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Original research

Osmotic dehydration combined with hot air convective drying of Aloe vera (*Aloe barbadensis* Miller) gel

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A B S T R A C T

Aloe vera gel is widely used as a functional ingredient in food processing. This gel is very unstable due to its high water activity. Drying process can be used to obtain shelf stable products. Response surface methodology was used to assess the effects of solution concentration (30-60% w/w), solution temperature (30-50°C) and immersion time (4-6h) at atmospheric pressure and constant solution to sample ratio (10:1) on water loss (WL), solid gain (SG), weight reduction (WR), vitamin C content (VCC) and the total phenolic content (TPC) of Aloe vera gel. The optimized conditions of osmotic dehydration combined with hot air drying were 60% (w/w) solution concentration, 30.2°C solution temperature and 349 min immersion time. Under these conditions water loss, solid gain and weight reduction found to be 72.67 (g/100g), 5.33(g/100g) and 67.34 (g/100g), respectively. The vitamin C and TPC of the osmo-air dried gel were 12.70 mg/100g DM and 11.33 mg GAE/100g DM, respectively.

Keywords: Aloe vera gel, Osmotic dehydration, Hot air convective drying, Total phenolic content, Vitamin C

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1. Introduction

An Aloe vera is a tropical and subtropical plant of *Liliaceae* family with turgid green leaves that contains a transparent mucilaginous jelly which is referred to as Aloe vera gel. Gel contains over 98–99% water and more than 60% of the dry matter is made up of polysaccharides (McAnalley, 1993). Aloe vera gel is a colorless, odourless hydrocolloid with several natural beneficial substances and has a long history in disease treatment. Due to its therapeutic and functional properties and hence its beneficial effects on humans, the use of Aloe vera gel in the formulation of food products has increased (Vega-Galvez et al., 2011). In view of highly perishable nature of Aloe vera gel, application of preservation methods to increase the shelf-life of that seems to be necessary. Aloe vera gel is always commercialized as a dried powder. Improper drying procedures of Aloe vera gel may cause irreversible modifications to the polysaccharides, affecting their original structure, which may promote important changes in the proposed physiological and pharmacological properties of these active ingredients (Eshun & He, 2004). Drying is an old process of food preservation (Borchani et al., 2011). The concept of water activity (a_w) has been very useful in food preservation and on that basis many processes could be successfully adapted and new products designed. Food drying process and decreasing a_w cause an increase in the shelf-life of foods (Akpinar, 2006; Al-Harahsheh et al., 2009). Hot-air drying is the most common method of food drying. Nevertheless, its low energy efficiency and long process time during the falling rate period lead to the degradation of the nutritional value, organoleptic properties, texture and color destruction and reduction in the rehydration capacity of food products (Maskan, 2001). Combinations of the hot-air drying with different pretreatments have been reported by scientists for the reduction of the drying time as well as improvement in the quality of dried products (Perez & Schmalko, 2009). One of the most applicable of pretreatments is osmotic dehydration (Noshad et al., 2011). Osmotic dehydration is defined as a partial drying method, by immersing the fruits and vegetable in hypertonic solutions. It is employed as a pretreatment for many food processes such as freezing, freeze drying, vacuum drying, microwave drying, and hot air drying (Md.Shafiq et al., 2010). During osmotic dehydration, two kinds of mass transfer phenomena take place simultaneously, including the diffusion of water from food into osmotic solution,

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and the second flow is diffusion of solutes from osmotic solution to the food (Rastogi & Raghavarao, 2004). In osmotic dehydration, partial water removal from a food product with mild heat treatment lead to low energy consumption, stabilization of color parameters, reduction of nutrient degradation, prevention of loss of volatile compounds and improvement of texture (Lazarides et al.,1995; Rodrigues et al., 2003; Falade et al., 2007; Vieira et al., 2012; Derossi et al., 2015). In spite of the above-mentioned benefits of osmotic dehydration, one basic problem regarding to this pretreatment is the loss of functional components like vitamins, minerals, phenolic compounds etc. by water removal and hence, the reduction in the nutritional value which has been reported in many researches (Stojanovich & Silva, 2007; Giovanelli et al., 2013; Kucner et al., 2013). The rate of mass transfer in osmotic dehydration depend on many factors, including the temperature, concentration of the osmotic solution, time, shape and size of the food material, solution-to-sample ratio, osmotic solution circulation, etc. The effective variables on this process are important to obtain optimum conditions for production of dried product with high quality and appropriate nutritional value (Yadav & Singh, 2012; Derossi et al., 2015).

