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EFEECTS OF PROCESS VARIABLES ON MASS TRANSFER DURING OSMOTIC DEHYDRATION OF GINGER SLICES USING CARBOXYMETHYL CELLULOSE AS AN EDIBLE COATING MATERIAL

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Abstract: Fresh ginger (*Zingiber officinale*) has a high moisture content, which makes it susceptible to deteriorate. Therefore, drying is an important processing method for water removal that reduces water activity and extends the shelf life of food products. On top of that, it can help to ensure a more sustainable food system. Edible coating prior osmotic dehydration has the potential to enhance the drying performance of ginger slices. In this study, the effect of carboxymethyl cellulose (CMC) as an edible coating on mass transfer of ginger slices during osmotic dehydration was quantitatively investigated. The influences of CMC concentrations (1-3% w/w) as edible coating solution, sucrose concentrations (40-60% w/w) with 2% w/w salt as osmotic solution, and immersion time (30-150 min) on water loss and solute gain during osmotic dehydration of ginger slices were investigated using response surface methodology. It was found that only CMC concentration had no significant effect on the water loss whereas in solute gain, all the process variables were significant. The optimum conditions to obtain water loss of 53% with a solute gain of 4.5% were found to be at 2.7% w/w CMC coating, immersed in 50% w/w sucrose solution with 2% salt solution for 150 min. The establishment of this process condition can provide useful information for further drying to achieve the energy-saving process.

Keywords: Sustainable, mass transfer, carboxymethyl cellulose; osmotic dehydration, coating.

Introduction

Utilization of local food resources efficiently plays a role in contributing to the achievement of sustainable development goals (SDGs). Ginger (*Zingiber officinale)*, a member of the Zingiberaceae family and is generally cultivated in South Asia, East Africa and the Caribbean (Rehman *et al*., 2011). In Malaysia, the production of ginger has increased steadily since 2011 and reaches about 14,278 tonnes in 2017 (Department of Agriculture Malaysia, 2018). Commonly, ginger is consumed locally as fresh. However, fresh ginger is perishable owing to its high moisture content (Lv *et al*., 2016). Thus, without proper storage, it is susceptible to microbial contamination which leads to product deterioration. Hence, drying is one of the key unit operation required to prolong the shelf life by inhibiting microbial growth. Dried ginger is commonly used as a spice and medicine (Deshmukh *et al*., 2014; An *et al*., 2016). In spite of that, a wide variety of dried ginger product can be developed through innovative drying technologies such as snack food and also as an additional ingredient in ready-to-eat soup, energy bar and cereals, which in turn help to increase and diversify its availability and generate more income to the ginger producers.

Osmotic dehydration is a water removal process that utilizes the osmosis phenomenon when the food products immersed in a hypertonic solution. This process will not result in sufficiently low moisture content to be considered as a shelf-stable product. Hence, it has often been applied as a pre-treatment step before the finish drying process. Water removal without a phase change can enable less energy requirement which has becomes a major concern in the drying process in terms of economic consideration (Chua & Chou, 2014). Lewicki & Lenart (1995) reported that osmotic dehydration requires only 0.1-2.4 MJ per kg of removed water compared to convective drying that needs about 5 MJ per kg of evaporated water. The previous study done by Md Salim *et al*. (2016b) shows that application of osmotic dehydration as pre-treatment can reduce about 68 % in energy input required for the production of dried broccoli stalk compared to the untreated process. Furthermore, osmotic dehydration was found advantageous in improving the quality of the food product. Dermesonlouoglou *et al*. (2018) reported that by applying osmotic pre-treatment before air drying of goji berry results in improved texture characteristics and higher retention in colour, antioxidant capacity and total phenolic content. Meanwhile, Cano-Lamadrid *et al.* (2017) in their study found that osmotic dehydration improved the rehydration rate, antioxidant capacity, colour and the sensory profile of dried pomegranate arils.

