

Optimization of the production of a self-stable powder from date fruit variety 'Shahani'

Zahra RAFIEE DARSANGI^{1,2}

Fojan BADI¹ (✉)

Mania SALEHIFAR²

Summary

The objective of this study was to prepare a self-stable powder from date paste. The concept of glass transition temperature (T_g) was used to predict the stability of powder at room temperature. The effect of maltodextrin (MD) as a drying additive in the range of 28 - 55%, tricalcium phosphate (TCP) as an anti-caking agent and glycerol monostearate (GMS) as a flowability agent in the range of 0.3 - 3% on some physico-chemical and thermal properties of the date powder was studied, using response surface methodology (RSM). Proposed mathematical models were successfully capable to fit the experimental results for different physico-chemical properties of the powder such as moisture content, bulk density, solubility, T_g , hygroscopicity and color index (L^* , a^*) with high R^2 values ($R^2 > 0.9$). The optimal combination of additives was determined as 35% MD, 3% TCP and 3% GMS with the overall desirability of 82.3%.

Key words

date powder, glass transition, optimization, Response surface methodology (RSM), stickiness

¹ Agricultural Engineering Research Institute (AERI), Agricultural Research, Education and Extension Organization (AREEO), Karaj, Iran

² Department of Food Science and Technology, Islamic Azad University, Shahre Ghods, Iran

✉ Corresponding author: f.badii@areeo.ac.ir, fjbadii@gmail.com

Received: November 3, 2019 | Accepted: May 14, 2020

Introduction

Date palm (*Phoenix dactylifera* L.), is the major fruit in many countries located in the Middle East, Southern Asia and some parts of Africa. It contributes largely to agricultural production and export in these regions (Sablani et al., 2008). This fruit is very popular among the local inhabitants in the date growing countries and has a great impact on their economy. There are different varieties of dates including dry, semidry and soft cultivars. Date dry solid of all the varieties mainly consists of low molecular weight sugars like fructose, glucose and sucrose. Date palm is a good source of minerals and some bioactive components with good antioxidant properties (Farahnaky et al., 2016). This fruit is primarily consumed as fresh. However, as a result of several reasons such as non-uniform ripening and poor postharvest management leading to physical damages, a great number of date fruits are not suitable for fresh consumption. Therefore, it is necessary to find alternative ways to use low quality dates for human consumption. Recently, a number of new products have been developed industrially from low quality dates such as date syrup, date concentrate, jam, bar and date liquid sugar. Production of powder with high nutritional value from low quality dates can be considered as a sugar substitution in different food formulations like confectionary, bakery products and drinks.

The major challenge of producing fruit powders is their stickiness, due to the presence of low molecular weight sugars and organic acids. During drying and rapid removal of water, these molecules change to amorphous or partially amorphous states. These amorphous materials are very hygroscopic and as a result of water plasticization, their glass transition temperature (T_g) drops and undesirable physical changes like stickiness and caking occur (Roos, 2003). Therefore, stickiness and caking of sugar-enriched powders and products are time dependent phenomena that occur in amorphous hygroscopic powders above their T_g , which affect product stability and shelf-life leading to significant effects on the sensory properties of the materials. Thus, the T_g is considered as a reference temperature to predict the stability, control quality and shelf-life of foods with low and intermediate water content (Roos, 2003; Champion et al., 2000; Blanshard, 1995; Slade et al., 1991; Roos and Karel, 1991).

Different high molecular weight materials such as maltodextrin (MD) with different dextrose equivalent (DE), proteins and gum Arabic are added to the fruit paste or juice in order to produce free flowing powders and overcome the problem of stickiness (Sablani et al., 2008; Farahnaky et al., 2016; Abadio et al., 2004; Manickavasagan et al., 2015; Mosquera et al., 2012; Shavakhi et al., 2011; Jaya and Das, 2004; Jaya et al., 2006; Papadakis et al., 2006). These substances have higher T_g and they are added to the fruit pulp as drying aids in order to increase the average T_g of the mixture resulting in reducing stickiness and improving the free flowing property of the final powders. The combination of MD as a drying additive, tricalcium phosphate (TCP) as an anti-caking agent and glycerol monostearate (GMS) as a flowability agent were successfully used to produce mango powder (Jaya and Das, 2004) and honey powder (Sahu, 2008). Sablani et al., (2008) produced date powder granules from date paste by adding MD as a drying aid. They suggested that at least 50% MD should be added to the date paste to achieve a self-stable powder. Previously, date powder was produced from date syrup by adding MD as an

antiplasticizing agent (Farahnaky et al., 2016). Manickavasagan et al. (2015) obtained date powder through a pilot scale spray dryer by adding MD and gum Arabic. However, in the present work, date powder was produced from whole fruit after pitting. Since the powder is obtained from fruit flesh, the final powder would have high nutritional value with a good source of dietary fibers and bioactive components. Besides, by adding this powder to food products, the amount of pure sugar can be reduced in the formulation and more importantly, the flavor of date can be maintained in the final products. Therefore, the aim of this study was to prepare a free-flowing and stable powder at room temperature from date paste with reduced level of MD.

Materials and Methods

Materials

Dates variety 'Shahani' were purchased from a local garden in Jahrom (Fars Province, Iran) and kept in cold store at $4 \pm 1^\circ\text{C}$ before the experiments. Maltodextrin (MD) with the dextrose equivalent (DE) of 7, tricalcium phosphate (TCP) and glycerol monostearate (GMS) were purchased from Sigma-Aldrich Co. (Germany). Other chemicals were of analytical grade and obtained from Merck Co. (Germany).

