SG in pear increased as the conc. of the osmotic solution increased (Mundada*et al.*, 2010). Cell plasmatic membrane allows solute to enter the pear slice because with the increase of the osmotic pressure gradient there is the loss of functionality of cell plasmatic membrane. Studies on osmotic dehydration of apple, pineapple (Sujata and Das, 2005), mango (Duduyemi, *et al.*, 2013) and cranberry (Shamaei, *et al.*, 2012) have shown similar results. There is non-significant effect ($p>0.05$) of temp. and calcium salt interaction and reveals that SG increases more with increase in temp. than increase in calcium salt conc.. The calcium salt conc. has non-significant ($p>0.05$) effect on the SG of pear with respect to immersion time. The multiple regression equation at 5% level of significance and neglecting the non-significant terms provided a good fit for describing the relationship between the process variables and response. The uncoded process variable formof the developed model is as follows:

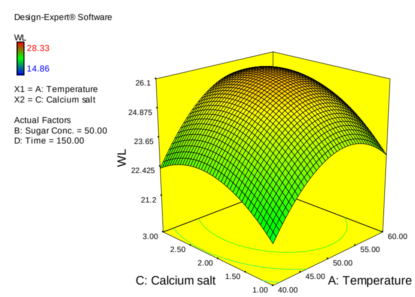
$$SG = -50.58 + 0.55 * Temp. + 0.95 * Sugar\ Conc. + 1.66 * Calcium\ salt + 0.20 * Time + 0.000725 * Temp. * Sugar\ Conc. - 0.05 * Sugar\ Conc. * Calcium\ salt - 0.0005 * Sugar\ Conc. * Time - 0.006 * Temp.^2 - 0.008 * Sugar\ Conc.^2 + 0.22 * Calcium\ salt^2 - 0.0005 * Time^2 \quad (2)$$

3.4. Influence of variables on weight reduction

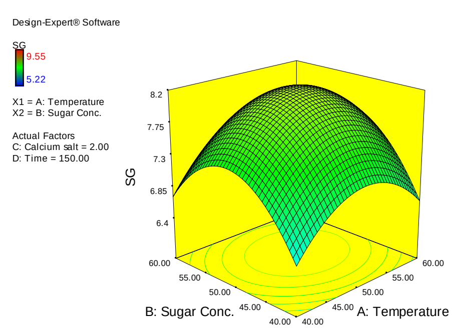
The range of WR varied from 9.6 to 19.33% (Fig 1c). Experiment no. 9, resulted in highest WR which corresponded to 60 °C temp., 50°B sugar conc., 2% calcium salt conc. and 180 min immersion time while experiment no. 25 resulted in minimum WR which corresponded to 50 °C temp., 40 °B sugar conc., 2% calcium salt conc. and 120 min immersion time (Table 2). The process variables which were expressed as the linear and quadratic terms had significant ($p<0.05$) effect on WR as shown from the analysis of variance. The values of the regression coefficient of the fitted second order polynomial was used for the interpretation of relationship between responses and variables. Itcan be seen that the effect on WR in descending order of the process variables on the basis of the values of regression coefficient are immersion time (a_4=2.87), sugar conc. of ternary solution (a_2=1.35), temp. of ternary solution (a_1=0.90) and calcium salt conc. (a_3=0.57). These results indicated that time of immersion and conc. of ternary solution are more important variables influencing the WR of pear slices in comparison to the temp. and calcium salt conc. (Table 3). WR increases more with the increase in sugar concentration than increase in temperature as a_2 is higher than a_1 while WR also increases with the increase in the osmosis time. The interaction of process variables showed a significant effect on WR during OD ($p<0.05$) (Table 3). Three dimensional response surfaces were plotted for the WR. Fig. 1c shows the most significant interaction plot between process variables. It is selected on the basis of high correlation coefficient among all interaction of the variables. Other plot between immersion time versus temperature and immersion time versus sugar conc. and immersion time versus calcium salt conc. showed that immersion time is the most significant of all factors affecting weight reduction (data not reported).

