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ORIGINAL ARTICLE

Using RSM for Optimization of Osmotic Solution Formulations based on Performance Coefficient in the Osmotic Dehydration of Quince

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ABSTRACT

Osmotic dehydration is used for partial removal of water from materials such as fruits and vegetables by immersing in aqueous solutions of high osmotic pressure such as sugar and salts. Optimization has been used in food engineering for the efficient operation of processing systems and unit processes yielding a highly acceptable product. Response surface methodology (RSM) is a set of statistical techniques for building models, designing experiments, searching the optimum conditions and evaluating the effects of factors. RSM was used for optimization of osmotic solutions (containing fructose, calcium chloride and citric acid) based on maximum performance ratio (WL/SG) in the osmotic dehydration of quince. The food tosolution ratio was kept constant at 1:10 throughout the osmotic dehydration process. The results showedthe effects of fructose and calcium cloride on WL/SG are quadratic and linear, respectively. The WL/SG was increased by decreasing fructose concentration in quadratic manner and by increasing citric acid in linear manner. The quadratic term of citric acid concentration has positive and significant effect.

Keywords: *quince, edible coating, response surface methodology,osmotic dehydration.*

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INTRODUCTION

The simplest and economic method for dehydration of foods is hot air-drying in conventional tray, cabinet or vaccum dryers but these dehydrated products have fewer acceptances since the products quality is considerably reduced. The problems associated with products obtained by air-drying are woody texture, slow or substantial amount of water loss. It also brings about undesirable changes in color, texture, flavor and loss in nutritive value [15]. Osmotic dehydration is used for partial removal of water from materials such as fruits and vegetables by immersing in aqueous solutions of high osmotic pressure such as sugar and salts. The most commonly used osmotic agents are sucrose for fruits and sodium chloride for vegetables. Osmotic dehydration can be conducted at low temperature and is less energy intensive than convective drying or freezing. The main advantages of osmotic dehydration include better colour, texture and flavor retention along with minimum heat damage. Osmotic dehydration is affected by several factors such as osmotic agent, solute concentration, temperature, time, size, and shape and tissue compactness of the material, agitation and solution/sample ratio[14].

To control the amount of solute uptake into the food, we can use edible coating before osmotic dehydration, which acts like a barrier that decreases solute uptake without having neg-ative effect on the rate of water removal. Edible coatings are made of one to four major materials including: lipids, polysaccharides, resins and proteins and also a mixture of these materials forms the new composite edible coatings, that can limit lipid, oxygen, water vapor and flavormigration between food and the surroundings. Coating solutions such as low-methoxylpectinate, high- methoxylpectinate, methylecellulose, carboxyl methylecellulose, maltodexterin, potato starch, corn starch, sodium alginate, chitosan and ethylecellulose can be used on vegetables and fruits to prevent solid gain and improve organoleptic properties, shelf-life and nutritive properties during osmotic dehydration [6].

Optimization has been used in food engineering for the efficient operation of processing systems and unit processes yielding a highly acceptable product. [13]Response surface methodology (RSM) is a set of statistical techniques for building models, designing experiments, searching the optimum conditions and evaluating the effects of factors [1].In multifactor experiments, RSM can be used to comprehensively examine variousparameters with minimum experimental times and determine the most relevant factors and their influence ranges aswell as interactions among the factors. This experimental strategy has been widely used in the development of food processes [4, 3]. Quince fruit (Cydoniaoblonga), a member of the Rosaceae family, is known for its characteristic and pleasant odor and distinctive taste. However, like other fruits, they are perishable; therefore drying is fairly advantageous, reducing water activity of the material, thus diminishing the microbiological activity to a level preventing deterioration. Even though the drying is one of the most common methods used to improve [10]; Ingredients of quince 83.8% water and 15.3% carbohydrates (wet basis) are the main constituents of quince. Minor ingredients of quince are proteins (0.4%, wet basis) and fats (0.1%, wet basis). It is presumed to be a good source of fiber, potassium, and vitamin C. The mean of the last 10 years' (1998–2008) world production of quince is estimated to be 510,000 t [13].

