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Vacuum drying of concentrated malta juice for production of malta powder using RSM

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Abstract

The malta powder was produced using a vacuum dryer under various drying conditions. Fresh concentrated malta juice was added with methyl cellulose, whole milk powder, tricalcium phosphate and glycerol monostearate at 0.08 to 0.016, 0.02 to 0.06, 0.0025 to 0.0045 and 0.005 to 0.015kg/kg of malta solid before exposing to the different drying temperatures at 65, 70, 75, 80, and 85°C in different time intervels as 0, 20, 60, 100, 140, 260, 380, 500, 620, 740 minutes. The drying rate of sample 24 was found higher due to added ingredients of methyl cellulose, tricalcium phosphate and glycerol monostearate. The different models were fit ed to the experimental data to predict the drying time, the generalized exponential model was found best, the predicted drying time at temperature (85°C) and EMC (1.49%db) was 322 minutes for the sample 24 to produce good quality malta powder.

Highlights

- The concentrated malta juice was dried in vacuum dryer to produce malta powder

Keywords: Concentrated malta juice, drying time, drying temperature, TCP and GMS

Citrus fruits occupy a predominant place in the fruit industry of Ut arakhand state. Total production of citrus fruits in Ut rakhand is 723.6 thousand metric tonne in area of 193.8 thousand hectare in 2009-2010 (NHB) out of which sharing about 14.38 percent of the total area under fruits and contributing about 17.75 percent to the total fruit production (Anonymous, 2006). The most important commercial citrus cultivar is sweet orange. Among these cultivars "malta Common" is grown on a large scale in Punjab, Haryana, Ut arakhand and Western district of Ut ar Pradesh. "Malta Common" one of the most important cultivar of citrus fruit, is grown in most of the hilly districts of the state Ut rakhand like Ghat, Mandal, Nagnath, Pakhari area of district Chamoli, Quiti, Thal, Berinag, Didihat of district Pithoragarh, and some areas of districts Rudraprayag and Almora. The high acceptability of "Malta Common" is due to its at ractive colour, distinctive flavor and taste. The excellent quality fruits are generally available for only one or two months. However owing to its poor shelf-life, fruits cannot be stored for longer period under ambient conditions and cannot be transported to distant places. Therefore, to supply this commodity throughout the year, the surplus during the season has to be processed into a variety of dehydrated and value added products (Saxena and Arora, 1997; Srinivasan *et al.* 2000).

Foam is defined as a mass of small gas cells separated by thin films of liquid and formed by the juxtaposition of bubbles, giving a gas dispersed in a liquid (BIS, 1983). In foam mat drying, a liquid material is converted into stabilized foam by whipping after the addition of edible foaming agents. The foam is then spread out in a sheet or mat and dried by means of heated air at atmospheric pressure. Many tiny bubbles in the foaming mass expose to larger surface area for moisture evaporation. The rapid drying is due to the moisture movement by capillarity in the liquid films separating foam bubbles. The foaming renders the drying mass extremely porous and more amenable to drying to its inner most layers (Berry *et al.* 1965; Hart *et al.* 1963; Morgan *et al.* 1961 and Venkataraman, 1996).

Foam mat drying technique can be used for heat sensitive, sticky, viscous and high sugar content food products (Chandak and Chivate 1972; Labelle, 1984 and Srinivasan, 1996). The method of drying used for this study was vacuum drying that has been successfully applied to many fruits and vegetables and other heat sensitive foods. In this method since, the drying takes place in the absence of oxygen, the oxidative degradation e.g. browning is low in the final product. The rate of drying is fast due to the creation of frothy or puffed structure in the juice concentrate (Jaya and Das 2004). Vacuum dried materials are characterized by better quality retention of nutrients and volatile aroma. However, the cost of the process is high (Tsami *et al.* 1999). In view of the above, present study was undertaken with the objective as (a)To study the effect of Carboxy methyl cellulose, tricalcium phosphate, glycerol monosterate, whole milk powder and drying temperature of concentrated malta juice.

Materials and Methods

Raw materials

Fresh, well graded, good quality fully ripe Malta (sweet orange) of variety Valencia was purchased from the Haldwani Sabji Mandi Dist. Nainital Uttrakhand and was used for the production of free flowing malta powder. Standard chemicals were used

Preparation of sample

The malta fruits were sorted and the fleshy portion of malta were separated manually with the help of stainless steel knife and the juice was extracted using juicer machine (Modern Scientific Sales Corporation Delhi, Model No. 626C). The malta juice was concentrated upto 40° Brix using vacuum rotary evaporator (JSGW) at 80°C for 4 hrs at constant speed of variac. The malta concentrate juice was kept in containers and stored under refrigerated conditions (5°C) till further use. The values of independent variables are given in Table 1.

Design of experiments

When the process variables satisfy an important assumption that they are measurable, continuous, and controllable by experiments, with negligible errors, the RSM procedure was carried out as follows:

(1) A series of experiments were performed for adequate and reliable measurement of the response of interest.

(2) A mathematical model of the second order response surface with the best fit was developed.

(3) The optimal set of experimental parameters producing the optimum response value was determined.

