PAPER

MODELING AND OPTIMIZATION OF PROCESS PARAMETERS FOR IMPROVING OSMOTIC DEHYDRATION OF KIWIFRUIT

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ABSTRACT

Osmotic conditions for kiwifruit dehydration were optimized using central composite rotatable design and response surface methodology. The optimal conditions included osmotic time of 4.29 h, sucrose concentration of 70 %, and osmotic temperature of 50 °C. At these optimum values, water loss (WL) exhibited a response value of 45.64 %. The optimized condition was validated and found to be fitted with the experimental values. Quadratic regression equations describing the effects of these factors on WL were developed. The osmotic dehydration of kiwifruit was significantly influenced by osmotic temperature, osmotic time, and sucrose concentration. Moreover, osmotic process at relatively high temperatures caused a significantly depletion of V_c content in kiwifruit.

Keywords: kiwifruit, osmotic dehydration, optimization, response surface methodology, water loss rate

1. INTRODUCTION

Kiwifruit is one of the delicious fruits originating from China. Although with full of phytochemicals, vitamins, and minerals, it has a relatively short shelf-life due to its highly perishable nature (KAYA *et al*., 2010). The short shelf life of fresh kiwifruit after harvest is becoming one of the main factors that affect the rapid development of kiwifruit processing industry. Osmotic dehydration is a potential preservation technique to reduce postharvest losses of fruits and vegetables and produce high-quality intermediate-moisture products (AHMED *et al*., 2016). It is widely used for partial removal of water from food materials as a pretreatment before further processing to improve texture characteristics, sensory, functional and nutritional properties (CHIRALT *et al*., 2001; TORREGGIANI and BERTOLO, 2001; TALENS *et al*., 2002; RASTOGI and RAGHAVARAO, 2004). The use of osmotic dehydration can prolong the shelf life of the kiwifruit, as the water content reduction slows down deteriorative reactions.

In osmotic dehydration process, food materials are used to immerse in concentrated solution creating a concentration gradient between the osmotic solution and food materials, the simultaneous mass transfer phenomena mainly include flow of water from the product to the solution, transfer of solute into the product, and leaching of the components of the product. The water is mainly removed by capillary flow and diffusion; meanwhile, leaching and solute uptake occur through diffusion (SHI and XUE, 2009). The rate of mass transfer during osmotic dehydration can be influenced by many factors, such as type and concentration of osmotic agents, temperature, agitation/circulation of solution, food to solution ratio, food structure, shape and size, thickness of food material, and pre-treatment (DA CONCEICAO *et al*., 2012; AKBARIAN *et al*., 2013). Osmotic temperature and solution concentration are the important factor, which affects osmotic mass transfer (TORTOE, 2010). LOMBARD *et al*. (2008) investigated the influence of the process temperature, pressure and osmotic concentration on the mass transfer process during the osmotic dehydration of South African grown Cayenne type pineapple pieces, and the results showed water loss and solids gain increased with temperature and concentration. FALADE *et al*. (2007) studied the osmotic mass transfer phenomenon of water melon slabs using three different concentrations of sucrose solution (40, 50 and 60°Brix). The water loss and solid gain of the watermelon slabs treated with the higher osmotic solution concentration were found to be higher. CAO *et al*. (2006) found that the optimal conditions for osmotic dehydration of kiwifruit slice were 60% sucrose concentration, 30–40° C osmotic temperature, 150 min osmotic time, and 8 mm slice thickness. Meanwhile, the influence of each factor or interactions among the factors should be determined to understand the behavior involved in mass transfer during osmotic dehydration. Individual screening of these factors at a time is laborious and requires much experimental work (FERNANDES *et al*., 2006). Therefore, an optimization technique for osmotic dehydration parameters must be established.

Response surface methodology (RSM) is an effective mathematical and statistical tool. It not only defines the effect of independent variables but also their interaction effects (MYERS and MONTGOMERY, 1995). The present study aims to determine the optimal osmotic dehydration conditions of independent variables (osmotic temperature, osmotic time, and sucrose concentration) for kiwifruit and validate the optimized conditions based on water loss rate by using RSM coupled with central composite rotatable design. In addition, the effects of different sucrose concentrations on kiwifruit water loss (WL) and solid gain (SG) rates were analyzed.

