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Osmotic Dehydration of Toddy Fruit Cubes in Sugar Solution Using Response Surface Methodology

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Abstract The response surface methodology (RSM) was applied to optimize the effects of immersion time (60, 90 and 120 min), temperature (35, 45 and 55°C) and concentration of sucrose solution (30, 40 and 50°Brix) in osmotic dehydration of toddy fruit tubes (1cm^3). Box-Behnken Design was used with water loss (WL, %), solid gain (SG, %), and weight reduction (WR, %) as responses. The models obtained for all the responses were significant ($P \leq 0.05$) without a significant lack of fit. The optimum conditions were temperature (45°C), immersion time (120min), concentration of sucrose solution (40°Brix) in order to obtain WL of (33.867g/100g initial sample), SG of (4.478g/100g initial sample) and WR of 29.39 g/100g initial sample, respectively.

Keywords: *response surface methodology, Box-Behnken Design, osmotic dehydration, toddy fruit tubes*

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1. Introduction

In Africa, South Asian (e.g. Sri Lanka and India), Southeast Asia (e.g. Myanmar, Cambodia, Malaysia, Indonesia, Vietnam and Thailand), Palmyra Palm, sugar palm or toddy palm (Borassus flabellifer Linn.) is commonly available [1,2]. Their all parts can be used for several purposes and the mesocarp or pulp ripe is sweet with abundant carotenoid. When it becomes ripe, it had yellow orange and can be used for foods such as cakes, jelly, ice cream, jams, cordials, beverages and toffee [3]. The important sources of digestible and indigestible minerals, carbohydrates and certain vitamins, particularly vitamins A and C are contained in fruits. The moisture in most of the fruits above 75% and fruits are prone to spoilage by molds and yeasts and this moisture content of fruits are prone to spoilage by molds and yeasts [4]. Fresh nipa palm fruits or fruits in syrup have short shelf-life because they have high moisture content (MC) and water activity (a_w). Too high MC and aw will affect the product by microorganism and bad qualities. Osmotic dehydration (OD) and drying processes are promising food preservation preservative techniques that may lead products to low MC and a_w [5].

The utilization of appropriate methods of processing and preservation would definitely reduce the spoilage of fruits and vegetables. Osmotic dehydration (OD) is one of the simple, low cost, energy efficient and enhancing the shelf-life of fruits and vegetables, method based on the removal of natural water content from fruits and vegetables by immersing them in hypertonic osmotic solutions prepared with sugars, salts, alcohols and starch solutions [6]. The OD process kinetics is influenced by the nature of the osmotic solute and it has a great impact on the nutritional and sensorial properties of the final product [7]. Rastogi and Raghavararo (1997) reported that osmotic dehydration reduced up to 50% weight of fresh vegetables and fruits [8]. The different types of osmotic agents such as glucose, sorbitol, sucrose and salts are used according to the final products [9]. However combination of different solutes can be used [10]. Water loss from vegetables and fruits took place in first two hours and maximum sugar gain within 30 minutes [11]. Temperature and concentration of osmotic syrups increased the rate of water loss during osmotic dehydration. Although higher temperature has the significant effect on the structure of tissues [12] and also cause deterioration of flavour and enzymatic browning at temperature above 45°C.

The response surface methodologies (RSM) are very useful techniques for optimization and applied in different food processes among that is osmotic dehydration [13,14,15,16,17]. The main advantage is that they reduce the number of experiments needed to obtain statistically valid results and are faster and more informative than traditional assessments which evaluate one variable at a time [16].

The objective of this work was to study the osmotic dehydration of toddy fruit cubes as a function of sugar concentration, temperature and immersion time through Response surface methodology (RSM) in order to identify process conditions for a high water loss at maximum solid uptakes and to optimize the osmotic dehydration as a pretreatment.

2. Materials and Methods

2.1. Raw Materials

In this research work, good, sound and unmatured toddy fruits were obtained from Hmawbi Township, Yangon Region. Sugar and potassium sorbate of commercial grade were purchased from local markets.

