Print ISSN : 0972-8813
e-ISSN : 2582-2780

[Vol. 19(2), May-August, 2021]

Pantnagar Journal of Research

(Formerly International Journal of Basic and Applied Agricultural Research ISSN : 2349-8765)

G.B. Pant University of Agriculture & Technology, Pantnagar

PANTNAGAR JOURNAL OF RESEARCH

Vol. 19(2) May-August, 2021

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To study the effect of maltodextrin, tricalcium phosphate, glycerol monostearate and drying temperature on vacuum foam mat quality parameters of papaya powder

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ABSTRACT: Vacuum foam-mat drying is a process by which a liquid concentrate along with a suitable foaming agent is used to whip to form stable foam and is subjected to dehydration in the form of a thin mat of foam at relatively low temperature. Response surface methodology (RSM) has been used to find out the effect of variable on the responses. The initial value of moisture content of raw papaya pulp was 856.023 % (db.). The total soluble solid (TSS) was tested by hand refractrometer i.e., 12.8ºBrix. Parameters such as whipping time (10 minute), papaya pulp thickness (4 mm) and vacuum oven pressure (25 inch Hg), pH value 5.56 and TSS(10º Brix) remain constant throughout the work. In view of the above, present study was undertaken with objectives of studying the effect of maltodextrin (0.15-0.75kg/kg papaya pulp solid), tricalcium phosphate (0.5-2.5%), glycerol monostearate (0.5-2.5%) and drying temperature (55-75 ºC) on vacuum foam mat drying characteristics of papaya pulp and to ascertain various quality characteristics of dried papaya powder. The flowability time of papaya powder ranges from 15.65 to 23.62 s. Its value is minimum at experiment run number 28 and maximum at experiment run number 1 with the combination of process conditions of temperature 65°C, maltodextrin 0.45 (w/w), glycerol monostearate 1.5% and tricalcium phosphate 1.5% and temperature 60°C, maltodextrin 0.30 (w/w), glycerol monostearate 1.0% and tricalcium phosphate 2.0% respectively. Hygroscopicity starts from 2.24 (experiment no. 30) to 6.84 (experiment no. 28) while the degree of caking is in between 1.83 (experiment no. 30) to 5.45% (experiment no. 28). On the basis of different experimental results it was found the minimum drying time was obtained (660 min.) at temperature (60ºC), maltodextrin (0.60 gm/110gm), GMS(1.0%) and TCP(2.0 %). It was also found that flowability time has significantly affected by temperature ($P<0.01$), maltodextrin ($p<0.05$) and glycerol monostearate ($P<0.1$), at linear level. The effect of temperature and tricalcium phosphate on hygroscopicity was highly significant ($P<0.01$) while maltodextrin was significant at 10% level of significance.

Key words: Drying rate, papaya, response surface methodology, shelf life, Vacuum foam mat drying

Papaya (*Carica papaya* L.) is one of the important fruits of tropical and subtropical regions grow well in the country upto 1000 meter above sea level. Papaya was originally derived from the southern part of Mexico; papaya is a perennial plant which is distributed over the whole tropical and subtropical area. It is one of the most consumed fruits. The interior flesh of the fruit goes through color changes from green (immature) to yellow (ripe) and when it is to overripe (McGrath and Karahadian, 1994). Total yearly world production is estimated at 11 million tones of fruits. India leads the world in papaya production with an annual output of about 4 million tonnes. Other leading producers are Brazil, Mexico, Nigeria, Indonesia, China, Peru, Thailand and Philippines (FAOSTAT 2012a, 2012b).

Papaya (*Carica papaya*) is a plant that belongs to the family of Caricaceae. It is a herbaceous succulent plant with self-supporting stems (Dick, 2003).The fruit is rich in β-carotene, vitamin-A and C, iron, calcium, protein, carbohydrates, phosphorous and good source of energy (Gopalan *et al.,* 1972). Papaya can be made into jam, jelly, nectar, dried into slabs, canned in the form of slice and the fruit powder can be used for preparation of nectar, ice cream flavour, ready to eat fruited cereals. Most fruits including papaya have high moisture content and are highly perishable, cannot be preserved for longer period of time results massive losses. Pantastico (1979) estimated for the Philippines that papaya postharvest loss ranged from 20 to 26%, with 8 – 12% of the loss being due to decay, 2 – 4% due

to over ripening and 10% due to mechanical injury. The total postharvest losses of papaya worked out to 25.49% (Gajanana *et al.,* 2010). Developed by Morgan *et al.* (1961) foam-mat drying is a process by which a liquid concentrate along with a suitable foaming agent is used to whip to form stable foam and is subjected to dehydration in the form of a thin mat of foam at relatively low temperature. Drying occurs in multiple constant rate periods due to periodic bursting of successive layers of foam bubbles, thus exposing new surfaces for heat and mass transfer as the drying progresses (Chandak and Chivate, 1972). This method is suitable for any heat sensitive, sticky and viscous materials which cannot be dried by spray drying (Hart *et al.,* 1963 and Berry *et al.,* 1965). Drying occurs in multiple constant rate periods due to periodic bursting of successive layers of foam bubbles, thus exposing new surfaces for heat and mass transfer as the drying progresses (Hart *et al.,* 1963 and Martin *et al.,* 1992). This method is suitable for any heat sensitive, sticky and viscous materials which cannot be dried by spray drying. The foam-mat dried products have better reconstitution properties because of their honeycomb structure and are superior to drum and spray dried products (Chandak *et al.,* 1974). This method is suitable for any heat sensitive, sticky and viscous materials which cannot be dried by spray drying. The foam-mat dried products have better reconstitution properties because of their honeycomb structure and are superior to drum and spray dried products (Chandak *et al.,* 1974). Renewed interest in foam-mat drying could be due to its simplicity, cost-effectiveness, rapid drying rate and enhanced product quality. Foaming of liquids and semi liquid materials has long been recognized as one of the methods to shorten drying time. Unlike other drying methods, foam-mat drying does not involve a large capital outlay. The product is also reduced to a light and porous form which, when packaged in polyethylene material, allows for good stability. Vacuum drying takes place in the absence of oxygen, the oxidative degradation e.g. Browning is low in the final product. The temperature range used for vacuum drying is usually kept within 65-75^{o}C (Copley *et al.,* 1956). The drawback of this method is the throughput of the dryer as the moisture is removed from the thin layer of the foam hence the material spread per unit surface of drying area is very small (Kudra and Ratti, 2006).

On the other hand, nutritional properties, pharmacological benefits, chemical composition and the forms of preparation of edible species have been increasingly studied with the growing search for natural and health foods (Takahashi *et al*., 2019). Characterization of drying is of paramount importance as it determines drying time and control measures can be taken to obtain energy efficient process that produces quality product. Response surface methodology (RSM) has been used to develop products and find out the effect of variable on the responses (Jaya and Das, 2004, Hymvathi and Khader, 2004a). It is used to get an optimum process conditions considering single response or multiple responses. It encompasses statistical and mathematical techniques. In view of the above, present study was undertaken with the objective to ascertain the quality characteristics namely flowability time, hygroscopicity, degree of caking, solubility, ascorbic acid, beta carotene and colour values of the dried papaya powder.

MATERIALS AND METHODS

The papaya fruits were purchased in Pantnagar's local market. The fresh papaya pulp was hand peeled using a stainless steel knife, and the ripened pieces were pulped using a mixer grinder. In the Process and Food Engineering laboratory of the Department of Post-Harvest Process and Food Engineering, College of Technology Pantnagar, preliminary work was done to set the parameters for the production of papaya powder with and without the addition of ingredients using the vacuum foam mat drying technique. To complete this work different type of experimental setups were required. The equipment's used for experiments were stain less steel knife, vacuum oven, centrifuge, vacuum pump, food processor, electronic balance, hot air oven, refrigerator etc. The list and specifications of these equipment's and the apparatus used are given in the Table 2.1.