2. MATERIALS AND METHODS

2.1. Sample preparation and osmotic treatment

Fresh kiwifruits of *xuxiang* cultivar were obtained directly from a producer from qinyuan orchard located at *Mei* county (*Shaanxi*, China). The average values of single weight, titratable acidity, and total soluble solid contents in the kiwifruits were 94.2±0.2g, 1.32± 0.34% and 16.2±0.65 Brix, respectively. The kiwifruits were washed and cut into cubes (1 cm × 1 cm × 1 cm) to prepare samples. Then the cube samples were subjected to osmotic dehydration under different osmotic temperatures, osmotic times, and sucrose concentrations based on the experimental design shown in Table 1. The ratio of the sample to the osmotic solution was 1:5 (wt/wt). In order to ensure concentration of the osmotic solution did not change significantly during the experiment, the osmotic system in a vessel was covered with a wrap to prevent evaporation without agitation. The temperature was controlled using a constant temperature water bath. After the osmotic treatment, the samples were removed from the osmotic solution, washed with distilled water, and blotted gently with a tissue paper to remove adhering water for the next analysis (ALI *et al.*, 2010; TYLEWICZ *et al.*, 2011).

2.2. Central composite rotatable design for optimizing process parameters during kiwifruit osmotic dehydration

A central composite rotatable design was used to optimize the conditions for osmotic dehydration of kiwifruit cubes. Osmotic temperature (23-57°C), osmotic time (2.3-5.7h), and sucrose concentration (43-77%, w/w) were taken as independent variables to optimize WL rate and determine the efficiency of osmotic dehydration. The experimental data were fitted using multiple linear regression in Equation (1) (BAŞ and BOYACI, 2007; PENG *et al.*, 2015):

$$Y = b_0 + \sum_{i=1}^{3} b_i X_i + \sum_{i=1}^{3} b_{ii} X_i^2 + \sum_{i=1}^{3} \sum_{j=(i+1)}^{3} b_{ij} X_i X_j, \quad (1)$$

where Y is the WL rate, *i and j* are the linear and quadratic coefficients, respectively, X_i and X_j represent the independent variables, and b_0, b_i, b_{ii}, and b_{ij} are the regression coefficients.

2.3. Mass transfer determination

The process kinetic variables of WL and SG rates of the samples were calculated as described by SINGH *et al.* (2007) and FALADE *et al.* (2007) by using Equations (2) and (3) with some minor adjustments:

$$WL\% = \frac{(M_0 - m_0) - (M_t - m_t)}{M_0} x100\% \quad (2)$$

$$SG\% = \frac{m_t - m_0}{M_0} x100\% \quad (3)$$

where M_0 and m_0 are the initial mass weights of the kiwifruit samples and the dry solid mass in the samples (g), respectively; M_t and m_t are the mass weights of the samples and the dry solids (g) in the samples after the osmotic dehydration time *t*.

2.4. Analytical determination

Moisture content was determined gravimetrically using a vacuum oven by drying to constant weight (AOAC, 1997).
Ascorbic acid (Vitamin C, V_c) in kiwifruit is the most important vitamin for human nutrition. A standard ascorbic acid solution method was used to determine V_c of kiwifruit based on the titration of ascorbic acid with 2,6-dichloroindophenol in acidic solution by the AOAC's official titrimetric method (AOAC, 1990). The analysis was done in triplicate, and the result for each sample was averaged.

2.5. Statistical analysis

All tests were run in triplicate. Analysis of variance (ANOVA; Origin software, OriginLab Corporation, Northampton, MA, USA) was used to indicate significant differences among tests. Differences were considered significant at the $p \leq 0.01$ level.