Introducing an osmotic dehydration preliminary step into the conventional stabilizing process may also lead to substantial energy savings (Collignan et al., 1992; Lewicki & Lenart, 1992). In fact, the product is processed in liquid phase, giving generally good heat and mass exchange coefficients, and water is removed from the product without phase change (Bolin et al., 1983). Globally, an osmotic dehydration pretreatment decreases time of convection drying necessary to obtain water activity between 0.6 and 0.8 (intermediate moisture food) (Lewicki & Lenart, 1992), and also the water load to the drier (Huxsoll, 1982). Despite its technological advantages, osmotic dehydration is a difficult process to control, because of the number of variables that influence mass transfer such as the sample size, solution-to-sample ratio, process time and temperature, concentration and osmotic solution circulation, and also the cell structure, which differs from one tissue to another (Monnerat et al., 2010; Tedjo et al., 2002; Andres et al., 2007; Rincon & Kerr, 2010; Azoubel & Silva, 2008).

Tortoe et al. (2007), Lombard et al. (2008) and Panades et al. (2008), have investigated the effect of temperature and solution concentration on the kinetics of the osmotic dehydration of different food materials. García-Segovia et al. (2010) have studied the effect of osmotic dehydration on water loss, solid gain, weight reduction and effective diffusivity of Aloe vera gel. The effect of heat treatment and dehydration at different temperatures (30-80°C) on physico-chemical changes of acemannan, a bioactive polysaccharide from Aloe vera parenchyma, was evaluated by Femenia et al. (2003). Chang et al. (2006) have investigated the effects of heat treatment on bioactive substances including polysaccharide and barbaloin of Aloe vera.

Response surface methodology (RSM) consists of a series of statistical techniques for process optimization (Vieira et al., 2012). In recent years, many researches have been aimed at optimizing the osmotic dehydration of various agricultural products through RSM (Ozdemir et al., 2008; Md.Shafiq et al., 2010; Singh et al., 2010; Derossi et al., 2015). However, a few of research has been done on the optimization of osmotic dehydration of Aloe vera gel prior to drying process. The objective of this study was to find the optimal conditions of the osmotic dehydration of Aloe vera gel combined with hot air convective drying in order to achieve a dried product with high quality.

2. Material and Methods

2.1. Materials

Fresh leaves of Aloe vera (*Aloe barbadensis* Miller) obtained from Sabzevar city (khorasan razavi province, Iran) were used as the raw material in all experiments. The leaves (23-28 cm in length), washed with water to remove adhering soil and other debris and cut vertically side down and the epidermal tissue (green crust) was separated from the gel, then, the gel was cut into cubic pieces with the dimensions of 1×1×1cm. The average moisture content of the gel was determined to be 98% on a wet basis (AOAC, 2000). Sugar, the osmotic agent, was purchased from a local supermarket. The osmotic solution is prepared by mixing the sugar with proper amount of pure water.

2.2. Osmotic dehydration pretreatment

The osmotic dehydration was conducted in a 500 mL Erlenmeyer flask. Agitation with Magnetic stirrer was given during osmosis for reducing the mass transfer resistance at the surface of the samples and for good mixing and close temperature control in osmotic medium. Aloe vera cubes weighed and then placed into dehydrating vessel containing osmotic solution of varying concentrations (30–60%), solution temperature of 30-50°C and immersion time of 4-6h at atmospheric pressure and constant solution to sample ratio (10:1). After the osmotic dehydration process, Aloe vera cubes were taken out and then gently blotted with adsorbent paper and weighed. In each of the experiments fresh osmotic syrup was used. All the experiments were done in triplicate and the average value was taken for calculations.

In order to follow adequately the osmotic dehydration kinetics, individual analysis for each sample was carried out and from which, weight reduction (WR), solid gain (SG) and water loss (WL) data were obtained, according to equations (1–3) respectively.

$$WL = \frac{W_i X_i - W_f X_f}{W_i} \times 100 \quad (1)$$

$$SG = \frac{W_f (1 - X_f) - W_i (1 - X_i)}{W_i} \times 100 \quad (2)$$

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

where W_i is the initial weight (g) and W_f stands for the sample weight after osmosis. X_i (%) and X_f (%) are the initial moisture content and the moisture content after osmosis, respectively.

