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Optimization of carboxy methyl cellulose-pectin and ascorbic acid based edible coating formulations for performance of osmotic dehydration of quince by RSM

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Abstract

In recent years, edible coatings are extensively applied for improving processing and preservation of food stuffs. The using of edible coating as a pretreatment in osmotic dehydration is an efficient method for decreasing of solids diffusion from osmotic solution to food textures. In this research, optimization of active-blend edible coating formulation was investigated for using in osmotic dehydration of quince by response surface methodology (RSM). For this purpose, central composite design with three variable (concentration of pectin, carboxymethyl cellulose and ascorbic acid) three replicate and 18 treatments were used .Osmotic dehydration efficiency coefficient and water loss (WL) are selected as model responses. For osmotic dehydration, optimized osmotic solution that resulted from previous research works (fructose 50%, calcium chloride 5%, acid citric 3%) was used. On the base of maximum osmotic efficiency coefficient, the coating solution containing 1.49% carboxymethyl cellulose, 1.49% pectin and 0.58% ascorbic acid was determined as best coating solution by RSM modeling. The study of dehydration kinetics and mass transfer was carried out with osmotic solution of fructose 50%, calcium chloride 5%, acid citric 3%) (w/v), and weight reduction, water loss and solids gain were measured. CMC- pectin coatings improved the efficiency of osmotic dehydration process, increasing the water loss and decreasing the solids gain.

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Introduction

Quince (Cydonia oblonga Mill) is a member of pomes fruit family; 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. (Noshad *et al*., 2011; FAO, 2010).

Edible coating is defined as edible material (protein, polysaccharide or lipid) that is used as a thin layer on the surface of foods (Azarakhsh *et al*., 2012; Gonzalez-Aguilar *et al*, 2010). It can be applied to providing a selective barrier to oxygen, carbon dioxide and moisture, preserving fresh-cut vegetables and fruits, improving textural and mechanical properties, preventing flavour loss and carrying food additives (Azarakhsh *et al*., 2012; Tapia *et al*., 2008). Many carbohydrates pose advantages for tissue engineering applications due to their hydrophilicity, degradability, and availability of chemical groups for modification. For example, carboxymethylcellulose (CMC) is a water-soluble cellulose derivative that is degradable by cellulase. CMC is cellulose ether, produced by reacting alkali cellulose with sodium monocloroacetate under rigidly controlled conditions. Aqualon CMC is soluble in either hot or cold water. Viscosity of CMC solutions depends on temperatures (Reeves *et al*., 2010). Pectin is commercially produced from citrus peel as a by-product from extraction of lime, lemon and orange juices; or from apple pomace, the dried residue remaining after extraction of apple juice. Pectin is a heteropolysaccharide in its native state, but extraction with hot mineral acid removes most of the neutral sugars such as rhamnose, galactose, arabinose, etc., that comprise the branched or"hairy" regions of the polymer. Thus, commercial pectin consists of a homopolymeric linear chain of a -(1_4)-d -galacturonic acid units, where the uronic acid group may be either free or esterified with methanol (Embuscado and Huber 2009; Pérez *et al*., 2003) . By definition, for use in food and pharmaceuticals, pectin contains at least 65% galacturonic acid and methyl galacturonate. By convention, if the degree of methyl esterification (DE) is greater than 50%, the pectin is called high methoxyl (HM) grade or high ester, while if it is less than 50%, it is called low methoxyl (LM) or low ester pectin. Depending on how the extraction process is controlled and how much de-esterification occurs, pectin can have a degree of esterification as high as 77% or as low as 20%. Low ester pectin with amidation is also produced commercially (Embuscado and Huber, 2009).

Osmotic dehydration is an operation used for the partial removal of water from plant tissues by immersion in a hyper-tonic (osmotic) solution. Water removal is based on the natural and nondestructive phenomenon of osmosis across cell membranes. The driving force for the diffusion of water from the tissue into the solution is provided by the higher osmotic pressure of the hyper-tonic solution. The diffusion of water is accompanied by the simultaneous counter diffusion of solutes from the osmotic solution into the tissue. Since the cell membrane responsible for osmotic transport is not perfectly selective, solutes present in the cells (organic acids, reducing sugars, minerals, flavors and pigment compounds) can also be leached into the osmotic solution, which affect the organoleptic and nutritional characteristics of the product. The rate of diffusion of water from any material made up of such tissues depends upon factors such as temperature and concentration of the osmotic solution, the size and geometry of the material, the solution-to-material mass ratio and, to a certain level, agitation of the solution (Renu *et al*., 2012). During the osmotic process, besides the desirable water loss, another, relatively undesirable, mass flux is observed; the uptake of osmotic solids. Thus, a major concern in osmotic dehydration is to minimize the uptake of osmotic solids, as it can severely alter organoleptic and nutritional characteristics of the product; besides, a surface layer of solids adds an extra barrier to the water removal process (Mitrakas, 2008).

