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Optimization of Osmotic Dehydration of *Aloe vera* using Response Surface Methodology

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Abstract Response surface methodology (RSM) was used to determine the optimum processing conditions that yield maximum water loss and mass reduction and minimum solid gain during osmotic dehydration process of *Aloe vera*. Sugar concentration (30, 40, 50, 60 and 70° Brix), process temperature (30, 40, 50, 60 and 70°C) and syrup to fruit ratio (3:1, 4:1, 5:1, 6:1 and 7:1) were the factors investigated with respect to water loss (WL), mass reduction (MR) and solid gain (SG). Experiments were designed according to central composite rotatable design with these three factors each at five different levels. The quadratic models were fitted to all responses because of highest R^2 value. Models developed for all responses were significant without significant lack of fit. Analysis of variance (ANOVA) was performed to check the adequacy and accuracy of fitted models. Applying desirability function method, optimum operating condition was found to be sugar concentration 68.25 °Brix, process temperature 60.48°C and STFR (syrup to fruit ratio) of 6.65. It was predicted the best condition by RSM design for osmotic dehydration of *Aloe vera*. At this optimum point water loss, mass reduction and solid gain were found to be 93.43, 77.66 and 13.18% respectively.

Keywords Osmotic dehydration, Sugar concentration, Water loss, Response surface methodology, *Aloe vera*.

Introduction

Aloe vera is a traditional medicinal plant used in food, pharmaceutical and cosmetic industries. *Aloe vera* grows in arid climates and is widely distributed in Africa, India, and other arid areas. *Aloe vera* is a perennial plant of Liliaceae family with turgid green leaves joined at the stem in a rosette pattern [1]. Accordingly to a report on *Aloe vera* cultivation, published by IASC (International Aloe Science Council).

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Table 1. Coded and actual values of variables in RSM experiment. X_1=Sugar Concentration (°Brix), X_2=Process Temperature (°C) and X_3=Syrup to Fruit Ratio.

Level	Coded Factor-1	 Factor-2	 Factor-3	Actual Factor-1 X_1	 Factor-2 X_2	 Factor X_3
-1.682	-1.682	-1.682	-1.682	30	30	3:1
-1.0	-1	-1	-1	40	40	4:1
0	0	0	0	50	50	5:1
+1.0	+1	+1	+1	60	60	6:1
+1.682	+1.682	+1.682	+1.682	70	70	7:1

Table 2. Experimental design for the optimization experiments as computed using RSM. X_1=Sugar Concentration (°Brix), X_2=Process Temperature (°C) and X_3=Syrup to Fruit Ratio.

Std. No.	Coded values Factor 1 X_1	 Factor 2 X_2	 Factor 3 X_3	Actual values Factor 1 X_1	 Factor 2 X_2	 Factor 3 X_3
1	-1	-1	-1	40	40	4:1
2	1	-1	-1	60	40	4:1
3	-1	1	-1	40	60	4:1
4	1	1	-1	60	60	4:1
5	-1	-1	1	40	40	6:1
6	1	-1	1	60	40	6:1
7	-1	1	1	40	60	6:1
8	1	1	1	60	60	6:1
9	-1.682	0	0	30	50	5:1
10	1.682	0	0	70	50	5:1
11	0	-1.682	0	50	30	5:1
12	0	1.682	0	50	70	5:1
13	0	0	-1.682	50	50	3:1
14	0	0	1.682	50	50	7:1
15	0	0	0	50	50	5:1
16	0	0	0	50	50	5:1
17	0	0	0	50	50	5:1
18	0	0	0	50	50	5:1
19	0	0	0	50	50	5:1
20	0	0	0	50	50	5:1