2.3. Hot-air-drying

Osmotically dehydrated samples were dried in a laboratory tray dryer (Hi Tech Dryer – FD-02, Iran). The dryer consisted of a drying chamber, electric heater, fan and a temperature controller. Drying was conducted at 70ºC air temperature and at a constant airflow velocity of 1± 0.2 m/s. After the dryer reached the set conditions, cubes of raw aloe vera samples (150 g) were uniformly spread in each tray and kept in dryer. The drying was continued until reaching the final moisture content of 10% (wet basis).

2.4. Analytical determinations

The moisture content was determined in triplicate using a gravimetric method by drying in a vacuum oven at 60ºC, to constant weight (AOAC, 2000). The vitamin C content of dried Aleo vera gel was determined according to standard (AOAC, 2000) methodology, immediately after processing. The results were reported as the mg vitamin C/100 g dry matter. Total phenolic contents of dried Aleo vera gel were obtained according to Stojanovich and Silva (2007), and quantified by the Folin–Ciocalteu spectrophotometric method using gallic acid (GA) as the standard. Total phenolic compounds were expressed as mg of gallic acid equivalents (GAE) per 100g of dry matter (DM).

2.5. *Experimental Design*

Response surface methodology (RSM) was used to estimate the main effects of osmotic dehydration process on water loss (WL), solid gain (SG), weight reduction (WR), vitamin C content (VCC) and total phenolic contents (TPC) of the Aloe vera gel. A face-centered central composite design was used with solution temperature (30–60°C), solution concentrations (30–50% w/w) and immersion time (4–6 min), being the independent process variables (Table 1). For the generated 20 experiments including 6 replicates for the center points (Table 2), RSM was applied to the experimental data using design expert 7.1.5 (Stat-Ease Inc., Minneapolis, USA).

In order to examine the linear, quadratic and interactive effects of the independent variables on the responses, the polynomial full quadratic model (Eq.4) was fitted to the empirical data.

$$Y = b_0 + b_1A + b_2B + b_3C + b_{11}A^2 + b_{22}B^2 + b_{33}C^2 + b_{12}AB + b_{13}AC + b_{23}BC \quad (4)$$

where Y is the response, b_0 represents the model intercept; b_i, b_{ii} and b_{ij} denote the regression coefficients for the linear, quadratic and interactive effects, respectively. A, B and C are osmotic solution concentration (% w/w), solution temperature (°C) and immersion time (h), respectively.

Table 1. The levels of different process variables in coded and uncoded form.

Independent variable	-1	0	+1
Solution concentration (%)	30	45	60
Solution temperature (°C)	30	40	50
Immersion time (h)	4	5	6

3. Results and Discussion

The appropriate model was selected regarding the significance of the F-test ($p \leq 0.05$), insignificance of lack of fit ($p > 0.05$), and R^2. Considering the analysis of variance (ANOVA) (Table 3), it was found out that the fitted model was significant in the case of all responses ($p < 0.01$). In order to develop the model, its non-significant terms ($p > 0.05$) were removed. The fitted polynomial models are presented in equations 5-9 based on the coded levels.

Analysis of variance (Table 3) revealed that all linear, interactive and quadratic terms of the model had significant effects on the WL ($p < 0.05$). The linear term of the osmotic solution concentration (A) followed by the linear term of of process time (C) had the highest positive impact on this response. The linear and quadratic terms of solution temperature (B and B^2) also had positive effects on WL. This indicated that as the solution concentration, solution temperature and dehydration time increased, WL increased, too. In addition, by further increase of solution temperature to higher levels, WL has been increased. Fig. 1 shows the effect of temperature and process time on WL.

The direct correlation between the solution temperature and WL could be attributed to the turgidity and plasticization of the cell membrane as well as the improvement in mass transfer on the surface of the product due to the lower viscosity of the osmotic solution at higher temperatures (Vieira et al., 2012). The positive effect of the concentration increase could also be attributed to the rise in the osmotic pressure arisen from the concentration difference among the interior of the cell and the osmotic solution in higher concentrations (Azoubel & Murr, 2004).