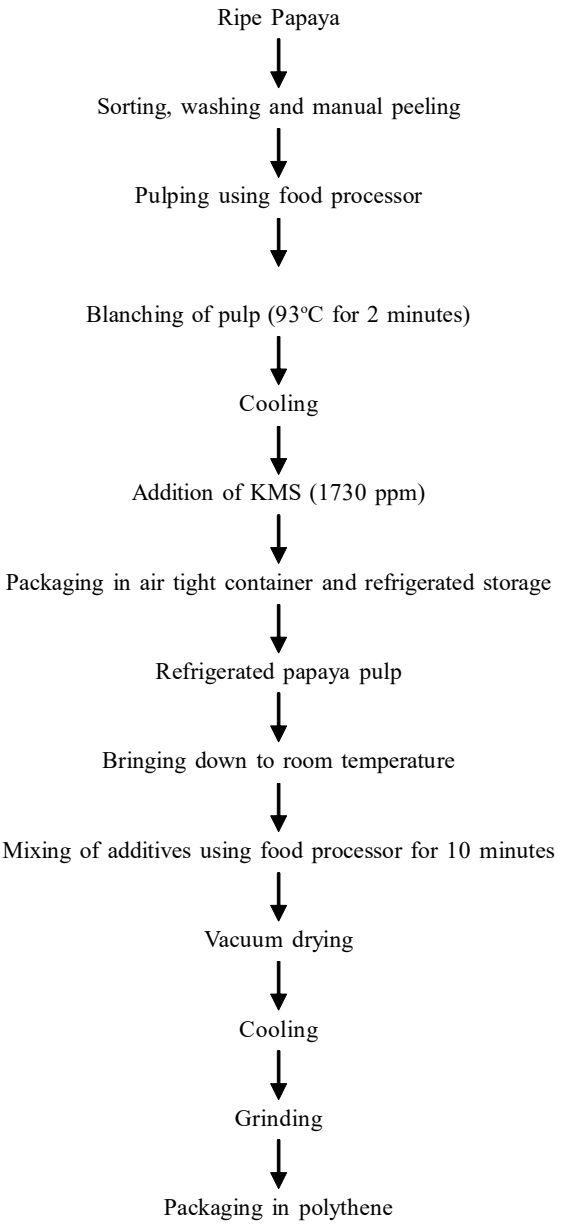

Fig. 2.1: Process flow chart for preparation of papaya powder in vacuum dryer

Experimental Design

Experimental design based on the review was according to Response Surface Methodology (RSM), the different variables was used in this methodology were temperature, maltodextrin, tricalcium phosphate and glycerol monostearate. The number of experiments at five levels was found by using a second order central composite rotatable design (CCRD). Experiments also were done at centre point. The design is rotatable which means that all the points in the design area are at equal distance from the central. The number of design points in (CCRD) is based upon a complete 2^k factorial design.

The total numbers of experiments are,

$$N= 2^k+2K+L \quad \ldots (2.1)$$

Where N= Total Number of experiments, K= Numbers of Factors and L= number of replicates of the central points

The details of the independent variable in the experimental plan and design of experiments are given in the Table 2.2, Table 2.3 and Table 2.4 respectively. All the experiments were done using software tool named as Response Surface Methodology (RSM), was 30 in number. To find out the effect of independent variables on the quality of powder a multiple linear regression analysis was used and the data was fitted as a second order equation. The equation is given by

$$Y=\beta_0+\sum_{t=1}^{k}\beta_t X_i+\sum_{t=1}^{k}\beta_{ii} X_i^2+\sum_{t=1}^{k-1}\sum_{j=i+1}^{k}\beta_{ij} X_i X_j \ldots..(2.2)$$

Where, β_0, β_i, β_{ii}, β_{ij} are regression coefficients ,X_i and X_j are independent variables in coded form and K is number of independent variables and Y is response.

Sample Preparation

The papaya was cleaned by fresh water and placed at room temperature until the desired peel colour is obtain. Fully ripened papaya was peeled manually using a stainless steel knife and the flesh portion was pulped by using a mixer grinder. The pulp was blanched at 93°C for 2 minute. 1730 ppm potassium met bisulphate was mixed into this. Now the pulp was stored in refrigerated condition inside a steel airtight container until its next use. The pulp was bring down at room temperature i.e., 27°C before going through drying procedure. The distilled water mixed into papaya pulp to get appropriate pulp

concentration (10° Brix). Different types of materials added as agents through different process. These materials are maltodextrin, glycerol monostearate and calcium phosphate. Maltodextrin used as drying agent, glycerol monostearate as foaming agent as well as stabilizer and tricalcium phosphate added as anti-caking agent. All materials used as limited in Prevention of food Adulteration Act (1955) of the Government of India. Now the pulp was ready to characterize for chemical parameters such as moisture content, ascorbic acid (mg/100 gm.) and beta- carotene (mg/100 gm.).

Vacuum foam mat drying for the preparation of papaya powder

In laboratory model vacuum oven (MSW-218) the papaya pulp was dried. The mixture after suitable addition of drying aids was spread evenly on Petri dish and steel tray (coated with aluminium foil), having dimension of 10x15x1.5 cm. After this wards the tray was kept inside the vacuum oven dryer shelves and the pressure of vacuum inside the chamber was reduced until its reached to 25" Hg. Five different drying temperature via 55, 60, 65, 70, 75°C (Jadhav, 2008) and drying time 1 hr. on each trial basis was selected to carry out vacuum drying. During vacuum foam mat drying process the initial quantity of the papaya pulp was kept constant. Five different drying temperatures *viz.*, 55, 60, 65, 70, 75°C and drying time *viz.*, 0, 60, 120, 180, 240, 300, 360, 420, 480, 540, 600, 660, 720, 780 and 840 minutes were selected to conduct foam mat drying for each sample. The study of drying behaviour was done in form of moisture content (%db.) with respect to time and temperature. The dried papaya pulp was grinded into a fine particulate powder using a food processor at medium speed for 5min and packed in polythene. The prepared papaya powder from foam mat drying method was used for analysis of physico-chemical characteristics *viz.*, moisture content (%), flowability time (second), hygroscopicity (%), degree of caking.

Quality Analysis Techniques

Flowability time

It consists of a stainless steel drum of diameter 120 mm and length 90 mm. One end of the drum was fitted with a transparent lid, while its other end was rigidly fixed to the shaft of motor. The drum had two slots, each having 4 mm width and 70 mm length on the surface of the drum. Powder sample weighing 25 times of its bulk density in g/cm^3 was kept in the drum. The drum was rotated at 30 rpm by a geared motor and the powder was allowed to flow through the slits. Time was noted for all the powder samples to come out of the slits provided on the drum. Flowability time was expressed as the time in seconds necessary for the powder to leave the rotary drum (Jaya and Das, 2006).

Hygroscopicity

Hygroscopicity is expressed as the final moisture content attained after exposing the powder in humid air having 85.11%relative humidity. The apparatus used for hygroscopicity measurement has been shown in Plate 2.1.A saturated solution of potassium chloride salt (equilibrium relative humidity = 85.11$\pm$ 2% at 20°C) was kept in glass wash bottle having two passages for air inlet and outlet. Powder sample weighing 0.5 gram, which had passed through 500 micron sieve, was spread uniformly on a filter paper. The diaphragm type vacuum pump was use to suck the air through salt solution and the sample at the flow rate of 300 to 400 ml per min. Increase in weight of the sample at every 15 min was noted. This measurement was continued till the difference between two successive weightings did not exceed

Plate 2.1: Hygroscopycity measurement apparatus

by 0.5 %.The hygroscopicity of the powder was calculated using the relation given in Eq. 3.3. Generally, a powder having the hygroscopicity value less than 10 % is considered as good 'non hygroscopic' powder.

$$HG=\frac{\frac{b}{a_h}+W_i}{1+\frac{b}{a_h}} \quad \text{.....(2.3)}$$

Where, HG=hygroscopicity b (g) is the increase in weight of powder, a_h (g) is the amount of powder taken for the measurement and W_i is the initial weight of sample (% wb) is the free water present in the powder before allowing it in to the humid air environment(Jaya and Das, 2006).