3. RESULTS

3.1. Model fitting

In this study, central composite rotatable design coupled with RSM was used to optimize osmotic dehydration for kiwifruit cubes. The response of WL rate was selected on the basis that the response directly influenced the following drying efficiency of the product. The three independent variables, namely, osmotic time, sucrose concentration, and osmotic temperature (coded *A*, *B*, and *C*, respectively) were used to optimize the response of WL rate coded *Y*. The experimental design and obtained values are shown in Table 1. Regression analysis of the response was conducted by fitting a suitable quadratic model in the case of the response variable to assess how well the model represented the data. The results of the analysis of variance (ANOVA) are shown in Tables 2 and 3. According to the estimated regression coefficients of the quadratic polynomial model in Table 2, non-significant factors were removed. The regression model equation in terms of coded value was obtained to express the relationship between the investigated factors and WL rate:

$$Y=37.32 + 2.54 \times A + 2.86 \times B + 8.24 \times C - 0.21 \times A \times B - 0.98 \times \times A \times C - 0.34 \times B \times C - 2.31 \times A^2 - 0.82 \times B^2 - 1.82 \times C^2 \quad (4)$$

The regression model was a function of changes in sucrose concentration, osmotic temperature and time. The *F*-value of 38.80 implied that the model was very significant ($p < 0.01$) and accurately predicted the WL rate of the samples. Moreover, the R^2 of 0.7884 for the model is in reasonable agreement with the adjusted R^2 of 0.9471, and the adequate precision of 21.532 indicates that the model has an adequate signal to noise ratio (Table 3).
As shown in Table 2, osmotic time, sucrose concentration, and osmotic temperature significantly affected the kiwifruit dehydration rate ($p < 0.01$); the model of $Prob > F$ and less than 0.01 indicated that the regression equation exhibited high significance and reliability. Meanwhile, the R^2 of the regression model was found to be 0.972, greater than 90%, indicating the significant relationship between the independent variable and the response value (Table 3).

Table 1. Experimental design and measured values of WL rate for osmotic dehydration of kiwifruit.

No.	*A*-Osmotic time /h	*B*-Sucrose concentration/%	*C*-Osmotic temperature/°C	*Y*-Water loss rate*/%
1	4.00(0)	76.82(+1.68)	40.00(0)	41.20±1.84
2	4.00(0)	60.00(0)	40.00(0)	37.31±1.53
3	5.00(+1)	50.00(-1)	50.00(+1)	40.44±1.97
4	3.00(-1)	50.00(-1)	50.00(+1)	38.41±1.25
5	4.00(0)	60.00(0)	23.18(-1.68)	20.65±1.13
6	5.00(+1)	50.00(-1)	30.00(-1)	23.23±0.89
7	2.32(-1.68)	60.00(0)	40.00(0)	24.64±1.54
8	4.00(0)	43.18(-1.68)	40.00(0)	29.09±1.32
9	4.00(0)	60.00(0)	56.82(+1.68)	44.02±1.28
10	4.00(0)	60.00(0)	40.00(0)	37.31±1.05
11	5.68(+1.68)	60.00(0)	40.00(0)	37.26±1.57
12	4.00(0)	60.00(0)	40.00(0)	37.31±1.44
13	5.00(+1)	70.00(+1)	50.00(+1)	43.81±1.58
14	3.00(-1)	70.00(+1)	30.00(-1)	23.23±1.88
15	3.00(-1)	50.00(-1)	30.00(-1)	17.67±1.05
16	4.00(0)	60.00(0)	40.00(0)	37.31±2.15
17	3.00(-1)	70.00(+1)	50.00(+1)	43.01±1.86
18	4.00(0)	60.00(0)	40.00(0)	37.31±1.43
19	5.00(+1)	70.00(+1)	30.00(-1)	28.34±1.62
20	4.00(0)	60.00(0)	40.00(0)	37.31±1.45

( ) Coded levels for actual values of different parameters during osmotic dehydration of kiwifruit.
*Each combination with triplicate and water loss rate expressed by average value ± standard deviation.

Table 2. ANOVA of WL rate regression model for osmotic dehydration of kiwifruit.