Some factors have been employed to speed up water transfer such as using a high concentration of osmotic solution, low molecular weight of osmotic agent, high processing temperature, stirring process or some pretreatment techniques. However, another concern in osmotic dehydration is currently to minimise the uptake of osmotic solids, as it can severely alter organoleptic and nutritional characteristics of the product. Numerous studied have attempted to reduce large solute uptake by using edible coating material prior to osmotic dehydration (pishut, 2012,Khin *et al.*, 2007; Garcia *et al.*, 2010; Jalaee *et al.*, 2010; Singh *et al.*,2010). The advantages of coating materials applied for osmotic dehydration process may include the following: (1) it may reduce the extensive solute uptake, (2) it may reduce losses of desired constituents such as colourant, flavour compounds and nutrients, (3) coating may provide greater product integrity and physical strength to food pieces, which can withstand mixing (throughout processing) and physical impact (during handling, storage and transportation), (4) it may also minimise microbial contamination and oxidation activity and (5) it may give greater esthetic appeal, especially for products with clear polysaccharide coatings (Phisut, 2012; Matuska *et al.*, 2006). For the purposes of the osmotic membrane process, edible coatings should have the following properties: good mechanical strength (gel strength), satisfactory sensory properties, easy and rapid film formation with simple techniques, high water diffusivity and maintenance (of the coating) in the intact state without dissolving into the osmotic solution (Phisut, 2012).

Response surface methodology is a set of statistical techniques for building models, designing experiments, searching the optimum conditions and evaluating the effects of factors (Manivannan and Rajasimman, 2011).

Several studies have been done to optimize the edible coating formulations for vegetables and fruits (Azarakhsh *et al.,* 2012; Tapia *et al.*, 2008; Ribeiro *et al.*, 2007; Rojas-Grau *et al.*, 2007; Avena-Bustillos *et al.*, 1994). The results of Chaiwong and Pongsawatmanit (2011) suggest that using 0.5 κ-Carrageenan as coating material in papaya cubes can lower sugar uptake and lower total soluble solid (TSS) in the product during osmotic dehydration process and in final dried product. García *et al.* (2010) showed that chitosan coatings(chitosan coatings at 1% (w/v) in lactic acid 1% (v/v) and Tween 80 at 0.1% (v/v); and with chitosan coatings at 1% (w/v) in lactic acid 1% (v/v), Tween 80 at 0.1% (v/v) and oleic acid at 2% (v/v)) improved the efficiency of osmotic dehydration process (osmotic solution of sucrose (40 $^{\circ}$ Brix), increasing the water loss and decreasing the solids gain. In both ripening stages, the water loss was higher in coated fruits.

The aim of this study was to determine the effect of LMP, CMC based active blend coatings containing ascorbic acid on the mass exchange: determining WL/SG, WL, SG and WR. Furthermore, the optimization of active-blend edible coating formulation was investigated for using in osmotic dehydration of quince by response surface methodology (RSM). However based on our knowledge, no article was published on using RSM for optimization of edible coating formulations for quince in osmotic dehydration.

Material and methods

Material

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 methoxyl pectinate (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 and edible coating solutions

Optimization of active-blend edible coating formulation was investigated for using in osmotic dehydration of quince by response surface methodology (RSM). 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 (Tapia *et al,* 2008 Azarakhsh *et al,* 2012). The overall volume for each formulation was 1000 ml and this includes different amounts of CMC, pectine and ascorbic acid (Tables 1) 0.2%(w/v) glycerol and the rest was distilled water (Montero-Calderon *et al.*, 2008). Then they were dried at 55-60°C for 5-10 minutes, in order to fix the coating on the samples. The different concentrations of CMC, pectine and ascorbic acid based on the experimental design were shown in Tables 1.