Degree of caking

After the determination of hygroscopicity, the Gooch filter along with the wet sample obtained at the end of hygroscopicity measurement was placed in a drying oven set at 102 °C ± 2 °C for one hour to measure degree of caking. After cooling the dried sample, it was weighed and transferred into a sieve of 500 µm size. The sieve was then vibrated for 5 min in a shaking apparatus. The weight of the powder remaining in the sieve was measured. Degree of caking, DC (%) was calculated by using equation:

$$DC=\frac{C}{d}x\ 1000 \quad \text{... (2.4)}$$

Where, d (g) is amount of the powder used for sieving and c (g) is amount of the powder left on the sieving after sieving.

Normally, the degree of caking between 5 to 20% is called as 'slightly caking' powder (Jaya and Das, 2006).

Data Analysis

Measurement of moisture content

The initial and final moisture content after vacuum drying was determined by hot air oven drying method as described by Ranganna (2004) for fruits and vegetables. After complete drying when weight of the samples shown constant values. The moisture content (%) on dry basis and drying rate were determined as described by Chakravarty (1997).

$$\text{Moisture Content (\%d.b.)} = \frac{W1-W2}{Wd} \text{x } 100 \quad \text{...(2.5)}$$

Where, W1= weight of sample before drying in gram; W2 = weight of sample after drying in gram; $W_{d=}$ weight of solid.

Equilibrium moisture 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 Henderson and Perry (1976), in which last three moisture content readings of drying experiment were taken. Equation was used to determine the equilibrium moisture content.

$$Me=\frac{M_1 x\ M_3 - (M_2)^2}{M_1 + M_3 - 2M_2} \quad \text{…(2.6)}$$

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

Moisture content should be taken with the following condition $(t_3 - t_2) = (t_2 - t_1)$.

Moisture ratio and drying rate

Moisture Ratio (MR) is defined by using following relation,

$$MR=\frac{M_t - M_3}{M_0 - M_e} \quad \text{…. (2.7)}$$

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; Me–Equilibrium moisture content (% db.)

Drying Rate is defined by using following relation as,

$$\frac{dm}{dt} = \frac{M_2 - M_1}{\Delta t} \quad \ldots\ldots (2.8)$$

Where, Δt – difference in time.

To study the drying characteristics of papaya pulp, moisture ratio and drying rate at different time intervals were calculated as by using equation 2.5 and 2.8.

Optimization of independent variables

Full second order model was fitted in different drying quality parameters and characteristics using least square regression analysis. Data analysis of quality parameters of powder was done by using Design-Expert 8.0.6 trial version statistical software. The optimization of independent variables was also done using Design-Expert 8.0.6 trial version statistical software. Effect of independent variables on the drying quality parameters and characteristics of sample was interpreted using the models.

RESULTS AND DISCUSSION

Drying Behaviour

The value of moisture content is obtained from 242.99 to 515.59% (db), these values based on the amount of added ingredients before drying, while it has in the range of 2.03 to 4.71 % (db) after drying in vacuum oven. The result of work in the vacuum foam mat drying of papaya pulp as a function of weight of testing materials and time. From experiments the drying data is as follow, the minimum drying time was obtained (660 min.) with the combination of the ingredients, temperature (60°C), maltodextrin (0.60 gm/110gm), GMS (1.0%) and TCP (2.0 %) of dried papaya powder. The minimum drying time was due to the high amount of maltodextrin because it has the tendency to reduce the moisture content quickly from the product. It was also found after experiments that drying time decreased due to increase in the amount of tricalcium phosphate (2%) in the materials so it help to reduce the moisture content of the products.

In Fig. 3.1 shows a relation among moisture ratio versus drying time. Graphical relationship between moisture ratios with drying time is obtained throughout the work with different 30 experiments shown by Fig. 3.1 (a), (b), (c), (d), (e), (f), these figures shows as the drying time increase, a non-linear decrement takes place in the moisture ratio for all sample. With very small value of drying time the moisture ratio of each sample decreased rapidly but as the drying time increase the rate of decrements of moisture ratio become very slow.

Effect of Drying Temperature and added Ingredients on Quality Parameters of Papaya Powder

Table 3.2 represents the measured quality parameters of papaya powder at each experimental run. The flowability time of papaya powder ranges from 15.65 to 23.62 s. Its value is minimum at experiment run number 28 and maximum at experiment run number 1 with the combination of process conditions of temperature (65°C), maltodextrin (0.45 w/w), glycerol monostearate (1.5%) and tricalcium phosphate (1.5%) and temperature (60°C), maltodextrin (0.30w/w), glycerol monostearate (1.0%) and tricalcium phosphate (2.0%) respectively. Hygroscopicity is in between 2.24 (experiment no. 30) to 6.84 (experiment no. 28), while the degree of caking is in between 1.83 (experiment no. 30) to 5.45% (experiment no. 28).

Effect of variables on flowability time

The regression analysis is presented in Table 3.2 and shows the regression coefficients in the model and significance of each term. A second order mathematical model (equation no. 3.1 and 3.2) was used into the flowability time data to examine the effect of variables. The coefficient of determination (R^2) of the regression was found for flowability time was 95.38 %, which implies that the model could account for 95.38% data. The model showed highly significant ($P<0.01$) with F-value as 22.12. Therefore, it was found that the second order model adequately describes the flowability time. Positive coefficient of the model at linear level indicates an increase in flow time with increase in level of its variables and vice versa. Negative coefficients of

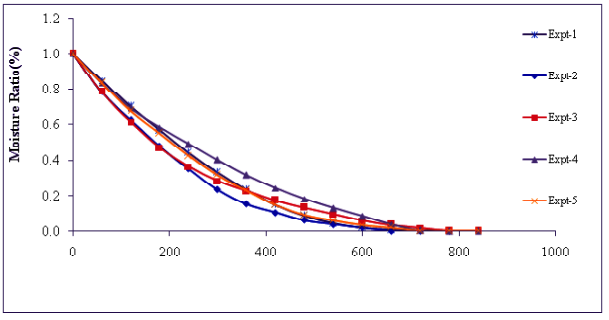

Fig. 3.1(a): Variation of Moisture ratio with drying time

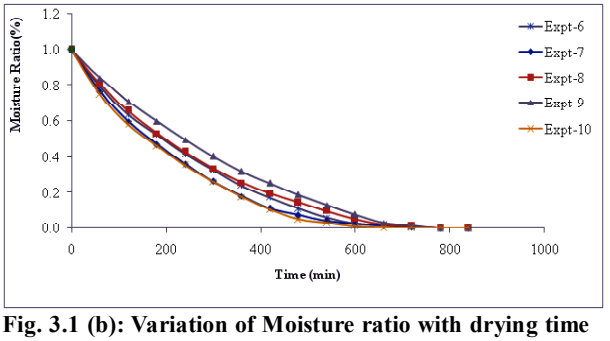

Fig. 3.1 (b): Variation of Moisture ratio with drying time

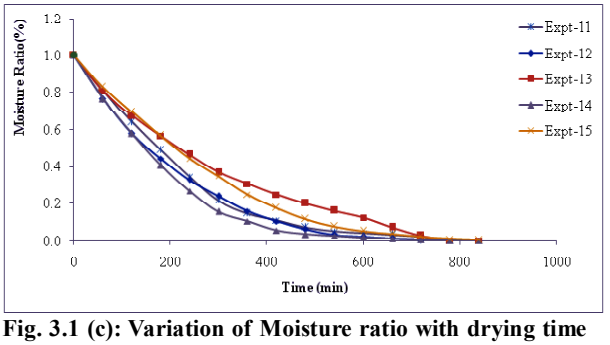

Fig. 3.1 (c): Variation of Moisture ratio with drying time

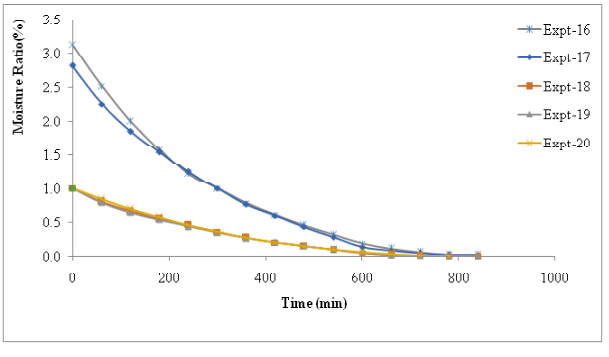

Fig. 3.1 (d): Variation of Moisture ratio with drying time

quadratic terms indicated that the maximum of flowability time was at the centre point while positive quadratic term gives the minimum response. Negative coefficients interactive term suggested that

