

Application of intermittent vacuum treatment on the osmotic dehydration of black cherry tomatoes (*Solanum lycopersicum* cv. OG)

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ABSTRACT

Osmosis pre-treatment process helps to reduce partially the moisture of fruits and vegetables before drying. The application of low pressure during the first few minutes of osmosis pushes the trapped gases out and facilitates the penetration of hypertonic solution into the food, thereby improving mass transmission efficiency. In this work, the effects of sucrose concentration (52–68°Brix), vacuum level (516–684 mmHg), and its application time (5–15 min) on water loss (WL) and solid gain (SG) of black cherry tomatoes (cv. OG) were investigated. The response surface methodology with a factorial experimental central composite design was used for the optimization. Results indicated that the application of a vacuum level of 627.22 mmHg every first 11.61 min of 1 h osmosis in a sucrose solution of 59.38°Brix increased the WL and reduced the osmosis time. The maximum WL was 28.60% of the tomatoes weight after 4 h of osmosis and the SG was 2.94%. Meanwhile, the corresponding values for the control sample (without vacuum application) were 15.62% and 1.66% after 5.5 h of osmosis dehydration. This proves the effectiveness and potential of the vacuum application in the osmotic dehydration process of black cherry tomatoes.

1. INTRODUCTION

The black cherry tomato is a new variety of tomatoes that has appeared in Vietnam in recent years and received much attention from the consumers. Similar to other tomato varieties, black cherry tomatoes contain bioactive compounds such as lycopene, phenolic compounds, and Vitamin C [1], however, this variety also synthesizes anthocyanin, which is mainly concentrated in the skin and outer tissue [2]. Because of their many health benefits, in addition to being eaten fresh, black cherry tomatoes can also be processed into many common products [3] such as tomato ketchup, tomato juice, tomato powder, dried tomatoes, picked tomatoes... Drying is one of the most effective and timehonored methods for the preservation of foods [4]. The reduction of water activity of food materials by removing the moisture inactivates potential pathogenic microorganisms or inhibits their growth and reproduction, slows enzyme activity, and minimizes many moisturerelated adverse reactions [5]. Although drying effectively prolongs the shelf-life of agricultural products, loss of organoleptic and nutritional quality is inevitable during conventional drying due to undesirable changes in structure and biochemical characteristics [5]. Especially bioactive compounds in black cherry tomatoes such as anthocyanin, lycopene, Vitamin C, and phenolics are very susceptible to heat [6-10].

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Department of Food Technology, Faculty of Agriculture and Natural Resources, An Giang University, An Giang Province, Vietnam. E-mail: htnha @ agu.edu.vn Osmotic dehydration is a significant stage often applied in the processing of dried fruit and vegetable products. The materials are soaked in a hypertonic solution with two diffusion fluxes occurring simultaneously and in opposite directions, water flows out of the food into the solution and water-soluble solids from the solution enter into the food. A third flux corresponds to the migration of natural solutes from the food into the solution which is less expressive [11]. Although the products from the osisture, their water activity is still high, so it is necessary to continue to carry out other processes for preservation such as drying [12]. Partial moisture reduction results in overall energy savings by shortening the time of subsequent drying [13]. Furthermore, a shorter exposure time to high temperature while still obtaining a dried product with the same moisture content is a beneficial approach for foods containing heat-sensitive nutrients [13].

Osmotic dehydration is a diffusion process. The diffusivity of water and solid depends on many factors such as temperature, pressure, concentration and viscosity of the osmotic solution, and food solution contact [14]. Traditional osmotic dehydration is often performed at ambient temperature and atmospheric pressure [12]. The application of low pressure for only the 1st min or during this process is known as vacuum osmotic dehydration, which removes gases in the intercellular spaces and further facilitates the penetration of the hypertonic solution in the extracellular spaces [15]. The action of the hydrodynamic mechanism promoted by pressure change causes the expansion and subsequent compression of the gases trapped in the capillaries, which leads to this exchange [15]. The agents commonly

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used for osmotic dehydration are hypertonic solutions of sucrose and sodium chloride and, also, a mixture of them [11].