Before preparation of samples, quinces, all containers, cutting board, knives and other utensils in contact with quince were washed. After washing, quinces were peeled manually and cut with a sharp knife into cylindrical samples with 40 mm diameter and 2 mm long (40×2 mm — D×L).

Coating and osmotic treatments

The coating treatment was applied prior to osmotic treatment. The cylindrical quinces were dipped in the edible coating formulation (CMC +pectin +ascorbic acid glycerol) for 1 min and then the excess coating materials samples were permitted to drip off. After that, coated samples were dipped in the calcium chloride solution 1% (w/v) for 2 min to have a good cross-linking between calcium and the coating material. Finally, the samples were washed with water to remove the excessive $CaCl_2$ and blotted with filter paper (Khin *et al.,* 2007).

Osmotic dehydration was carried out in optimized osmotic solution that resulted from our previous research works by RSM (fructose 50%, calcium chloride 5%, acid citric 3%) (w/v), under temperature of 25 °C (The temperature was monitored by the thermocouple and was set at 25 °C). A sample to solution ratio of 1:10 (w/w) was used in order to avoid excessive dilution of the osmotic solution during processing (Khin *et al.,* 2006). Samples were withdrawn from the osmotic solution at the 15th, 30th, 45th, 60th, 75th, 90th ,105th, 120th, 135th, 150th, 165th, 180th, 195th, 210th, 225th and 240th minute, respectively blotted with filter paper and analyzed for their water loss (WL) and solid gain (SG). Non-coated quinces were also dehydrated osmotic ally under the same conditions as for coated quinces, for comparing their mass transfer behaviors during osmotic dehydration.

Analytical methods (calculations)

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 weight reduction (WR) was calculated by the following equations (Garcia *et al.*, 2010). The WL was the net loss of water from quince cylinders at time (θ) on an initial mass basis.

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

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

$$SG\%=[W_\theta(1-X_\theta)-W_i(1-X_i)]/W_i \quad (2)$$

$$PerformanceRatio(pr)=WL/SG \quad (3)$$

$$WR=WL-SG \quad (4)$$

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 (Pisalkar *et al*, 2011; lazarides *et al*, 2007).

Statistical analysis and experimental design

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 edible coating process on water loss (WL) and Performance Ratio (WL/SG) in quinces culinders. The center composite design (CCD) was used for optimization of edible coating 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 (Mirhosseini *et al.*, 2008). A rotatable central composite design was used with CMC concentration (X_1, %w/v) (0.00, 0.3, 0.745, 1.49, 1.49), pectin concentration (X_2, %w/v) (0.00, 0.3, 0.745, 1.49, 1.49) and ascorbic acid percent (X_3, %w/v) (0.00, 0.12, 0.295, 0.47, 0.589) 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:

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{\substack{i=1 \\ i<j}}^{k-1} \sum_{j=1}^{k} \beta_{ij} X_i X_j \quad (5)$$

In this model, Y is a calculated response (i.e., WL, WL/SG, %) X_i and X_j are factors (i.e., concentration of pectin, carboxymethyl cellulose, and ascorbic acid β_i ،β_{ii} and β_{ij} are linear, quadratic and interaction coefficients, respectively and β_0 is a constant. Software's of SAS 9.1 (England) and Statistical 9 (USA) were used for analysing data and drawing response 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). To verify the adequacy of the regression models the fitted values predicted by the models were compared with experimental data (Azarakhsh *et al*, 2012).

Results and discussion

Response surface analysis for CMC, pectin and ascorbic acid coating

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 edible coating components on each studied response variable. RSM suggested response surface models to show the relationship between independent variables and experimental data.

Table 1. Experimental design used for CMC, pectine and ascorbic acid based edible coating.