A central composite design (CCD), which is very efficient design tool for fitting second order models (Montgomery, 2001), was selected for use in this study. The number of tests required for a central composite design include: the standard 2^k factorial points, 2k axial point fixed at a distance, say α, from the center to generate quadratic terms, and replicate tests at the centre of experimental region; where k is the number of variable. Replicates of the test at the center are very important as they provide an independent estimate of the experimental error. A central composite design for 5 factors (CMC, Temperature, WMP, TCP and GMS), with 6 replicates at the center resulting in total 2^{k-1}+2K+L i.e. 2^{5-1}+2×5+6=32 (Khuri and Cornell, 1987). A CCD is made rotatable by the choice of α. A value of α= $2^{(k-1)/4}$=$2^{(5-1)/4}$=2 assures rotation of the CCD (Box and Hunter, 1957). In this study k was 5 factors, therefore α become 2. The codes were calculated as functions of the range of interest of each factor, as shown in Table 2. In order to determine if a relationship existed between the factors and the responses investigated, the collected data was analyzed statistically using regression analysis. A regression design is employed to model a response as a mathematical function of a few continuous factors and 'good' model parameter estimates are desired (Montgomery, 2001). Each response of Y can be represented by a mathematical equation that correlates the response surface. The responses can be expressed as a second order polynomial equation, according to Eq. (1).

$$Y = f(x) = \beta_0 + \Sigma_i^k = {}_1\beta_i X_i + \Sigma_i^k = {}_1\beta_{ii} X_i^2 + \Sigma_{i=1}^{k-1}\beta_{ii}\Sigma_{j=i+1}^{k}\beta_{ij} X_i X_j \quad (2.1)$$

Where Y is the predicted response used as a dependent variables; k the number of independent variables, x_i (i = 1, 2, 3, 4, 5) the input predictors or controlling variables: β_0 the constant coefficient, β_i, β_{ij}, and β_{ii} the coefficients of linear, interaction and quadratic term, respectively. The coefficient parameters were estimated using a multiple linear regression analysis employing the software Design –expert (version 8.0.1).

Vacuum drying for the preparation of malta powder

The malta concentrate mixture was dried in a laboratory model vacuum dryer (MSW-218).The mixture after suitable addition of drying aids was spread evenly into petri dishes, having diameter of 15 cm. The petri dishes were then placed on the shelves of the vacuum dryer and the pressure was reduced until the vacuum inside the chamber reached to 25″ Hg. Five different drying temperatures (65, 70, 75, 80, 85°C) and drying time (0, 20, 140, 260, 380, 500 and 600 minutes) were selected to carry out vacuum drying on the basis of preliminary trials. The drying behavior was studied in terms of moisture content (% db) with respect to time and temperature. The dried malta concentrate was powdered into a fine particulate powder using a food processor at medium speed for 5min.

Data analysis

Equilibrium moistiure content

Hygroscopicity is a fundamental characteristic of biological materials. When such material exposed to a given atmosphere, they have a tendency to lose or gain moisture depending on temperature and relative humidity of surrounding atmosphere and their own moisture content. Equilibrium Moisture Content was required for calculations of moisture ratio (MR). It was determined using a method developed by Chakerverty, (1988) in which last three moisture content readings of drying experiment were taken. Equation was used to determine the equilibrium moisture content.

$$Me = \frac{M_1 \times M_3 - (M_2)^2}{M_1 + M_3 - 2M_2} \quad \ldots(2.2)$$

Where, M_1–Moisture content (%db) at time t_1, M_2-Moisture content (%db) at time t_2 and M_3–Moisture content (% db) at time t_3

Moisture ratio and drying rate

Moisture Ratio (MR) is calculated by using this formula.

$$MR = \frac{M - M_e}{M_o - M_e} \quad \ldots(2.3)$$

Where, M–Average moisture content (% db) at time t (min) during drying, M_0–Moisture content (% db) at the initiation of drying i.e. at 0 time and Me–Equilibrium moisture content (% db)

Drying Rate is calculated by using this formula

$$\frac{dm}{dt} = \frac{M_2 - M_1}{\Delta t} \quad \ldots(2.4)$$

Where, ∆t – difference in time.

Empirical modeling

The mathematical modeling of drying process helps in understanding the physics of drying. Drying behavior of the sample can be shown graphically. However, there is a need to develop mathematical models to predict the drying time. Many at empts have been made to develop analytical and empirical models. The moisture ratio curve data were fit ed into Page's, generalized exponential and modified page model in order to select the best predictive model for vacuum drying of concentrated malta juice. The following models are used as:

Page's Model

$$MR = \frac{M - M_e}{M_o - M_e} = e^{-Kt^N} \quad \ldots (2.5)$$

Where, t- Drying time, min and K, N- constants of page's equation

Generalized Exponential Model

$$MR = \frac{M - M_e}{M_o - M_e} = e^{-(Kt)^N} \quad \ldots (2.6)$$

Where, K, A-drying constants

Modified Page's Model

$$MR = \frac{M - M_e}{M_o - M_e} = e^{-(Kt)^N} \quad \ldots (2.7)$$

Where, K, N- constants of page's equation

Results and Discussion

The drying behavior of Malta (sweet orange) concentrated juice was analyzed for the production of malta powder in vacuum foam mat drying at different temperature (65-85°C) and at different levels of ingredients of drying aid-carboxy methyl cellulose (4-8%), whole milk powder (1-3%), anticaking agent-tricalcium phosphate (0.125-0.225%) and foam stabilizer-glycerol monostearate (0.25-0.75%). The weight of malta concentrated juice was recorded at an interval of 0, 20, 60, 100, 140, 260, 380, 500, 620 and 740 minutes. The moisture content on dry basis was calculated using the moisture loss data and then moisture ratio and drying rate were determined. The selected mathematical models namely Page's, Generalized exponential and Modified Page's were used to describe the drying behavior of concentrated malta juice. The goodness of fit of the model was tested using coefficient of determination (R^2), Fisher's F-test and standard error of estimation (SEE).