Methods

Chemical composition

Ash, protein, dietary fibers and fat contents of fresh dates were determined by standard methods (AOAC, 1997). Ashing was analyzed after combustion of a 3 g sample in a muffle furnace (Carbolit, UK) at 550°C for 8 h. Protein was calculated by Kjeldahl method (Gerhardt - 2000, Germany), using a factor of 6.25 for N to crude protein conversion. Fat was measured by weight loss by extraction at 60°C for 8 h with hexane using a Soxhlet apparatus (Gerhardt - Vapodest 30, Germany).

Sugar analysis by HPLC

Sugar analysis was performed by a HPLC instrument (Knauer-RF-10A XL, Germany), equipped with a Eurokat column, K1001 pumps and refractive index (RI) detector model K2301. After drying at 70°C for 72 h, dates were grinded and sieved. Date powder in amount of 0.03 g was shaken severely with 1.5 mL of 80% ethanol for 5 min followed by centrifuging at 3000 rpm for 10 min (Universal 32 centrifuge, Froilabo, SW14R, England). Liquid phase was poured in a 15 mL test tube and kept at 50°C until ethanol was evaporated completely. After ethanol evaporation, 10 mL of distilled water (acidified distilled water, pH 2.5) was added and the tube was shaken severely until all sugars were completely dissolved in water. In order to remove impurities, 0.5 mL of 5% zinc sulphate and 0.47 mL of 0.3 N barium hydroxide were added to the tubes and centrifuged at 3000 rpm for 10 min. Clear supernatant was filtered through a $0.45 \mu\text{m}$ polyethersulfone syringe filter and injected to a Eurokat column. Calibration curves were obtained using standard solutions of glucose, fructose and sucrose. Types and concentrations of sugars in the sample were determined by comparison with retention times of the standards (Bernárdez, 2004).

Preparation of date powder

A smooth paste of dates was obtained as described by Sablani et al. (2008) with slightly modification. After peeling and pitting, date fruits were minced using a laboratory mincer (MK-G20NR, National, Japan). Maltodextrin (drying additive), TCP (anti-caking agent) and GMS (flowability agent) were added to the date paste and mixed using a laboratory scale mixer (National, Japan). Different formulations of date paste were spread to a thickness of 5 mm on a plastic screen and dried in an oven dryer (Memmert-UE600, Germany) at 70°C and relative humidity (RH) level of 45% for approximately 16 h. After drying, the mixture was ground and passed through a testing sieve with the pore size of 500 µm. The collected date powders were stored in the airtight plastic containers. The following physico-chemical and thermal properties of the different date powders were determined.

Moisture content (MC)

Moisture content was measured by drying 3 g date powder at 70°C to constant weight (Farahnaky et al., 2016).

Bulk density (BD)

Bulk density of the date powder was measured by determining the mass and volume. Date powder was filled in a cylinder with known dimensions (36 mm diameter and 40.11 mm height). The cylinder was tapped several times and the excess powder was removed by sliding a ruler over the edge of the cylinder. Then mass of the date powder inside the cylinder was measured by an analytical balance (Sartorius, Germany) and the volume of the cylinder was calculated from its known dimensions (Sablani et al., 2008).

Color index

Color characteristics of the date powders were obtained using a Hunter lab colorimeter (Hunterlab – DP-9000, USA). The three dimensions of L* (lightness or brightness), a* (redness or greenness) and b* (yellowness or blueness) values of the powders were measured.

Solubility (S)

One g of powder was added to 100 mL distilled water and mixed with a high speed magnetic stirrer (Heidolph, MR Hei-Standard, Germany). To remove undissolved materials, the mixture was centrifuged at 7500 rpm for 10 min. Then, 25 mL of supernatant was dried at 105°C for 5 h and the solubility (%) was measured based on Eq. (1) (Cano-Chauca et al., 2005):

$$S = \frac{(m_1 - m_2)}{m_i} \times \left(\frac{25}{100}\right) \times 100 \quad (1)$$

where S is the solubility (%), m_1 is the weight of the container after drying, m_2 is the weight of the empty container and m_i is the initial weight.

Hygroscopicity (HG)

Hygroscopicity (HG) was measured based on the method described by Jaya and Das (2004). It was defined as the final

moisture content obtained after equilibrating the powders with a humid air at the RH level of 80.9%. Hygroscopicity was obtained by Eq. (2):

$$HG = \frac{\Delta m / (M + M_i)}{1 + \Delta m / M} \quad (2)$$

where Δm is the increase in weight of powder after equilibration, M is the initial weight of powder before equilibration and M_i is the initial moisture content of powder.

Glass transition temperature (T_g)

An approximately 5 mg powder was hermetically sealed in the 40 µL aluminum pan. T_g was determined by heating from -90 to 100°C at 10°C/min, using a Mettler Toledo, DSC1 (Switzerland) calibrated for temperature and enthalpy with Indium ($T_{m, onset} = 156.6^\circ\text{C}$, $\Delta H = 28.45\text{J/g}$). The reference was an empty aluminum pan. After the first heating, the samples were cooled at a rate identical to the rate of heating (10°C/min), and then the second heating cycle was performed. Glass transition temperature (T_g) was determined from the midpoint of the step-change in the specific heat of the sample in the second heating run with STARE system software (Badii et al., 2014).