These findings conformed to the results of Falade et al. (2007) and Ispir and Togrul (2009) who worked on watermelon and apricot, respectively. Devic et al. (2010) also reported that the WL increased as the immersion time increased and it was also accelerated at higher temperatures.

In contrast, the quadratic term of the osmotic solution concentration (A^2) and process time (C^2) influenced the WL negatively; nevertheless, with regard to the values of their coefficients, the impacts of these terms are negligible as compared to the linear terms of concentration and process time. The negative quadratic effects of concentration and process time explain that the elevated levels of process time and concentration of the osmotic solution reduced the WL. Giraldo et al. (2003) in osmotic dehydration of mango reported that the extreme increase in the concentration of osmotic solution led to the reduction of water loss. This observation could be because of the decreased mass transfer resulted from the increased viscosity in concentrated sugar solutions (Vieira et al., 2012). Rapid removal of water in the early stages of osmotic dehydration due to the high osmotic driving force between the fresh sample and concentrated solution has been reported by several researchers (Eren & Kaymak-Ertekin, 2007; Ganjloo et al., 2014). The determination coefficient (R^2) of the model was equal to 0.9952, furthermore, the lack of fit of the model was insignificant ($p > 0.05$) demonstrating the adequacy of the fitted model.

Table 3 reveals that merely the linear terms of the osmotic solution concentration (A), solution temperature (B) and process time (C) affected the SG significantly ($p < 0.05$); however, all quadratic (A^2, B^2 and C^2) and interactive (AB, AC and BC) terms had no significant effect on this response ($p > 0.05$). Thus, these terms were removed from the model. Among the linear effects, solution temperature (B) followed by process time (C) had the highest positive impact on SG. The linear term of solution concentration (A) also had positive effect on SG. Fig. 2 shows the positive effect of temperature and concentration on SG during osmotic dehydration of Aloe vera gel. Mass transfer enhancement due to the temperature and concentration increase could be considered the reason for the elevated SG by the gel. Rastogi and Raghavarao (2004) suggested the osmotic pressure difference and concentration difference as the propulsions of mass transfer for moisture removal and solid gain, respectively. The positive effect of the temperature increase on the simultaneous increase of WL and SG has been previously cited Lazarides et al. (1995) in the case of apple and Mundada et al. (2011) in the case of pomegranate aril. The lack of fit of SG was not significant ($p > 0.05$). The determination coefficient (R^2) of the model was equal to 0.9637 revealing the good agreement between the actual and predicted results.

Table 2. Experimental conditions and observed response values.

Run	Solution concentration (%)	Solution temperature (°C)	Immersion time (h)	WL (%)	SG (%)	WR (%)	VCC (mg/100g DM)	TPC (mg GAE/100g DM)
1	45	40	5	68.108	5.268	62.84	13.16	10.36
2	45	40	5	68.104	5.38	62.724	14.57	10.59
3	45	40	6	69.1	6.362	62.738	11.03	13.07
4	30	50	6	67.287	7.1	60.187	10.7	12.14
5	60	50	4	70.914	5.631	65.283	10.68	9.84
6	45	40	4	65.209	4.79	60.419	14.28	9.26
7	45	40	5	67.825	5.242	62.583	14.57	9.79
8	45	40	5	68.108	5.265	62.843	13.25	8.58
9	60	30	6	72.752	5.488	67.264	11.96	13.07
10	45	40	5	67.218	4.868	62.35	14.33	9.68
11	45	40	5	68.11	5.247	62.836	14.4	10.36
12	60	30	4	69.262	4.285	64.977	19.68	10.41
13	30	30	6	66.087	4.791	61.296	12.8	12.61
14	60	50	6	71.396	7.96	63.436	4.43	10.44
15	45	30	5	68.101	3.765	64.336	11.86	9.22
16	30	30	4	59.42	3.87	55.55	21.25	13.23
17	45	50	5	69.504	6.798	62.706	8.58	8.65
18	30	40	5	64.115	4.868	59.247	16.42	13.18
19	60	40	5	70.53	5.754	64.776	8.49	12
20	30	50	4	63.898	5.905	57.993	15.25	12.89

Table 3. Analysis of variance (ANOVA) of polynomial full quadratic model for osmotic dehydration of Aloe vera gel.