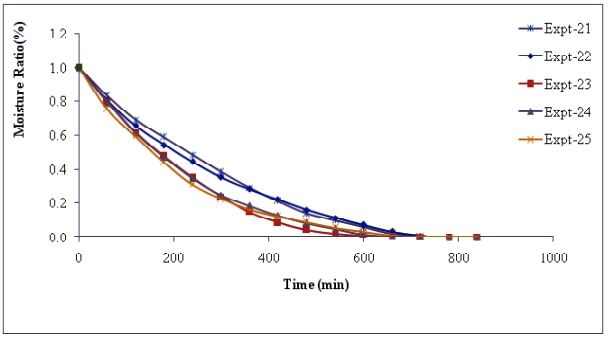

Fig.: 3.1(e): Variation of Moisture ratio with drying time

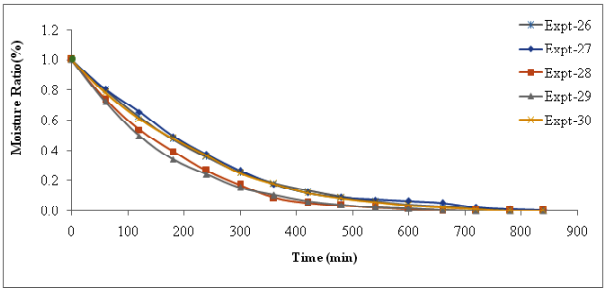

Fig. 3.1(f): Variation of Moisture ratio with drying time

the level of one variable of the interaction can be increased while the other decreased simultaneously. It can be seen from Table 3.1 that flowability time has significantly affected by temperature ($P<0.01$), maltodextrin (<0.05) and glycerol monostearate ($P<0.1$) at linear level. The coefficient of temperature, maltodextrin, and glycerol monostearate and tricalcium phosphate are negative and therefore with increase in the levels of these, the flowability time decreases. Effect of ingredients at linear, quadratic and interactive levels is reported in table 3.3.It shows that the effect of ingredient on flowability time at linear has highly significant ($P<0.01$), but interactive and quadratic levels has significant at 5% and 10% level of significance respectively.

Total effect of individual parameter on flowability time was calculated by using the sequential sum of squares, and it is given in the Table 3.4. It was observed that temperature and maltodextrin affected the flowability time significantly at ($P<0.01$) level of significance but there is no significance of tricalcium phosphate and glycerol monostearate on flowability time. Effect of temperature and maltodextrin was found highest as compared to that

of other variable parameters.

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

$$Y=19.11-2.09X_1-0.56X_2-0.24\ X_3-0.22\ X_4+0.15\ X_1X_2+0.014\ X_1X_3+0.26\ X_1X_4+\ 0.16\ X_2X_3\ -\ 0.19X_2X_4+0.12X_3X_4+0.005833X_1^2+0.16X_2^2\ -\ 0.17\ X_3^2-0.083X_4^2 \quad \ldots (3.1)$$

Significant predictive equation for flowability time is given blow:

$$Y=19.11-2.09X_1-0.56X_2-0.24\ X_3 \quad \ldots.. (3.2)$$

Where, Y is flowability time (sec) and X_1, X_2, X_3, X_4 are coded variables for temperature, maltodextrin, and glycerol monostearate and tricalcium phosphate.

Figure 3.2 (a) depicts the variation of flowability time with maltodextrin at optimum point of temperature (59.05 °C), glycerol monostearate (1.78%) and tricalcium phosphate (2.5%) at linear level. It was observed that decreasing the flowability time from 21.5 seconds to 19.1 seconds gradually by increasing the levels of maltodextrin and free flowing behaviour of papaya powder depends upon the amount of maltodextrin. Minimum flowability time (19.1 sec) was found at maltodextrin w/w (0.65) and maximum (21.5 sec) at maltodextrin w/w (0.30).

Figure 3.2 (b) shows the effects of glycerol monstearate on flowability time at optimum values of temperature (59.05°C), maltodextrin (0.55w/w) and tricalcium phosphate (2.5%) at linear level. It was concluded that the flowability time increased slightly from 19sec to 19.2 sec with increased the level of glycerol monostearate up to 1.54 % after that it has slightly decreased with increased the level of glycerol monostearate (2.0%). It means that little effect of glycerol monostearate on flowability time was observed.

Effect of variables on hygroscopicity

The regression analysis is presented in Table 3.1 and shows the regression coefficients in the model and

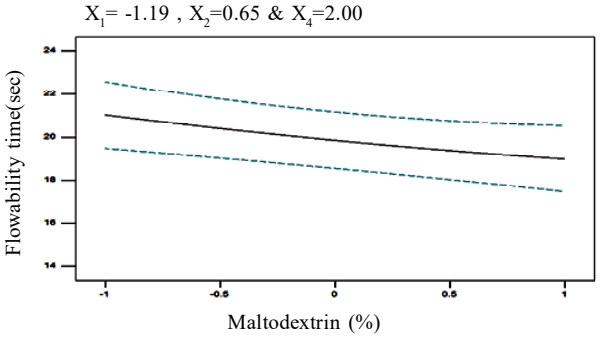

Fig. 3.2 (a): Variation of flowability time (sec) with maltodextrin (%) at optimum points (-1.19, 0.56 & 2.00)

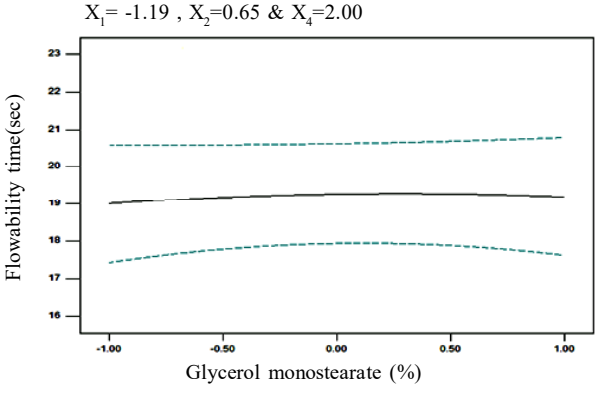

Fig. 3.2 (b): Variation of flowability time (sec) with glycerol monostearate (%) at optimum points (-1.19, 0.65 & 2.00)

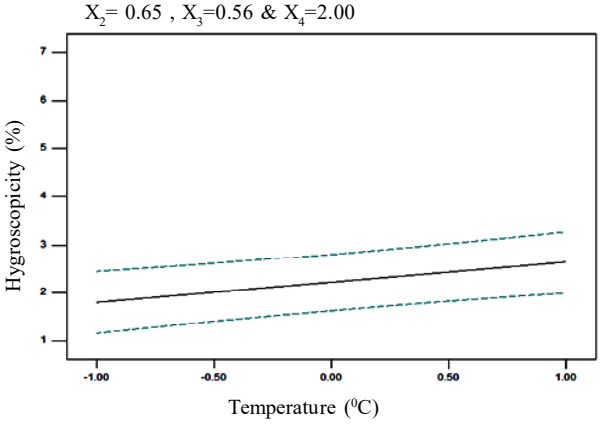

Fig. 3.3 (a): Variation of hygroscopicity (%) with temperature (0C) at optimum points (0.65, 0.56 & 2.00)