Vacuum osmotic dehydration has been effectively applied to many products of fruit and vegetable, including pineapple [16], red cherry tomato [17], pumpkin [15], and sliced red tomato [11]. However, the application on black cherry tomatoes has not previously been studied yet. Therefore, the purpose of this study was to determine the effect of sucrose solution concentration, vacuum level, and vacuum treatment time on water loss (WL) and solid gain (SG) of black cherry tomato fruits (*Solanum lycopersicum* cv. OG) during osmotic dehydration, from that determining the optimal parameters to help the mass transfer process achieve the highest efficiency. The loss of bioactive compounds of the chosen optimal sample and the control sample after drying to the same moisture content was also compared to confirm the effectiveness of the vacuum application.

2. MATERIALS AND METHODS

2.1. Preparation of Black Cherry Tomato Fruits

Black cherry tomatoes (cv. OG, American) were grown on the farm of Nam Long Production and Trading Facility, in Vinh Long province, Vietnam. Fruits were harvested from the 30^{th} to the 32^{nd} day after fruit formation (fully ripeness), with the fruit diameter of 23.28-26.94 mm, the fruit firmness of 854-944 g force, the total soluble solid content of 6.05-6.29%, and the pH value of 4.37-4.49.

After harvesting, the tomatoes were packed into perforated cardboard boxes and transferred immediately to the Food Technology Laboratory (Can Tho University, Vietnam) within 1 h.

At the laboratory, the sorting was done to remove diseased or damaged fruits. After that, the tomatoes were washed with tap water 3 times and surface disinfected by soaking 1500 g fruits in 3000 mL ozonated water for 15 min using a Z755 ozone-generating equipment (made in Vietnam) with a production capacity of 80.4 mg/h [18]. Fruits were then treated in 2.08% CaCl, solution at 62°C for 23 min before blanching in water at 90°C for 1 min to improve fruit firmness. Both CaCl, treatment and blanching were performed by placing 500 g of tomatoes in a rectangular stainless steel mesh basket (25.5 cm long; 10 cm wide; 6.5 cm high with square holes of 0.5 cm size) and immersing in a thermostatically controlled water bath (Rex C-90, Memmert, Germany), in which the ratio of fruits and solution was 1:2 [19]. Then, the fruits were cooled rapidly in cold water (10°C) for 1 min to prevent further damage by heat, drained, and punctured small holes on the surface (hole diameter of 1 mm, a density of 20 holes/cm²) because tomatoes have a waxy skin that makes it difficult for mass transfer between the fruits and the hypertonic solution [20].

2.2. Experimental Design of Vacuum Osmotic Dehydration

In this study, the Portable Statgraphics Centurion software (version 15.2.11.0, USA) was used to design an optimized experiment for vacuum osmotic dehydration process with three operating variables of sucrose solution concentration (X_1) , vacuum level (X_2) , and its treatment time (X_3) . The Response Surface Methodology (RSM) based on the factorial Central Composite Design (CCD) was applied [21]. Before designing the optimization experiment, a preliminary investigation was carried out based on the literature for wide ranges of sucrose concentration $(50-70^{\circ}\text{Brix})$, vacuum level (500-700 mmHg), and vacuum treatment time (5-15 min) to find out an efficient range for the further study. As a result, the narrower investigation ranges were chosen as $52-68^{\circ}\text{Brix}$, 516-684 mmHg, and 7-13 min. The

actual and coded values of each variable are shown in Table 1. The coded values set for each variable in five levels: -1.68179 (-alpha), -1 (minimum), 0 (central), +1 (maximum), and +1.68179 (+alpha). The experiment was carried out with six replications of the central point, so there were a total of 20 runs.