there are close to 23,600 hectares of *Aloe vera* being cultivated worldwide. In India total production of *Aloe vera* is estimated to be about 1,00,000 tonnes [2]. Reports credit that *Aloe vera* has anti-tumor and anti-diabetic properties [3]. In addition it has efficacy in healing wounds and burns and also help in treatment of gastric ulcers [4]. Unfortunately, because of improper processing procedure, many of these so-called aloe products contain very little or virtually no active ingredient [5]. The simplest and most economic method for dehydration of foods is hot air-drying in conventional tray, cabinet or vacuum dryers. The problems associated with products obtained by air drying are woody texture, slow or incomplete re-hydration, considerable shrinkage caused by collapsing of cells due to substantial water loss. It also brings about undesirable changes in color, texture, flavor and loss in nutritive value [6]. Osmotic dehydration is the process of water removal, with low energy consumption at low temperature. This provides minimum thermal degradation of the nutrients due to low temperature water removal process. Some of the advantages of osmotic process are (1) minimized heat damage, (2) least discolouration of product by enzymatic browning, (3) increased retention of volatile matter, flavour and aroms, (4) improved textural quality and (5) lower energy consumption than the air drying. However, osmotic process does not give a high quality product of sufficiently low moisture content to be considered as shelf stable and therefore, osmosed product needs to be further dried through air, vacuum or freeze drying [7]. The effects of osmotic pre-treatment on drying rates have been investigated by several authors [8] and vary according to the raw material used and the drying conditions. During osmotic dehydration, water removal from the product is accompanied by the simultaneous counter diffusion of solutes from the osmotic solution into the tissue. Among different solutes, sucrose is considered one of the best osmotic solute, especially when the osmotic dehydration (OD) is employed before drying. The presence of this sugar on the surface of the dehydrated sample is an obstacle for the contact with oxygen thus reducing the oxidative reactions but also posing an additional resistance to mass exchange and lowering the rates of complementary (vacuum, convection and freeze) dehydration [8]. In such situations, it becomes more important to determine the optimum processing conditions that yield maximum water loss and mass reduction and minimum solid gain during osmotic dehydration process of *Aloe vera* [9, 10]. In this study, it was therefore aimed to investigate the effect of temperature, processing time, sucrose concentration on the mass transfer phenomena

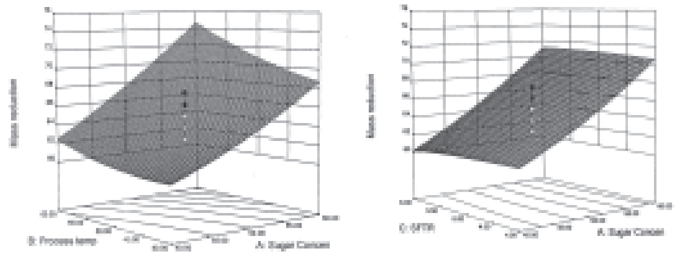

Fig. 1. Response surface plot for mass reduction (%) during osmotic dehydration of *Aloe vera.*

during osmotic dehydration of *Aloe vera* in sucrose solutions, to model water loss, mass reduction and solid gain as a function of the process variables and to find the optimum operating conditions that maximize water loss and mass reduction and minimize the solid gain.

Materials and Methods

Raw meterial

Fresh whole *Aloe vera* leaves, of between 30 and 50 cm of length, were collected from the medicinal plants section of Chaudhary Charan Singh Haryana Agricultural University, Hisar (Haryana), India. The whole *Aloe vera* leaves were thoroughly washed with distilled water to remove adhering impurities. The spikes, placed along their margins, were removed before slicing the leaves. The thick epidermis (or skin) is carefully separated from the parenchyma using a stainless steel cutter. The fillets were cut into 1.5 × 1.5 × 1.5 cm cubes with the help of sharp stainless steel cutter.

Analytical determinations

The moisture content of the fresh as well as osmotically dehydrated *Aloe vera* samples was determined by using AOAC [11] method. All moisture content values were expressed on a wet basis. Total soluble solids of the prepared syrup solutions were measured by hand refractometer which depicts the reading directly in degree brix (°Brix) [12].