During the osmotic dehydration process, osmotic pressure acts as a driving force across the cell wall of the food products. This pressure results in cell membrane disintegration that caused the occurrences of mass transfer processes (Rastogi *et al*., 2002). The two major mass transfer occurs are the water outflow from the product and the solute inflow from solution to product concurrently. The rate of mass transfer was depended upon various operating variables such as the temperature, type of osmotic agent, osmotic solution concentration, immersion time, size and geometry of the food sample, the ratio of solution to product and the level of agitation. There are numerous publications in a different type of food materials that have been carried out to evaluate the mass transfer. However, it is still difficult to generalize the operating conditions as it depends not only on the operating variables but also on the cellular tissue type of the sample itself (Eren & Kaymak-Ertekin, 2007). Therefore, optimization on the osmotic dehydration processing variables are becomes an essential step to achieve the desired output of the process (Zhao *et al*., 2014; da Costa Ribeiro *et al*., 2016; de Mendonça *et al*., 2016; Md Salim *et al*., 2016a; Liu *et al*., 2018).

Apart from that, the other challenge in osmotic dehydration process is the excessive invasion of solute from the osmotic solution into the food itself gives negative impact on the final product especially in the textural and organoleptic qualities, which often becomes the limitation for this technique to be applied in food industries (Rahimi *et al*., 2013; Jansrimanee & Lertworasirikul, 2017). To overcome this problem, edible coating before osmotic dehydration has been studied extensively for lowering the solute gain. The coating serves as an extra barrier to the mass transfer during the osmotic dehydration process. The various edible coating is available nowadays and carboxymethyl cellulose (CMC) is one of the potential edible coatings that have been implemented for minimizing the solute gain without affecting the performance of water loss (Dehghannya *et al*., 2006; Khin *et al*., 2007). CMC is a water-soluble cellulose derivative which is known for their good film-forming ability, biocompatibility, biodegradability, hydrophilicity and also nontoxicity (Su *et al*., 2010). To date, studies on CMC coating on performance of mass transfer during osmotic dehydration of ginger slices are still limited in the literature.

This study aimed is to investigate the effects of CMC concentration, sucrose concentration and immersion time on the mass transfer during osmotic dehydration process of ginger slices and to find the optimum operating conditions that maximize the water loss and minimize the solid gain.

Material and Methods

Ginger was purchased from a local market in Kuala Nerus, Terengganu, Malaysia. The ginger was washed, peeled and sliced to a uniform shape with a thickness of 4 mm and diameter of 15 mm. Moisture content of samples were determined by drying in a hot air oven at 105 °C for until constant weight is reached (AOAC, 2000). The coating solution was prepared by dissolving the CMC (Acros Organics, Geel, Belgium) with distilled water as described by Rahimi *et al.* (2013) at three different concentration, 1, 2 and 3 % w/w. The samples were individually dipped in the CMC solution before placed in a hot air oven at 70 °C for 10 min to decrease the surface moisture. The osmotic solutions were prepared by mixing the sucrose concentration (40, 50 and 60 % w/w) with 2 % w/w salt solution. The selection of CMC and sucrose concentration was based on the preliminary study. The weighed CMC coated ginger slices were then immersed into the container filled with sucrose and kept within the required time. Samples were then removed from the osmotic solution and rinsed immediately in flowing water, drained on a tissue paper to remove surface moisture. The weight and moisture content of the sample before and after the osmotic process were determined. In each experiment, fresh sucrose solution was used. A ratio of sample to the solution was kept constant at 1:10.

Mass Transfer Evaluation

The evaluation of mass transfer of the sample and solution between coated ginger slices and osmotic solution during the dehydration process known as water loss (WL) and solute gain (SG), respectively. The WL and SG were calculated according to the following equations (Hawkes & Flink, 1978):

$$WL\,(\%) = \frac{w_{wo} - (w_t - w_{st})}{w_{wo} + w_{so}}\; x\; 100 \qquad (1)$$

$$SG\,(\%) = \frac{w_{st} - w_{so}}{w_{wo} + w_{so}}\; x\; 100 \qquad (2)$$

Where, W_{wo} is the mass of water in sample before dehydration (g), W_t is the mass of the sample after dehydration (g), W_{so} is the mass of the solids in the sample before dehydration (g) and W_{st} is the mass of the solids in the sample after dehydration (g).