Vacuum osmotic dehydration was carried out by putting 500 g of tomatoes in a plastic box (size of $17 \text{ cm} \times 11.5 \text{ cm} \times 8.5 \text{ cm}$, the capacity of 1200 mL) along with sucrose solution at different concentrations, the ratio of tomatoes and sucrose solution was 1:1. After that, the plastic boxes containing the samples were placed into the vacuum generator (Rocker 400, Laftech, Uc), adjusted and kept at vacuum levels for the time as designed. The vacuum generator needed about 5-7 s to reach the required vacuum level. Sucrose solutions of different concentrations (expressed in °Brix) were prepared by dissolving sucrose in water, adding 0.15% (w/v) citric acid. After vacuum removal, the mixture was kept at atmospheric condition until 1 h was reached. The vacuum application and osmotic dehydration at atmospheric pressure were repeated until equilibrium was reached between the fruits and the solution, which meant, the total soluble solid content of the sucrose solution did not change after 0.5 h of osmosis. The whole process was carried out at room temperature ($30 \pm 2^{\circ}$ C). After every 0.5 h, the fruits were taken out from the sucrose solution and soaked in water for 10 s to remove excess osmotic solution on the surface. The surface of the tomato fruits was drained using a centrifugal vegetable rotating basket. A control sample was prepared by soaking the fruits in a sucrose solution at the selected optimum sucrose concentration until equilibrium was reached but without vacuum application.

2.3. Experimental Validation

After completing the data analysis, the vacuum osmotic dehydration of black cherry tomato fruits was performed 3 times following the above procedure but with the chosen optimal parameters to validate the results under experimental conditions. Two responses (WL and SG) were determined and compared with the estimated values from the regression equations.

2.4. Determination of WL and SG

The tomato samples were weighed by an analytical balance with the accuracy of 0.01 g (JJ200, G&G, China) and the moisture content was determined in a drying oven at 105°C to a constant weight [22]. WL and SG were calculated as % of the weight of black cherry tomatoes before osmosis (Equations 1 and 2) [23].

$$WL(\%) = \frac{x_0^w M_0^o - x_f^w M_f^o}{M_0^o} \times 100$$
(1)

$$SG(\%) = \frac{x_f^{ST} M_f^o - x_0^{ST} M_0^o}{M_0^o} \times 100$$
(2)

Where, M_o^0 was original sample weight (g), M_f^0 was sample weight after osmotic hydration (g), χ_o^w was original moisture content (% w/w), x_f^w was the moisture content after osmotic hydration (% w/w), χ_0^{ST} was the original solid content (% w/w), and x_f^{ST} was the solid content after osmotic hydration (% w/w).

2.5. Statistical Analysis

The obtained experimental data were processed by the Portable Statgraphics Centurion statistical software (version 15.2.11.0, U.S.A.). The standardized Pareto charts were used to express the influence of

Run	Sucrose solution concentration	Vacuum level	Vacuum treatment time	Water loss (% w/w)	Solid gain (% w/w)
1	65 (+1)	650 (+1)	12 (+1)	24.51	2.07
2	60 (0)	600 (0)	10 (0)	27.37	2.86
3	60 (0)	600 (0)	10 (0)	28.17	2.76
4	65 (+1)	550 (-1)	8 (-1)	19.61	1.38
5	60 (0)	600 (0)	10 (0)	28.67	2.90
6	60 (0)	600 (0)	13 (+1.68179)	28.47	2.88
7	60 (0)	516 (-1.68179)	10 (0)	24.29	1.83
8	55 (-1)	550 (-1)	8 (-1)	22.15	1.86
9	60 (0)	600 (0)	7 (-1.68179)	25.72	2.36
10	60 (0)	684 (+1.68179)	10 (0)	27.48	2.63
11	55 (-1)	650 (+1)	8 (-1)	24.32	2.41
12	65 (+1)	550 (-1)	12 (+1)	21.61	1.69
13	68 (+1.68179)	600 (0)	10 (0)	17.42	1.29

Table 1: Water loss and solid gain of black cherry tomatoes at different vacuum osmotic dehydration conditions.

Units of sucrose solution concentration, vacuum level, and vacuum treatment time were "Brix, mmHg, and min, respectively. Numbers in parentheses were coded values. Water loss and solid gain were calculated on the weight of black cherry tomatoes before osmosis

each independent variable (sucrose solution concentration, vacuum level, and vacuum treatment time), quadratic values, as well as the interaction of factors on WL and SG at a 5% significance level (P < 0.05). From the results, a second-order polynomial response surface model (equation 3) was developed to predict the specific optimum condition, where, Y was the estimated response; β_0 was an intercept; β_i , β_{ii} , and β_{ij} were the regression coefficients of the variable for linear, quadratic, and interaction terms, respectively; and X_i and X_j are independent variables [24]. The fitness of polynomial equations was expressed by the coefficient of determination ($R^2 > 0.8$) and the non-significant lack-of-fit test (P < 0.05) [25]. The two-dimensional surfaces and their respective contour plots were constructed to illustrate the effects of each pair of independent variables on each response [24].