Drying behavior

Moisture content of the sample was in the range of 84 to 140% (db) depending on the quantity of added ingredients before drying, while it was in the range of 1.22 to 5.86% (db) af er drying in vacuum dryer. The values of moisture content, moisture ratio and drying rate were calculated. The drying curves of samples in relationship between moisture ratio versus drying time, drying rate versus drying time and drying rate versus moisture content are given in Figs. 1-3. The relationship between moisture ratios with drying time for all 32 experiments is shown graphically in Figs.1 (a), (b), (c), (d), (e), (f), (g) and (h), which exhibit a non linear decrease of moisture ratio with drying time of each sample. Initially, moisture ratio of each samples decreased rapidly while very slowly towards the end. As expected, the drying time varied with drying temperature and level of added ingredients.

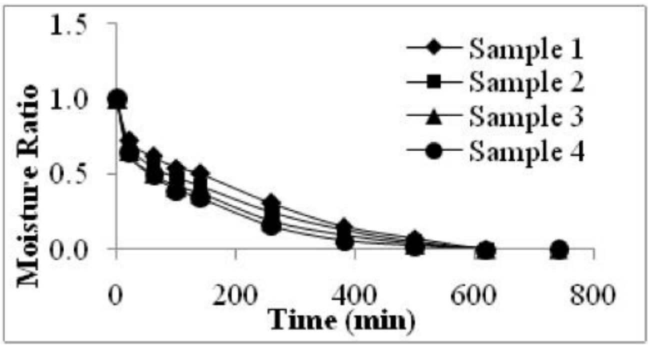

Fig.1(a) Variation of Moisture ratio with drying time for sample 1, 2, 3 and 4.

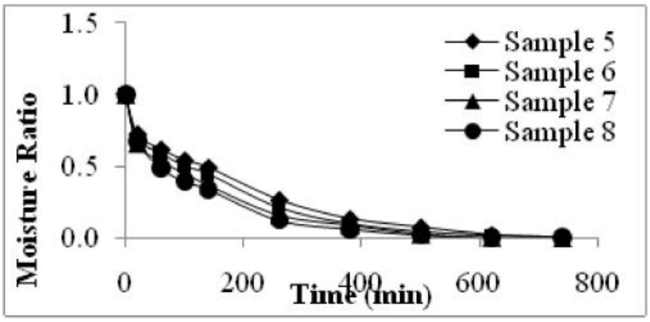

Fig. 1(b) Variation of Moisture ratio with drying time for sample 5, 6, 7 and 8.

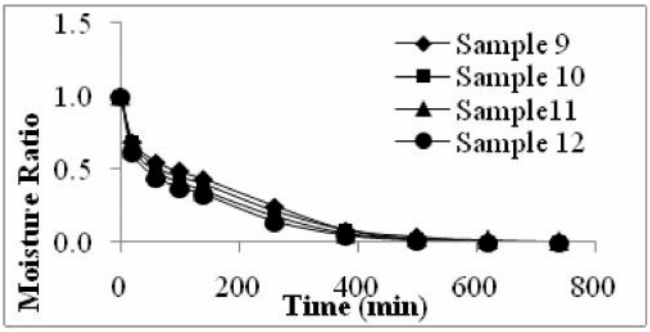

Fig. 1(c) Variation of Moisture ratio with drying time for sample 9, 10, 11 and 12.

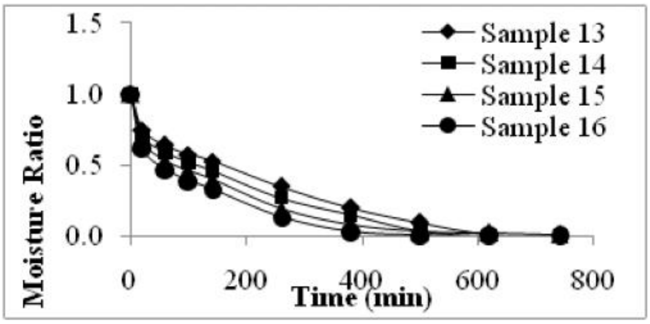

Fig. 1 (d) Variation of Moisture ratio with drying time for sample 13, 14, 15 and 16.

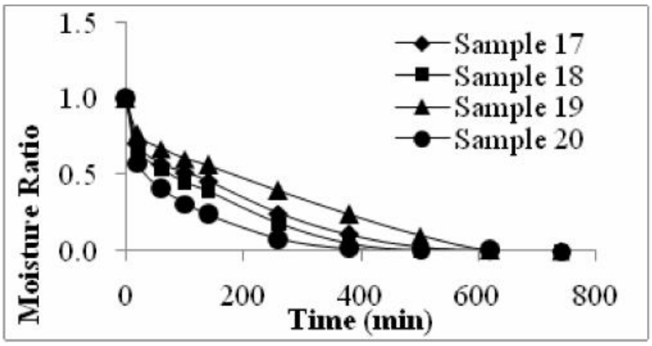

Fig. 1 (e) Variation of Moisture ratio with drying time for sample 17, 18, 19 and 20.