Experimental design and statistical analysis

The response surface methodology (RSM) was applied to evaluate the effects of three different additives on the physico-chemical properties and the stability of date powder. Central composite rotational design (CCRD) with three numerical factors was used (Šumić et al., 2016). Independent variables were the amount of MD (X_1), TCP (X_2) and GMS (X_3). These variables had five levels and were coded according to Table 1. Design had twenty experimental runs, including six replicates at the central point (Table 2). The minimum and maximum levels for each variable were obtained based on the preliminary experiments. The response variables were different physico-chemical and thermal properties of date powder. A second order polynomial equation (Eq. 3) was used to describe relationship between the responses and the independent variables:

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 \quad (3)$$

where Y is the response variable; X_1 , X_2 and X_3 are independent variables; β_0 is the regression coefficient for intercept; β_1 , β_2 and β_3 are the regression coefficients for the linear effects; β_{12} , β_{13} and β_{23} are the regression coefficients for the interaction effects, and β_{11} , β_{22} and β_{33} are the regression coefficients for the quadratic effects of the independent variables.

Table 1. Coded levels for the independent variables

Variables	Coded levels				
	-1.68	-1	0	+1	+1.68
MD (%)	28.2	35	45	55	61.8
TCP (%)	0.32	1	2	3	3.68
GMS (%)	0.32	1	2	3	3.68

Table 2. Experimental design and responses to the independent variables

Treatments	MD	TCP	GMS	MC (%)	L*	a*	b*	BD (kg.m ⁻³)	Solubility (%)	HG (%)	T _g (°C)
1	0	2	0	2.65	72.90	7.63	28.19	890.20	73.50	5.31	23.61
2	1	-1	1	2.36	79.90	5.45	21.94	897.09	67.43	4.57	33.90
3	-1	1	1	2.73	69.42	8.77	26.70	895.31	74.97	5.98	33.11
4	-1	-1	-1	2.78	72.66	8.68	23.77	855.8	78.95	6.30	22.52
5	0	0	0	2.60	73.84	7.58	25.58	891.21	73.34	5.34	22.98
6	1	1	1	2.28	82.60	6.3	20.00	919.86	66.90	4.31	40.39
7	0	2	0	2.56	76.33	7.53	22.40	896.20	71.93	4.98	36.91
8	-1	1	-1	2.70	73.17	7.9	20.09	861.20	75.90	5.90	27.73
9	0	0	0	2.62	73.72	7.69	25.72	887.64	74.69	5.35	22.67
10	0	0	0	2.59	73.56	7.70	25.84	895.30	73.12	5.33	23.18
11	0	-2	0	2.65	71.34	7.85	25.37	872.20	75.20	5.50	24.40
12	0	0	0	2.60	72.53	7.69	23.72	890.69	72.94	5.30	21.20
13	0	0	1.68	2.62	74.55	7.70	21.24	881.16	69.70	5.40	27.85
14	1.68	0	0	1.98	83.60	4.57	18.00	927.42	63.20	3.76	42.77
15	0	0	-1.68	2.57	74.50	7.47	25.78	893.40	75.30	5.20	27.53
16	1	-1	-1	2.32	75.89	6.70	27.26	902.28	68.12	4.48	38.92
17	-1.68	0	0	2.90	67.87	9.01	25.00	824.30	80.00	6.71	18.83
18	-1	-1	1	2.83	68.43	8.26	25.95	854.20	77.50	6.32	21.08
19	0	0	0	2.60	71.40	7.32	23.36	893.22	73.52	5.36	21.94
20	1	1	-1	2.25	76.24	5.82	21.02	922.30	69.97	4.24	37.55
Control	0	0	0	3.19	55.07	15.24	34.81	-	91	7.93	-8.80

The optimum combination of independent variables for obtaining the optimum values for each response was achieved using the desirability method (Derringer and Suich, 1980). Statistical analysis, desirability and the 3-D response surface plots were obtained using Design-Expert software v.7 (Stat-Ease, Inc, MN, USA). Data was analyzed by analysis of variance (ANOVA) at the significant level of 0.01 ($P < 0.01$). The adequacy of the model was assessed by the coefficient of determination (R^2), model p-value and lack of fit testing.

Results and Discussion

Chemical composition of date fruit variety 'Shahani' is shown in Table 3. Date fruit contains 1.15% fat, 2.15% protein and 1.13% ash. It can be considered as a good source of fibers with approximately 15% of dry solid. Sugars are the major components of the date dry solid content. The main sugar molecules determined in the date flesh were glucose, fructose and sucrose

with 30%, 28.5% and 9.84%, respectively. The presence of these low molecular weight sugars has a significant adverse effect on the stability of the powder at temperatures above their T_g .

Table 3. Chemical composition of date flesh. Numbers are the mean \pm standard errors of three replications

Chemical composition	%	Chemical composition	%
Moisture	49.22 \pm 0.84	Total sugar	68.64 \pm 0.7
Lipid	1.15 \pm 0.02	Reducing sugar	58.8 \pm 0.15
Protein	2.15 \pm 0.06	Glucose	30 \pm 0.09
Ash	1.13 \pm 0.15	Fructose	28.5 \pm 0.1
Fiber	15 \pm 0.6	Sucrose	9.84 \pm 0.05

As it is shown in Table 2, T_g of the control date powder was approximately -8.8°C (far below the room temperature) and it shows that the powder is not self-stable at room temperature. Thus, keeping the control date powder at this condition may lead to physico-chemical deteriorative changes such as stickiness and caking of the powder.