With the passage of immersion time during OD, the tissue allows penetration of calcium salt having lower molecular weight and the sugar having more molecular weight might have got deposited on the surface of the tissue which may have posed an additional resistance to mass exchange and lowering the rates of WR. Similar trends have been reported by Ganjloo, *et al.*, 2014 for seedless guava and Jokic, *et al.*, 2007 for sugar beet. Due to the high concentration of the osmotic solution, the pear having soft tissues floated in the solution which created hindrances of contact between pear slices and osmotic solutions and thereby reducing the rate of mass transfer and ultimately the rate of WR also lowered. El-Aouar, *et al.* (2006) have also reported the reduction of mass transfer rates similarly during the osmotic dehydration of papaya. The multiple regression equation at 5% level of significance and neglecting the non-significant terms provided a good fit for describing the relationship between the process variables and response. The uncoded process variable form of the developed model, is as follows:

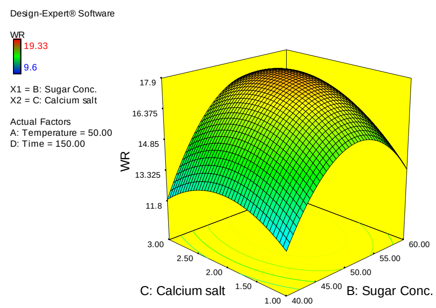
$$WR = -133.97 + 1.32 * Temp. + 3.08 * Sugar\ Conc. + 1.25 * Calcium\ salt + 0.36 * Time - 0.005 * Temp. * Sugar\ Conc. + 0.02 * Temp. * Calcium\ salt + 0.0005 * Temp. * Time + 0.06 * Sugar\ Conc. * Calcium\ salt + 0.0009 * Sugar\ Conc. * Time + 0.007 * Calcium\ salt * Time - 0.01 * Temp.^2 - 0.03 * Sugar\ Conc.^2 - 1.39 * Calcium\ salt^2 - 0.001 * Time^2 \quad (3)$$



(a)



(b)



(c)

Figure 1. Response surface plot for the effect of process variables on water loss (a), solid gain (b), and weight reduction (c) of pear slices

3.5. Influence of variables on color values

The range of *L*- value varied from 38.71 to 69.55 (Fig 2a). Experiment no. 1, resulted in highest *L*- value which corresponded to 40 ^oC temp., 50 ^oB sugar conc., 2% calcium salt conc. and 120 min immersion time while experiment no. 29 resulted in minimum *L*- value which corresponded to 50 ^oC temp., 60 ^oB sugar conc., 2% calcium salt conc. and 180 min immersion time (Table 2). The process variables which were expressed as the linear and quadratic terms had significant ($p<0.05$) effect on *L*- value as shown from the analysis of variance. The values of the regression coefficient of the fitted second order polynomial were used for the interpretation of relationship between responses and variables. It can be seen that the effect on *L*- value in descending order of the process variables on the basis of the values of regression coefficient are immersion time (a_4=-8.43), sugar conc. of ternary solution (a_2=-4.87), temp. of ternary solution (a_1=-3.04) and calcium salt conc. (a_3=-2.33). These results indicated that time of immersion and conc. of ternary solution are more important variables influencing the *L*- value of pear slices in comparison to the temp. and calcium salt conc. (Table 3). The interaction of process variables showed a non-significant effect on *L*- value during OD ($p<0.05$) (Table 3).

Fig. 2a describes the surface plot for the effect of pretreatment variables calcium salt conc. and sugar conc. at centre values of time and temp. on *L*- value of Pear slices. It is selected on the basis of high correlation coefficient among all interaction of the variables. It shows that *L*- value decreased with increase in sugar conc. and calcium salt conc.other significant plot between temp. and calcium salt conc. showed that as temp. and calcium salt conc. increased the *L*- value increased (data not reported). As the same time the surface plot between variables and immersion time showed horse saddle plot. Saxena *et al*. (2015) reported similar result for jackfruit bulb that visual colour is mainly a function of blanch. sol. conc. *L*- value also increased with increase in immersion time for osmosis and increase in calcium salt conc. of osmotic solution. The reason could be attributed to the impregnation of calcium salt and inward movement of solutes in the pear slice. Similar results have been reported that calcium salt conc. increased the visual colour by enhancing it. (Saxena *et al*., 2015). The multiple regression equation at 5% level of significance and neglecting the non-significant terms provided a good fit for describing the relationship between the process variables and response. The uncoded process variable form of the developed model, is as follows:

$$L\text{- value} = 624.80 - 10.74 * \text{Temp.} - 5.28 * \text{Sugar Conc.} - 30.27 * \text{Calcium salt} - 1.41 * \text{Time} + 0.1 * \text{Temp.}^2 + 0.04 * \text{Sugar Conc.}^2 + 5.55 * \text{Calcium salt}^2 + 0.004 * \text{Time}^2 \quad (4)$$