In this study, the optimization of osmotic solutions (containing fructose, calcium chloride and citric acid) based on maximum performance coefficient in the osmotic dehydration of quince were investigated using response surface methodology (RSM).

MATERIALS AND METHODS

Materials

Fresh quinces (varieties of Sharafkhane) were purchased at local market in Tabriz, Iran. carboxymethyl cellulose (Food chem, China, Viscosity 2280, Degree of substitution0.82), low methoxylpectinate (LMP, degree of esterification: 31.5%, Degussa, Pullach,Germany) and ascorbic acid (Northest pharmaceutical, China) were used as polysaccharide-based edible coatings. Glycerol (Sigma-Aldrich, Germany) was applied for plasticizer. Calcium chloride (Sigma-Aldrich, Germany) was added for gel forming and cross-linking. Fructose (Krueger, Germany), Calcium chloride (Sigma-Aldrich, Germany) and citric acid (Kaselcit, China, $C_6H_8O_7.H_2O$) were used as osmotic solution formulations.

Preparation of samples

quinces (varieties of Sharafkhane) were purchased from a local supermarket (produced in tabriz, Iran) and stored at 4 °C. A single batch of quinces was used in the experiments, which were restricted to a period of time. Because of the ripening of the quinces, it was always ensured that firm quinces were selected for dehydration experiments. For all experiments quinces were washed and sliced (40mm diameter, 2mm thickness) with two special cutting tools. Initially, in order to avoid undesirable enzymatic reactions and improve structural properties, quince slices were blanched in hot water (80 °C for 1min).

Coating treatment

The slices of quinces were immersed in optimized coating solutions resulted from our previous research works by RSM (1.49% carboxymethyl cellulose, 1.49% pectin and 0.58% ascorbic) (w/v) for 3 minutes. Then they were dried at 55-60°C for 5-10 minutes, in order to fix the coating on the samples. for prepration of coating solution, carboxymethyl cellulose, pectine and ascorbic acid powder was dissolved in distilled water by heating the mixtures using the stirring hot plate (70°C) until the solutions became clear and then glycerol as plasticizer was added to the solutions [17, 1].The overall volume for each formulation was 1000 ml and this includes amounts of CMC, pectine and ascorbic acid, 0.2%(w/v) glycerol and the rest was distilled water. For cross-linking of polymers a 1% (w/v) calcium chloride solution wasused [12].

Osmotic treatment

Optimization of osmotic solution formulation was investigated by using response surface methodology (RSM). fructose, calcium chloride and citric acid powder was dissolved in distilled water by heating the mixtures using the stirring hot plate (25°C). The overall volume for each formulation was 1000 ml and this includes different amounts of fructose, calcium chloride and citric acid (Tables 1).The different concentrations of fructose, calcium chloride and citric acid based on the experimental design were shown in Tables 1. Osmotic treatment was carried out at 25°C (the temperature was monitored by the thermocouple and was set at 25 °C). The optimom immersion time in osmotic solution was determind about 180 minutes by previous test (kinetic of osmose). A sample to solution ratio of 1:10 (w/w) was used in order to avoid excessive dilution of the osmotic solution during processing [8].

Analytical methods

After immersion time, the dehydrated quince samples were recuperated on a strainer and washed with tap water for few seconds to remove the adhering osmotic solution and gently blotted with tissue paper. Recuperation of samples and draining of excess water were carried out in a maximum time of 3 min, in order to minimize exchanges between the samples and the ambient air. Water loss (WL), solids gain (SG) and performance ratio(pr) were calculated by the following equations [8]. The WL was the net loss of water from quince cylinders at time (θ) on an initial mass basis.

$WL\%=(W_iX_i-W_\theta X_\theta)/W_i$ (1)

The dry matter gain is related to solid gain (SG) and hence, the SG was the net gain in total solids by quince cylinderson the initial mass basis.