Experimental Design and Statistical Analysis

Response surface methodology (RSM) was used to investigate the influence of different process variables on the mass transfer during the dehydration process and to optimize the process condition for osmotic dehydration of CMC coated ginger slices. The experiment was designed according to face centred composite design with three independent variables; CMC concentration (X_1), sucrose concentration (X_2), and immersion time (X_3) at three levels using commercial statistical package Design Expert 11 free-trial version (Stat-ease, Inc., Minneapolis, MN, USA) as shown in Table 1.

The WL (Y_1) and SG (Y_2) obtained from the experiment are the dependent variables, also referred as responses were subjected to an analysis of variance (ANOVA) to determine the significant effects of independent variables on each response. The following quadratic model was used for the statistical analysis:

Table 1: Process variables and the levels for the osmotic dehydration of CMC coated ginger slices

Independent Variables	**Symbol Coded *(Xi)***	**Range and levels**		
		Low Level -1	**Center 0**	**High Level +1**
CMC concentration (w/w %)	X_1	1	2	3
Sucrose concentration (w/w %)	X_2	40	50	60
Immersion time (min)	X_3	30	90	150

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{11}{X_1}^2 + b_{22}{X_2}^2 + b_{33}{X_3}^2 + b_{12}X_1X_2 + b_{13}X_1 + b_{23}X_2X_3 \quad (3)$$

Where, Y is the response (i.e. WL and SG); are the regression coefficients; X_1, X_2 and X_3 are the coded independent variables.

Results and Discussion

Influence of Process Variables on WL and SG

The experimental results obtained from the calculation (Equation 1 and 2) for different runs of osmotic dehydration conditions are presented in Table 2. The WL was a range from 22.11 to 57.51 with a ratio of maximum to a minimum is 2.60. Meanwhile, for SG, the range obtained was 1.47 to 7.92 with a ratio of maximum to minimum of 5.39.

The quadratic equation, as given in Equation 3 was fitted with the experimental data in order to predict the WL and SG as a function of CMC concentration, sucrose concentration and immersion time. The ANOVA of the quadratic model for the three independent variables on WL and SG are presented in Table 3 and Table 4, respectively. It can be seen that the regression coefficient using least square fit for WL is 0.99 and for SG is 0.98. These high correlation value obtained for both responses indicate a good fit of experimental data to Equation 3. The p-value less than 0.0500 indicate model terms are significant.

Based on the results in Table 3, WL was affected by sucrose concentration linearly.

Table 2: Experimental conditions with values of response variables

	Independent Variables			Dependent Variables	
Run #	CMC Concentration, X_1 (w/w %)	Sucrose Concentration, X_2 (w/w %)	Immersion Time, X_3 (min)	Water loss (WL)	Solute gain (SG)
1	1 (-1)	40 (-1)	30 (-1)	23.50	3.39
2	1 (-1)	60 (+1)	30 (-1)	33.83	4.56
3	3 (+1)	40 (-1)	30 (-1)	22.11	1.47
4	3 (+1)	60 (+1)	30 (-1)	32.96	3.99
5	1 (-1)	40 (-1)	150 (+1)	48.6	6.54
6	1 (-1)	60 (+1)	150 (+1)	57.18	7.92
7	3 (+1)	40 (-1)	150 (+1)	47.09	4.65
8	3 (+1)	60 (+1)	150 (+1)	57.51	5.31
9	2 (0)	40 (-1)	90 (0)	36.12	4.12
10	2 (0)	60 (+1)	90 (0)	48.99	5.88
11	1 (-1)	50 (0)	90 (0)	43.00	5.78
12	3 (+1)	50 (0)	90 (0)	43.43	4.19
13	2 (0)	50 (0)	30 (-1)	25.44	2.33
14	2 (0)	50 (0)	150 (+1)	55.11	4.74
15	2 (0)	50 (0)	90 (0)	44.05	3.79
16	2 (0)	50 (0)	90 (0)	44.75	4.13

Table 3: ANOVA of the quadratic model for WL in osmotic dehydration of CMC-coated ginger slices