2.2. Method of Preparation

Good, sound and unmature toddy fruits were washed, peeled with a sterile knife and cut in to uniform cubes ($1cm^3$ thickness) and steam blanched for 1minute. Then the cubes were dipped in the 0.1% potassium sorbate solution for 5 minutes and drained. Osmotic dehydration was done in sucrose solution with different concentrations such as 30, 40 and 50°Brix. The sample to solution ratio was constant 1:5 (w/w). The toddy fruit cubes was weighed and submerged in salt solution at 30, 40 and 50°C. The temperature was maintained constant using a hot water bath and the samples were removed from the solution at different time intervals of 60, 90 and 120 min. In each of the experiments, fresh osmotic solutions were used. After removing from the sugar solution, the samples were drained and the excess solution at the surface was removed with filter paper for subsequent weight measurement. After dehydration the samples were dried in hot air oven at 40°C about 6 hours until equilibrium moisture content was obtained. All experiments were done triplicates and the average value was taken for calculation.

2.3. Methodology

The methodology involved osmotic dehydration with different concentration of sucrose solution, determination of water loss, solid gain, weight reduction and optimization of response parameters with RSM.

2.3.1. Water Loss, Solid gain and Weight Reduction

Water Loss (WL), Solid Gain (SG) and Weight Reduction (WR) were calculated and given in Equations (1, 2 and 3) [18].

$$\text{Water Loss} = \text{Solid Gain} + \text{Weight Reduction} \tag{1}$$

$$\text{Solid Gain} = \frac{(m - m_o)}{M_o} \times 100 \tag{2}$$

$$\text{Weight Reduction} = \frac{(M_o - M)}{M_o} x100 \tag{3}$$

Where, M_o- Initial mass of the samples (g)
M-Mass of sample after dehydration (g)
m_o- Initial mass of the solids in sample(g)
m- Mass of the solids in the sample after dehydration (g)

2.4. Design of Experiment

The Response Surface Methodology (RSM) is a statistical modeling technique applied for multiple regression analysis using quantitative data obtained from properly designed experiments. The Box-Behnken Design (BBD) of three variables and seventeen trials were used for designing the experiments of osmotic dehydration [19].

Table 1. Codes and Actual Levels of the Independent Variables for the Design of Experiment

Independent Variables	Notations	Coded Levels		
		-1	0	+1
Duration of osmosis (min)	A	60	90	120
Temperature of solution (°C)	B	30	40	50
Sugar Concentration (ºBrix)	C	30	40	50

The response surface methodology assumes that there is a polynomial function that relates the responses to the independent variables namely Duration of osmosis (A), Temperature of the solution (B) and Salt concentration (C) in the process. Therefore, the experimental data obtained from the design (Table 1) were fitted to a polynomial of the form found in equation 4 [20].

$$\begin{aligned} Response\ (Y) = {} & a_0 + a_1 A + a_2 B + a_3 C + a_{11} A^2 + a_{22} B^2 \\ & + a_{33} C^2 + a_{12} AB + a_{13} AC + a_{23} BC \end{aligned} \tag{4}$$

where, the response (Y) is (WL, SG and WR %), the a_n are constants and A, B, C are independent variables.

2.5. Optimization

Optimization was carried by attempting to combine various factors that simultaneously satisfy the requirements placed on each of the response and factors. There are several response variables describing the quality characteristics and performance measurements of the system, are to be maximized while some are to be minimized. RSM was applied to determine the optimum conditions for producing a model for osmotic dehydration of toddy fruit cubes with maximum water loss, weight reduction and minimum solid gain.

3. Results and Discussion

3.1. Effect of Variables on Water Loss, Solid Gain and Weight Reduction

The effects of variables such as osmotic solution temperature, osmotic solution concentration and duration on water loss, solid gain and weight reduction were studied and a second order polynomial equation was fitted with the experimental data.

3.2. Statistical Analysis on Model Fitting

The experimental responses as a function of process variables such as Time (A), Temperature (B) and Sugar Concentration (C) during osmotic dehydration of toddy fruit cubes are shown in Table 2.