The range of *a*- value varied from -1.99 to -3.52 (Fig 2b). Experiment no. 8, resulted in highest *a*- value which corresponded to 50 ^oC temp., 50 ^oB sugar conc., 2% calcium salt conc. and 150 min immersion time while experiment no. 9 resulted in minimum *a*- value which corresponded to 60 ^oC temp., 50 ^oB sugar conc., 2% calcium salt conc. and 180 min immersion time (Table 2). The process variables which were expressed as the linear and quadratic terms had significant ($p<0.05$) effect on *a*- value as shown from the analysis of variance. The values of the regression coefficient of the fitted second order polynomial were used for the interpretation of relationship between responses and variables. It can be seen that the effect on *a*- value in descending order of the process variables on the basis of the values of regression coefficient are immersion time (a_4=0.340), temp. of ternary solution (a_1=0.173), calcium salt conc. (a_3=-0.104) and sugar conc. of ternary solution (a_2=0.102),. These results indicated that time of immersion and temp. of ternary solution are more important variables influencing the *a*-value of pear slices in comparison to the sugar conc. and calcium salt conc. (Table 3).

The interaction of process variables showed a non- significant effect on *a*- value during OD ($p<0.05$) (Table 3). Fig. 2b describes the surface plot for the effect of process variables time and sugar conc. at centre values of temp.and calcium salt conc. on *a*- value of pear slices. The figure was selected on the basis of highest value of regression coefficient of interaction variables. It shows that *a*- value increased with the increase in immersion time and with the increase in sugar conc. The reason of increase in *a*- value with immersion time could be attributed to increase in solid content in the pear slice. Similar result has been reported that use of calcium salt increased the visual color of the products (Saxena *et al*., 2015). The multiple regression equation at 5% level of significance and neglecting the non-significant terms provided a good fit for describing the relationship between the process variables and response. The uncoded process variable form of the developed model is as follows:

$$\text{a value} = 20.32 - 0.45 * \text{Temp.} - 0.03 * \text{Sugar Conc.} - 1.07 * \text{Calcium salt} - 0.16 * \text{Time} + 0.005 * \text{Temp.}^2 + 0.0003 * \text{Sugar Conc.}^2 + 0.24 * \text{Calcium salt}^2 + 0.0006 * \text{Time}^2 \quad (5)$$

The range of *b*- value varied from 16.981 to 17.6 (Fig 2b). Experiment no. 17, resulted in highest *b*- value which corresponded to 50 ^oC temp., 50 ^oB sugar conc., 3% calcium salt conc. and 180 min immersion time while experiment no. 24 resulted in minimum *b*- value which corresponded to 50 ^oC temp., 40 ^oB sugar conc., 1% calcium salt conc. and 150 min immersion time (Table 2). The process variables which were expressed as the linear and quadratic terms had significant ($p<0.05$) effect on *b*- value as shown from the analysis of variance. The values of the regression coefficient of the fitted second order polynomial were used for the interpretation of relationship between responses and variables. It can be seen that the effect on *b*- value in descending order of the process variables on the basis of the values of regression coefficient are calcium salt conc. (a_3=0.11), conc. of ternary solution (a_2=0.09), immersion time (a_4=0.01), temp. of ternary solution (a_1=0.01), and These results indicated that calcium salt conc. (a_3=0.11) and sugar conc. of ternary solution (a_2=0.09)are more important variables influencing the *b*- value of pear slices in comparison to the sugar conc. and calcium salt conc. (Table 3).The interaction of process variables showed a non- significant effect on *a*-value during OD ($p<0.05$) (Table 3).Fig. 2c describes the surface plot for the effect of pretreatment variables sugar conc. and temp. at centre values of immersion time and calcium salt conc. on *b*- value of pear slices. It shows that as temp. and sugar conc increased the *b*-value increased. The figure was selected on the basis of highest value of regression coefficient of interaction variables. The multiple regression equation at 5% level of significance and neglecting the non-significant terms provided a good fit for describing the relationship between the process variables and response. The uncoded process variable form of the developed model is as follows:

$$\text{b value} = 19.65 + 0.06 * \text{Temp.} + 0.07 * \text{Sugar Conc.} + 0.58 * \text{Calcium salt} - 0.085 * \text{Time} - 0.0004 * \text{Temp.} * \text{Sugar Conc.} - 0.004 * \text{Temp.} * \text{Calcium salt} + 0.0001 * \text{Temperature} * \text{Time} - 0.000490417 * \text{Temp.}^2 - 0.0003 * \text{Sugar Conc.}^2 - 0.07 * \text{Calcium salt}^2 + 0.0003 * \text{Time}^2 \quad (6)$$

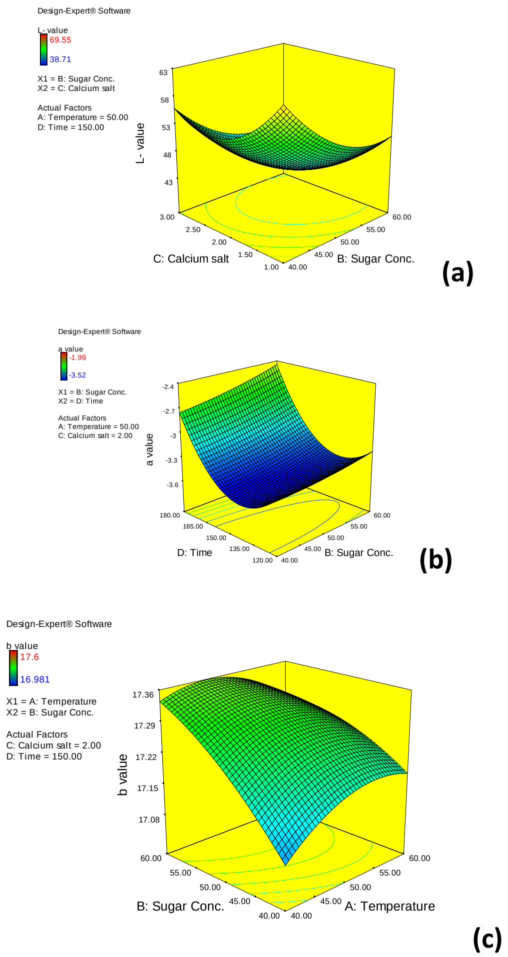


Figure 2. Response surface plot for the effect of process variables on *L*- value (a), *a*- value (b), *b* value (c) of pear slices