Source	*df*	Coefficients	Sum of Squares	F-value	p-value
Model	9	43.54	1949.12	73.46	<0.0001
Linear					
X_1	1	-0.30	0.91	0.31	0.5994
X_2	1	5.30	281.43	95.46	<0.0001
X_3	1	12.77	1629.45	552.72	<0.0001
Quadratic					
X_1^2	1	-0.10	0.03	0.01	0.9286
X_2^2	1	-0.56	0.83	0.28	0.6147
X_3^2	1	-2.84	21.28	7.22	0.0362
Interactions					
$X_1 X_2$	1	0.30	0.70	0.24	0.6442
$X_1 X_3$	1	0.14	0.15	0.05	0.8314
$X_2 X_3$	1	-0.27	0.59	0.20	0.6693
Residual	6		17.69		
Lack of Fit	5		17.44	14.24	0.1984
Pure Error	1		0.25		
Corrected Total	15		1966.81		

df= degree of freedom

Meanwhile, the immersion time is affected linearly and quadratically. On the other hand, it was found that all the independent variables were affected by linearly and quadratically for SG, as shown in Table 4. However, no significant interaction effects for both responses were observed in this study. To visualize the effects of each variable on WL and SG, response surface curves were generated (Figures 1 and 2). This surface plot reflects the influence of two independent variables on WL and SG, while the third variable was kept constant at their central point.

The effect of CMC and sucrose concentration on WL and SG can be seen in Figure 1. For CMC concentration, no significant effects were observed on WL during the osmotic dehydration process which in line with results reported by Rahimi *et al.* (2013). In contrast, in this study, the SG was found to decrease as the CMC concentration increased. The decrease in SG is much more pronounced at higher CMC concentrations which presented an improved physical barrier through the film formation. This result was similar to the findings reported by Dehghannya *et al.* (2006) on apple slices, where the SG was decreased and WL was minimal as the CMC concentration increased. Thus, these results confirmed that CMC can be used in relation to the formation of barriers for solute penetration without affecting the performance of WL during the osmotic dehydration process. Meanwhile, the WL and SG rise steadily as the sucrose concentration increased. This is due to the high osmotic driving force between the osmotic solution and the sample results in

Table 4: ANOVA of the quadratic model for SG in osmotic dehydration of CMC-coated ginger slices

Source	*df*	Coefficients	Sum of Squares	F-value	p-value
Model	9	4.20	35.46	26.19	0.0004
Linear					
X_1	1	-0.86	7.36	48.94	0.0004
X_2	1	0.75	5.61	37.29	0.0009
X_3	1	1.34	18.01	119.72	< 0.0001
Quadratic					
X_1^2	1	0.66	1.16	7.74	0.0319
X_2^2	1	0.68	1.22	8.09	0.0294
X_3^2	1	-0.79	1.63	10.81	0.0166
Interactions					
$X_1 X_2$	1	0.08	0.05	0.33	0.5866
$X_1 X_3$	1	-0.25	0.51	3.36	0.1166
$X_2 X_3$	1	-0.21	0.34	2.26	0.1833
Residual	6		0.90		
Lack of Fit	5		0.84	2.92	0.4160
Pure Error	1		0.06		
Corrected Total	15		36.37		