Source	Sum of Squares	Degree of Freedom	Mean Square	*F* Value	*p*-value*
Model	1251.66	9	139.07	38.80	<0.0001
A	88.29	1	88.29	24.63	0.0006
B	111.41	1	111.41	31.08	0.0002
C	926.79	1	926.79	258.54	<0.0001
AB	0.35	1	0.35	0.098	0.7602
AC	7.68	1	7.68	2.14	0.1739
BC	0.91	1	0.91	0.25	0.6251
A^2	76.60	1	76.60	21.37	0.0009
B^2	9.75	1	9.75	2.72	0.1302
C^2	47.52	1	47.52	13.26	0.0045
Residual	35.85	10	3.58		
Lack of Fit	35.85	5	7.17		
Pure Error	0.000	5	0.000		
Total	1287.51	19			

A: Osmotic time (h); B: Sucrose concentration (%); *C*: Osmotic temperature (℃).
*p-Values less than 0.01 indicate model terms are significant, and values greater than 0.1 indicate the model terms are not significant.

Table 3. ANOVA for response surface quadratic model.

Terms	Values
Standard deviation	1.89
Mean	33.94
Coefficient of variation (%)	5.58
R^2	0.9722
Adjusted R^2	0.9471
Predicted R^2	0.7884
Adequate precision*	21.532

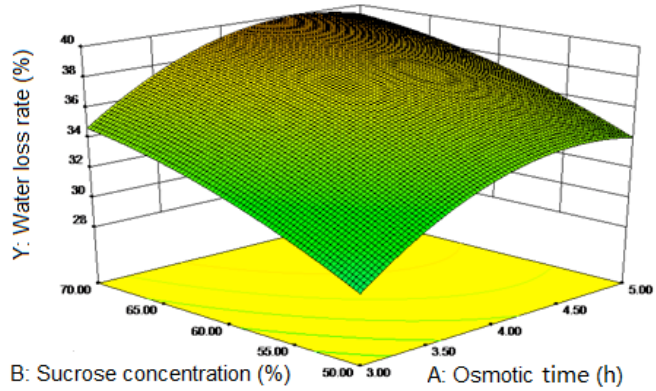
*Adequate precision measures the signal to noise ratio. A ration greater 4 is desirable.

3.2. Linear effect of osmotic variables on WL rate for kiwifruit dehydration

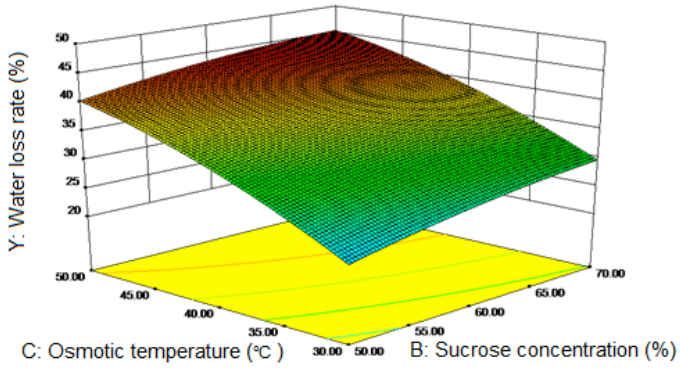
Osmotic time, sucrose concentration, and osmotic temperature significantly affected ($p < 0.01$) the WL rate of the samples at the linear level (Table 2). The coefficients of linear terms in the regression equation (Equation 4) indicated that the WL rate of the samples was mainly influenced by osmotic temperature ($p \leq 0.01$), followed by sucrose concentration ($p \leq 0.01$) and osmotic time ($p \leq 0.01$). In addition, the quadratic terms of osmotic temperature and time ($p < 0.05$) had significant effects, while the interaction of factors had no significant effect ($p > 0.05$) on WL rate within the investigated range (Table 2).