Osmotic dehydration (OD)

The osmotic solutions (OS) were prepared by mixing the sucrose with distilled water on a weight to weight basis. The sugar concentrations selected for study were 30, 40, 50, 60 and 70°Brix. The process temperature was kept at 30, 40, 50, 60 and 70°C along with syrup to fruit ratio (STFR) of 3:1, 4:1, 5:1, 6:1 and 7:1. *Aloe vera* was dehydrated in osmotic solution for 4 hour duration. The prepared samples (Aloe vera cubes) were weighed for every experiment and immersed in the sugar syrup (30, 40, 50, 60 and 70 °Brix) contained in a glass beaker. The beakers were placed inside the constant temperature water bath and stirred regularly. One beaker was removed from the water bath at designated time (30 min) and the *Aloe vera* samples were immediately rinsed with water and placed on absorbent paper to remove the surface moisture. The samples were weighed and their moisture contents were determined.

Mass transport in osmosis

The water loss WL), mass reduction (MR) and solid gain (SG) were calculated by using the following mass balanced equations [13]. The water loss (WL) is defined as the net weight loss of the fruit on initial weight basis and is estimated as follows.

$$WL = \frac{W_i X_i - W_\theta X_\theta}{W_i} \times 100$$

The solid gain is the net uptake of solids by the

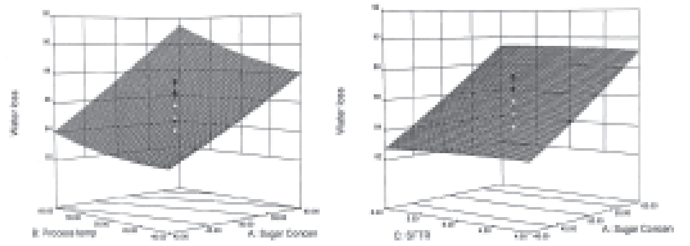

Fig. 2. Response surface plot for water loss (%) during osmotic dehydration of *Aloe vera*.

samples on initial weight basis. It is computed using following expression:

$$SG = \frac{\boldsymbol{W}_{\theta}\,(1 - X_{\theta}) - W_i\ \ (1 - X_i)}{W_i} \times 100$$

The mass reduction (MR) can be defined as the net weight loss of the fruit on initial weight basis

$$MR = \frac{W_i - W_{\theta}}{W_i} = WL - SG$$

Where, WL=Water loss (g per 100 g mass of sample), SG = Solid gain (g per mass of sample), MR = Mass reduction (g per 100 g mass of sample), W_{θ} = Mas of samples after time θ, g, W_i = Initial mass of samples, g, X_{θ} = Water content as a fraction of mass of samples at time θ,and X_i = Water content as a fraction of initial mass of samples.

Results and Discussion

Optimization

The experimental plant consists of 3 process independent variables (X_1, X_2 and X_3) (sugar concentration (°Brix), process temperature (°C) and syrup to fruit ratio at 5 different levels have been drawn as shown in Table 1. The response surface methodology was used to estimate the main effects of process variables on the response functions (Y_1, Y_2 and Y_3) i.e. water loss (%), mass reduction (%) and solid gain (%). A Central Composite Rotatable Design (CCRD) was used for designing the experimental data [14]. The CCRD design constituted of 20 experiments which were carried out in randomized order. The results are presented in Table 2.

The data generated were analyzed using Design Expert Software (8.07.1 version) and a polynomial

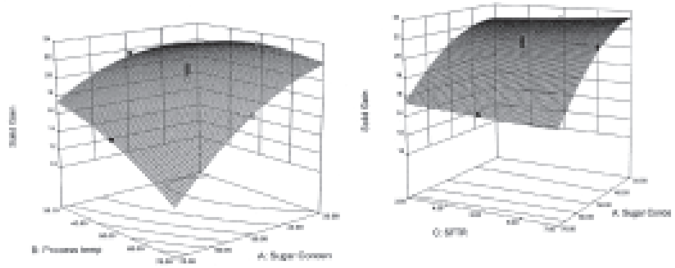

Fig. 3. Response surface plot for solid gain (%) during osmotic dehydration of *Aloe vera*.

Table 3. Experimental design of process variables and values of experimental data for osmotic dehydration. *The total of 20 treatments were carried out in a random order, **Figures are average of three replications.