	Coded variables			Uncoded variables		
Treatment	X_1	X_2	X_3	CMC	Pectin	Ascorbic acid
1	-1	-1	-1	0.33	0.33	0.12
2	-1	-1	1	0.33	0.33	0.47
3	-1	1	-1	0.33	1.19	0.12
4	-1	1	1	0.33	1.19	0.47
5	1	-1	-1	1.19	0.33	0.12
6	1	-1	1	1.19	0.33	0.47
7	1	1	-1	1.19	1.19	0.12
8	1	1	-1	1.19	1.19	0.47
9	-1.682	0	0	0.036	0.745	0.295
10	1.682	0	0	1.49	0.745	0.295
11	0	-1.682	0	0.745	0.036	0.295
12	0	1.682	0	0.745	1.19	0.295
13	0	0	-1.682	0.745	0.745	0.0006
14	0	0	1.682	0.745	0.745	0.58
15	0	0	0	0.745	0.745	0.295

16	0	0	0	0.745	0.745	0.295
17	0	0	0	0.745	0.745	0.295
18	0	0	0	0.745	0.745	0.295

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 3.1 ± 0.41 to 6.78 ± 2.18%. Relatively high correlation coefficients (i.e. R^2) was obtained for WL/SG indicating good fit of experimental data to Eq. (2) (Table 4). That lack of fit was not significant for WL/SG (0.27) at P = 5% level. Obtained summarized model to predict the effects of CMC, pectin and ascorbic acid on WL/SG, after excluding non-significant factors, is as follows:

$$Y=4.48-0.73X_1-2.83X_2+1.95X_2^2 \quad (6)$$

The analysis of variance for final reduced models (Table 4) showed that WL/SG was mainly affected linearly by CMC concentration whereas the quadratic effect of CMC concentration was not significant at 5% level. The quadratic effect of pectin concentration was significant at 99% and the main effect of pectin concentration was significant at 5% level on WL/SG (Table 4). The effect of changing CMC and pectin concentration on the percent performance ratio (WL/SG) of coated samples is given in Fig. 1.a. The WL/SG is increasing with pectin and CMC concentrations (Fig. 1. a). The effects of CMC and pectin on WL/SG are quadratic and linear, respectively. The results of Lazaridis *et al.*, 2007, Khin *et al.,* 2007 and Jalaee *et al,* 2010 Khin *et al.*, (2007) reported that performance ratio, defined as the ratio of the amount of water loss to the amount of solute uptake, for coating materials, was initially investigated by Carmirand, Krochta, Pavlath, Wong and Cole (1992).

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
CMC concentration	g/mlit	X_1	0.00	0.33	0.745	1.19	1.49
Pectin concentration	g/mlit	X_2	0.00	0.33	0.745	1.19	1.49
Ascorbicacid concentration	g/mlit	X_3	0.00	0.12	0.295	0.47	0.58

It was 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 methoxyl pectinate (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.

Jalaee *et al,* (2010) 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 (lowmethoxyl pectinate (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 and 2) (Chin and Law, 2012).

Table 3. Responses for CMC, pectin and ascorbic acid- based edible coating.

Run	Responese	
	WL (gr/gr%)	WL/SG(%)
1	29.86 ± 1.81	4.02 ± 0.23
2	27.74 ±1.23	3.24 ± 0.20
3	30.13 ± 0.35	3.86 ± 0.71
4	28.84 ± 1.26	4.39 ± 0.72
5	32.28 ± 0.73	3.53 ± 0.41
6	31.99 ± 3.14	4.18 ± 0.16
7	37.10 ± 0.17	5.45 ± 0.11
8	40.32 ± 1.78	5.63 ± 0.83
9	32.76 ± 2.46	3.10 ± 0.41
10	37.83 ± 1.95	4.87 ± 1.27
11	30.62 ± 0.19	3.20 ± 0.19
12	40.62 ± 3.59	6.78 ± 2.18
13	38.05 ± 2.17	4.14 ± 0.05
14	35.59 ± 6.17	3.31 ± 0.05
15	31.75 ± 0.94	4.45 ± 0.81
16	38.99 ± 1.46	4.17 ± 0.01
17	33.81 ± 4.10	3.7 ± 0.62
18	39.58 ± 4.33	3.6 ± 0.02