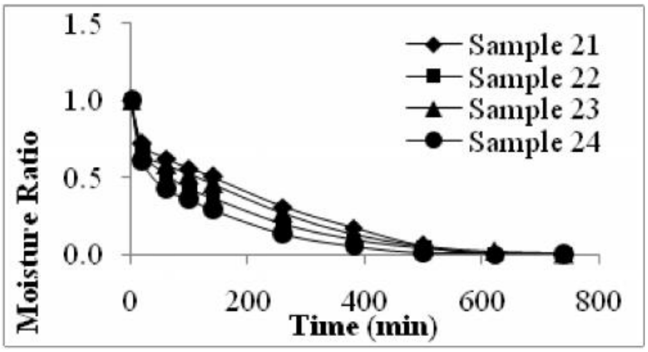

Fig. 1 (f) Variation of Moisture ratio with drying time for sample 21, 22, 23 and 24.

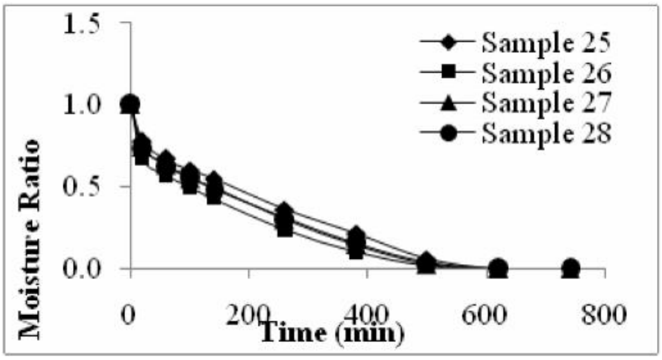

Fig. 1 (g) Variation of Moisture ratio with drying time for sample 25, 26, 27 and 28.

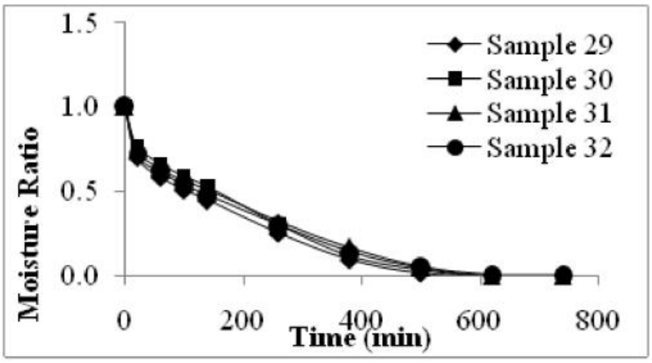

Fig. 1 (h) Variation of Moisture ratio with drying time for sample 29, 30, 31 and 32.

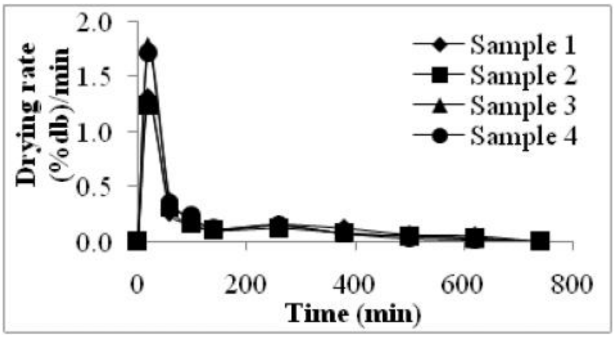

Fig. 2 (a) Variation of drying rate with drying time for sample 1, 2, 3 and 4.

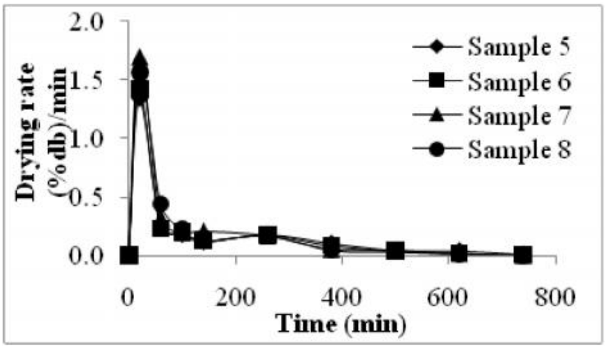

Fig. 2 (b) Variation of drying rate with drying time for sample 5, 6, 7 and 8.

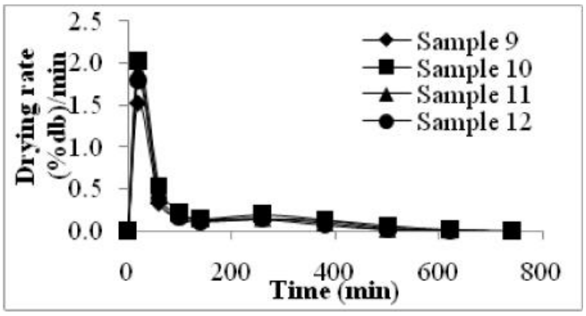

Fig. 2 (c) Variation of drying rate with drying time for sample 9, 10, 11 and 12.