	Factors			Responses**		
Standard	Sugar concentration (°Brix)	Process temperature (°C)	STFR	Mass reduction (%)	Water loss (%)	Solid Gain (%)
1	40	40	4:1	60.00	82.22	22.22
2	60	40	4:1	68.75	88.20	19.45
3	40	60	4:1	62.50	84.09	21.59
4	60	60	4:1	73.75	90.93	17.18
5	40	40	6:1	61.25	83.66	22.41
6	60	40	6:1	70.00	88.93	18.93
7	40	60	6:1	61.25	83.17	21.92
8	60	60	6:1	75.00	91.53	16.53
9	30	50	5:1	57.50	80.24	22.74
10	70	50	5:1	77.50	92.64	15.14
11	50	30	5:1	62.50	84.09	21.59
12	50	70	5:1	72.50	90.29	17.79
13	50	50	3:1	65.00	85.83	20.83
14	50	50	7:1	63.75	84.97	21.22
15	50	50	5:1	66.25	86.65	20.40
16	50	50	5:1	63.75	84.97	21.22
17	50	50	5:1	62.50	84.09	21.59
18	50	50	5:1	66.25	86.65	20.40
19	50	50	5:1	65.00	85.83	20.83
20	50	50	5:1	67.50	87.44	19.94

Table 4. Regression coefficients and ANOVA of fitted quadratic model for the three responses. *Significant at $p \leq 0.05$, **Significant at $p \leq 0.01$, ***Significant at $p \leq 0.001$ ns non significant.

	Estimated *p*-values		
Parameters	Mass Reduction (%)	Water Loss (%)	Solid Grain (%)
Model	<0.0001***	<0.0001***	<0.0001***
A-Sugar Concentration	<0.0001***	<0.0001***	<0.0001***
B-Process temperature	0.0004**	0.0013**	<0.0001***
C-STFR	1	0.9738	0.9317
AB	0.1173	0.2274	0.02350*
AC	0.5804	0.797	0.2305
BC	0.5804	0.4372	0.9979
A^2	0.0759	0.4838	0.0007***
B^2	0.0759	0.1421	0.0182*
C^2	0.5928	0.6453	0.4907
p-value for Lack of fit	0.8224^{ns}	0.7407^{ns}	0.9376^{ns}
R^2	0.96	0.9428	0.97

equation was obtained for each response. The second-order polynomial equation was fitted to the experimental data of each dependent variable as given below:

$$Y_k = B_{ko} + \sum_{i=1}^{n} B_{ki} x_i + \sum_{i=1}^{n} B_{kii} x_i^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} B_{kij} x_i x_j + e_k \qquad (2)$$

Where Y_k is the response variable (Y_1 = mass reduction; Y_2 = water loss; Y_3 = solid gain), x is the factor, x_i are regression coefficient for linear effect, x_i^2 is the regression coefficient for quadratic effect, $x_i x_j$ is the regression coefficient for interaction effect and B_k is the coefficient for each term calculated by multiple regression analysis lack of fit was calculated [15].

Model considered adequate when *F* (adequacy ratio) was more than Table *F* value and the coefficient of determination (R^2) that reflects the proportion of variability in data explained or accounted by the model and a larger R^2 values suggest a better fit of model data. The results indicate the adequacy of quadratic model for all responses because of high R^2. The relative effect of each process parameter on individual response was compared from the $p \leq 0.01$ indicate model terms are significant [9]. 3D plots and contour graphs were developed using 2nd order polynomial model. The response surface plots were generated for different interaction of any two independent variables, while holding the value of third variable as constant. Such three-dimentional surfaces could give accurate geometrical representation and provide useful information about the behavior of the system within the experimental design. The center runs provide a means for estimating the experimental error and a measure of lack of fit. The axial points were added to the factorial design to provide for estimation of curvature of model. The effect of different process variables on the individual responses as per experiment designed are presented in Table 3. The significance of coefficient of fitted quadratic model was evaluated by using *F-test and* p-value. The results of regression analysis for mass reduction, water loss and solid gain are given in Table 4.