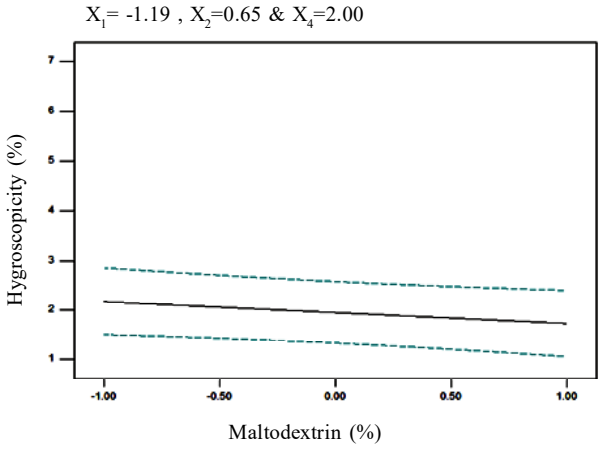

Fig. 3.3 (b): Variation of hygroscopicity (%) with maltodextrin (%) at optimum points (-1.19, 0.56 & 2.00)

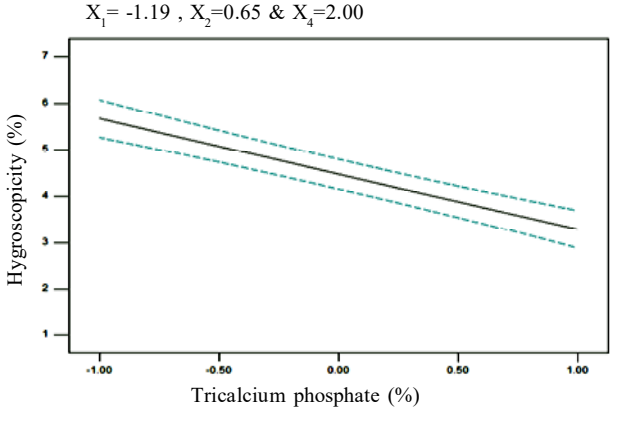

Fig. 3.3 (c): Variation of hygroscopicity (%) with tricalcium phosphate (%) at optimum points (-1.19, 0.65 & 0.56)

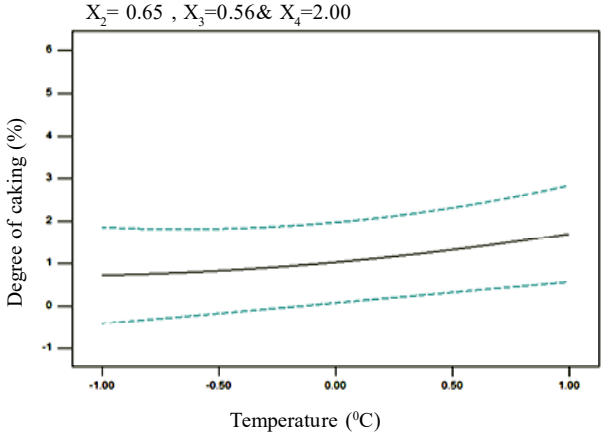

Fig. 3.4 (a): Variation of degree of caking (%) with maltodextrin (%) at optimum points (0.65, 0.56& 2.00)

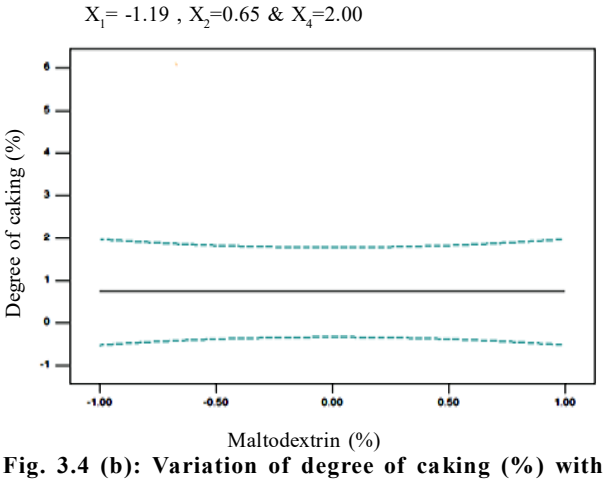

Fig. 3.4 (b): Variation of degree of caking (%) with maltodextrin (%) at optimum points (-1.19, 0.56 & 2.00)

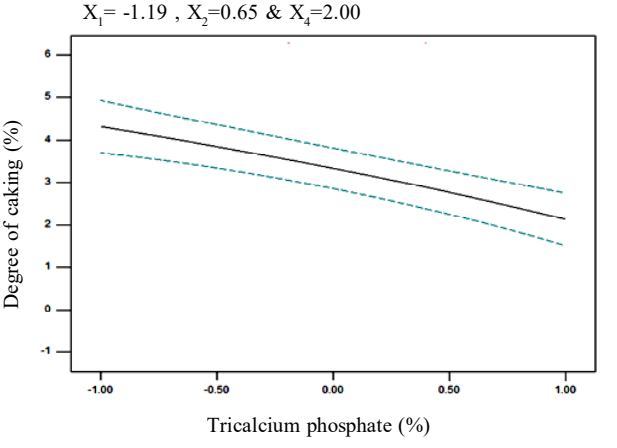

Fig. 3.4 (c): Variation of degree of caking (%) with tricalcium phosphate (%) at optimum points (-1.19, 0.65 & 0.56)

significance of each term. A second order mathematical model (equation no. 3.3 and 3.4) was used into the hygroscopicity data to examine the effect of variables. The coefficient of determination (R^2) of the regression model for hygroscopicity is 88.80%, which implies that the model could account 88.80% variability in data. The model was satisfactory because the calculated F-value (8.5) is higher than tabulated F-value (3.56 at 1% & 2.42 at 5%), although the lack of fit is significant. The model was found suitable for describing the hygroscopic behaviour because of higher value of R^2 and F.

It was observed from Table 3.5 that the effect of

Table 2.1 Specification of experimental equipment / apparatus

Equipments/ apparatus	Specification	Make
Balance electronic	Capacity - 300gLeast count:0.01g	Winsor
Vacuum Oven	Temperature:40 - 130°C Vacuum:0-760mm Hg Voltage:220-230V	Macro Scientific Works
Vacuum pump	Capacity: 150 lit/min Oil required:0.5lit Motor-H.P.:0.3 3Volt:220AC Phase 1 RPM:1420	Macro Scientific Works
UV Spectrophotometer	200-1000nmAccuracy:3nm	Beckman
Centrifuge	R8C,11-32,3000-6000rpm	Remiequipments
Water bath	No:1188, 220 volt, 50Hz	JSGW
Beaker	Capacity: 100,250,500, 1000ml	Borosil
Conical flack	Capacity: 25, 50, 100, 250ml	Borosil
Measuring cylinder	50 mm diameter	Borosil
Funnel	Capacity: 100, 250, 500ml	Borosil
Volumetric flask	Glass,	Borosil
Sieve	Dia:500 micron	Associate Instrument Ltd
Pipette	Capacity: 1, 5, 10ml	Borosil
Test tube	25,50ml	Borosil
Refrigerator	310 liter	Kelvinator
Filter Paper	Whatman No.1,dia-125mm	AXIVA, Sichem Biotech
Food Processor	No of speed-3, D_1/Q_0/M/2632,810w	Sujata ltd
Digital Camera	2.0 mega pixels	Sony
Stop watch	Range 15min, Least count 0.01sec	Timax
Thermometer	Range:0-110°C 300 mm	Sonar Laboratory
Desiccator	Diameter: 150 and 300 mm	Borosil
Petri dish	Disposable	Borosil