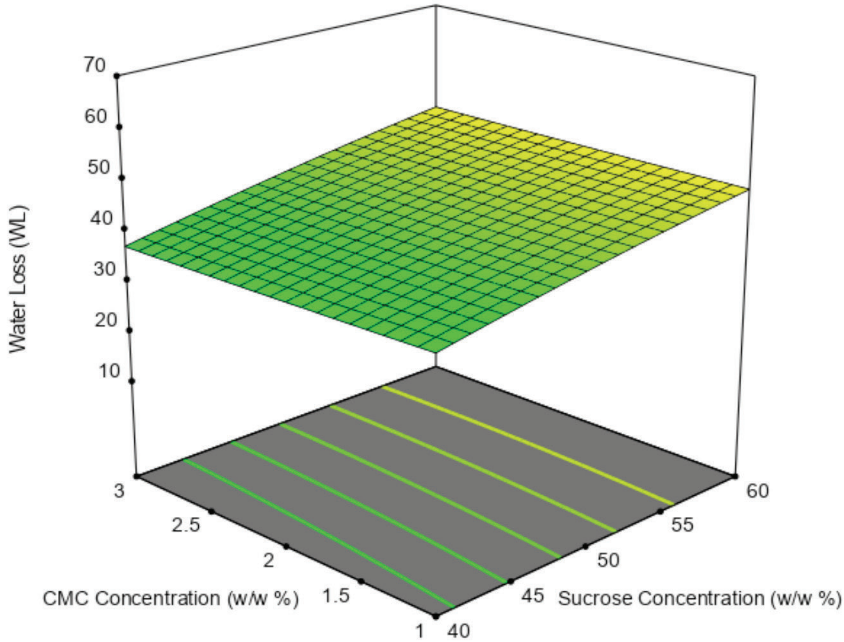
df= degree of freedom

membrane swelling effect, which expedite the water removal process and thus improves the cell membrane permeability to sucrose molecules. This finding is consistent with the past studies reported by Corrêa *et al.* (2016).

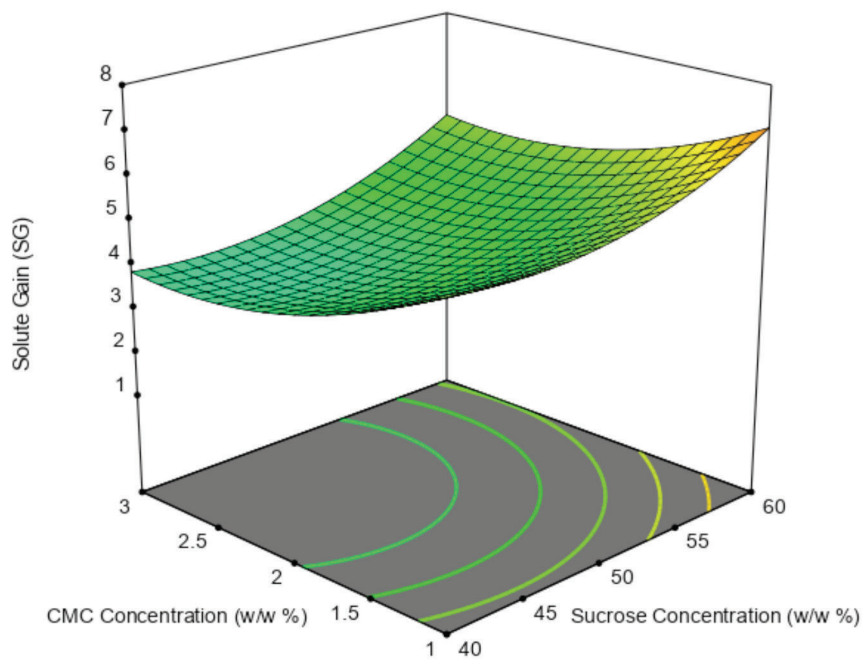
The effects of immersion time on WL and SG can be clearly seen in Figure 2 (a) and (b), respectively. The rapid mass transfer was observed during the early stage of the process, after which it was slowed down. In accordance with the results, previous studies has showed that higher water removal from the sample at the beginning of the process reduces the concentration gradient around the sample and consequently decreases the driving force, thus lowering the mass transfer process at further processing times (Campos *et al.*, 2012; Md Salim *et al.*, 2016a). This mass exchange is commonly referred as the dynamic period during the osmotic process which takes place until it reached the equilibrium period, where no further changes in weight or sample composition were observed (Li & Ramaswamy, 2010).

Predictive Model for WL and SG

The predictive model for WL and SG was obtained with considering their significance level was less than 5 % were presented in Equation 4 and 5, respectively. The correlation value of these models for WL ($R^2 = 0.99$) and SG ($R^2 = 0.95$) indicate a good fit of experimental data. Both models are valid within the applied range of the experimental processing variables used in this study. The ANOVA for the reduced