The value of water loss (%), solid gain (%) and weight reduction (%) were within the ranges of 16.4-32.6, 2.4-4.8 and 13.0-27.9 respectively. Regression analysis and ANOVA results are shown in Table 3. The model F values of three responses such as WL, SG and WR were 18984.66, 107.21 and 17409.73 implying that the model is significant. At the same time WL, SG and WR showed that they possess non -significant lack- of- fit. These values indicated that the models were fitted and reliable. The adequacy of the model is further checked by Coefficient of determination (R^2) was found to be 1.0000, 0.9928 and 1.0000 for WL, SG and WR respectively. As the calculated R^2 was found to be approximately equal to 1 it was considered to be high enough for predication purposes and the predicted R^2 for WL, SG and WR of 0.9995, 0.9279 and 0.9997 were in reasonable agreement with adjusted R^2 of 0.9999, 0.9835 and 0.9999. The values of R^2 and adjusted R^2 obtained in the study implied that the predicted values are in good agreement with the experimental values. The values of Adeq precision are 358.4607, 30.1582 and 352.1277 for WL, SG and WR respectively. The values of Adeq precision obtained in this study are greater than 4.0 indicating that these responses had better precision and reliability. The values of coefficient of variation (C.V %) were 0.2240, 2.87 and 0.2429 for WL, SG and WR respectively which showed that the deviations between experimental and predicted values are low.

3.3. Effect of Process Variables on Water Loss

In (Figure 1), the increase in mass transfer properties (WR, SG and WL) of osmotic dehydration of toddy cubes during the early stages when osmotic solution concentration increased from 30 to 50^{o}Brix. Spiazzi, E. A., & Mascheroni, R. H. (1997) said due to the fact that penetration of smaller molecular weights of the solute (sugar) into the plant tissue and the outflow of water from the inner tissue rapidly enchance to the osmotic solution and also increased the WR and WL [21]. Moreover, enhances in osmotic pressure gradient because of higher concentration of the solution. The loss of functionality of plasmatic membrane of the cell which allow the solute to penetrate into the plant tissues and improved the SG through the early stages of osmotic dehydration (Figure 1). However, the mass transfer properties of toddy cubes decreased beyond 45^{o}Brix level of sugar concentration (Figure 1) which described that the diffusion strength of osmotic solution (sugar solution) into the plant material and the comparative moisture content on the outside of product were independent on the concentration of the solution and hence reduced the moisture transport phenomenon of the toddy cubes [22].

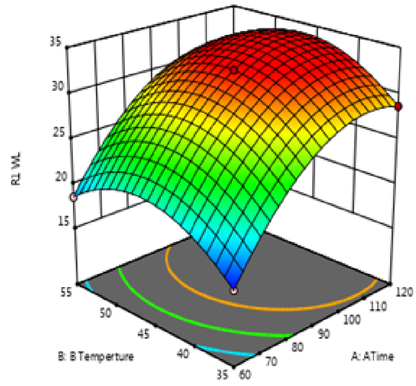
$$\begin{aligned} \textit{Water loss} &= +32.58 + 6.04*A + 0.99*B + 0.565*C \\ &\quad -4.76*A^2 - 4.29*B^2 - 4.4*C^2 \\ &\quad -0.085*A*B + 0.425*A*C + 0.905*B*C \end{aligned} \tag{5}$$

Table 2. The Box-Behnken Design for Osmotic Dehydration of Toddy Fruit Cubes

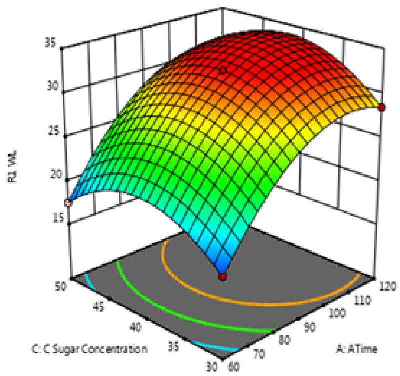
Run	F 1	F 2	F 3	R 1	R 2	R 3
	A:time	B:Temp	C:Sugar	WL	SG	WR
1	120	45	30	28.5	3.58	24.92
2	90	55	30	23.38	2.5	20.88
3	60	45	50	17.5	3.1	14.4
4	90	45	40	32.5	4.6	27.9
5	120	35	40	28.7	3.9	24.8
6	90	35	50	22.6	3.2	19.4
7	90	35	30	23.2	3.1	20.1
8	60	55	40	18.54	2.9	15.64
9	90	45	40	32.6	4.8	27.8
10	60	35	40	16.4	3.4	13
11	90	45	40	32.6	4.7	27.9
12	60	45	30	17.3	2.4	14.9
13	120	55	40	30.5	3.8	26.7
14	90	45	40	32.6	4.8	27.8
15	90	55	50	26.4	3.3	23.1
16	90	45	40	32.6	4.8	27.8
17	120	45	50	30.4	3.2	27.2