3.3. Interactive effect of osmotic variables on WL rate for kiwifruit dehydration

Considering the interactive effect of osmotic variables, Fig.1 shows the response surface plot and contour plot of kiwifruit WL rate under the effects of input parameters of osmotic time, sucrose concentration, and osmotic temperature. Some profiles for the quadratic response surface plot in the optimization of the two parameters were obtained by keeping the other parameter at zero levels for WL rate. As shown in Fig.1a, the WL rate first gradually increases with increasing osmotic time and sucrose concentration and subsequently maintains a steady state. This trend may be rationalized by considering that the intracellular free water movement speed in kiwifruit accelerates with increasing sucrose concentration. The WL rate will gradually decrease with decreasing amount of free water. When the osmotic pressure between the solution and the internal kiwifruit cells reach the equilibrium, the WL rate will not change. Fig. 1b and 1c demonstrate the same trends that the WL rate first increases and subsequently maintains a steady state under the interaction between two parameters. At lower sucrose concentration with increasing osmotic temperature, the WL rate increases gradually, but as the sucrose concentration increases, the WL rate increases rapidly with increasing osmotic temperature (Fig.1b). Similarly, the interaction between osmotic temperature and time showed similar positive correlation (Fig.1c).This might be due to higher temperature led to swelling and plasticizing cellular membrane and rapider release of moisture from the kiwifruit cells, and viscosity of the sucrose solution was lower at higher temperature, which improved water loss from common surface of kiwifruit and osmotic solution.



A



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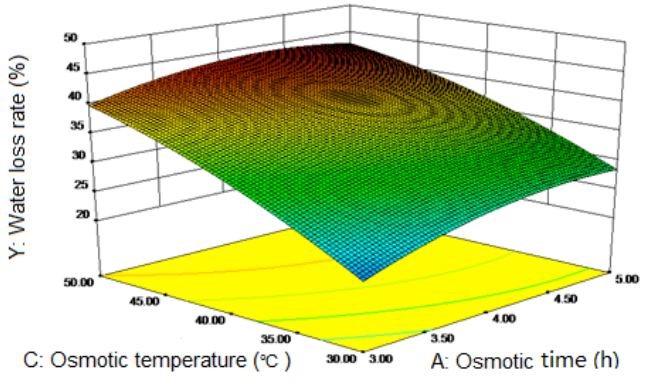

C

Figure 1. Response surface and contour plots for response of kiwifruit water loss rate during osmotic dehydration (**a**: the interaction between the osmotic time and sucrose concentration; **b**: the interaction between the sucrose concentration and the osmotic temperature; and **c**: the interaction between the osmotic time and temperature).

3.4. Determination and experimental validation of optimal conditions

Process parameters can be optimized by finding the stationary point of the model equation in the ranges of tested independent parameters (PENG *et al.*, 2015). The optimal conditions were determined by maximizing the desirability of the response using Design Expert

software (version 6.0.4 by Stat-Ease, Inc., MN, USA). The optimal conditions included osmotic time of 4.29 h, sucrose concentration of 70 % , and osmotic temperature of 50 ℃ with a predicted response value of 45.64 % for WL rate. A confirmation test was conducted using the optimum parameters identified by RSM to verify the adequacy of the regression models. The fitted values predicted by the models were compared with the experimental data. Under these optimal conditions, the experimental value of WL rate is consistent with the predicted value with 3.89 % standard deviation (Table 4). These values did not show any significant difference ($p > 0.05$). Response surface method is reasonable and effective for optimization of WL rate of kiwifruit.

Table 4. Optimal conditions and validation.

Osmotic time (h)	Osmotic temperature (°C)	Sucrose concentration (%)	Predicted water loss rate (%)	Experimental water loss rate (%)*	Standard deviation (%)
4.29	50	70	45.64	43.81	3.89

*Experimental water loss rate expressed by average value with triplicate to eliminate the errors.