Figure 1. b depicts the interactive effect of the CMC and ascorbic acid concentration on WL/SG. The WL/SG was increased by increasing ascorbic acid concentration in quadratic manner. The coated quince had maximum WL/SG at mean concentrations of ascorbic acid (0.25 to 0.45 w/v%). The results suggested that the WL/SG of the samples increased linearly with increasing CMC concentration. Khin *et al.*, (2007) also found that the WL/SG of the fruits increased with using coating. Khin *et al.*, (2007), explored a novel approach to monitoring solids uptake during osmotic dehydration, the combination of product coating with alternative scenarios of product/solution contacting ("flow") was investigated. Potato was used as a model plant material for short term (i.e. 3 h) osmotic treatment in a series of sucrose solutions with decreasing or increasing concentrations to simulate co-current or counter-current product/solution contacting (flow), respectively. A mixed-level full factorial experimental design was used. Data wereanalyzed using multiple linear regression procedures. Counter-current product/solution contacting contributed to faster water loss and slower solids uptake. In both flow-types, initial solids had a significant impact on both water loss and solids uptake. Sodium alginate coating yielded significantly decreased solids uptake, without negatively affecting water removal. It was concluded that "dehydration efficiency" was drastically improved (up to 77%) by combined coating and counter-curenrt contacting. The effects of pectin and ascorbic acid on WL/SG are shown in figure 1.c. The quadratic term of pestin concentration has positive and significant effect, whereas the ascorbic acid concentration has no significant effect on WL/SG.

The results of optimization based on WL/SG indicated that the optimized formulations for coating were [1.49% (w/v) CMC, 1.49 % (w/v) pectin and 0.58% (w/v) ascorbic acid].

Effect of CMC, pectin and ascorbic acid water loss (WL)

As shown in Table 3, water loss of edible coated quince cylinders durng osmotic dehyfrstion varied from 27.74 ± 1.23% to 40.62 ± 3.59. Uncoated quince cylinders at the same condition exhibited a water loss of 46.80±0.52%. Relatively low correlation coefficients (i.e. R2) was obtained for WL, don't indicate good fit of experimental data to Eq. (3) (Table 5). That lack of fit was not significant for WL (0.62) at P = 5% level. The quadratic effects of CMC and pectin concentrations on Performance Ratio were not significant at 95%. obtained Summarized model to predict the effects of CMC, pectin and ascorbic acid on WL/SG, after excluding non-significant factors, is as follows:

$$Y=36.21+2.46X_1+2.29X_2 \quad (7)$$

As for water loss, CMC and pectin concentrations were found significant for linear effects at 5% level. (Table 5). Water loss is almost similar to Weight loss because other components like gaseous products of respiration, aroma or flavour are practically undetectable in terms of weight (Olivas and Barbosa-Canovas, 2005; Azarakhsh *et al.*, 2012). Edible coatings have potential to control the water loss of fresh-cut fruits or during osmotic dehydration (Gonzalez-Aguilar *et al.*, 2010).

As can be seen from figure 2.a, the WL was predominantly affected by the CMC and pectin concentrations. Highest CMC and pectin concentrations seem to Highest water loss through osmotic dehydration. Similar research indicated that gellan and sodium alginate-based coatings used on osmotic dehydrated apples were effective to reduce the water loss when sunflower oil was applied in coating formulation as lipid source (lazarides *et al.*, 2007).

Table 4. Regression equation coefficients for performance ratio (WL/SG) during osmotic dehydration of quince cylinders Analysis results of variance for the osmotic dehydration basis on WL/SG.

Sources	Regression Coefficience	(df)	(SS)	(MS)	F	p
X_1	- 0.731072	1	2.87	2.87	10.81	0.011036*
X_2	- 2.837472	1	7.90	7.90	29.72	0.000607**
X_3	- 1.11385	1	0.04	0.04	0.18	0.682205
X_1^2	0.031472	1	0.00	0.00	0.00	0.968967
X_1X_2	1.604089	1	0.70	0.70	2.64	0.142404
X_1X_3	1.80142	1	0.14	0.14	0.55	0.478401
X_2^2	1.959502	1	1.66	1.66	6.24	0.036998*
X_2X_3	1.386899	1	0.08	0.08	0.32	0.582728
X_3^2	- 2.793118	1	0.09	0.09	0.34	0.571473
Model	-	9	13.79	1.53	5.76	0.010807*
Linear	-	3	10.82	3.60	13.57	0.001666**
Quadratic	-	3	2.02	0.67	2.54	0.129408
Cross Product	-	3	0.93	0.31	1.17	0.377871
Error	-	8	2.12	0.26	-	-
Lack of fit	-	5	1.67	0.33	2.22	0.2714
Pure Error	-	3	0.45	0.15	-	-
Total	-	17	15.92	-	-	-
R^2	86.64	-				
CV	12.25	-				

R^2=86.64 R^2_{adj}=71.61، R^2_{pre}=79.82 CV=12.25

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

Table 5. Regression equation coefficients for WL during osmotic dehydration quince cylinders.