In this study, the physico-chemical and thermal properties of date powder produced by adding different amount of MD, TCP and GMS were modeled using response surface methodology. Eq. (3) was fitted to the experimental data for eight responses shown in Table 2. The values of coefficient of determination (R^2) for each response are presented in Table 4. Based on high R^2 values for all the responses ($R^2 > 0.9$), except b^* (0.63), the regression models were successfully fitted to the experimental data. Since the value of R^2 was low for b^* chromaticity parameter result, the fitted model obtained by RSM was not adequate for this response and thereby, the results of statistical analysis for b^* value was not shown in Table 4. All the responses had relatively low CV (less than six) that represented small variation in the values of means and very high reproducibility of the experiments. Furthermore, non-significant lack of fit for the responses indicated that the mathematical models

were successfully capable to fit with the experimental results. The following mathematical model equations for seven responses after neglecting insignificant coefficients were obtained and successfully used to describe the effects of independent variables (MD, TCP and GMS) on these responses within experimental range of variables.

$$MC = 2.60 - 0.25MD - 0.04TCP - 0.06MD^2 \quad \text{Eq.4}$$

$$BD = 890.48 + 28.15MD + 6.81TCP - 2.32GMS + 4.04MD \times TCP + 4.96MD^2 - 1.94TCP^2 \quad \text{Eq.5}$$

$$L^* = 73.61 + 4.20MD + 0.95TCP + 2.29MD \times GMS + 0.89MD^2 \quad \text{Eq.6}$$

$$a^* = 7.59 - 1.23MD - 0.15MD \times GMS + 0.38TCP \times GMS - 0.31MD^2 \quad \text{Eq.7}$$

$$T_g = 22.45 + 6.34MD + 3.18TCP - 1.52MD \times TCP + 1.84TCP \times GMS + 3.27MD^2 + 3.22TCP^2 + 2.17GMS^2 \quad \text{Eq.8}$$

$$HG = 5.32 - 0.87MD - 0.15TCP + 0.04GMS + 0.03MD \times TCP - 0.03MD^2 - 0.03TCP^2 \quad \text{Eq.9}$$

$$S = 73.25 - 4.62MD - 0.71TCP - 1.14GMS + 0.86MD \times TCP - 0.65MD^2 \quad \text{Eq.10}$$

Table 4. Analysis of variance and R^2 values for response variables

Regression coefficient	Responses						
	MC (%)	L^*	a^*	BD (kg.m^{-3})	Solubility (%)	HG (%)	T_g ($^\circ\text{C}$)
β_0	2.60	73.61	7.59	890.48	73.25**	5.32	22.54
Linear							
β_1	-0.25**	4.2**	-1.23**	28.15**	-4.62**	-0.87**	6.34**
β_2	-0.035**	0.95**	n.s.	6.81**	-0.71**	-0.15**	3.18**
β_3	n.s.	n.s.	n.s.	-2.32**	-1.14**	0.04**	n.s.
Interactions							
β_{12}	n.s.	n.s.	n.s.	4.04**	0.86**	0.03**	-1.52**
β_{13}	n.s.	2.29	-0.15	n.s.	n.s.	n.s.	n.s.
β_{23}	n.s.	n.s.	0.38	n.s.	n.s.	n.s.	1.84**
Quadratic							
β_{11}	-0.061**	0.89**	-0.31**	4.96**	-0.65**	-0.03**	3.27**
β_{22}	n.s.	n.s.	n.s.	-1.94**	n.s.	-0.03**	3.22**
β_{33}	n.s.	n.s.	n.s.	n.s.	n.s.	n.s.	2.17**
Model (F-value)	311.99**	68.74**	240.58**	180.39**	65.79**	2647.16**	64.81**
R^2	0.98	0.95	0.98	0.98	0.97	0.99	0.97
Lack of fit (p)	0.16	0.36	0.43	0.23	0.17	0.37	0.10
CV	1.22	1.42	2.11	0.38	1.17	0.49	5.33

n.s.: not significant; **: significantly different ($P < 0.01$)

Moisture content

Moisture content (MC) is a decisive factor for different deteriorative microbial and chemical reactions that have impacts on physical stability and storage life of dried powders. Moisture content of the pure date powder (3.19%) was significantly higher than those of powders obtained by adding different amounts of MD, TCP and GMS (Table 2). As can be seen from Table 2, the lowest moisture content was obtained for the powder with 61.8% MD, 2% TCP and 2% GMS, while the highest MC was obtained with 28.2% MD, 2% TCP and 2% GMS. Table 4 represents the effects of three independent variables on the moisture content of the final date powders. The proposed model (Eq. 4) showed that MD and TCP had negative significant linear effects on moisture content ($p < 0.01$). Maltodextrin had greater impact on moisture content of the dried powder and the linear effect of MD was approximately six times higher than that of TCP. Maltodextrin also showed a quadratic effect on moisture content. As an antiplastcizing agent, MD reduced the final moisture content of the date powder more effectively. However, adding GMS did not show significant effect on the amount of MC of the date powders. Moisture content is a very important quality indicator for powders and it is desirable to keep it low during storage. Negative influences of MD and TCP on the moisture content of final powders show that they reduce the susceptibility of powders to water. Fig 1a shows that MC of powder decreased by increasing the percentage of MD and altering the amount of TCP had smaller effect on MC, compared to MD. As a drying additive, MD had the same effect on date syrup powder (Farahnaky et al., 2016), freeze dried Camu-Camu pulps (Silva et al., 2006) and pineapple pulp powder (Gabas et al., 2007). The moisture content of spray dried date powder with 40% MD was in the range of 1.5 to 4.4% (Manickavasagan et al., 2015).