3.6. Influence of variables on water activity

The range of water activity (a_w) varied from 0.576 to 0.855 (Fig 2b). Experiment no. 28, resulted in highest a_w which corresponded to 40 ºC temp., 60 ºB sugar conc., 2% calcium salt conc. and 150 min immersion time while experiment no. 18 resulted in minimum a_w which corresponded to 50 ºC temp., 50 ºB sugar conc., 2% calcium salt conc. and 150 min immersion time (Table 2). The process variables which were expressed as the linear and quadratic terms had significant (p<0.05) effect on a_w as shown from the analysis of variance. The values of the regression coefficient of the fitted second order polynomial were used for the interpretation of relationship between responses and variables. It can be seen that the effect on a_w in descending order of the process variables on the basis of the values of regression coefficient are temp. of ternary solution (a_1=-0.0263), calcium salt conc. (a_3=0.0239), immersion time (a_4=-0.0148) and sugar conc. of ternary solution (a_2=-0.0063). These results indicated that temp. of ternary solution (a_1=-0.0263) and calcium salt conc. (a_3=0.0239) are more important variables influencing the a_w of pear slices in comparison to the sugar conc. and calcium salt conc. (Table 3). The interaction of process variables showed a significant effect on *a*-value during OD (p<0.05) (Table 3). Fig. 2c describes the surface plot for the effect of pretreatment variables immersion time and calcium salt conc. at centre values of immersion time and temp. on a_w of pear slices. It shows that as time and calcium salt conc. increased the a_w decreased. The figure was selected on the basis of highest value of regression coefficient of interaction variables. These results indicated that a_w the decreased with increase in immersion time for osmosis and increase in calcium salt in comparison to sugar conc. and temp. of osmotic solution. The reason could be attributed to the impregnation of calcium salt in comparison to sugar due to it lower molecular weight than sugar and lowering the a_w of the sample (Table 3). Restriction of gain in sugar by the addition of calcium salt have been observed by various researchers; for guavas which were osmotically dehydrated in maltose solutions but not for papaya in sucrose solutions by Pereira *et al*., 2006 and the reason reported for the same was the specific tissue structure of each fruit. In another research on osmotic dehydration of apples, solute gain reduced by the addition of 0.6% calcium lactate to solution, and reason reported for the same was a reduction in cell wall porosity (Mavroudis*et al*., 2012). Silva *et al*., 2014, conducted experiments for pineapple by OD and also attributed the reason for limited transfer of sucrose into pineapple tissue to the present pectin and enzymes in the fruit. Pectin methyl esterase, an important enzyme present in the pineapple, hydrolyses the pectin methyl esters (Silva *et al*., 2011a and Silva *et al*., 2011b) and generates carboxyl groups that interact with calcium (Guillemin *et al*., 2008) and promotes the cross-linking of pectin polymers which repairs the cell walls (Anino*et al*., 2006). The cuts and injuries to the tissue releases enzymes which reacts with the calcium forming the calcium pectate around the cut surfaces and would act as a partial barrier to the diffusion of larger molecules such as sucrose in to the tissue (Barrera *et al*. 2009; Silva *et al*., 2014). This shows that loss of water also influences water activity. Water activity is a critical indicator for shelf life of the pear because of microbial stability (Guiamba*et al*., 2016). Three dimensional response surfaces for a_w were also plotted. Figure 3a shows the surface plot with the most significant ($p<0.05$) interaction for the effect of process variables of temp. and time at centre values of sugar conc. and calcium salt conc. on a_w of pear slices. as the temp. and time increases, $a_w$decreases which is the desirable effect for the stability of the final product.The multiple regression equation at 5% level of significance and neglecting the non-significant terms provided a good fit for describing the relationship between the process variables and response. The uncoded process variable form of the developed model, is as follows:

$$aw = -3.79 + 0.15 * Temp. + 0.02 * Sugar\ Conc. - 0.06 * Calcium\ salt + 0.01 * Time + 0.0002 * Temp. * Sugar\ Conc. + 0.003 * Temp. * Calcium\ salt - 0.0001 * Temp. * Time - 0.002 * Sugar\ Conc. * Calcium\ salt - 0.00003 * Sugar\ Conc. * Time + 0.0012 * Calcium\ salt * Time - 0.002 * Temp.^2 - 0.0001 * Sugar\ Conc.^2 - 0.035 * Calcium\ salt^2 - 0.00001 * Time^2 \quad (7)$$

3.7. Influence of variables on overall acceptability score

The overall acceptability (OAA) score profile for different combinations of process variables varied from 5.76 to 8.54 (Fig. 3b) for different combinations of treatments. Among them, combination no. 12 resulted in the lowest OAA score corresponding to 60°C temp., 50°B sugar conc., 1% calcium salt and 150 min immersion time while combination no. 22 gave the highest score corresponding to 50 °C temp., 40°B sugar conc., 3% calcium lactate salt and 150 min immersion time (Table 2). The experimental variables temp., sugar conc., calcium salt and immersion time were found to have significant ($p<0.05$) effect on OAA score profile. The analysis of variance of the responses indicated that all linear terms as well as quadratic terms have significant effect ($p<0.05$) on OAA score. To understand the positive effects in ascending order of the variables on OAA score, the values of the regression coefficients of the fitted second-order polynomial representing the relationship between the responses and variables can be used for interpretation. The magnitude of coefficients indicates maximum positive effect of temp. (a_1=-0.26), calcium salt conc. (a_3=0.24), immersion time (a_4=-0.15), followed by sugar conc. (a_2=-0.06) (Table 3). These results indicate that OAA score was affected by calcium salt conc. and immersion time of the pear slices. Saxena *et al.*(2012) reported sensory attributes of jackfruits bulbs to be effected by calcium salt conc. and immersion time. Figure 3b shows the significant ($p<0.05$) interaction surface plot for the effect of pretreatment variables sugar conc. and temp. at centre values of immersion time and calcium salt conc. on OAA score of pear slices. The multiple regression equation at 5% level of significance and neglecting the non-significant terms provided a good fit for describing the relationship between the process variables and response. The uncoded process variable form of the developed model, is as follows:

$$OAA\ score = -38.58 + 1.49 * Temp. + 0.13 * Sugar\ Conc. - 0.60 * Calcium\ salt + 0.11 * Time + 0.002 * Temp. * Sugar\ Conc. + 0.03 * Temp. * Calcium\ salt - 0.001 * Temp. * Time - 0.02 * Sugar\ Conc. * Calcium\ salt - 0.0003 * Sugar\ Conc. * Time + 0.01 * Calcium\ salt *$$

$$\text{Time} - 0.02 * \text{Temp.}^2 - 0.001 * \text{Sugar Conc.}^2 - 0.32 * \text{Calcium salt}^2 - 0.0002 * \text{Time}^2 \quad (8)$$

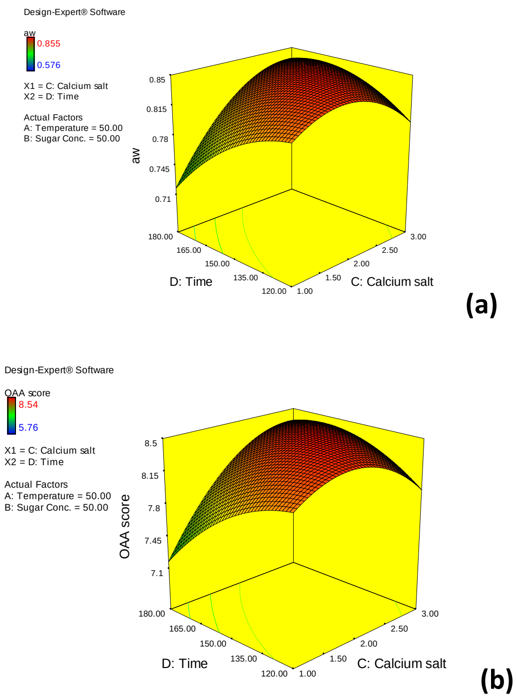


Figure 3. Response surface plot for the effect of process variables on water activity (a) and overall acceptability (b) of pear slices

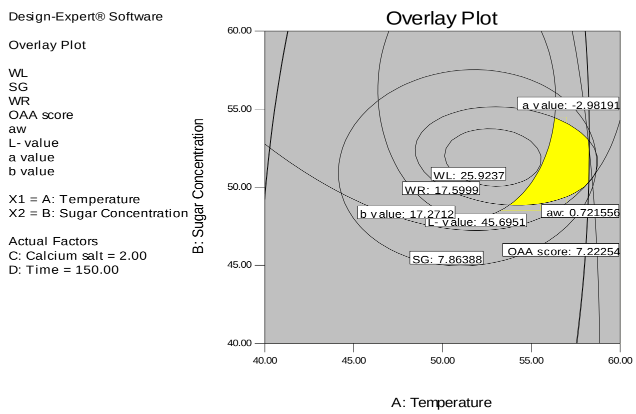
3.8. Optimization of osmotic dehydration pre-treatment

Maximization goal was kept for WL, WR and OAA score while minimization goal was kept for SG and a_w to obtain optimum conditions for OD for pear slices. The importance of variables were maximum for WL, WR, OAA score, a_w, *L*- value, *a*- value and *b*- value. Specified optimum dehydration conditions, were determined by fitting second order polynomial models obtained in the study, for each response. The regression models were only valid in the selected experimental domain. Therefore, the criteria for optimization were selected based on different parameters which included economical and product quality related attributes (Eren and Kaymak-Ertekin, 2007, Noshad*et al*, 2012).The optimum covering criteria deduced from the study was 59.57°C for temp., 53.73°B for sugar conc., 2.27% calcium salt conc. and 151.9 min immersion time by applying the desirability function method.