Sources	Regression Coefficience	(df)	(SS)	(MS)	F	p
X_1	2.469714	1	82.8256	82.8256	6.489517	0.03431*
X_2	2.295337	1	71.95205	71.95205	5.637557	0.04494*
X_3	- 5.652936	1	1.570206	1.570206	0.123028	0.73483
X_1^2	- 1.04534	1	13.82216	13.82216	1.082988	0.328456
X_1X_2	7.961872	1	17.33781	17.33781	1.358445	0.277379
X_1X_3	10.54168	1	5.03411	5.03411	0.394431	0.547485
X_3^2	- 5.036023	1	10.96756	10.96756	0.859326	0.381043
X_2X_3	7.195265	1	2.34529	2.34529	0.183757	0.679473
X_3^2	- 0.506857	1	3.249609	3.249609	0.254612	0.627448
Model	-	9	201.9676	22.44084	1.758276	0.219309 0.0048*
Linear	-	3	156.3477	52.11589	4.083362	0.049522*
Quadratic	-	3	20.90272	6.967574	0.54592	0.664591
Cross Product	-	3	24.71721	8.23907	0.645544	0.607254
Error	-	8	102.1039	12.76298	-	-
Lack of fit	-	5	57.53876	11.50775	0.77467	0.626415
Pure Error	-	3	44.56511	14.85504	-	-
Total	-	17	304.0715	-	-	0.03431

R^2	66.42	-	0.04494
CV	10.40	-	

66.42 R^2= 10.40 CV= *, **: significant at P < 5% and P < 1%, respectively.

Figure 2. b showed the effects of CMC and ascorbic acid on WL. These results indicate a quadratic increase in water loss with an increase in CMC concentration (Fig. 2b). Figure 2.c shows that different pectin and ascorbic acid concentrations used in coating, influenced the WL of the quince cylinders during osmotic dehydration. Pectin and ascorbic acid concentrations have a quadratic and linear effect on WL, respectively. WL increased with increasing pectin concentration.

The results of optimization based on WL revealed that the optimized formulations for coating were [0.84% (w/v) CMC, 1.68 % (w/v) pectin and 0.84% (w/v) ascorbic acid].

Table 6. Predicted and experimental data for the responses at optimum point for edible coating based on WL/SG in osmotic dehydration of quinces.

Responces	Predicted value	Experimental value[a]
Performance ratio (WL/SG)	3.504	3.91 ± 0.162
Water loss (WL)	42.1236	29.56 ± 2.35

[a] Mean ± S.D. [b] No significant (p>0.05) difference between experimental and predicted value.

Optimization Procedure

For determination of the optimum levels of independent variables, multiple response optimizations were used. For better visualizing the variation of WL and WL/SG as function of main edible coating components, the 3D surface plot was used. Results obtained show that, in terms of WL/SG studied, the overall optimized region was predicted to be 1.49% (w/v) CMC, 1.49 % (w/v) pectin and 0.58% (w/v) ascorbic acid respectively. For optimized WL/SG for edible coating were predicted to be 3.504% (table 6). The predicted results indicated that the overall optimized region based on WL for CMC and pectin-based edible coating containing ascorbic acid was achieved by formulation comprising of 0.84% (w/v) CMC, 1.68 % (w/v) pectin and 0.84% (w/v) ascorbic acid. Under the optimized condition, water loss of coated quinces was predicted to be 42.13% (Table 6) (Azarakhsh *et al*., 2012).

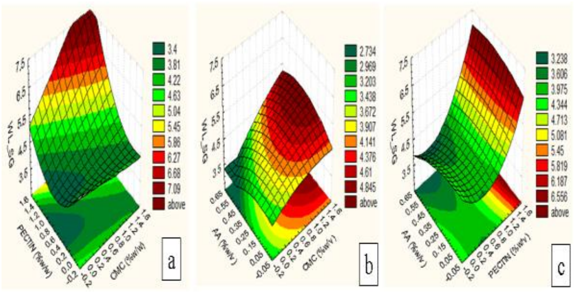

Fig. 1. Profile of response surface and contour plots for Performance ratio (WL/SG) during osmotic dehydration of coated quince cylinders as function of (a) CMC and pectine concentration (w/v%) (b) CMC and ascorbic acid concentration (w/v%) (c) pectin and ascorbic acid concentration (w/v%).