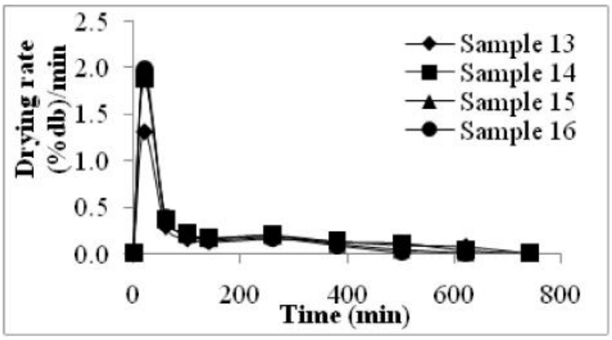

Fig. 2 (d) Variation of drying rate with drying time for sample 13, 14, 15 and 16.

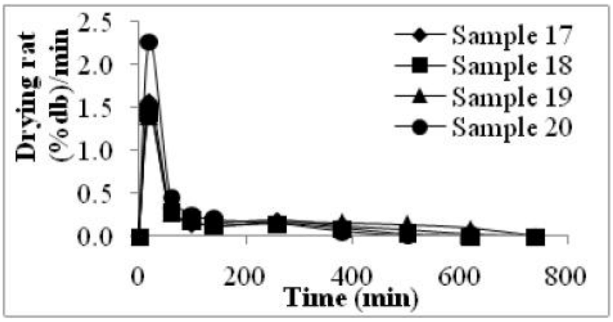

Fig. 2 (e) Variation of drying rate with drying time for sample 17, 18, 19 and 20.

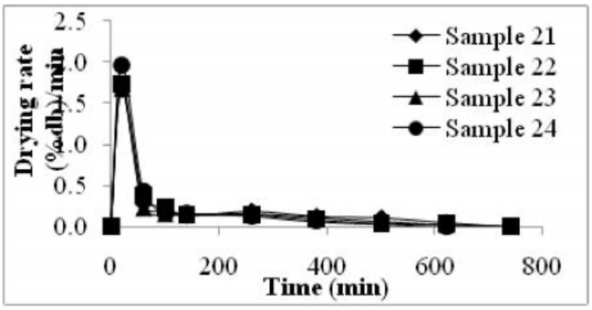

Fig. 2 (f) Variation of drying rate with drying time for sample 21, 22, 23 and 24.

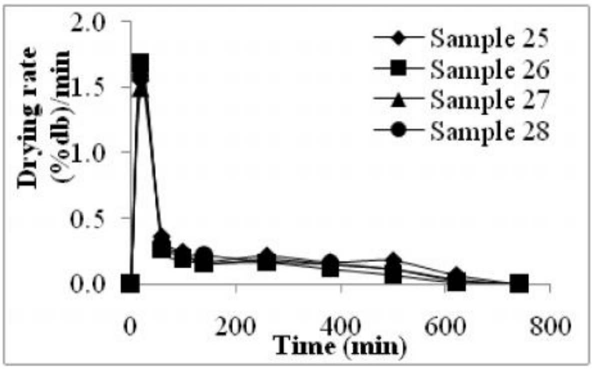

Fig. 2 (g) Variation of drying rate with drying time for sample 25, 26, 27 and 28.

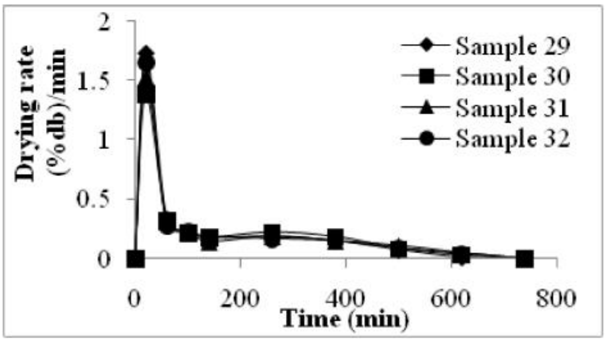

Fig. 2 (h) Variation of drying rate with drying time for sample 29, 30, 31 and 32.

The relationship between drying rates with drying time is shown in Figure 2 (a), (b), (c), (d), (e), (f), (g) and (h). The rate of drying of every sample as expected decreased continuously with increase in time, being faster at higher temperatures. The decrease in drying rate with drying time was non-linear. It was observed that the maximum drying rates were at ained in first 20 min of drying of each sample. The fast decrease trend of drying rate of samples were found in next 2 h then very slow trend was observed. It was also observed that although the moisture content was relatively high, constant rate drying was absent and the drying took place in falling rate period in all the cases. The drying rates for expt. no.4, 8, 12, 16, 20, 24 and 26 of the respective Figs. 2(a), (b), (c), (d), (e), (f), (g) and (h) were found to be higher in comparison to other experiments.

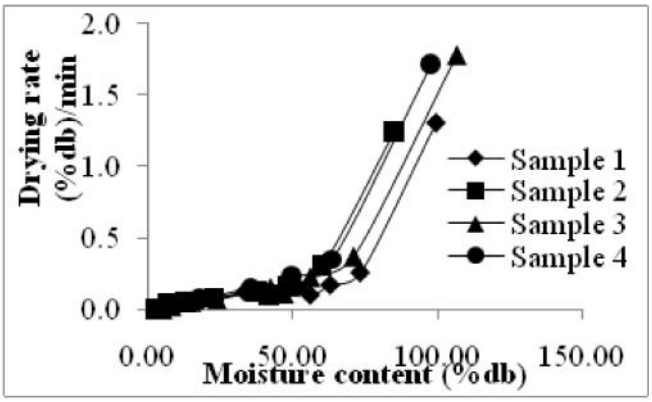

Fig. 3 (a) Variation of drying rate with moisture content for sample 1, 2, 3 and 4.