Bulk density

Bulk density of powders is a key factor for optimizing packaging methods and handling practices (Santhakshmy et al., 2015). Bulk density of the date powder was a function of additives and increased by adding heavier molecules like MD (Sablani et al., 2008; Jaya and Das, 2004). The results of analysis of variance showed that both MD and TCP had significant positive linear effect, while GMS showed a negative linear effect on the bulk density of the powder. Heavier molecules occupy the spaces between the particles more easily, resulting in a denser powder with a higher bulk density value (Santhakshmy et al., 2015). The interaction of MD and TCP was significant and both MD and TCP showed negative significant effects on their quadratic terms ($p < 0.01$) (Table 4). Figures 1b and 1c show bulk density of the powders obtained by adding different percentage of MD, TCP and GMS. The values of BD ranged from 927.42 to 824.30 kg/m^3 for the powder containing 61.8% MD, 2% TCP, 2% GMS and 28.2% MD, 2% TCP, 2% GMS, respectively. The obtained regression equation (Eq. 5) after excluding insignificant coefficients shows that MD has a highly significant effect on bulk density compared to other independent factors. The addition of bigger sized and heavier molecules such as MD to the mixture resulted in producing denser powders with higher bulk density (Sablani et al., 2008). In contrast, GMS as a flowability agent reduced bulk density. Date powders obtained by a spray dryer (Manickavasagan et al., 2015) and a drum dryer (Farahnaky et al., 2016) had lower bulk density compared to that of obtained by an oven dryer (Sablani et al., 2008).

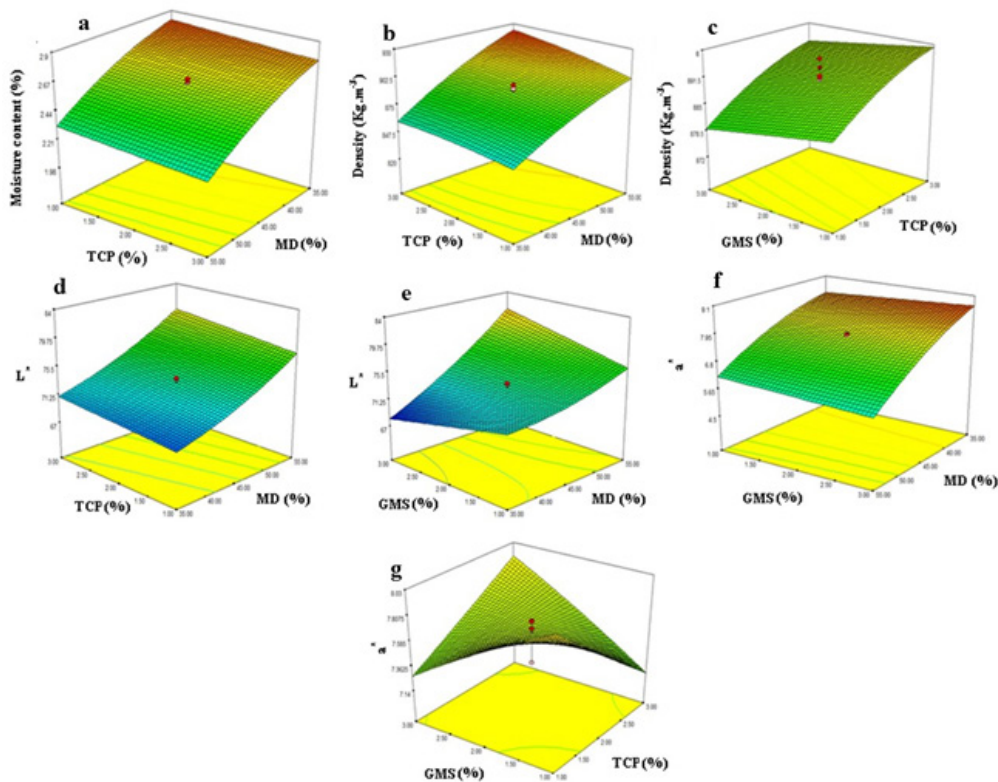


Figure 1. Three dimensional plots for moisture content (a), bulk density (b, c), L^* (d, e) and a^* (f, g) of date powder as functions of maltodextrin, tricalcium phosphate and glycerol monostearate