Verification of the models

The comparison between fitted values predicted by the response regression models and experimental values indicated the adequacy of the response surface equations. Tables 6 show the predicted and experimental values. These values did not show any significant (p>0.05) difference. The predicted values were indicated to be in agreement with the experimental response values. (Azarakhsh *et al*, 2012).

Effect of process time on mass transfer

The general behavior of material transfer shows (Fig. 3) the predominance of water removal, as compared to soluble exchanges. The kinetics of material transfer can be divided into three phases: (1) a starting phase (0–2 h) during which exchange rates grow until 2 h; (2) an acceleration phase (2–5 h) during which the material exchange reaches its maximum value; and (3) a decreasing phase where exchange values decrease. The observed behavior could be due to that in the initial stage it exists more difference between the chemical potential of the fruit and osmotic solution and, for consequence, it exists more water loss and solids gain, permitting that the diffusion of the molecules is quicker. The decreasing rates could be attributed to a decrease in the concentration gradient and structural changes that occur in the tissues, slowing the diffusion process. The used solution: samples mass relation avoid changes in osmotic solution concentration during the dehydration process, therefore, changes in concentration gradient are due to the changes in the fruit (Garcia *et al.*, 2010).

Process efficiency

The main characteristic of an osmotic dehydration process is the loss of water; however, the solids gain is a parameter to consider, because the process efficiency depends on these two parameters. The weight reduction is also considered an important parameter in order to measure the efficiency of the osmotic process.

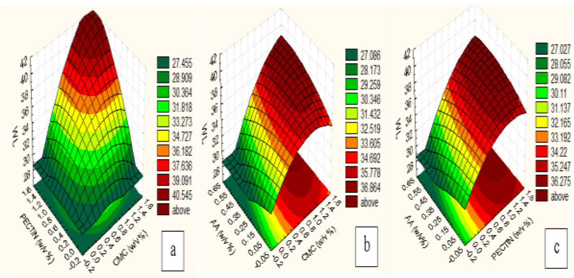

Fig. 2. Profile of response surface and contour plots for Performance ratio (WL/SG) during osmotic dehydration of coated quince cylinders as function of (a) CMC and pectin concentration (w/v%) (b) CMC and ascorbic acid concentration (w/v%) (c) pectin and ascorbic acid concentration (w/v%).

Water loss (WL)

Fig. 3. a. shows the changes in the water loss during the osmotic dehydration process of quince samples with and without coatings. During the process it was observed a similar behavior in the water loss values in all evaluated treatments, observing an increment in the values of this parameter in the time, reaching the high rate of water loss during the first three hours of the process, in correspondence with Barbosa and Vega (2000), who concluded that the high water loss of in food occurs in the first 6 h of the process, being the two initial hours, those of high rate of water removal. This kinetic tendency was also reported by Nowakunda, Andrés, and Fito (2004) in osmodehydrated banana slices. In samples, it is observed that uncoated fruit lost less water than coated samples during the osmotic process. It is agree with the results obtained by Garcia *et al.*, (2010) and Díaz (2003).

Misljenovic *et al.*, (2011) studied the osmotic dehydration process of carrots in terms of water loss and solid gain. Initial high rate of water removal and solid uptake, followed by slower removal and uptake in the later stages was observed. Rapid loss of water and solid gain in the beginning is apparently due to the large osmotic driving force between the dilute sap of the fresh carrot and the surrounding hypertonic solution. Water loss and solid gain were most intensive in the first two hours of osmotic dehydration process.

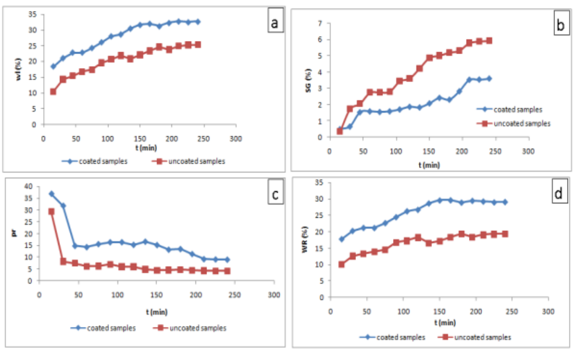

Fig. 3. Variation of water loss (a), solid gain (b), performance ratio (c) and weight reduction (d) of quince samples during osmotic dehydration.