$$Y = \beta_o + \sum_{i=1}^{3} \beta_i X_i + \sum_{i=1}^{3} \beta_{ii} X_i^2 + \sum_{i=1}^{2} \sum_{j=2}^{3} \beta_{ij} X_i X_j$$
(3)

3. RESULTS AND DISCUSSION

3.1. Effect of the Vacuum Osmotic Dehydration on WL and SG

Table 1 shows the effects of sucrose solution concentration, vacuum level, and vacuum treatment time on WL and SG of black cherry tomatoes after the osmotic.

The linear, quadratic, and interaction effects on the WL and SG were presented graphically on two standardized Pareto charts in Figure 1a and 1b, respectively. The length of each bar was proportional to the absolute value of its associated regression coefficient and the order in which the bars displayed from top to bottom of charts corresponded to the order of the levels of the effects, with the strongest effect on the top and the weakest effect on the bottom.

The chart also included a blue vertical line that corresponded to the 95% confidence limit (P = 0.05) indicating statistical significance. The horizontal bar of any effect crossed this vertical line meant that the effect was statistically significant and vice versa [26]. It could be observed that all three independent variables (sucrose solution concentration, vacuum level, and vacuum treatment time) had a significant effect on the WL and SG of black cherry tomato fruits after osmosis because P < 0.05. In which, for the WL, the vacuum level was





the most important influence, followed by the sucrose concentration, on the contrary, the SG was affected by the concentration of sucrose solution more significantly. The vacuum treatment time had the least effect on both responses. Meanwhile, the effects of the interactions were not significant (P > 0.05) in the models.

3.2. Predicted Models for WL and SG

From the data obtained for WL and SG under vacuum osmotic dehydration conditions, regression models were established to predict the change of these two responses according to three independent variables (Equations 4 and 5 in Table 2). The R^2 coefficient of determination, which gave the proportion of the total variation that the selected model accounted for each response, was used to evaluate how well a model explained and predicted future outcomes. The higher value of R^2 indicated that the fitness was better correlated with the empirical data. The R^2 value should be at least 0.8 and as close to 1 as possible [27]. Regression analysis for two responses (WL and SG) exhibited good correlations between experimental and estimated results from the second-order polynomial models with high correlation coefficient (R^2) values of more than 0.97. Besides, the lack-of-fit test was also performed by comparing the variability of the current model residuals with the variability between observations at the replicate setting of the variables to check whether the selected model was adequate to describe the empirical data or a more complicated model should be used. Since *P*-values for lack of fit in the ANOVA tables were greater than 0.05, the developed models appeared to be appropriate to the observed data at the 5% significance level [25]. Therefore, the chosen mathematic formulations could be effectively applied to forecast the change of WL and SG based on sucrose concentration, vacuum level, and vacuum treatment time with high accuracy.

3.3. Response Surfaces, Contour Plots, and Main Effects Plots for Responses

The three-dimensional response surface graphs and two-dimensional contour plots at different vacuum osmotic dehydration conditions from

the predicted models for WL and SG are illustrated in Figures 2 and 3. These plots reflected the effects of two independent variables while the third was fixed at the central point. The response surface graphs were used to determine the direction to increase the desired response. While in the contour plots, curves of equal response values were drawn on a plane whose coordinates represented the levels of the independent variables and the center of these curves represented the optimum conditions.

The amount of released water and the amount of entered solid tended to increase with increasing concentration of sucrose solution in the range of 52–60°Bx. This variation was due to the outflow of water from the tomato fruits into the sucrose solution and the diffusion of the solute from the osmotic solution into the tomato tissue [15]. At higher sucrose concentrations, the dynamics of this process would be greater. This increase was also observed in red cherry tomatoes [17] and sliced red tomatoes [11]. However, if the concentration of sucrose solution continued to be increased from 60 to 68°Bx, the mass transfer efficiency decreased, and the penetration of liquid from the outside was limited due to the high solution viscosity and fruits hardening effect by rapid dehydration of the outer cells at a too high concentration of sugar [28].