Color

The original date powder was a dark reddish powder with L^* , a^* and b^* values of 55.07, 15.34 and 34.81, respectively (Table 2), while MD, TCP and GMS were whitish powders with L^* values above 90. Therefore, the color profile of the mixture significantly varied by changing the proportion of the additives. Adding of these powders to the date paste resulted in a rise in the whiteness (L^*) and a reduction in the redness (a^* values) of the final date powder. As can be seen from Table 4, the linear and quadratic terms of MD and the linear term of TCP had positive significant effects on the L^* value of the powders. Besides, interaction effect of MD and GMS was significant and positive. Therefore, the polynomial Eq. (6) was best fitted to the experimental data of L^* . Figures 1d and 1e show that the L^* values increase by adding MD and TCP. However, the impact of MD was more pronounced. The highest value of L^* was obtained at the experimental point with the highest amount of MD (61.8% MD, 2% TCP and 2% GMS). In contrast, for the sample containing lowest amount of MD, the minimum brightness was obtained (28.2% MD, 2% TCP and 2% GMS). The color of MD was white whereas the date paste was dark red, thus, by increasing the proportions of this whitish powder in the date paste, the color profile of the mixture was changed, and the whiteness (L^* value) of the powder increased, and subsequently, the redness (a^* value) of the powder decreased (Sablani, et al., 2008).

Table 4 shows that the linear and quadratic effects of MD had negative significant effects on a^* value, while the interaction effects of MD and GMS (negatively) and TCP and GMS (positively) influenced the response. Therefore, the polynomial Eq. (7) was developed for predicting a^* values of the powder as a function of these additives over the investigated experimental range. It can be seen from Fig 1f and 1g that the lowest a^* was observed on the experimental point of 61.8% MD, 2% TCP and 2% GMS, while the highest a^* was obtained for powder containing 28.2% MD, 2% TCP and 2% GMS. The date powders containing 45% MD (Table 2) had comparable color values to the spray dried date powder with 40% MD (Manickavasagan et al., 2015).

Glass transition temperature (T_g)

Several important powder properties like stickiness, solubility and caking are governed by T_g . The results of analysis of variance in Table 4 show that the linear terms of MD and TCP had significant positive effects on T_g , while the interaction effects of MD and TCP (negatively) and TCP and GMS (positively) influenced the response. Interestingly, the quadratic effects of all the variables positively influenced T_g (Table 4). Therefore, the polynomial Eq. (8) was developed for describing the effects of adding MD, TCP and GMS on the T_g of the final powder and predicting its value over the investigated experimental range. T_g of control date powder was -8.8°C , while the T_g values of MD and TCP were obtained 79.4 and 61.86°C , respectively. High molecular weight substances such as MD act as antiplasticizing agents and increase the T_g of the fruit powder and reduce the stickiness and hygroscopicity of the powder. Therefore, adding different proportions of these additives to the date paste increased T_g significantly and altered it from 18.83°C to 42.77°C . As can be seen from Figures 2a and 2b, the lowest T_g value was observed on the experimental point of 28.2% MD, 2% TCP and 2% GMS, while the highest T_g was obtained for powder containing 61.8% MD, 2% TCP and 2% GMS.

Table 1 shows that about 68% of date dry matter consists of sugars. The presence of low molecular weight amorphous sugars such as glucose and fructose in date powder reduced the T_g to around -9°C , far below room temperature (Table 3) and this explains why control date powder is sticky and highly hygroscopic at room temperature. In order to overcome the stickiness of sugar enriched date powder at room temperature, the T_g of the powder should be increased. Since MD and TCP have higher values of T_g , adding these molecules can increase the overall T_g and sticky point of the powder, and improve the stability and storage quality of the powder at room temperature.

Hygroscopicity

Statistical analysis showed that the linear terms of MD and TCP had negative impact, whereas the linear term of GMS exhibited positive influence on the hygroscopicity of the powders. Besides, interaction effect of MD and TCP was significant and positive. Quadratic terms of MD and TCP had negative significant effect on HG (Table 4). Thus, Eq. (9) was proposed for describing the effects of adding MD, TCP and GMS on the hygroscopicity of the date powder.

By increasing the T_g , hygroscopicity of the obtained powders declined. According to Fig 2c and 2d, the lowest amount of HG was observed for the powder with the highest T_g (61.8% MD, 2% TCP and 2% GMS) and the highest value of HG was at the experimental point with the lowest T_g (28.2% MD, 2% TCP and 2% GMS). The hygroscopicity of the date powder declined by increasing the amount of MD. Since the majority of the date powder consists of low molecular weight sugars with amorphous structure that are very hygroscopic, the addition of MD as a macromolecule with high molecular weight reduces the overall hygroscopicity of the powder, which leads to improved stability and storage-life of the date powder. The hygroscopicity of the powders obtained in our study and that of obtained by Sablani et al. (2008) are in good agreement. However, the hygroscopicity of the spray dried date powder was considerably lower (Manickavasagan et al., 2015). The presence of MD can modify the balance of hydrophilic/hydrophobic sites in the date powder. Therefore, the surface stickiness of the low molecular weight sugars and their hygroscopicity decrease (Farahnaky et al., 2016).