$SG\%=[W_\theta(1-X_\theta)-W_i(1-X_i)]/W_i$(2)PerformanceRatio(pr)=WL/SG (3)

where, W_θ=mass of quince cubes after time θ, g, W_i= initial mass of quince cylinders, g, X_θ=water content as a fraction of the weight at time 'θ', and X_i=water content as a fraction of initial weight of quince cylinders [16, 9].

Experimental design and statistical analysis

Response surface methodology (RSM CC0318, Central composite design with three variables at five levels (-1.682, -1, 0, +1, +1.682) was used to estimate the main effects of osmotic dehydration on performance ratio (WL/SG) in quinces cylinders. The center composite design (CCD) was used for optimization of osmotic solution formulations. The type of CCD was axial with 4 blocks and eighteen experimental runs. (Tables 1). For evaluation the repeatability of methods, the center point was repeated six times [11]. A rotatable central composite design was used with fructose concentration (X1, %w/v)(12.38, 21.92, 35.91, 49.91, 59.44), chloride calcium (X2, %w/v) (0.98, 1.8, 2.99, 4.19, 5) and citric acid concentration (X3, %w/v) (1, 1.51, 2.25, 2.99, 3.49) being the independent process variables.The linear, quadratic and interaction terms of independent variables in the response surface models were predicted by multiple regressions. For evaluation the relationship between the response and independent variables the generalized polynomial model was usedas below:

$$Y=\beta_0+\sum_{i=1}^{k}\beta_i X_i+\sum_{i=1}^{k}\beta_{ii}X_i^2+\sum_{\substack{i=1\\i<j}}^{k-1}\sum_{j=2}^{k}\beta_{ij}X_iX_j \qquad (4)$$

In this model, Y is a calculated response (i.e. WL/SG, %) X_i and X_j are factors (i.e., concentration of fructose, calcium cloride, citric acid β_i '$\beta_{ii}$and β_{ij} are linear, quadratic and interaction coefficients, respectively and β_0 is a constant Softwares of SAS 9.1 (England) and Statistica 9 (USA) were used for analysing data and drawing responce surface graphs.

Verification and optimization procedures

Numerical and graphical optimization procedures were applied to determine the optimum level of three independent variables (X_1 , X_2 and X_3) [1].

Table 1: Experimental design used for fructose, calcium chloride and citric acid based osmotic solution

	Coded variables			*Uncoded variables		
Treatment	X_1	X_2	X_3	Fructose	Calcium chloride	Citric acid
1	-1	-1	-1	21.9285	1.8	1.51
2	-1	-1	1	21.9285	1.8	2.99
3	-1	1	-1	21.9285	4.19	1.51
4	-1	1	1	21.9285	4.19	2.99
5	1	-1	-1	49.91	1.8	1.51
6	1	-1	1	49.91	1.8	2.99
7	1	1	-1	49.91	4.19	1.51
8	1	1	-1	49.91	4.19	2.99
9	-1.682	0	0	12/38	2.99	2.25
10	1.682	0	0	59/44	2.99	2.25
11	0	-1.682	0	35/91	0.98	2.25
12	0	1.682	0	35/91	5.00	2.25

13	0	0	-1.682	35/91	2.99	1/00
14	0	0	1.68	35/91	2.99	3/49
15	0	0	0	35/91	2.99	2.25
16	0	0	0	35/91	2.99	2.25
17	0	0	0	35/91	2.99	2.25
18	0	0	0	35/91	2.99	2.25

Uncodedvariables:Fructose ((w/v%)), calcium chloride ((w/v%)) and citric acid ((w/v)%)*

Table 2: Variables of central composite experimental design and coded levels

Type of variable	Unit	Variable mathematical symbol	Coded levels of variable				
			-1.682	**-1**	**0**	**+1**	**+1.682**
Fructose concentrations	**g/lit**	X_1	12.38	21.92	35/915	49.91	59/44
calcium chloride concentration	**g/lit**	X_2	0.98	1.8	2.995	4.19	5/00
Citric acid concentration	**g/lit**	X_3	1.00	1.51	2.25	2.99	3/49

RESULTS AND DISCUSSION

Response surface analysis for fructose, calcium cloride and citric acid concentration

The results of experimental data obtained by the response variables were shown in table 3.Response surface methodology has the ability to determine main, quadratic and interaction effects of two osmotic solution components on each studied response variable. RSM suggested response surface models to show the relationship between independent variables and experimental data.