Solid gain (SG)

Fig. 3. b. shows the percentage of soluble solids gain during the osmotic dehydration process of quince samples. All evaluated treatments for quinces, presented an increment in the values of soluble solids gain. It was observed that coated quinces presented a minor soluble solids gain than quince samples without coating. In the case of the treatments with coated quince, the solids accumulation on the coatings surface, limited its penetration inside the fruits, which it did not happen in samples without coatings, where a great amount of solubles penetrated inside the fruits (Garcia *et al.*, 2010; Díaz, 2003). The solid accumulation, together with the use of coatings, may create a crust which constitutes a barrier to mass transfer, limiting the dehydration regime and consequently the solubles gain. The above assumptions could explain the difference of dehydration regimes and material transfer between the coated and uncoated quince samples. The gain of solubles by the samples is comparable to weight loss, particularly in the first phase of transfer phenomenon (0–2 h). In the second phase (2–5 h), the weight loss becomes higher than gain of solubles. As weight loss is the balance between water removal and soluble gain, it could be concluded that, gain of soluble is compensated by the water removalg (Garcia *et al.*, 2010).

Jalaee *et al.*, (2010) studied the influence of different edible coating materials such as lowmethoxyl pectinate (LMP), carboxyl-methyl cellulose (CMC), corn starch, and an osmotic sucrose solution with two concentrations of 50% and 60% (w/w) on mass transfer of apple rings. Experimental results showed that coating on apple could be a solution for reducing the solid gain without affecting much on the water removal in comparison with uncoated samples. Apple coated with LMP, CMC and corn starch and osmotic dehydrated in 50% and 60% sucrose solution had lower solid gain than the uncoated sample in the same conditions. Misljenovic *et al.*, (2011) revealed that solid gain, during the osmotic dehydration (sugar beet molasses) of carrot, showed a tendency to increase with increasing the immersion time.

Process efficiency index (pr)

The values of the process efficiency index (Pr =WL/SG) are used for evaluation of efficiency of osmotic dehydration process (Fig. 3. c), due to their easy interpretation, because if Pr increases it could mean one of this three possibilities: (i) the process is favoring the water loss and solids gain, but mostly the water loss; (ii) the process limits the solids gain; and (iii) the process favors the water loss. It was observed, in general, that the treatments with coatings presented higher values of Pr during the osmotic dehydration process, what could be due to that in these treatments the water loss was favored, while the soluble solids gain was limited. At the beginning of the process, the changes in the Pr value can be attributable mainly to the water loss, and when the process time increases, the solids gain has more influence on the Pr value. This indicates that upon designing an osmotic dehydration process for this fruit, the time of contact will be defined in function of the pursued objective (Garcia *et al.*, 2010).

Weight reduction (WR)

Fig. 3. d. shows the evolution of weight reduction of the coated and uncoated quince samples during dehydration process. All samples, coated and uncoated samples, showed a similar behavior for weight reduction, observing, of general way, that coated quince samples presented higher values for this parameter than uncoated fruits, which it could due to that the fruits treated with coatings lost more water (Fig. 3. a) and won less solids than fruits without coatings (Fig. 3.b). It is agree with the results obtained by Garcia *et al.,* (2010), Díaz (2003), Argaiz *et al.*, (2003).

Conclusions

Response surface methodology was applied in this study to optimize the carboxymethyl cellulose and pectin- based active blend edible coating formulations (containing ascorbic acid) for osmotic dehydration of quince. Regression models were obtained for predicting the effects of CMC, pectin and ascorbic acid concentrations on WL and WL/SG coated quinces in osmotic dehydration. All models were fitted significantly ($p < 0.05$). The lack of fit for response variables in this study, was not significant ($p > 0.05$). It shows the accuracy of proposed models is sufficient to evaluate the variability of responses. The optimum formulation predicted for edible coating based on WL/SG, was 1.49% (w/v) CMC, 1.49 % (w/v) pectin and 0.58% (w/v) ascorbic acid. On the other hand, edible coating based on WL, the predicted optimum formulation [0.84% (w/v) CMC, 1.68 % (w/v) pectin and 0.84% (w/v) ascorbic acid. Furthermore, the study showed that carboxymethyl cellulose-pectin based active blend coatings containing ascorbic acid, improved the efficiency of osmotic dehydration process, increasing the water loss and decreasing the solids gain.

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