Table 3. Regression Coefficients for Osmotic Dehydration of Toddy Fruit Cubes

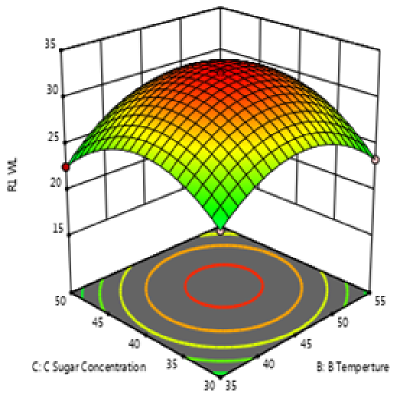
Variables/Factor	DF	Water Loss (%)		Solid Gain (%)		Weight Reduction (%)	
		Sum of Squares	F-value	Sum of Squares	F-value	Sum of Squares	F-value
Model	9	590.69	18984.66	10.63	107.21	472.30	17409.73
A-time	1	292.34	84560.06	0.8978	81.51	260.83	86532.21
B-Temp	1	7.84	2268.00	0.1512	13.73	10.17	3373.95
C-Sugar	1	2.55	738.70	0.1860	16.89	1.36	451.60
AB	1	0.0289	8.36	0.0400	3.63	0.1369	45.42
AC	1	0.7225	208.99	0.2916	26.47	1.93	640.98
BC	1	3.28	947.63	0.1225	11.12	2.13	707.17
A^2	1	95.30	27566.18	1.50	136.48	72.87	24173.45
B^2	1	77.40	22388.62	1.74	157.81	55.94	18558.68
C^2	1	81.42	23552.16	4.84	439.72	46.55	15443.13
Lack of Fit		0.0162		0.0451		0.0091	
R^2		1.0000		0.9928		1.0000	
Adjusted R^2		0.9999		0.9835		0.9999	
Predicted R^2		0.9995		0.9279		0.9997	
Adeq Precision		358.4607		30.1582		352.1277	
Std. Dev.		0.0588		0.1049		0.0549	
Mean		26.25		3.65		22.60	
C.V. %		0.2240		2.87		0.2429	



(a) immersion time and temperature



(b) sugar concentration and time



(c) sugar concentration and temperature

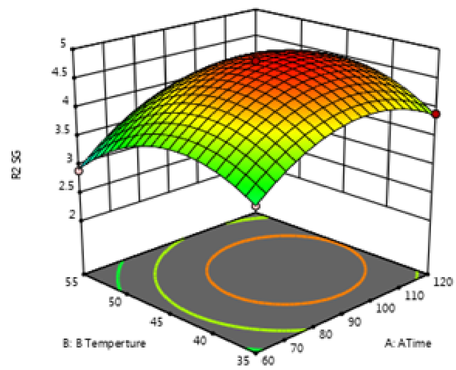
Figure 1. Effect of Process Variables on Water loss

3.4. Effect of Process Variables on Solid Gain

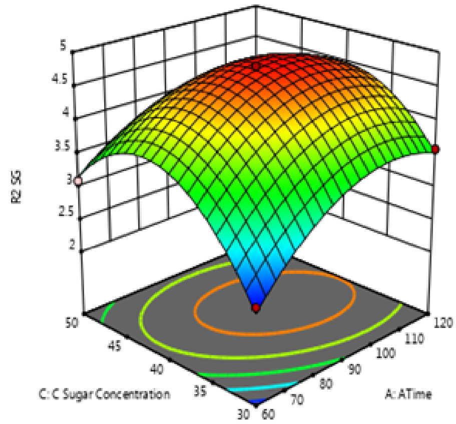
One of the vital parameters which control the osmotic dehydration properties of the toddy cubes is the temperature of the osmotic solution. The effect of osmotic solution temperature on the mass transfer properties of the toddy cubes and the consequences were depicted in Figure 2. It was observed that there was an increment in water loss from the plant materials and simultaneous uptake of solids (Figure 2) [23,24] through swelling and plasticizing of cell membranes [25,26] while the temperature of the osmotic solution was enhanced from 35 to 45℃. In (Figure 2), after osmotic solution temperature of 45℃, the mass transfer properties of toddy cubes decreased which may be due to decreased the viscosity of osmotic solution and increased the exchange of solids into the plant material.