Source	Sum of square	DF	F-value	Prob > F
WL				
Model	170.21	9	229.64	< 0.0001
A	115.92	1	1407.51	< 0.0001
B	5.44	1	66.08	< 0.0001
C	32.11	1	389.87	< 0.0001
A^2	0.76	1	9.28	0.0123
B^2	2.5	1	30.31	0.0003
C^2	1.33	1	16.14	0.0024
AB	3.62	1	43.96	< 0.0001
AC	4.63	1	56.18	< 0.0001
BC	4.94	1	59.97	< 0.0001
Lack of Fit	0.18	5	0.28	0.9041
SG				
Model	19.65	9	29.48	< 0.0001
A	0.67	1	9.01	0.0133
B	12.53	1	169.19	< 0.0001
C	5.21	1	70.37	< 0.0001
Lack of Fit	0.59	5	3.79	0.0850
WR				
Model	135.08	9	133.38	< 0.0001
A	98.99	1	879.74	< 0.0001
B	1.46	1	12.95	0.0049
C	11.45	1	101.73	< 0.0001
A^2	0.93	1	8.31	0.0163
B^2	2.36	1	20.98	0.0010
C^2	2.84	1	25.23	0.0005
AB	2.95	1	26.20	0.0005
AC	7.03	1	62.49	< 0.0001
BC	7.38	1	65.62	< 0.0001
Lack of Fit	0.93	5	4.76	0.0559
VCC				
Model	234.08	9	7.24	0.0024
A	44.86	1	12.49	0.0054
B	77.90	1	21.69	0.0009
C	91.32	1	25.43	0.0005
Lack of Fit	33.73	5	15.52	0.0046
TPC				
Model	42.17	9	5.62	0.0063
A	6.87	1	8.24	0.0167
A^2	14.62	1	17.52	0.0019
B^2	5.01	1	6.00	0.0343
Lack of Fit	5.64	5	2.09	0.2191

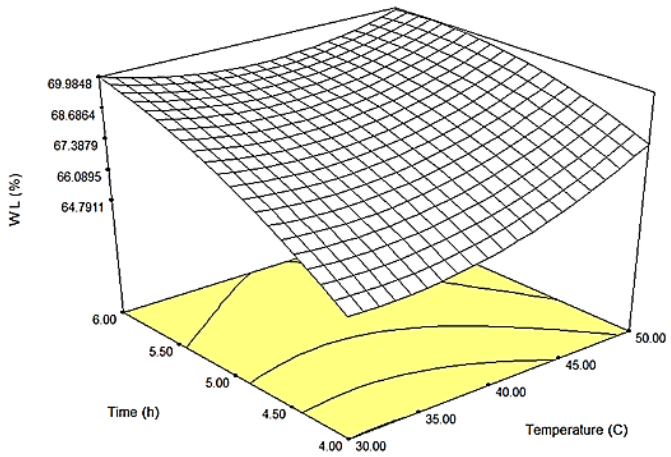

Fig. 1. Response surface for water loss (%) as a function of solution temperature and immersion time.

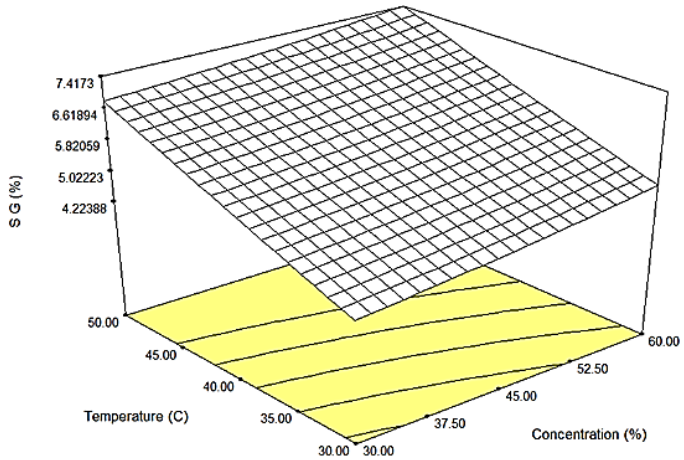

Fig. 2. Response surface for solid gain (%) as a function of solution concentration and temperature.