3.5. Effect of sucrose concentration on WL and SG rates

Change in WL and SG rates for osmotic dehydration of kiwifruit under different sucrose concentrations was showed in Fig. 2. The results showed that the WL and SG rates have similar trends in 50%, 60%, and 70% sucrose concentrations. Osmotic time had a substantial effect on mass transfer kinetics. Increasing the time increased the percentage of water loss and solid gain. From Fig. 2a, WL rate rapidly increased in the first 5 h of osmosis, then increasing slowly. This phenomenon is due to the largest pressure difference between the kiwifruit cells and the surrounding hypertonic solution, thereby promoting the osmotic dehydration of kiwifruit in the initial stage of the penetration process and inducing rapid diffusion of the water molecules. As osmotic time continues, the pressure difference gradually decreases and the structural changes in kiwifruit tissues gradually occur, the mass transfer tends to reach the dynamic equilibrium state. The WL rate increases with increasing sucrose concentration, and higher concentrations of osmotic solution could facilitate the removal of moisture from the texture of food product and resulted in lower moisture contents and higher WL rate from the texture, consistent with some other reports. LENART (1992) reported that increasing the concentration of an osmotic solution led to high WL rate until the equilibrium level was achieved; by contrast, low-concentrated sucrose solution led to small WL and SG rates (TORTOE, 2010). Similarly, RAMASWAMY (2005) studied the effect of osmotic time on mass transfer, and the results showed that mass exchange occurred at a faster rate within the initial 2h followed by a reduction in drying rate during further processing time.

The kiwifruit SG rate showed similar trends in 40%, 60%, and 80% sucrose concentrations (Fig. 2b). The SG rate increased continuously throughout the osmotic time in the test range, and the increase in the sucrose concentration could raise the SG rate. High concentration promotes sucrose mass transfer from the solution to the kiwifruit cells. The concentration of an osmotic agent affects the mass transfer kinetics during osmotic dehydration (HERMAN-LARA *et al.*, 2013). The difference in osmotic potential between the solution and the fruit sample resulted in a high diffusion rate of the solute and water (AZOUBEL and MURR, 2004; PHISUT, 2012). Similarly, LAZARIDES (1994) reported that apple processed at a temperature of 30 and 50°C resulted in higher sugar gain (up to 55%)

compared to room temperature condition. It is due to the swelling of membrane and plasticizing effect, which enhances the permeability of the membrane.