Mass reduction

Regression model fitted to experimental result of mass reduction showed the *p*-value for lack of fit as 0.8224 implies the lack of fit was non-significant. The value of coefficient of determination (R^2) was found to be 0.96. Regression analyses showed that mass reduction was significantly ($p<0.05$) affected by the linear effect of sugar concentration (A) and process temperature (B) whereas it was non significantly ($p>0.05$) affected by STFR. The quadratic terms of all process parameters had a negative effect on mass reduction. It implies that increased mass reduction with increase of sugar concentration and process temperature. Figure 1 indicates an increase in mass reduction with increase in sugar concentration and process temperature. Mass reduction in osmosis increased with increase in syrup temperature and concentration of solution while STFR had the negligible effect on mass reduction. At initial stage increasing temperature and sugar concentration of solution raised water loss more than solid gain which caused an increase in mass reduction. However later increase in solid gain blocked the surface layers of the product, which further reduced the concentration gradient between the product and osmotic solution, posed as additional resistance to mass exchange and lowered the rates of water loss and consequently mass reduction. Similar results have been reported earlier [10, 16].

Water loss

Regression model fitted to experimental result of water loss showed the *p*-value for lack of fit as 0.7407 which implies the lack of fit was non-significant. The value of coefficient of determination (R^2) was found to be 0.94. Regression analyses showed that water loss was significantly ($p<0.05$) affected by the linear effect of sugar concentration (A) and process temperature (B) whereas it was non significantly ($p>0.05$) affected by STFR. The quadratic terms of all process parameters had a negative effect on mass reduction. It implies that increased water loss with increase of sugar concentration and process temperature. Figure 2 indicates an increase in water loss with increase in sugar concentration and process temperature. Water loss in osmosis increased with increase in sugar concentration, this was because an increase in the concentration of sugar solution resulted in osmotic gradient, which increased the driving force for water removal between solution and fruit, and thereby giving higher mass transfer rates. Similar results have been reported earlier [14]. Also it was observed that water loss increased with increase in syrup temperature. This might be due to change in semi-permeability of cell membrane of the gel, allowing more water to diffuse out in a shorter period. Earlier [15, 16, 17] similar results were reported regarding effect of temperature. Syrup to fruit ratio had no significant effect on water loss. It might be due to the short durations and high temperatures employed during osmotic dehydration.

Solid gain

Regression model fitted to experimental result of solid gain showed the p-value for lack of fit was 0.9376 implies the lack of fit was non-significant. The value of coefficient of determination (R^2) was found to be 0.97. Regression analyses showed that solid gain was significantly ($p < 0.05$) affected by the linear effect of sugar concentration (A) and process temperature (B) and also it was affected by interaction of process variables viz. sugar concentration, process temperature (AB) and quadratic term of sugar concentration (A^2) and process temperature (B^2). Whereas it was non significantly ($p>0.05$) affected by STFR (Fig. 3). The values of solute gain were much lower than the water loss for all the process parameters during osmotic dehydration, because sucrose having larger ionoc radius could not diffuse easily through the cell membrane and thus the approach to osmotic equilibrium was achieved primarily by flow of water from cell. Similar results were reported earlier [14]. Solid gain values showed variability among the treatments. Solid gain in osmosis decreased with increase in sugar concentration and syrup temperature, it might be due to high viscosity of more concentrated sugar solution, which imprated resistance to the solute penetration at solution and fruit interface.

Conclusion

Response surface methodology was used to determine the optimum operating conditions that yield maximum water loss, mass reduction and minimum solid gain in osmotic dehydration of *Aloe vera*. Analysis of variance has showed that the effects of process variables like sugar concentration and process temperature were statistically significant, while the syrup to fruit ratio has negligible effect. Second order polynomial models were obtained for predicting water loss, mass reduction and solid gain. The optimal conditions predicted as the best condition by RSM design for osmotic dehydration of *Aloe vera* for maximum water loss, mass reduction and minimum solid gain were found to be sugar concentration 68.25 °Brix, process temperature 60.48°C and syrup to fruit ratio (STFR) of 6.65. At this optimum point water loss, mass reduction and solid gain were found to be 93.43, 77.66 and 13.18% respectively.

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