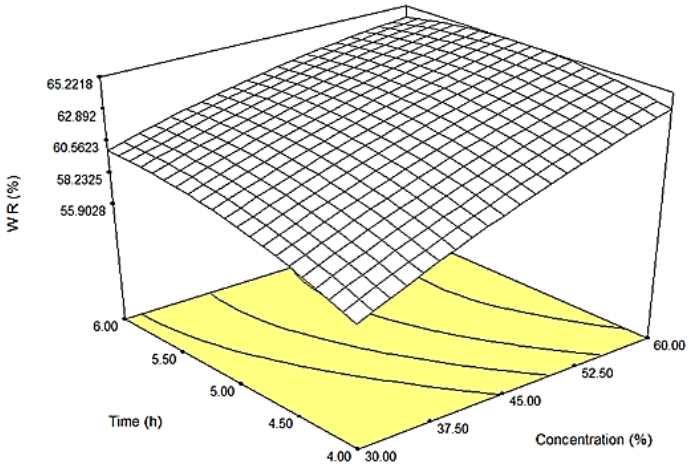

Fig. 3. Response surface for solid gain (%) as a function of solution concentration and temperature.

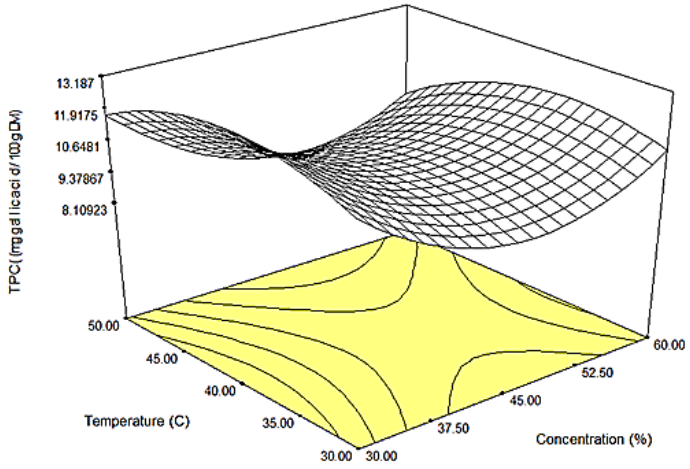

Fig. 4. Response surface for total phenolic content (mg GAE/100g DM) as a function of solution concentration and temperature.

All linear, quadratic and interactive terms of the model had significant effects on the WR ($p < 0.05$) (Table 3). The linear term of the osmotic solution concentration (A) had the highest positive impacts on WR. The linear effects of process time (C) and quadratic effect of solution temperature (B^2) also had positive impacts on WR. In osmotic dehydration, WR is defined as the difference between WL and SG. Similar to linear effects of concentration and process time in addition to the quadratic effect of solution temperature on the rise of WL, the increment in WR could be elucidated by the increase in concentration, time and temperature to higher levels. The quadratic term of process time (C^2) had the highest negative effect on this response. In addition, the linear effect of temperature (B) and quadratic effect of concentration (A^2) had negative effects on WR, too. Fig. 3 shows the effect of concentration and process time on WR.

As shown in the ANOVA table, merely the linear terms had significant effects on the vitamin C ($p < 0.05$) whereas the other terms had no significant effect ($p > 0.05$) and consequently were removed from the model. Among the significant terms, process time followed by the solution temperature and osmotic solution concentration had the highest negative effects on the response. Azoubel et al. (2009) also reported the loss in the vitamin C content during the osmotic dehydration of cashew apple. Considering the high solubility of vitamin C in water, it is expected that the loss in its content would be accompanied by the water removal out of the gel. The R^2 of the fitted model for the vitamin C content was 0.867; nevertheless, the significance of the model lack of fit ($p < 0.05$) exhibited no good fit for this response. Response surface plot of vitamin C was not shown due to the lack of fit to the model.