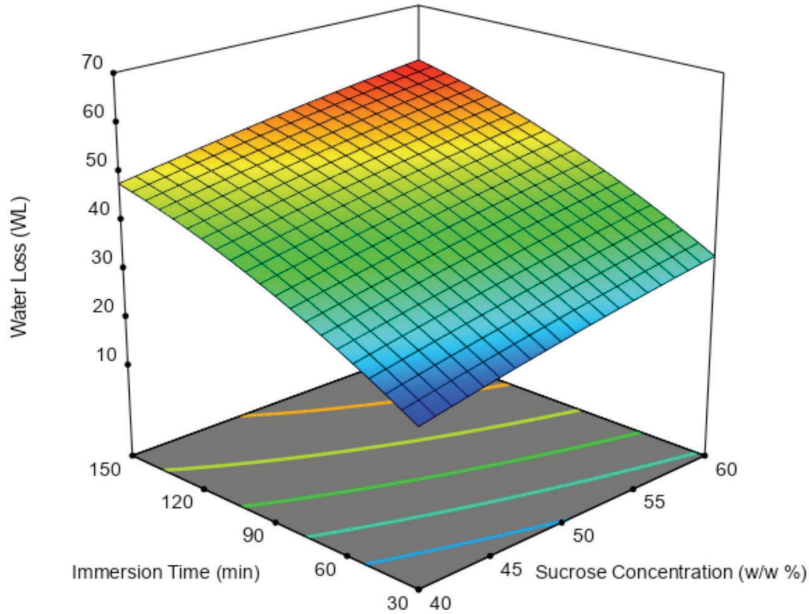


(a)

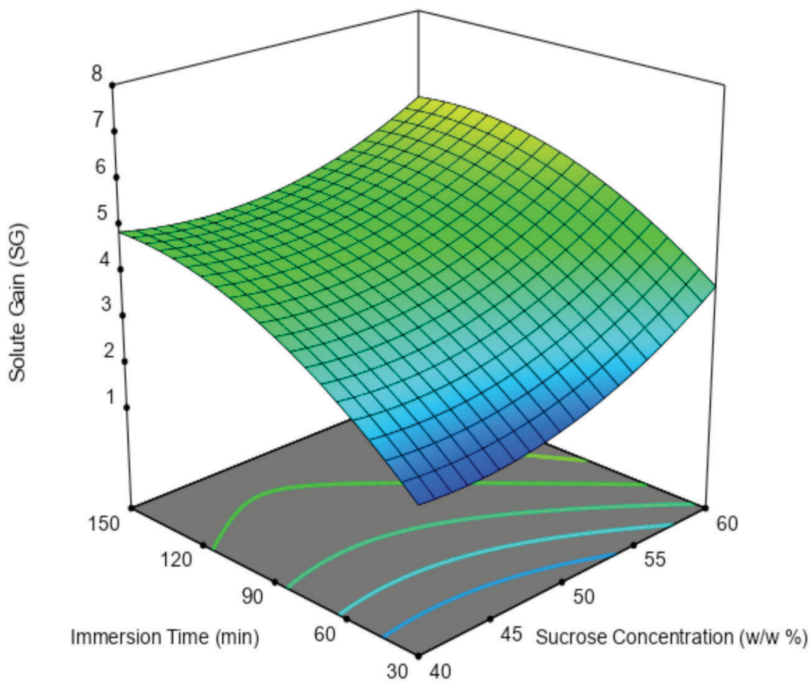


(b)

Figure 1: 3D surface plot as a function of CMC concentration and sucrose concentration during osmotic dehydration of CMC coated ginger slices on (a) Water Loss (WL); (b) Solute Gain (SG)



(a)



(b)

Figure 2: 3D surface plot as a function of sucrose concentration and immersion time during osmotic dehydration of CMC coated ginger slices on (a) Water Loss (WL) ; (b) Solute Gain (SG)

quadratic model is shows in Table 5. A predicted against the actual experimental data for WL and SG is shown in Figure 3 and 4, respectively.

$$WL = 43.39 + 5.30\,X_2 + 12.76\,X_3 - 3.06\,{X_3}^2 \qquad (4)$$

$$SG = 4.20 - 0.86\,X_1 + 0.75\,X_2 + 1.34\,X_3 + 0.66\,{X_1}^2 + 0.68\,{X_2}^2 - 0.79\,{X_3}^2 \qquad (5)$$