Solubility

Solubility and other properties such as moisture content, bulk density and dispersion are of importance to determine the easiness of powder for reconstitution (Mahendran, 2011). Solubility of the original date powder was 91% (Table 2). Adding different amounts of MD, TCP and GMS had adverse effects on the solubility of the powder. Table 4 represents the effects of three independent variables on the solubility of the final date powders. Maltodextrin, TCP and GMS had negative significant linear effect on solubility ($p < 0.01$) and the linear effect of MD was significantly higher than those of two other factors. Also, MD showed a negative quadratic effect on powder solubility, while the interaction effect of MD and TCP influenced the response, positively. Thus, Eq. (10) was proposed for explaining the effects of adding MD, TCP and GMS on the solubility of the date powder. As can be seen from Fig 2e and 2f, all three additives reduced the solubility of date powder. The lowest solubility was obtained for the powder with 61.8% MD, 2% TCP and 2% GMS, while the powder containing 28.2% MD,

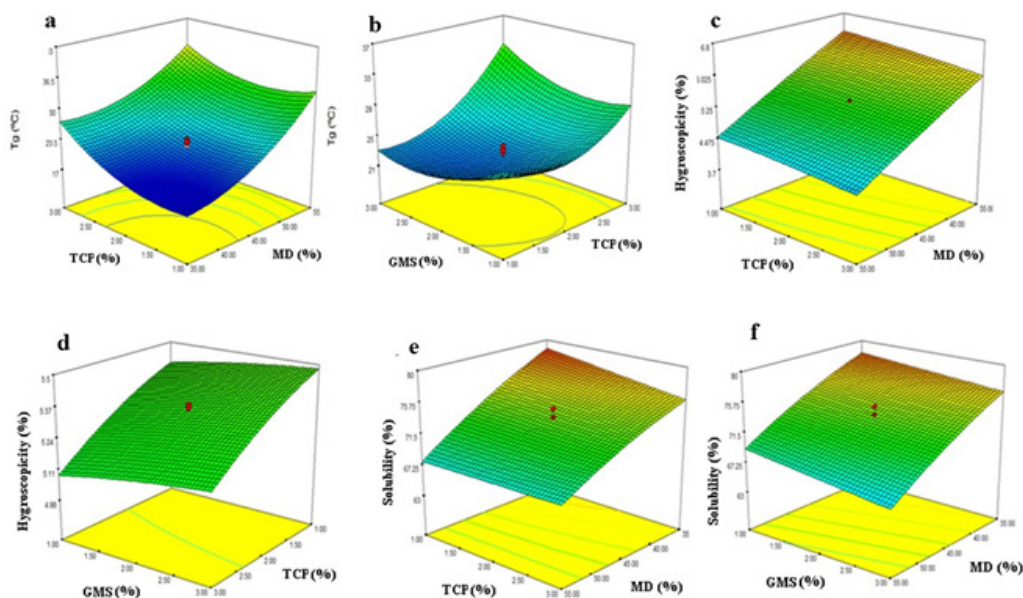


Figure 2. Three dimensional plot for T_g (a, b), hygroscopicity (c, d) and solubility (e, f) of date powder as functions of maltodextrin, tricalcium phosphate and glycerol monostearate

2% TCP and 2% GMS showed highest solubility. Maltodextrin had the same effect on the solubility of pineapple powder and guava powder (Gabas et al., 2007; Mahendran, 2011). Compared to other drying additives, MD and gum Arabic are considered as good carrier agents for drying fruit juices, as a result of having high solubility in water. However, since the original date powder consists of about 70% low molecular weight sugars that are easily soluble in water, by adding MD, TCP and GMS, the percentage of sugars and hydrophilic groups decreased in the powder leading to lower solubility. The solubility of spray dried date powder with 40% MD was in the range of 66-87% (Manickavasagan et al., 2015).

Optimization

Optimization of the date powder production was obtained using Design-Expert software, The main criteria parameter for designing the optimal conditions was the T_g , since the stickiness of dried sugar enriched powders normally occurs in the rubbery state when the transition from glassy to rubbery state takes place. Amorphous materials show sticky behavior at approximately 10 - 20°C above their T_g (Roos and Karel, 1991; Jaya and Das, 2009). Therefore, the T_g should be above 25°C (storing temperature) in order to make sure that the date powders (obtained in this study) are in the glassy state during storage at room temperature. At the same time, the obtained powder should have minimum hygroscopicity, moisture content and lightness and maximum solubility and redness. The optimal proportions of additives were achieved by Design-Expert software as 35% MD, 3% TCP and 3% GMS with the overall desirability of 82.3%. At this optimum condition, the responses were determined as T_g of 31.5°C, hygroscopicity of 6.03, Solubility of 74.46%, a^* value of 8.96 and lightness of 69.28. Therefore, a self-stable date powder at room temperature was obtained with the lowest amount of MD (35%) compared to the date powders obtained in other studies (Sablani et al., 2008; Farahnaky et al., 2016; Manickavasagan et al., 2015).

Conclusion

The stickiness of the date powder is mainly due to its low T_g as a result of the presence of low molecular weight sugars. To overcome the problem of stickiness and caking of the date powder, large molecular weight materials such as MD as drying aid are required. Date powder was successfully obtained by adding MD, TCP and GMS. Response surface methodology was applied to obtain optimal combinations of MD, TCP and GMS in order to achieve a self-stable date powder with the best quality. Optimal amount of additives was 35% MD, 3% TCP and 3% GMS with the overall desirability of 82.3%. Based on the satisfactory statistical analysis, the generated mathematical models were successfully used to obtain physico-chemical and thermal properties of the date powder. More research is being carried out currently on the technological applications and sensory properties of the date powder.

Acknowledgments

This study was supported by the Agricultural Engineering Research Institute (AERI).