Table 3: Responses for fructose, calcium cloride and citric acid- based osmotic solution

Runs	WL (gr/gr%)	SG (gr/gr%)	WL/SG (%)
1	26.06146 ± 7.89	1.061459066 ± 0.22	24.55249 ± 3.21
2	29.75001 ± 4.32	2.090436321 ± 1.08	14.23148 ± 1.57
3	32.88797 ± 5.66	2.118741199 ± 0.25	15.52241 ± 0.96
4	31.87894 ± 2.34	2.67102307 ± 1.14	11.93511 ± 1.06
5	32.7897 ± 2.26	4.982208836 ± 0.19	6.581357 ± 0.74
6	41.84364 ± 1.63	5.433387525 ± 0.72	7.701207 ± 0.60
7	44.61462 ± 1.03	5.528327207 ± 0.35	8.070185 ± 0.36
8	46.35717 ± 1.46	5.561153952 ± 0.49	8.335891 ± 0.89
9	22.66833 ± 2.16	0.903628239 ± 0.16	25.08591 ± 1.78
10	50.63876 ± 2.36	9.38875642 ± 0.96	5.393553 ± 0.35
11	33.36714 ± 4.6	3.955375254 ± 1.06	8.435897 ± 0.80
12	43.42486 ± 0.33	5.173497685 ± 2.22	8.393715 ± 2.28
13	39.67429 ± 2.67	6.545455251 ±0.04	6.061349 ± 0.36
14	49.2144 ± 2.24	7.737126031 ± 2.25	6.360811 ± 2.66
15	49.10342 ± 8.03	5.000851727 ± 0.11	9.819011 ± 1.42
16	44.4347 ± 0.19	5.304267509 ± 0.38	8.377161 ± 0.62
17	39.67825 ± 0.90	5.635694507 ± 0.98	7.040525 ±0.85
18	44.84141 ±2.54	5.247504218 ± 0.17	8.545284 ±0.73

Effect of CMC, pectin and ascorbic acid on water performance ratio (WL/SG)

Table 3 shows performance ratio (WL/SG) of coated quinces varied from 5.39 ± 0.35 to 25.08 ± 1.78%. High correlation coefficients (i.e. R^2) was obtained for WL/SG indicating good fit of experimental data to Eq. (5) (Table 4). That lack of fit was not significant for WL/SG at P = 5% level. Obtained Summarized model to predict the effects of fructose, calcium chloride and citric acid on WL/SG, after excluding non significant factors, is as follows:

$$Y = 75.1 - 2.11X_1 + 0.014X_1^2 + 0.1X_1X_2 + 0.18X_1X_3 \quad (5)$$

The analysis of variance for final reduced models (Table 4) showed that WL/SG was mainly affected linearly and quadraticiy by fructose concentration at 99% level.

Table 4: Regression equation coefficients for water loss (WL/SG) during osmotic dehydration of quince slices

Souece	Regression Coeficience	(df)	(SS)	(MS)	F	p
X_1	- 2.115743	1	345.3032	345.3032	61.81511	0.0001**
X_2	- 7.863281	1	6.297583	6.297583	1.127374	0.319335
X_3	- 7.468055	1	10.57778	10.57778	1.893602	0.206085
X_1^2	0.014542	1	102.7581	102.7581	18.39544	0.002655**
X_1X_2	0.100493	1	22.61269	22.61269	4.048055	0.079036
X_1X_3	0.183292	1	29.23778	29.23778	5.234058	0.051441
X_3^2	0.306182	1	2.418193	2.418193	0.432897	0.529045
X_2X_3	0.825525	1	4.321144	4.321144	0.773558	0.404762
X_3^2	- 0.616009	1	1.478633	1.478633	0.2647	0.620817
Model	-	9	532.7676	59.1964	10.59716	0.001443**
Lineae	-	3	362.1779	120.726	21.61199	0.000342**
Quadratic	-	3	114.4181	38.13937	6.827593	0.01348*
Cross Product	-	3	56.17162	18.72387	3.35189	0.076088
Error	-	8	44.68851	5.586064		
Lack of fit	-	5	40.8134	8.16268	6.319309	0.36
Pure Error	-	3	3.875113	1.291704	-	-
Total	-	17	577.4561	-	-	-
R^2	92.26%	-	-	-	-	-
R^2_{adj}	83.55%	-	-	-	-	-
R^2_{pred}	78.76%	-	-	-	-	--
CV	22.33879	-	-	-	-	-