In addition to the osmotic solution, the application of vacuum treatment also positively affected the mass transfer process, as shown

Table 2: Regression equations in terms of coded variables to predict the water loss and solid gain in the vacuum osmotic dehydration of black cherry tomatoes.

Reponses	Equation		R ²	P-value (lack of fit)
Water loss (% w/w)	$ \begin{array}{l} Y_1 =& -536.122 + 14.3365 A + 0.38223 B + 3.31254 C - 0.127819 A^2 + 0.00126 A \\ B + 0.0045 A C - 0.000349771 B^2 - 0.001575 B C - 0.111658 C^2 \end{array} \right. \tag{2}$	(4)	0.9770	0.4338
Solid gain (% w/w)	$ Y_2 = -100.399 + 2.07146A + 0.122237B + 0.82677C - 0.0175375A^2 - 0.0000 \\ 15AB - 0.000625AC - 0.0000940585B^2 - 0.0003875BC - 0.0243155C^2 $	5)	0.9831	0.3716

A: Sucrose solution concentration (°Brix); B: Vacuum level (mmHg); C: Vacuum treatment time (min); the water loss and solid gain were calculated on the weight of black cherry tomatoes before osmosis



Figure 2: Response surface and contour plots for the effect of (a) sucrose concentration and vacuum level, (b) sucrose concentration and vacuum treatment time, and (c) vacuum level and vacuum treatment time on the water loss of black cherry tomatoes. (The water loss and solid gain were calculated on the weight of black cherry tomatoes before osmosis).

by the increase in the amount of released water and the amount of entered solid when rising the vacuum level and vacuum application time. This could be explained by the hydrodynamic mechanism in the 1st min of osmosis. When vacuum application was performed, the gases in the intercellular space of the tomatoes were removed and when atmospheric pressure conditions were restored, a pressure gradient appeared between the inside of the tomatoes and the outside of osmotic solution, which promoted the penetration of liquid from the outside to fill these voids in the inside. This mechanism helped to increase the contact area between tomato tissue and the osmotic solution, thus improving the efficiency of mass transfer [15].

The changing trend of two responses according to the investigated variables was shown more clearly in the main effects plot [Figure 4]. Since WL and SG only increased to a certain limit when increasing the value of sucrose solution concentration, vacuum level, and vacuum treatment time, it was necessary to determine the value of the variables to achieve the optimum of the two required responses.

In osmotic dehydration, the optimal conditions were the conditions that helped the mass transfer process take place most effectively, which meant, the amount of released water was the highest. In addition, the product was also expected to have the least penetration of solutes (specifically, sucrose). Each of the two responses achieved optimal values at different vacuum osmosis dehydration conditions. However, when performing the simultaneous optimization of both responses by overlapping the two plots [Figure 5], the obtained optimal result was also the optimal point for the WL. Under the optimum conditions (sucrose concentration of 59.38°Brix, vacuum level of 627.22 mmHg, and its processing time of 11.61 first of each osmosis hour), the WL was predicted to reach the maximum value (28.60%) after 4 h of osmosis, then the amount of solid increased to 2.94%.

3.4. Empirical Validation of the Predicted Models

The obtained results were also verified by conducting vacuum osmotic dehydration at chosen optimal conditions (59°Brix, 630 mmHg, 12 min), the WL reached 27.96% (lower 2.24% compared with predicted value), and the SG was 2.88% (lower 2.04% than predicted value). This difference was still within the allowable limit (<5%). The reason was that the optimal value predicted from the model was decimal, but the performance condition was difficult to adjust to the number of decimals, so the natural number value was chosen. This confirmed the compatibility



Figure 3: Response surface and contour plots for the effect of (a) sucrose concentration and vacuum level, (b) sucrose concentration and vacuum treatment time, and (c) vacuum level and vacuum treatment time on the solid gain of black cherry tomatoes. (The water loss and solid gain were calculated on the weight of black cherry tomatoes before osmosis).



Figure 4: Main effects plot for (a) water loss and (b) solid gain of black cherry tomatoes in vacuum osmotic dehydration. (The water loss and solid gain were calculated on the weight of black cherry tomatoes before osmosis).