Where;

$$X_1 = \frac{CMC\ concentration - 2}{1}\ ;\ 1\,\%\,w/w \leq CMCconcentration \leq 3\,\%\,w/w$$

$$X_2 = \frac{Sucrose\ concentration - 50}{10}\ ;\ 40\,\%\,w/w \leq Sucrose\ concentration \leq 60\%\,w/w$$

$$X_3 = \frac{Immersion\ time - 90}{60}\ ;\ 30\ min \leq Immersion\ time \leq 150\ min$$

Table 5: ANOVA of the reduced quadratic model for WL and SG in osmotic dehydration of CMC-coated ginger slices

	WL				SG			
Source	***df***	**Sum of Squares**	**F-value**	**p-value**	***df***	**Sum of Squares**	**F-value**	**p-value**
Model	3	1945.93	327.80	<0.0001	6	34.57	28.85	<0.0001
Residual	12	20.88			9	1.80		
Lack of Fit	11	20.63	7.66	0.2754	8	1.74	0.76	0.3800
Pure Error	1	0.25			1	0.06		
Corrected Total	15	1966.81			15	36.37		

df= degree of freedom

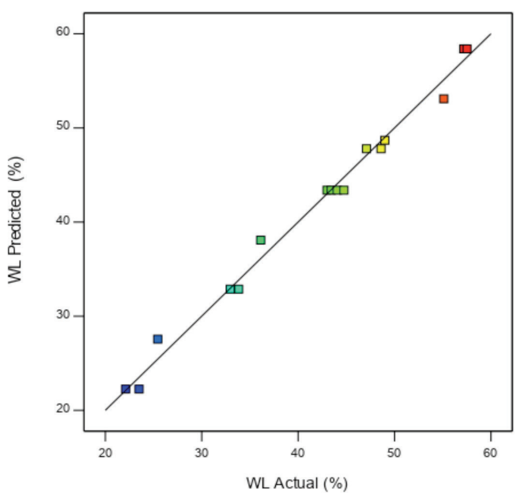


Figure 3: Predicted versus actual experimental data for WL of CMC-coated ginger slices

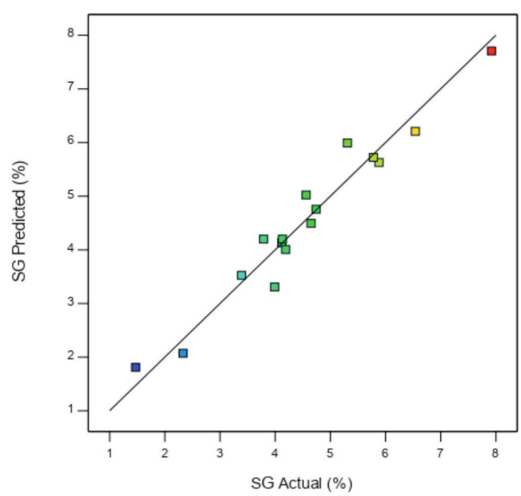


Figure 4: Predicted versus actual experimental data for SG of CMC-coated ginger slices

Optimum Condition

The optimum process variables for osmotic dehydration of ginger slices within the studied range were determined according to the software optimization step to obtain the following criteria: maximum WL and minimum SG. From the analysis, the optimum condition was found to be at a CMC coating concentration of 2.68 % (w/w) immersed in sucrose concentration of 50 % (w/w) with 2 % salt (w/w) within 150 minutes to achieve WL of 53 % with SG of 4.5 %.

Conclusion

Effects of three processing variables (CMC concentration, sucrose concentration, and immersion time) on the mass transfer during osmotic dehydration of ginger slices were studied. The findings of this research provide information that implementation of CMC coating prior to osmotic dehydration of ginger slices helps in minimizing the SG without affecting the performance of WL. The optimum condition obtained through response surface methodology to achieve WL of 53 % with SG of 4.5 % was found to be at 2.7 % w/w CMC coating, immersed in 50 % w/w sucrose solution with 2 % salt solution for 150 min. Through the establishment of this optimum process condition, it can help in reducing the overall energy requirement for the further drying process. On top of that, by promoting value-added products from ginger such as the dried product can help in providing more food availability which in turn increase our local producers' profitability, create employment and boosting local economies.

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