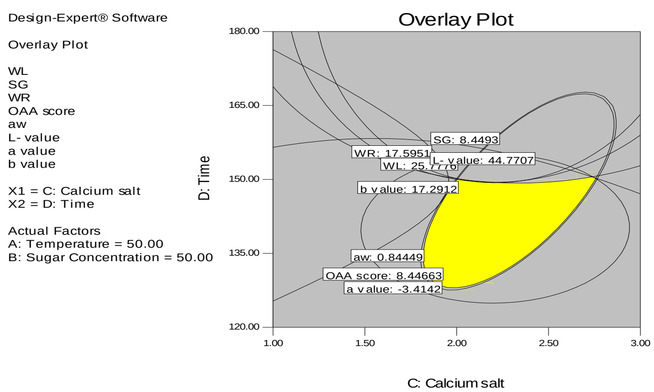
The graphical method of optimization was also adopted to deduce the workable range for optimized conditions. The effects whether main or interactive of the independent treatments on different responses were illustrated by the contour plots. In the set of four treatments, the contours were plotted between the two treatments while the other two were kept constant. The overlay plot for temp. and sugar conc. with calcium salt conc. and immersion time as its central value is described in Fig 4a and the overlay plot for calcium salt conc. and immersion time with temp. and sugar conc. as its central value is described in Fig 4b. The optimal zone for different combinations of treatments is represented by the shaded area highlighted within the overlay plots. The optimal ranges drawn from the overlay plat were found to be 54.3-59.1°C temp. and 48.3-54.5°B sugar conc. (Fig 4a). In the same way, the overlay plot between time and calcium salt conc., the optimal ranges from the overlay plot were found to be 127-150 min immersion time and 1.9-2.85 percent of calcium salt conc. (Fig 4b). The graphically and numerically obtained optimized conditions were in proximity. The optimization process was useful in ensuring the optimal OAA of the osmo-processed product.

3.9. Verification of the final models

The models obtained with the derived optimum conditions were further investigated under predictive optimum conditions. It was found that the predicted models were comparable to the experimental model shown in Table 4. This indicates that the second order polynomial model could be used to predict quality of osmotically dehydrated pear at different levels of temp., sugar conc., calcium salt and immersion time chosen as variable factors for the osmotic dehydration of pear.



(a)



(b)

Figure 4. Superimposed contours plot for the response variables of pear slices at varying temp. and sugar conc. (a), and at varying time and calcium salt conc. (b)

Table 4. Optimized independent variables and predicted and experimental

Variables	Optimum condition	
Temperature (^oC)	59.57	
Sugar (^oB)	53.73	
Calcium lactate (%)	2.27	
Treatment time (min)	151.9	
Response	Predicted value	Actual value
WL (%)	25.56	25.33±0.05
SG (%)	7.85	7.79±0.15
WR (%)	17.72	17.30±0.03
L- value	49.58	48.95±0.05
a value	-2.82	-2.79±0.05
b value	17.29	17.58±0.05
Water activity aw	0.693	0.71±0.05
OAA score	6.92	6.87±0.25

4. Conclusions

Osmotic dehydration was optimized by evaluating the effect of the sugar conc., temp., Ca salt conc., immersion time in an aqueous ternary solution on the water loss, solid gain, weight reduction, OAA score, water activity, and hunter color values such as *L*- value, *a*-value and *b*- value established using the response surface methodology (RSM). The process kinetics under the optimized conditions was also modeled. Sugar conc. was the most significant factor among all to affect the water loss and solid gain in the sample. Sugar gain and calcium impregnation were significantly affected by sugar and calcium lactate conc. Optimized conditions for the osmotic dehydration were process temperature 59.57°C, Sugar concentration 53.73°B, calcium salt 2.27 % and osmotic treatment time of 151.9 min were indicated during the course of study using the RSM. Although the optimal conditions for the osmotic dehydration process of pear was defined at an immersion time of 151.9 min using the RSM, the osmotic dehydration kinetics showed that process time of about 151.9 min will favor WL greater than 25.56% while keeping SG at approximately 7.85 % (Fig 6). Upper limit of the contact time according to experimental design was 180 min., however, it was observed that longer contact time up to 240 min were used to study the OD kinetics. Thus, OD was an effective pretreatment to obtain shelf stable pear slices with better overall characteristics. More detailed studies about the influence of calcium salt on the tissue micro-structure would be necessary to explain the changes in the mass transport studied in the present work.

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