*, **: significant at P < 5% and P < 1%, respectively.

The effect of changing fructose and calcium chloride concentration on the percent performance ratio (WL/SG) of coated quinces is given in Fig1. The WL/SG is decreasing with fructose and calcium chloride concentrations (Fig 1). The effects of fructose and calcium chloride on WL/SG are quadratic and linear, respectively.

[8] Reported that the performance ratio depended on the coating material, the concentration and type of osmotic agent. High performance ratio was obtained when the osmotic agent was sucrose and the coating material was low methoxylpectinate (LMP) or mixtures of LMP and other polymers such as methyl cellulose or pure corn starch. Furthermore, the highest performance ratio was obtained when the osmotic agent was glycerol and the coating material was ethyl cellulose.[6] showed the changes in water loss/solid gain of apples depend on the chemical potential or mass transfer driving force of water and solute between sample and osmotic solution. They concluded that the molecular structures of coating materials (lowmethoxylpectinate (LMP), carboxyl-methyl cellulose (CMC), corn starch) also influence the rate of water loss/solid gain ratio. The effects of coating with CMC, corn starch and LMP on the water

loss/ solid gain ratio are different, because the structures of these three edible coatings are also different and permeability of water and solute in these coatings are different. Coating of a sample with CMC and LMP can cause high water loss/solid gain ratio than starch coating, regardless of the concentration of the osmotic solution. This is for acting of CMC and LMP coatings as a good barrier that can decrease the solid gain and somewhat reduce water loss of the samples. Starch coated samples can decrease the level of water removal less than two other coated samples (CMC and LMP). This might be due to the starch coating solution produced low viscosity than CMC and LMP solution, thus it cannot produce good adhering layer to the surface of the samples and cannot improve barrier properties against the water and solid transfer.

To visualize the combined effect of the two factors on the response, the response surface and contour plots were generated for each of the models in the function of two independent variables, while keeping the remaining independent variable at the central value (Figure 1) [2].

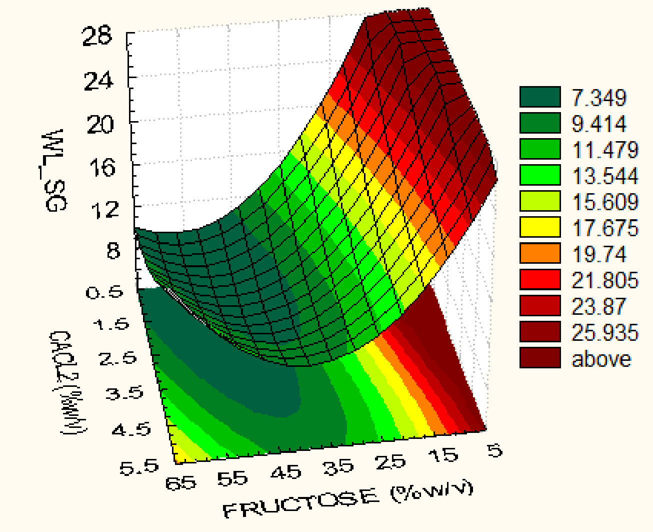

Fig. 1: Profile of response surface and contour plots for performance ratio (WL/SG) during osmotic dehydration of coated quince slices as function of) fructose and calcium chloride concentration (w/v%)

Figure 2 depicts the interactive effect of the fructose and citric acid concentration on WL/SG. The WL/SG was increased by decreasing fructose concentration in quadratic manner and by increasing citric acid in linear manner. The coated quince had maximum WL/SG at minimum concentrations of fructose and at maximum concentrations of citric acid. The results suggested that the WL/SG of the samples increased quadraticly with decreasing fructose concentration.