Table 2.2: Values of independent variables in coded and actual form

Independent variables		Coded levels				
Name	Code	-α	-1	0	+1	+α
		Actual Levels (%)				
Temperature (°C)	X_1	55	60	65	70	75
Maltodextrin (kg/kg papay pulp solid)	X_2	0.15	0.3	0.45	0.6	0.75
Glycerol monostearate (%)	X_3	0.50	1.0	1.50	2.0	2.5
Tricalcium phosphate (%)	X_4	0.50	1.0	1.50	2.0	2.5

Table 2.3: List of additives and their functions

Additives	Function
Maltodextrin (MD)$(C_6H_{10}O_5)_n$(LOBA chemie Pvt Ltd.)	▪ Used as a drying aids. ▪ MD treatment increases degree of solubility, up to 90% ▪ Reduces the stickiness of fruit powders ▪ Produces non sticky, free flowing powder ▪ Reduces the hygroscopicity of the powder
Tricalcium Phosphate (TCP)$(Ca_3\ O_8\ P_2)$ (LOBA chemie Pvt Ltd.)	▪ Used as an anticaking agent ▪ Reduces the hygroscopicity of powder ▪ Improve the flowability and inhibit the tendency to cake
Glycerol monosterate (GMS)$(C_{17}H_{35}COOCH_2CHOHCH_2OH)$ (LOBA chemie Pvt Ltd.)	▪ Act as a foam stabilizer ▪ For retention of puffed structure created in the initial stage of vacuum drying.

Table 2.4: Total numbers of experiments (designed by design expert trial version 8.0.6)

Expt No.	Coded values				Actual values of independent variables			
	X_1	X_2	X_3	X_4	Temp. °C	MD %	GMS %	TCP %
1	-2	0	0	0	55	0.45	1.5	1.5
2	1	-1	1	1	70	0.3	2	2
3	0	0	2	0	65	0.45	2.5	1.5
4	-1	1	-1	1	60	0.6	1	2
5	-1	1	1	1	60	0.6	2	2
6	0	0	0	-2	65	0.45	1.5	0.5
7	1	1	1	-1	70	0.6	2	1
8	0	0	0	0	65	0.45	1.5	1.5
9	-1	1	-1	-1	60	0.6	1	1
10	1	1	-1	1	70	0.6	1	2
11	0	0	0	0	65	0.45	1.5	1.5
12	1	1	-1	-1	70	0.6	1	1
13	-1	1	1	-1	60	0.6	2	1
14	1	-1	-1	-1	70	0.3	1	1
15	-1	-1	-1	1	60	0.3	1	2
16	0	-2	0	0	65	0.15	1.5	1.5
17	0	0	0	0	65	0.45	1.5	1.5
18	0	0	0	0	65	0.45	1.5	1.5
19	0	0	0	0	65	0.45	1.5	1.5
20	-1	-1	-1	-1	60	0.3	1	1
21	-1	-1	1	-1	60	0.3	2	1
22	-1	-1	1	1	60	0.3	2	2
23	0	0	-2	0	65	0.45	0.5	1.5
24	0	2	0	0	65	0.75	1.5	1.5
25	1	-1	-1	1	70	0.3	1	2
26	0	0	0	0	65	0.45	1.5	1.5
27	1	-1	1	-1	70	0.3	2	1
28	2	0	0	0	75	0.45	1.5	1.5
29	1	1	1	1	70	0.6	2	2
30	0	0	0	2	65	0.45	1.5	2.5

X_1= Temperature °C, X_2= Maltodestrin (w/w), X_3= Glycerol monosterate (%) and X_4= Tricalcium Phosphate (%)

temperature and tricalcium phosphate on hygroscopicity was highly significant ($P<0.01$) at linear level, while maltodextrin was significant at 10% level of significance. The coefficient of maltodextrin and tricalcium phosphate has negative that indicates with increase the level of these variables, hygroscopicity decreased. Effect of ingredients on hygroscopicity at linear, interactive and quadratic levels is reported in Table 3.7.It was clear from the table that the effect of ingredients on hygroscopicity at linear level was highly significant ($P<0.01$), but there is no significance in the quadratic and interactive levels. The sequential sum of squares method used to calculate total effect of individual parameter on hygroscopicity and depicted in Table 3.6. It was observed that temperature affected the hygroscopicity significantly at 1% level of significance while maltodextrin and tricalcium phosphate affected the hygroscopicity significantly at 5% level of significance but glycerol monostearate had no effect on hygroscopicity. It was seen from Figure3.3 (a) which depicts the effect of temperature on hygroscopicity at most efficient values of maltodextrin (0.55w/w), glycerol monostearate (1.78%) and tricalcium phosphate (2.5%) at linear level of papaya powder. It was observed that hygroscopicity of powder was increased gradually with increased the value of temperature. The minimum value of hygroscopicity (1.9%) was obtained at temperature 55°C. It was seen from Fig. 3.3(b) which depicts the effect of temperature on hygroscopicity at most efficient values of maltodextrin (0.55w/w), glycerol monostearate (1.78%) and tricalcium phosphate (2.5%) at linear level of papaya powder. It was seen that the value of hygrosopicity decreasing gradually with increasing the amount of maltodextrin. Fig. 3.3(c) shows the effect of tricalcium phosphate on hygrosiopicity at optimum values of temperature (59.05°C), maltodextrin (0.55w/w) and glycerol monostearate (1.78%) at linear level. Decreased the hygroscopicity with increased the levels of tricalcium phosphate. The minimum hygroscopicity of powder has obtained at highest level of tricalcium phosphate.

Effect of variables on degree of caking

The regression analysis is presented in Table 3.1 and shows the regression coefficients in the model and significance of each term. A second order mathematical model (equation no. 3.5 and 3.6) was used into the degree of caking data to examine the effect of variables. The coefficient of determination (R^2) of the regression model for degree of caking has 87.98%, which implies that the model could account 87.98 % variability in data. The second order model was significant at ($P<0.05$). The model was found suitable for describing the degree of caking behaviour because of higher value of R^2 and F. From Table 3.7 it is observed that the independent variable

Table 3.1: Results of regression analysis of papaya powder properties.

	Flowability Time(s)		Hygroscopicity (%)		Degree of caking (%)	
	Coeff.	P (%)	Coeff.	P (%)	Coeff.	P (%)
Cons	19.11	0.01*	4.703	0.01*	3.583	0.01
X_1	-2.085	0.01*	0.416	0.40*	0.302	1.00
X_2	-0.564	0.05**	-0.221	9.18***	-0.235	3.65
X_3	-0.235	8.30***	0.137	28.19	-0.035	73.70
X_4	-0.215	10.99	-1.188	0.01*	-0.983	0.01
X_1X_2	0.1523	34.07	-0.062	68.66	-0.055	66.70
X_1X_3	0.0134	93.05	-0.014	92.51	-0.010	93.75
X_1X_4	0.26	11.41	0.044	77.20	0.114	37.84
X_2X_3	0.16	31.82	0.109	47.83	0.095	46.01
X_2X_4	-0.194	23.04	0.098	52.41	0.064	61.84
X_3X_4	0.12	45.07	-0.044	77.20	-0.056	65.99
X_1X_1	0.006	96.13	0.212	8.46***	0.185	7.21
X_2X_2	0.156	20.77	-0.018	87.89	0.005	95.73
X_3X_3	-0.169	17.34	-0.035	76.28	-0.127	20.34
X_4X_4	-0.083	49.43	-0.214	8.21***	-0.109	27.46
R^2 (%)	95.38		88.80		88.59	
F	22.12		8.50		8.32	
LOF	NS		S		S	

*, **, *** Significant at 1, 5 and 10 % level of significance respectively, Cons= Constant and Coeff. = Coefficient

tricalcium phosphate was highly significant (P<0.01) at linear level, while temperature has significant at 5% level of significance. The quadratic term of temperature has found to be significant at 10% level of significance (P<0.1). The coefficients of interactive term like temperature, maltodextrin and tricalcium phosphate were negative indicating with the increase of the level of these variables, degree of caking decreased. Effect of ingredients on degree of caking at linear, quadratic and interactive levels is reported in Table 3.7.It shows to second order model and the effect at the linear level that was highly significant (P<0.01). There is no significant effect of interactive and quadratic level on degree of caking.