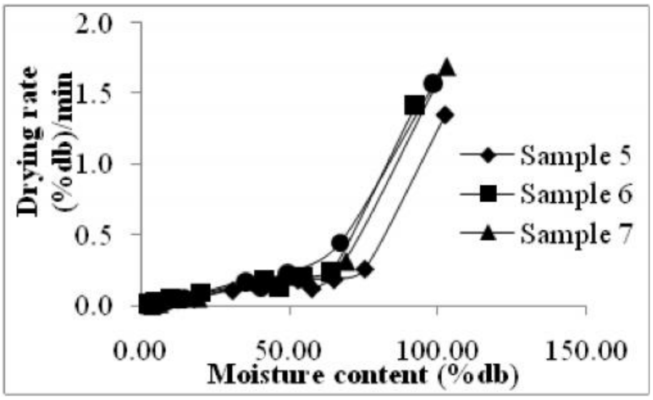

Fig. 3 (b) Variation of drying rate with moisture content for sample 5, 6, 7 and 8.

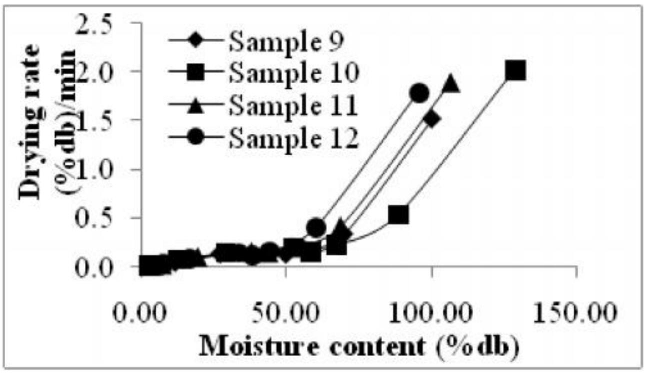

Fig. 3(c) Variation of drying rate with moisture content for sample 9, 10, 11 and 12.

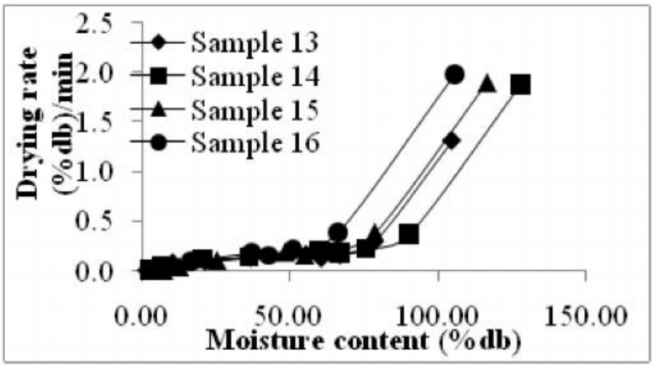

Fig. 3 (d) Variation of drying rate with moisture content for sample 13, 14, 15 and 16.

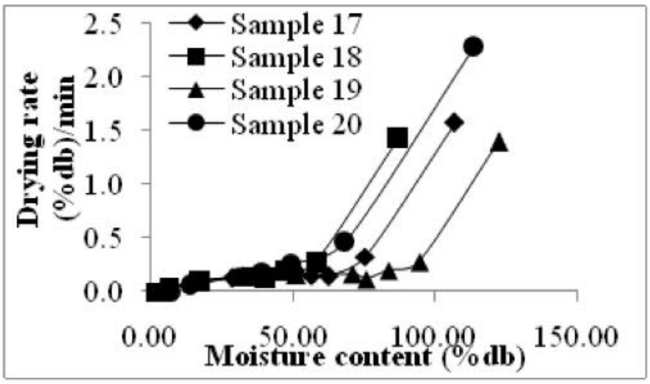

Fig. 3 (e) Variation of drying rate with moisture content for sample 17, 18, 19 and 20.

This was because of higher drying temperature, drying agent and foaming agent.

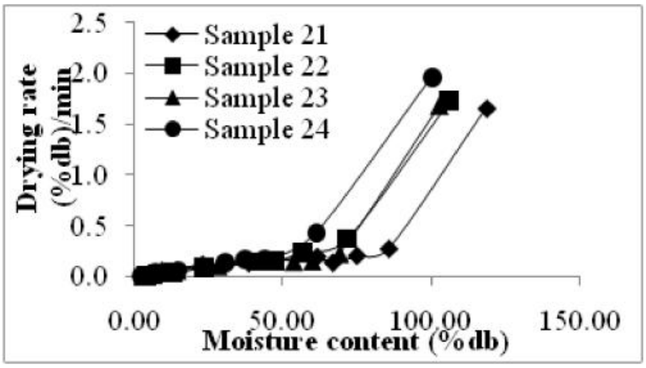

Fig. 3 (f) Variation of drying rate with moisture content for sample 21, 22, 23 and 24.