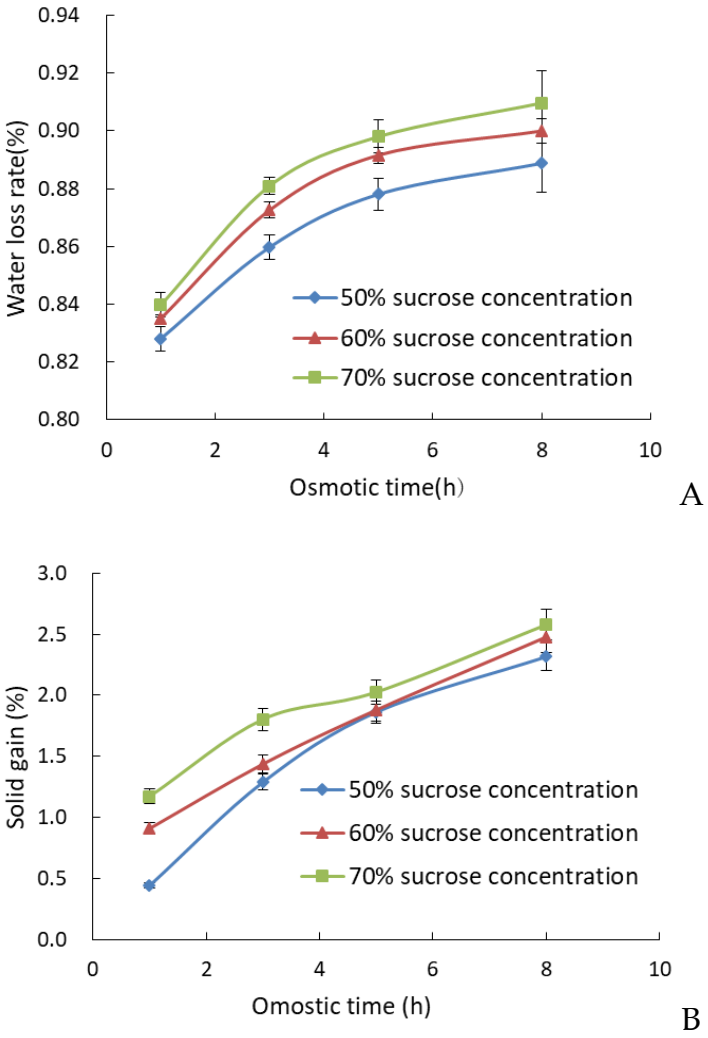

Figure 2. Variation of WL and SG rates with time during kiwifruit osmotic dehydration under different sucrose concentrations at 50℃.

3.6. Change of V_c content in untreated and osmotic treated kiwifruits

Comparison of ascorbic acid (V_c) content in untreated and osmotic treated kiwifruit samples at different osmotic temperatures was showed in Fig. 3. The V_c content of kiwifruit was significantly decreased by osmotic dehydration. It may be that V_c is transferred from the kiwifruit to the osmotic solution with the water molecule moving from the inside of the kiwifruit during the osmotic process. Moreover, the V_c content of osmotic treated kiwifruit was decreased significantly with increasing the osmotic temperature, this may be because the internal molecular movement in kiwifruit osmotic system at high temperature were much faster, which accelerated the loss of internal V_c molecules. This result is in agreement with the report by CAO *et al.*(2006), which osmotic temperature was the most significant factor affecting the ascorbic acid loss. However, CHAKRABORTY and SAMANTA (2016) found that the optimally dehydrated kiwifruit

demonstrated a significant increase in the ascorbic acid content by simultaneous osmotic dehydration using fructose as osmotic solution and vacuum drying under far - infrared radiation.

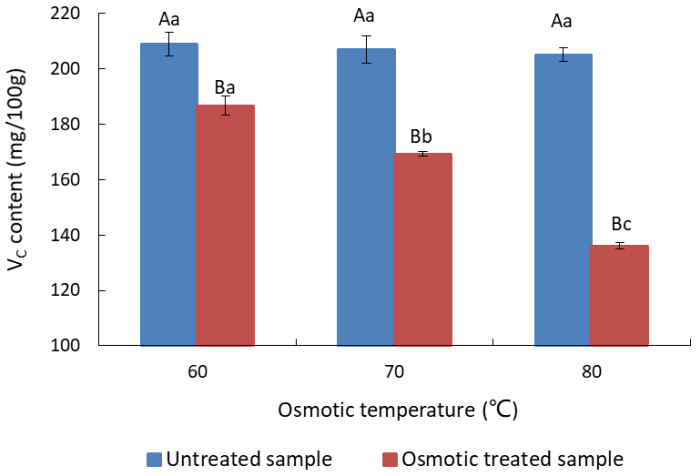

Figure 3. Comparison of V_c content in untreated and osmotic treated kiwifruit samples at different osmotic temperatures. Bars with different capital letters at each osmotic temperature are significantly different at $p< 0.05$. Bars with different small letters at treated samples are significantly different at $p< 0.05$.

4. CONCLUSIONS

The optimization of the osmotic conditions for kiwifruit dehydration was successfully examined using the RSM. The optimal conditions comprised osmotic time of 4.29 h, sucrose concentration of 70 %, and osmotic temperature of 50 ℃ with a response value of 45.64 % for the WL rate. The WL rate of the kiwifruit cubes was mainly influenced by osmotic temperature ($p \leq 0.01$), followed by sucrose concentration ($p \leq 0.01$) and osmotic time ($p \leq 0.01$). Moreover, the V_c content was decreased significantly with increasing the osmotic temperature. The optimized condition was validated and found to be fitted with the experimental values. Therefore, osmotic dehydration of kiwifruit highly depends on osmotic temperature, osmotic time, and solvent concentration. The predicted model for WL rate established by the response surface quadratic regression provided an adequate mathematical description of kiwifruit osmotic dehydration.

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