Total effect of individual parameter on degree of caking was calculated by using the sequential sum of squares method, and it is given in the Table 3.8. It was found that both temperature and tricalcium phosphate affected to the degree of caking significantly at 5% and 1 % level of significance respectively. Tricalcium phosphate had highest effect on degree of caking (P<0.01). At linear level, Fig. 3.4 (a) shows the variation of degree of caking with temperature at optimum values of maltodextrin (0.55%), glycerol monostearate (1.78%) and

Table 3.2: Quality parameters for papaya powder

ExptNo.	Coded levels				FL,sec	HG,%	DC,%
	X_1	X_2	X_3	X_4			
1	-2	0	0	0	23.62*	5.23	3.98
2	1	-1	1	1	17.38	3.47	2.42
3	0	0	2	0	18.52	5.13	3.35
4	-1	1	-1	1	19.35	2.65	1.87
5	-1	1	1	1	20.12	2.89	1.98
6	0	0	0	-2	20	6.42	5.25
7	1	1	1	-1	16.17	6.36	4.78
8	0	0	0	0	19.81	4.67	3.64
9	-1	1	-1	-1	21.09	4.94	4.31
10	1	1	-1	1	16.28	3.42	2.67
11	0	0	0	0	19.41	4.39	3.57
12	1	1	-1	-1	16.73	5.27	3.86
13	-1	1	1	-1	20.45	5.53	3.98
14	1	-1	-1	-1	17.12	6.23	4.97
15	-1	-1	-1	1	22.03	2.24	1.88
16	0	-2	0	0	21.64	6.27	5.03
17	0	0	0	0	18.98	4.81	3.43
18	0	0	0	0	18.58	4.78	3.74
19	0	0	0	0	19.12	4.46	3.36
20	-1	-1	-1	-1	22.18	5.31	4.29
21	-1	-1	1	-1	21.49	5.37	4.36
22	-1	-1	1	1	20.49	2.94	1.92
23	0	0	-2	0	19.35	4.96	3.58
24	0	2	0	0	18.83	3.96	2.96
25	1	-1	-1	1	17.36	3.72	3.08
26	0	0	0	0	18.76	5.11	3.76
27	1	-1	1	-1	16.06	6.32	4.57
28	2	0	0	0	15.65**	6.84*	5.45*
29	1	1	1	1	16	3.85	2.54
30	0	0	0	2	18.56	2.24**	1.83**

*- for maximum, **- for minimum, FL-flowability time, HG-hygroscopicity, DC-degree of caking

Table 3.3: ANOVA for flowability time

Source	DF	SS	MS	F_{Cal}	F_{Tab}
Model	14	118.99	8.5	22.12*	3.56
Linear	4	114.41	28.6	75.263*	4.893
Interactive	6	2.69	0.449	1.182	1.99
Quadratic	4	1.64	0.41	1.079	1.99
Residual error	15	5.76	0.38		
Total	**29**	**124.75**			

*, **, *** Significant at 1, 5 & 10% level of significance respectively. (Model- $F_{(14, 15, 0.01)} = 3.56$, $F_{(14, 15, 0.05)} = 2.42$ & $F_{(14, 15, 0.1)} = 1.99$), (Linear & Quadratic level- $F_{(4, 15, 0.01)} = 4.893$, $F_{(4, 15, 0.05)} = 3.056$ & $F_{(4, 15, 0.1)} = 2.36$) and (Interactive level-$F_{(6, 15, 0.01)} = 4.318$, $F_{(6, 15, 0.05)} = 2.79$ & $F_{(6, 15, 0.05)} = 2.208$)

tricalcium phosphate (2.5%). It has observed that decreased the degree of caking of powder with decreased the level of temperature. At linear level Fig. 3.4 (b) shows the variation of degree of caking with maltodextrin at optimum values of temperature (59.05°C), glycerol monostearate (1.78%) and tricalcium phosphate (2.5%). It has seen that no effect of maltodextrin on degree of caking has observed. At linear level Fig. 3.4 (c) relationship between degree of caking and tricalcium phosphate at optimum values of temperature (59.05°C), maltodextrin (0.55%) and glycerol monostearate (1.78%). It was seen from figures the degree of caking was totally affected by tricalcium phosphate. Minimum degree of caking was found at highest level of tricalcium phosphate

Optimization of ingredient variables for production of papaya powder

In this work the numerical optimization of independent variables was done carried out using Design–Expert 8.0.6 statistical tool. The main aim was fixed to be in the range for flowability time, hygroscopicity, degree of caking, solubility, ascorbic acid, beta carotene and colour. To archive aim this work was begins at random starting point and proceeds up and down the steepest slope on the response surface for a maximum or minimum value of the response respectively. The Table 3.9 represents to goal setup for optimization. Optimization has done according to above criteria. Optimization achieved through 45 solutions, out of which one most suited the criteria has selected. Appendix-V contains all optimum points obtained. Table 3.10 represents the most suitable optimum point.

SUMMARY AND CONCLUSION

Finding during work, represents the measured quality parameters of papaya powder at each experimental run. The flowability time of papaya powder ranges from

Table 3.5: ANOVA for hygroscopicity

Source	DF	SS	MS	F_{-Cal}	F_{-Tab}
Model	14	43.07	3.08	8.5*	3.56
Linear	4	39.65	9.913	27.53*	4.893
Interactive	6	0.468	0.078	0.2166	2.208
Quadratic	4	2.53	0.633	1.7588	2.36
Residual error	15	5.43	0.36		
Total	**29**	**48.5**			

*, **, *** Significant at 1, 5 & 10% level of significance respectively,
(Model- $F_{(14, 15, 0.01)} = 3.56$, $F_{(14, 15, 0.05)} = 2.42$ & $F_{(14, 15, 0.1)} = 1.99$), (Linear & Quadratic level- $F_{(4, 15, 0.01)} = 4.893$, $F_{(4, 15, 0.05)} = 3.056$ & $F_{(4, 15, 0.1)} = 2.36$) and (Interactive level-$F_{(6, 15, 0.01)} = 4.318$, $F_{(6, 15, 0.05)} = 2.79$ & $F_{(6, 15, 0.05)} = 2.208$)

Table 3.4: Total effect of individual parameters on flowability time

Source	DF	SS	MS	F_{-Cal}	F_{-Tab}
Model	14	118.99	8.5	22.12*	3.56
Temperature (X_1)	5	105.78	21.156	55.68*	4.556
Maltodextrin (X_2)	5	9.69	1.938	5.11*	4.556
Glycerol Monostearate (X_3)	5	2.753	0.55	1.448	2.273
Tricalcium Phosphate (X_4)	5	3.21	0.642	1.689	2.273
Residual error	15	5.76	0.38		
Total	**29**	**104.65**			

*, **, *** significant at 1, 5 & 10% level of significance respectively (Model- $F_{(14, 15, 0.01)} = 3.56$, $F_{(14, 15, 0.05)} = 2.42$ & $F_{(14, 15, 0.1)} = 1.99$) and (Independent variables-$FF_{(5, 15, 0.01)} = 4.556$, $F_{(5, 15, 0.05)} = 2.901$ & $F_{(5, 15, 0.1)} = 2.273$).