With regard to reduction of the total phenolic content (TPC), results showed that the quadratic term of solution temperature (B^2) had the largest impact followed by the linear term of concentration (A) which had a negative effect on this response ($p < 0.05$). At the same time, the quadratic effect of concentration (A^2) influenced the TPC positively ($p < 0.05$). The other terms had no significant effect on the response ($p > 0.05$) and were removed from the model. Fig. 4 shows the response surface plot for TPC of osmo-air dried Aloe vera gel. Aloe vera gel contains both hydrophilic and hydrophobic phenolic compounds (Nejatzadeh-Barandozi, 2013). It is hard for the hydrophobic ones to diffuse into water. As a result, loss of the phenolic compounds during the osmotic dehydration of Aloe vera gel could be described via the diffusion of the small hydrophilic phenolic compounds into the osmotic solution. This mechanism of the reduction of the phenolic compounds was reported by Devic et al. (2010) in the osmotic dehydration of apple. Kucner et al. (2013) also declared that increase in the diffusion flow rate leads to higher migration of phenolic compounds to the osmotic solution. With regard to this mechanism besides the negative effect of the quadratic term of concentration on WL, the greater retention of the phenolic compounds in higher concentrations could probably be attributed to the reduction of the WL rate and hence to the reduction of the leakage of the phenolic compounds out of the gel. Likewise, regarding the positive effect of the quadratic term of solution temperature and the linear term of concentration on WL, the greater reduction of the phenolic compounds in low concentrations and high temperature could be justified. The R^2 of the fitted model for the TPC was equal to 0.8348. The non-significance of the model lack of fit ($p > 0.05$) demonstrated its appropriateness.

3.1. Optimization

To achieve a dried product with high quality and nutritional value, the optimization of osmotic dehydration of Aloe vera gel was aimed at maximizing the water loss, weight reduction, TPC and vitamin C content and minimizing the solid gain. The importance of responses was 4, 4, 4, 5 and 5 for WL, SG, WR, vitamin C and TPC, respectively. In terms of health-promoting properties, vitamin C along with other antioxidants such as phenolic compounds, reduce the risk of many diseases, including cardiovascular diseases and certain cancers; stimulate the immune system delay the aging process (Borchani et al., 2011). Therefore, considering the presence of these compounds in Aloe vera gel, it is important that the optimal conditions of the osmotic dehydration of this gel be achieved in a way that most of these functional compounds would be preserved. As a result, maximum importance was given to the vitamin C and TPC. The optimum conditions of the process were found to be: solution temperature of 30.2°C, the osmotic solution concentration of 60% (w/w) and the process time of 349 min. Under these conditions, the amount of WL, SG and WR were predicted as 72.67 (g/100g), 5.33 (g/100g) and 67.34 (g/100g), respectively. The vitamin C and TPC of the osmo-air dried gel were 12.70 mg/100g DM and 11.33 mg GAE/100g DM, respectively.

In order to verify the adequacy of the polynomial full quadratic model, the predicted results were validated. Aloe vera gel was osmotically dehydrated under the optimal conditions and then dried at 70°C. The experimental and predicted results were compared. Experimental results for WL, SG, WR, vitamin C and TPC obtained, were 73.40 ± 0.94 (g/100g), 5.43 ± 0.12 (g/100g), 67.97 ± 1.02 (g/100g), 12.97 ± 0.14 mg/100g DM and11.62 ± 0.76 mg GAE/100g DM respectively. The coefficient of variation (CV %) for WL, SG, WR, vitamin C and TPC also were 1.009, 1.93, 0.93, 2.70 and 2.045 respectively.

4. Conclusion

The selection of proper drying condition is of prime importance for reduced thermal stress and to retain the key compounds in the rehydrated product. In this research, the effect of the osmotic solution concentration, solution temperature and immersion time were examined during the osmotic drying prior to hot air convective drying of Aloe vera gel and eventually the optimum conditions were determined. The polynomial full quadratic model was utilized to predict the behavior of the samples. The optimum conditions of the process were found to be: solution temperature of 30.2°C, the osmotic solution concentration of 60% (w/w) and the process time of 349 min which the preservation of functional ingredients such as vitamin C and phenolic compounds were maximized. In conclusion, results indicated that the fitted model was highly capable of predicting the studied parameters. Furthermore, it enables the manufacture of a product with a long shelf-life and high health-promoting properties. Therefore, the results obtained from this model could be applied in the industrial osmotic dehydration of Aloe vera gel.

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Conflict of interest

The authors declare that there is no conflict of interest.

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