References

- Abadio, F.D.B., Domingues, A.M., Borges, S.V., Oliveira, V.M. (2004). Physical properties of powdered pineapple (*Ananas comosus*) juice: effect of maltodextrin concentration and atomization speed. *J Food Eng* 64: 285-287.
- AOAC, (1997). Official Methods of Analysis. 16th edn, Association of Official Analytical Chemists, Washington DC.
- Badii, F., Farahnaky, A., Behmadi, H. (2014). Effect of Storage Relative Humidity on Physical Stability of Dried Fig. *J Food Process Preserv* 38: 477-483.
- Bernárdez, M.M., De la Montaña Miguélez, J., Queijeiro, J.G. (2004). HPLC determination of sugars in varieties of chestnut fruits from Galicia (Spain). *J Food Comp Anal* 17: 63-67.

- Blanshard, J.M.V. (1995). The glass transition, its nature and significance in food processing. In: Physico-chemical aspects of food processing (Beckett S.T., ed), Springer, US, pp. 17-48
- Cano-Chauca, M., Stringheta, P.C., Ramos, A.M., Cal-Vidal, J. (2005). Effect of the carriers on the microstructure of mango powder obtained by spray drying and its functional characterization. *Innov Food Sci Emerg Technol* 6: 420-428.
- Champion, D., Le Meste, M., Simatos, D. (2000). Towards an improved understanding of glass transition and relaxations in foods: molecular mobility in the glass transition range. *Trends Food Sci Technol* 11: 41-55.
- Derringer, G., Suich, R. (1980). Simultaneous optimization of several response variables. *J Quality Technol* 12: 214-219.
- Farahnaky, A., Mansoori, N., Majzoobi, M., Badii, F. (2016). Physicochemical and sorption isotherm properties of date syrup powder: Antiplasticizing effect of maltodextrin. *Food and Bioprod Process* 98: 133-141.
- Gabas, A.L., Telis, V.R.N., Sobral, P.J.A., Telis-Romero, J. (2007). Effect of maltodextrin and arabic gum in water vapor sorption thermodynamic properties of vacuum dried pineapple pulp powder. *J Food Eng* 82: 246-252.
- Jaya, S., Das, H. (2004). Effect of maltodextrin, glycerol monostearate and tricalcium phosphate on vacuum dried mango powder properties. *J Food Eng* 63: 125-134.
- Jaya, S., Das, H., Mani, S. (2006). Optimization of maltodextrin and tricalcium phosphate for producing vacuum dried mango powder. *Int J Food Prop* 9: 13-24.
- Jaya, S., Das, H. (2009). Glass transition and sticky point temperatures and stability/mobility diagram of fruit powders. *Food Bioprocess Technol* 2: 89-95.
- Mahendran, T. (2011). Physico-chemical properties and sensory characteristics of dehydrated guava concentrate: effect of drying method and maltodextrin concentration. *Tropical Agric Res Exten* 13: 48-54.
- Manickavasagan, A., Thangavel, K., Dev, S.R.S., Aniesrani, D.S., Nambi, E., Orsat, V., Raghavan, G.S.V. (2015). Physicochemical characteristics of date powder produced in a pilot scale spray dryer. *Drying Technol* 33: 1114-1123.
- Mosquera, L.H., Moraga, G., Martínez-Navarrete, N. (2012). Critical water activity and critical water content of freeze-dried strawberry powder as affected by maltodextrin and arabic gum. *Food Res Int* 47: 201-206.
- Papadakis, S.E., Gardel, C., Tzia, C. (2006). Spray drying of raisin juice concentrate. *Drying Technol* 24: 173-180.
- Roos, Y.H. (2003). Thermal analysis, state transitions and food quality. *J Therm Anal Calorim* 71: 197-203.
- Roos, Y., Karel, M. (1991). Amorphous state and delayed ice formation in sucrose solutions. *Int J Food Sci Technol* 26: 553-566.
- Sablani, S.S., Shrestha, A.K., Bhandari, B.R. (2008). A new method of producing date powder granules: Physicochemical characteristics of powder. *J Food Eng* 87: 416-421.
- Sahu, J.K. (2008). The effect of additives on vacuum dried honey powder properties. *Int J Food Eng* 4: 1-26.
- Santhalakshmy, S., Bosco, S.J.D., Francis, S., Sabeena, M. (2015). Effect of inlet temperature on physicochemical properties of spray-dried jamun fruit juice powder. *Powder Technol* 274: 37-43.
- Shavakhi, F., Boo, H.C., Osman, A., Ghazali, H.M. (2011). Effects of enzymatic liquefaction, maltodextrin concentration, and spray-dryer air inlet temperature on pumpkin powder characteristics. *Food Bioprocess Technol* 5: 2837-2847.
- Silva, M.A., Sobral, P.J.A., Kieckbusch, T.G. (2006). State diagrams of freeze-dried camu-camu (*Myrciaria dubia* (HBK) Mc Vaugh) pulp with and without maltodextrin addition. *J Food Eng* 77: 426-432.
- Slade, L., Levine, H., Reid, D.S. (1991). Beyond water activity: recent advances based on an alternative approach to the assessment of food quality and safety. *Crit Rev Food Sci Nutr* 30: 115-360.
- Šumić, Z., Vakula, A., Tepić, A., Čakarević, J., Vitas, J., Pavlič, B. (2016). Modeling and optimization of red currants vacuum drying process by response surface methodology (RSM). *Food Chem* 203: 465-475.