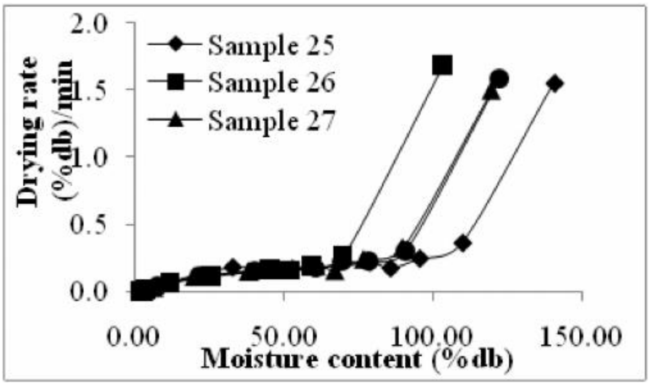

Fig. 3 (g) Variation of drying rate with moisture content for sample 25, 26, 27 and 28.

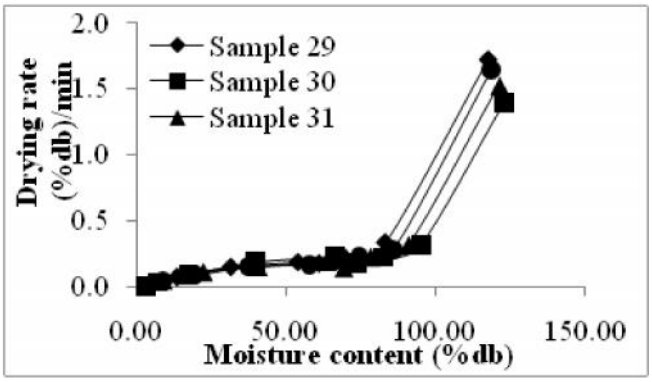

Fig. 3(h) Variation of drying rate with moisture content for sample 29, 30, 31 and 32.

The relationship between drying rate and moisture content is shown in Figure 3 (a), (b), (c), (d), (e), (f), (g) and (h). It is obvious that drying rate decreased with decrease in moisture content for all the experiments. Inflexion points for samples 19, 21, 25, 30, 31 and 32 were found from 90 to 100% moisture content and for all other experiments, inflexion points were observe from 60 to 80% moisture content. Inflexion points were found at higher moisture content because of higher quantity of whole milk powder and anticaking agent in the sample. Equilibrium moisture content for each experimental run is given in Table 6 and is varied between 0.54 and 5.21%. The moisture loss data were first analyzed for equilibrium moisture content (Chakerverty, 1988) and determined using Eqn 2.2. Moisture ratio was determined using Eqn 2.3.

Table 1. Values of independent variables in coded and actual form

Independent variables		Coded levels				
Name	**Code**	**-α**	**-1**	**0**	**+1**	**+α**
	Actual Levels (%)					
Carboxy Methyl cellulose	X_1	4	5	6	7	8
Temperature	X_2	65	70	75	80	85
Whole milk powder	X_3	1.0	1.5	2.0	2.5	3.0
Tricalcium phosphate	X_4	0.125	0.15	0.175	0.2	0.225
Glycerol monostearate	X_5	0.25	0.375	0.50	0.625	0.75

Table 2. Relationship between the coded and actual values of a factor

Code	Actual value of factor
-α	X_{min}
-1	
0	
+1	
+α	X_{max}

X_{max} and X_{min}: minimum and maximum value of X

Table 3. Statistical parameters for experimental models

Model	R^2		SEE	
	Minimum	Maximum	Minimum	Maximum
Page	0.981	0.997	0.0281	0.0773
Generalized	0.980	0.990	0.0583	0.0774
Modified Page	0.971	0.987	0.0660	0.0944

Table 4. ANOVA for drying time

Source	DF	SS	MS	F_{Cal}	F_{Tab}
Model	20	482063	24103.15	5.4*	4.10
Linear	5	286185	57237	12.83*	5.32
Quadratic	5	150353.3	30070.66	6.74*	5.32
Interactive	10	69300.24	6930.02	1.55**	2.85
Error	11	49071.78	4461.071		
Total	31	531134.8			

*, ** Significant at 1, 5% level of significance respectively

Table 5. Total effect of individual parameters on drying time

Source	DF	SS	MS	F_{Cal}	F_{Tab}
Model	20	482063	24103.15	5.4*	4.10
Carboxy Methyl Cellulose (X_1)	6	152105.9	25350.98	5.68*	5.07
Temperature (X_2)	6	170037.6	28339.6	6.35*	5.07
Whole Milk Powder (X_3)	6	44489.80	7414.96	1.66**	3.09
Tricalcium Phosphate (X_4)	6	154146.75	25691.12	5.75*	5.07
Glycerol Monostearate (X_5)	6	54315.67	9052.61	2.03**	3.09
Error	11	49071.78	4461.071		
Total	31	531134.8			