The present results are also in agreement with findings of [27] obtained during the optimization of the osmotic dehydration of peach slices. This positive interaction between process time and osmotic agent concentration was also reported by [28] during the osmotic dehydration studies on beetroot in salt solution. The regression model of solid gain as a function of process parameters for osmotic dehydration of toddy cubes are given in equation (6). The positive values of interaction term between A, B and C indicated that increase in their level increased solid gain. The negative values of quadratic terms of process variables for osmotic dehydration of toddy cubes indicated that higher values of these variables affected solid gain.

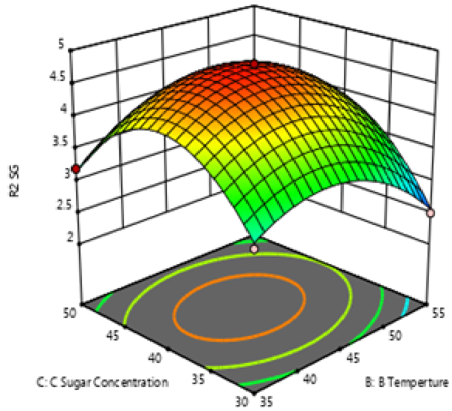
$$\begin{aligned} &\textit{Solid Gain} \\ &= +4.74 + 0.335 * A - 0.1375 * B + 0.1525 * C \\ &\quad - 0.5975 * A^2 - 0.6425 * B^2 - 1.07 * C^2 \\ &\quad + 0.1 * A * B - 0.27 * A * C + 0.175 * B * C \end{aligned} \tag{6}$$



(a) immersion time and temperature



(b) sugar concentration and time



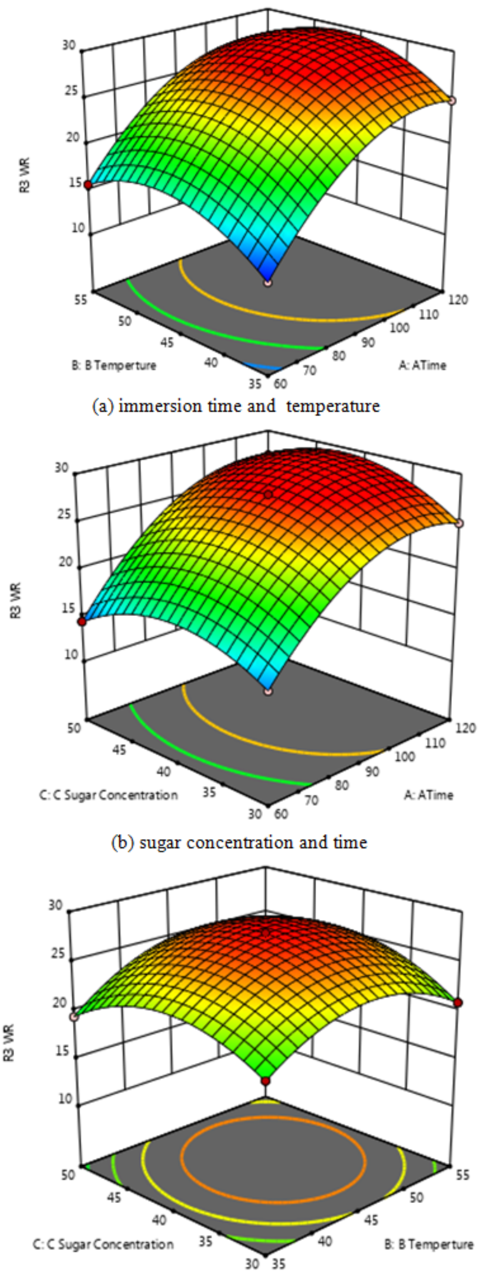
(c) sugar concentration and temperature

Figure 2. Effect of Process Variables on Solid Gain

3.5. Effect of Process Variables on Weight Reduction

Weight reduction indicates the amount of water loss by the sample during the osmotic dehydration process. The regression model of weight reduction as a function of process parameters is given in equation (7).