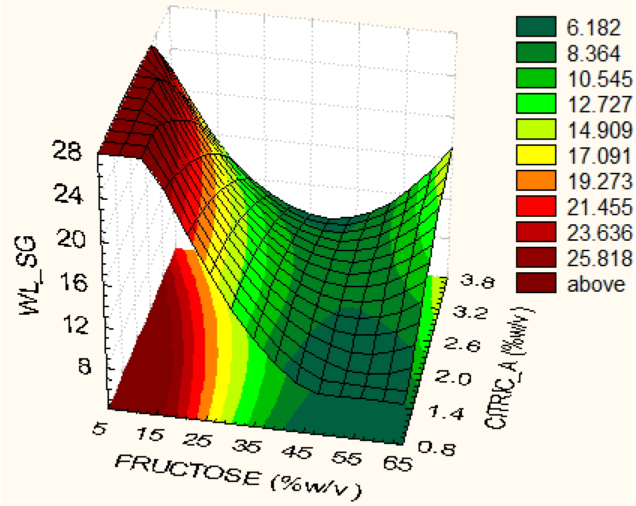

Fig. 2: Profile of response surface and contour plots for performance ratio (WL/SG) during osmotic dehydration of coated quince slices as function of fructose and citric acid concentration (w/v%)

The effects of calcium chloride and citric acid on WL/SG are shown in figure 3. The quadratic term of citric acid concentration has positive and significant effect, whereas the calcium chloride concentration has no significant effect on WL/SG.

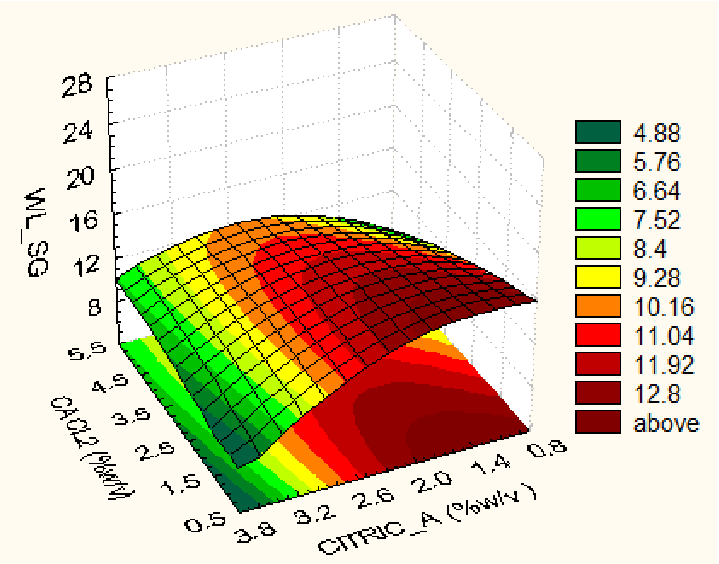

Fig. 3: Profile of response surface and contour plots for performance ratio (WL/SG) during osmotic dehydration of coated quince slices as function of calcium chloride and citric acid concentration (w/v%).

The results of optimization based on WL/SG indicated that the optimized formulations for coating were [12.39% (w/v) fructose, 5.00 salt % (w/v) and 3.49% (w/v) citric acid].

CONCLUSION

In this study, the optimization of osmotic solutions (containing fructose, calcium chloride and citric acid) based on maximum performance coefficient in the osmotic dehydration of quince were investigated using response surface methodology (RSM). The results showed that fructose had linear and quadratic significant effects ($p<0.5$) on the performance coefficient of osmotic dehydration, while citric acid and calcium chloride showed no significant effect at determined range of concentration. The results of optimization based on WL/SG indicated that the optimized formulations for coating were [12.39% (w/v) fructose, 5.00 salt % (w/v) and 3.49% (w/v) citric acid].

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