, * significant at 1, 5% level of significance respectively

Table 6. Experimental Moisture content and Predicted Drying time

Expt. No.	Initial MC (%db)	EMC (%db)	Final MC (%db)	Drying time to reach 7 % db MC (min)
1	99.62	4.68	4.69	706.06
2	84.86	2.44	2.43	504.03
3	106.50	4.65	4.70	712.83
4	97.79	2.86	2.89	408.60
5	102.40	5.21	5.86	724.60
6	92.43	0.96	1.61	457.00
7	102.76	3.09	3.18	457.89
8	98.40	2.74	2.77	386.31
9	100.00	3.58	3.59	559.56
10	128.66	2.20	2.24	504.11
11	106.44	1.05	1.76	388.67
12	95.57	3.50	3.50	392.90
13	104.71	1.33	1.33	649.08
14	127.93	2.56	2.56	620.42
15	116.76	4.21	5.56	562.24
16	105.74	3.08	3.07	385.09
17	106.69	2.90	2.91	568.91
18	86.66	1.92	1.97	396.75
19	122.20	3.68	3.69	711.02
20	113.40	4.79	4.93	330.05
21	118.59	2.52	2.53	679.45
22	105.89	3.00	3.07	487.01
23	102.65	0.54	1.29	519.39
24	100.22	1.49	1.73	322.00
25	140.84	2.55	2.54	724.40
26	103.46	1.15	1.22	481.39
27	119.88	3.00	3.00	666.80
28	122.49	3.00	3.01	681.22
29	117.86	2.99	2.99	573.92
30	122.93	3.00	3.01	677.92
31	121.23	2.85	2.86	699.36
32	118.86	2.99	2.99	657.99

Table 7. Optimum levels of ingredients

Independent variables	Coded levels	Actual levels
Carboxy Methyl Cellulose (X_1)	1.92	7.92 (%)
Temperature (X_2)	2.0	85 (°C)
Whole milk powder (X_3)	-0.90	1.55 (%)
Tricalcium phosphate (X_4)	2.0	0.225 (%)
Glycerol monostearate (X_5)	-1.83	)

Empirical modeling

Mathematical models namely Page's, generalized exponential and modified Page's model were selected for prediction of drying curves of concentrated malta juice undergoing foam mat drying for all experimental data. These models were fit ed to the experimental data in their linearized forms using regression technique to determine the drying constants of models. The comparison of the applicability of all models was done on the basis of standard error of estimation, coefficient of determination and residual errors. The minimum and maximum values of R^2 and SEE for each model are given in Table 3.

Although, Page's model had higher R^2 and lower SEE in comparison with that of generalized exponential and modified Page's models, generalized exponential model was found to be most suitable because this model had least residual errors as compared to other two models.

This model was also used to compute the drying time corresponding to the 7% moisture content (db) for all experiment runs. This was used to determine the effect of temperature and added ingredients.

Effect of Drying Temperature and added Ingredients on Drying Time

Full second order model was fit ed into drying time using multiple regression analysis. The coefficient of determination (R^2) for the regression model for drying time was 90.76%, which implies that the model could account for 90.76% variability in data. Lack of fit was non significant but model was highly significant ($P<0.01$) with F-value as 5.40. Therefore, second order model was adequate in describing drying time.

Effect of independent variables on drying time at linear, quadratic and interactive level is reported in Table 4. It shows that the effect of variables on drying time was highly significant ($P<0.01$) at linear level in comparison to quadratic and interactive level because higher value of sum of square and F-value.

Total effect of individual parameter on drying time was calculated using the sequential sum of squares, and it is given in the Table 5. It was observed that methyl cellulose, temperature, whole milk powder, tricalcium phosphate and glycerol monostearate affected the drying time significantly at ($P<0.01$) level of significance. Effect of temperature was maximum on drying time followed by tricalcium phosphate and methyl cellulose in decreasing order. Whole milk powder and glycerol monostearate did not show any significant effect on drying time.

Second order predictive quadratic equation for drying time (min) is given below

$$Y=652.52-60.28X_1-74.68X_2-13.29X_3-28.76X_4-41.35X_5+0.31X_1X_2+0.78X_1X_3+36.78X_1X_4+13.04X_1X_5-18.05X_2X_3-11.14X_2X_4-20.10X_2X_5+42.33X_3X_4-11.41X_3X_5+5.63X_4X_5-37.17X_1^2-27.74X_2^2-12.07X_3^2-52.71X_4^2-7.15X_5^2 \quad ...(2.8)$$

Significant predictive equation for drying time (min) is given below

$$Y = 652.52 - 60.28X_1 - 74.68X_2 - 28.76X_4 - 41.35X_5 + 36.78X_1X_4 + 42.33X_3X_4 - 37.17X_1^2 - 27.74X_2^2 - 52.71X_4^2. \quad ...(2.9)$$

Where, Y is drying time (min), X_1, X_2, X_3, X_4 and X_5 are coded variables for methyl cellulose, temperature, whole milk powder, tricalcium phosphate and glycerol monostearate.

Optimization of independent variables for production of malta powder

Numerical optimization of independent variables was carried out using Design–Expert 8.0.7.1 statistical sof ware. The most suitable optimum point is given in the Table 7.

Conclusion

From the study it was concluded that the total drying time considerably reduced by 53.6% with increase in drying temperature from 65 to 85°C. Drying time was mostly effected by temperature followed by Methyl celluloe and glycerol monostearate. Drying time decreased with increase in carboxy methyl cellulose and glycerol monostearate. Generalized exponential model was found to be best suitable for foam mat drying of concentrated malta juice. The best drying time predicted by generalized model was found to be 322 minutes. The results of a confirmation experiment were found to be in good agreement with the values predicted by the model. This demonstrates that to obtain a maximum amount of information in a short period of time, with the least number of experiments, RSM and CCD can be successfully applied for modeling and optimizing drying process parameters.

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