Figure 5: The overlay plot for simultaneous optimization of two responses (water loss and solid gain) of black cherry tomatoes according to (a) sucrose concentration and vacuum level, (b) sucrose concentration and vacuum treatment time, and (c) vacuum level and vacuum treatment time. (The water loss and solid gain were calculated on the weight of black cherry tomatoes before osmosis).

Table 3: Comparison of the content of bioactive compounds between two samples of black cherry tomatoes with osmotic dehydration at optimal vacuum conditions and at atmospheric conditions.

Amou	nt of loss after osmosis		Remaining content after drying at 70°C to moisture content of 25%			
Reponses	Optimal sample	Control sample	Reponses	Optimal sample	Control sample	
Anthocyanin (% w/w)	14.01*b±0.25**	9.79ª±0.21	Anthocyanin (mgCE/100 g)	15.33 ^B ±0.40	13.37 ^A ±0.31	
Lycopene (% w/w)	1.04 ^a ±0.03	1.09ª±0.02	Lycopene (µg/g)	276.17 ^B ±3.87	262.35 ^A ±4.46	
Vitamin C (% w/w)	36.25 ^b ±1.09	25.01ª±0.78	Vitamin C (mg/100 g)	167.26 ^B ±4.85	132.54 ^A ±4.24	
Total phenolic (% w/w)	22.84 ^b ±0.43	15.85ª±0.30	Total phenolic (mgGAE/100 g)	170.13 ^B ±3.57	151.60 ^A ±2.73	

*Average of three replications; **standard deviation; values with different superscripts within a row were significantly different at 5% significance level (P<0.05). The amount loss after osmosis was calculated on the weight of black cherry tomatoes before osmosis, the remaining content after drying was calculated on the weight of black cherry tomatoes after drying.

of the results predicted from the model and the experimental data. For the control sample (without vacuum treatment), the WL and the SG were significantly lower (15.62% and 1.66%, respectively) after a longer period of time (5.5 h) for equilibration.

3.5. Comparison of the Bioactive Compounds Loss

To better understand the effect of vacuum osmotic dehydration on the improvement of bioactive compounds in the dried product, the study compared the loss of anthocyanin, lycopene, Vitamin C, and total phenolic compounds after osmotic dehydration between the optimal and the control samples. Besides, this study also compared the remaining contents of these components after drying the two samples at the same temperature of 70°C until the same moisture content of 25% was reached. The results are presented in Table 3. Due to the effect of vacuum application on the efficiency of mass transfer, the contents of water-soluble bioactive compounds such as anthocyanin, Vitamin C, and other phenolics of the osmotic dehydration sample with vacuum application were higher than that of the osmotic dehydration sample at the atmospheric pressure (4.22%, 11.24%, and 6.99% higher, respectively). Meanwhile, the WL of the vacuum-treated sample (27.96%) was higher than that of the control sample (15.62%) to 12.34%. Because of the greater amount of water remaining, when drying to the same moisture content, the control sample had to be subjected to heat for a longer time, thereby leading to a lower content of bioactive compounds compared to samples with vacuum application.

4. CONCLUSION

RSM with the CCD applied in this study had proven to be an effective statistical tool in optimizing WL as well as SG during vacuum osmotic dehydration. The application of the vacuum technique in osmotic dehydration had greatly improved the mass transfer efficiency. The partial removal of moisture helped to limit the destruction of bioactive compounds in tomatoes during subsequent drying. This technique further could be applied in the processing of dried black cherry tomatoes in a larger scale to shorten the drying time and improve the product quality.

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6. AUTHORS' CONTRIBUTIONS

Conceptualization, Ho Thi Ngan Ha (H.T.N.H.) and Nguyen Minh Thuy (N.M.T.); formal analysis, H.T.N.H.; investigation, H.T.N.H.;

methodology, H.T.N.H. and N.M.T.; supervision, N.M.T.; writing – original draft, H.T.N.H.; and writing – review and editing, H.T.N.H. and N.M.T. All authors have read and approved the final version of the manuscript.

7. CONFLICTS OF INTEREST

The authors declare no conflicts of interest.

8. FUNDING SOURCES

This research received no external funding.

9. ETHICAL APPROVAL

This study does not involve experiments on animals or human subjects.

10. DATA AVAILABILITY

All data generated and analyzed are included within this research article.

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