$$\begin{aligned}&\textit{Weight reduction}\\&=+27.84+5.71*A-1.13*B+0.4125*C\\&\quad+0.73*B*C-4.16*A^2-3.64*B^2-3.33C^2\\&\quad-0.185*A*B+0.695*A*C\end{aligned} \quad (7)$$



(a) immersion time and temperature

(b) sugar concentration and time

(c) sugar concentration and temperature

Figure 3. Effect of Process Variables on Weight Reduction

The results revealed that, increase in the immersion time of the toddy cubes in the osmotic solution up to 120 minutes caused an improvement in the mass transfer parameters of the toddy cubes (Figure 3). The transport phenomena become very rapid during the early stages of dehydration and also helped to the progression of mass transfer of solutes due to the feasible membrane enlargement or plasticizing, which forced to increase the cell membrane permeability to the sugar molecules to infuse into the plant material [29] and advanced the loss of water from the plant tissues [30]. It was also visualized that after an osmotic dehydration time of 140 minutes, equilibrium level was attained between the sample and the osmotic solution which brought a negative trend in mass transfer rate and decreased the mass transfer properties of the slices [31]. The presence of negative values of interaction term between A, B and C indicated that increase in their level decreased weight reduction. The positive values of quadratic terms of process variables of osmotic dehydration of toddy cubes indicated that higher values of these variables reduced weight reduction. The response surface plot for osmotic dehydration of toddy cubes indicated in Figure 3 represents weight reduction as a function of time, temperature and concentration of the osmotic solution. Weight reduction increases with increase in sugar concentration and time as shown in Figure 3. The reason was that the viscosity of osmotic solution was lowered and the diffusion coefficient of water increases at high temperature.

3.6. Numerical Optimization of Process Parameters

The criteria variables were set such that the independent variables (Time, Temperature and Concentration) would be minimum from an economical point of view [29]. The main criteria for constraints optimization for osmotic dehydration of toddy cubes were maximum possible water loss and weight reduction. The desired goals for each factor and response are shown in Table 4. In order to optimize the process parameters for osmotic dehydration of toddy cubes by numerical optimization which finds a point that maximize the desirability function; equal importance of `3' was given to all the three process parameters and three responses.

Table 4. Criteria and Output for Numerical Optimization of Process Parameters

Criteria	Goal	Lower limit	Upper limit	Importance	Output
A: Time	maximize	60	120	3	120
B: Temp	is target = 45	35	55	3	45
C: Sugar	is equal to 40	30	50	3	40
Water loss (%)	maximize	16.4	32.6	3	33.867
Solid Gain (%)	minimize	2.4	4.8	3	4.478
Weight reduction (%)	maximize	13	27.9	3	29.39
Desirability					0.669

3.7. Verification of the Model for Osmotic Dehydration of Toddy Fruit Cubes

Osmotic dehydration experiments were conducted at the optimum process condition (A= 120 min, B=45°C and C=40%) for testing the adequacy of the model equations for predicting the response values. The observed experimental values (mean of three experiments) and values predicted by the equations of the model are presented in Table 5. The experimental values were found to be very close to the predicted values of process responses such as water loss, solid gain and weight reduction. Therefore, it could be concluded from above discussion that model are quite adequate to assess the behavior of the osmotic dehydration of toddy tubes.

Table 5. Predicted and Experimental Values of Response at Optimum Process Conditions for Osmotic Dehydration of Toddy Fruit Cubes

Response	Predicted Value	Observed Value
Water loss (%)	33.8675	33.78
Solid Gain (%)	4.4775	4.45
Weight reduction (%)	29.39	29.33

4. Conclusion

Response surface methodology was effective in optimizing process parameters for the osmotic dehydration of toddy tubes in osmotic aqueous solution of sugar having concentration in the range of 30-50, temperature 35-55°C and process duration 60-120min. The results exhibited that, all the independent variables have considerable influence on the osmotic dehydration process of toddy tubes. Three second order polynomial models were framed for the responses (WR, SG and WL) from the observed data. The regression equations obtained in this study can be used for optimum conditions for desired responses within the range of conditions in the study.

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