Table 3.6: Total effect of individual parameters on hygroscopicity

Source	DF	SS	MS	F_{-Cal}	F_{-Tab}
Model	14	43.07	3.08	8.5*	3.56
Temperature (X_1)	5	5.486	1.097	3.05**	2.901
Maltodextrin(X_2)	5	1.579	0.3159	0.8776	2.273
Glycerol Monostearate (X_3)	5	0.827	0.165	0.459	2.273
Tricalcium Phosphate (X_4)	5	7.068	1.414	3.927**	2.901
Residual error	15	5.43	0.36		
Total	**29**	**48.5**			

*, **, *** significant at 1, 5 & 10% level of significance respectively.
(Model- $F_{(14, 15, 0.01)} = 3.56$, $F_{(14, 15, 0.05)} = 2.42$ & $F_{(14, 15, 0.1)} = 1.99$) and (Independent variables-$FF_{(5, 15, 0.01)} = 4.556$, $F_{(5, 15, 0.05)} = 2.901$ & $F_{(5, 15, 0.1)} = 2.273$).

Second order predictive quadratic equation for hygroscopicity (%) is given below

$$Y=4.70+0.42X_1-0.22X_2+0.14X_3-1.19X_4-0.062X_1X_2-0.014X_1X_3+0.044X_1X_4+0.11X_2X_3+0.098X_2X_4-0.044X_3X_4+0.21X_1^2-0.018X_2^2 - 0.035\ X_3^2 -0.21X_4^2 \quad \ldots 3.3)$$

Significant predictive equation for hygroscopicity (%) is given below

$$Y=4.70+0.42X_1-0.22X_2-1.19X_4+0.21X_1^2 -0.21X_4^2 \quad \cdots (3.4)$$

Where, Y is hygroscopicity (%),X_1, X_2, X_3 and X_4 are coded variables for temperature, maltodextrin, and glycerol monostearate and tricalcium phosphate.

Table 3.7: ANOVA for degree of caking

Source	DF	SS	MS	F_{-Cal}	F_{-Tab}
Model	14	29.25	2.09	8.32*	3.56
Linear	4	26.749	6.687	26.74*	4.893
Interactive	6	0.516	0.086	0.344	2.208
Quadratic	4	1.7	0.425	1.7	2.36
Residual error	15	3.77	0.25		
Total	**29**	**33.02**			

*, **, *** Significant at 1, 5 & 10% level of significance respectively
(Model- $F_{(14, 15, 0.01)} = 3.56$, $F_{(14, 15, 0.05)} = 2.42$ & $F_{(14, 15, 0.1)} = 1.99$), (Linear & Quadratic level- $F_{(4, 15, 0.01)} = 4.893$, $F_{(4, 15, 0.05)} = 3.056$ & $F_{(4, 15, 0.1)} = 2.36$) and (Interactive level-$F_{(6, 15, 0.01)} = 4.318$, $F_{(6, 15, 0.05)} = 2.79$ & $F_{(6, 15, 0.05)} = 2.208$)

Table 3.8: Total effect of individual parameters on Degree of caking

Source	DF	SS	MS	F_{-Cal}	F_{-Tab}
Model	14	29.25	2.09	8.32*	3.56
Temperature (X_1)	5	105.78	21.156	2.815***	2.273
Maltodextrin(X_2)	5	9.69	1.938	1.267	2.273
Glycerol Monostearate (X_3)	5	2.753	0.55	0.528	2.273
Tricalcium Phosphate (X_4)	5	3.21	0.642	19.085*	4.556
Residual error	15	3.77	0.25		
Total	**29**	**33.02**			

*, **, *** significant at 1, 5 & 10% level of significance respectively. (Model- $F_{(14, 15, 0.01)} = 3.56$, $F_{(14, 15, 0.05)} = 2.42$ & $F_{(14, 15, 0.1)} = 1.99$) and (Independent variables-$FF_{(5, 15, 0.01)} = 4.556$, $F_{(5, 15, 0.05)} = 2.901$ & $F_{(5, 15, 0.1)} = 2.273$).

Second order predictive quadratic equation for degree of caking (%) is given below

$$Y=3.58+0.30X_1-0.24X_2-0.035X_3-0.98X_4-0.055X_1X_2-0.01X_1X_3+0.11X_1X_4+0.095X_2X_3 +0.064X_2X_4-0.056X_3X_4+0.19X_1^2 +0.005208X_2^2 - 0.13\ X_3^2 -0.11X_4^2 \quad \ldots.. (3.5)$$

Significant predictive equation for degree of caking (%) is given blow

$$Y=3.58-0.30X_1-0.24X_2-0.98X_4+0.19X_1^2 \quad \ldots\ldots (3.6)$$

Where, Y is degree of caking (%), X_1, X_2, X_3 and X_4 are coded variables for temperature, maltodextrin, and glycerol monostearate and tricalcium phosphate.

Table 3.9: Value of dependent variables for optimization

Name	Goal	Lower Limit	Upper Limit
Temperature (X_1)	is in range	-2	2
Maltodextrin(X_2)	is in range	-2	2
Glycerol Monostearate (X_3)	is in range	-2	2
Tricalcium Phosphate (X_4)	is in range	-2	2
Flowability Time	minimize	15.65	23.62
Hygroscopicity	minimize	2.24	6.84
Degree of Caking	minimize	1.83	5.45

Table 3.10: Value of ingredients for optimization

Independent variables	Coded levels	Actual levels
Temperature (X_1)	-1.19	59.05 (°C)
Maltodextrin(X_2)	0.65	0.55 (%)
Glycerol Monostearate (X_3)	0.56	1.78 (%)
Tricalcium Phosphate (X_4)	2.0	2.5 (%)

15.65 to 23.62 s. Its value is minimum at experiment run number 28 and maximum at experiment run number 1 with the combination of process conditions of temperature 65°C, maltodextrin 0.45 (w/w), glycerol monostearate 1.5% and tricalcium phosphate 1.5% and temperature 60°C, maltodextrin 0.30 (w/w), glycerol monostearate 1.0% and tricalcium phosphate 2.0% respectively. Hygroscopicity starts from 2.24 (Expt. No. 30) to 6.84 (Expt. No. 28) while the degree of caking is in between 1.83 (Expt. No. 30) to 5.45% (Expt. No.28). From experiments the drying data is as follow, the minimum drying time was obtained (660 min.) with the combination of the ingredients, temperature (60°C), maltodextrin (0.60 gm/110gm), GMS (1.0%) and TCP (2.0 %) of dried papaya powder. The minimum drying time was due to the high amount of maltodextrin because it has the tendency to reduce the moisture content quickly from the product. It was also found after experiments that drying time decreased due to increase in the amount of tricalcium phosphate (2%) in the materials so it help to reduce the moisture content of the products. The drying time increase, a non-linear decrement take place in moisture ratio for all sample. With very small value of drying time the moisture ratio of each sample decreased rapidly but as the drying time increase the rate of decrement of moisture ratio become very slow. Flowability time has significantly affected by temperature ($P<0.01$), maltodextrin (<0.05) and glycerol monostearate ($P<0.1$), at linear level. The coefficient of temperature, maltodextrin, and glycerol monostearate and tricalcium phosphate are negative and therefore with increase in the levels of these, the flowability time decreases. The effect of temperature and tricalcium phosphate on hygroscopicity was highly significant ($P<0.01$) at linear level, while maltodextrin was significant at 10% level of significance. The coefficient of maltodextrin and tricalcium phosphate has negative indicating with increase the level of these variables, hygroscopicity decreased. It was found that both temperature and tricalcium phosphate affected to the degree of caking significantly at 5% and 1 % level of significance respectively. Tricalcium phosphate had highest effect on degree of caking ($P<0.01$).The independent variable tricalcium phosphate was highly significant ($P<0.01$) at linear level, while temperature has significant at 5% level of significance. The quadratic term of temperature has found to be significant at 10% level of significance ($P<0.1$). The coefficients of interactive term like temperature, maltodextrin and tricalcium phosphate were negative indicating with the increase of the level of these variables, degree of caking decreased.

ACKNOWLEDGEMENTS

We are also deeply thankful to the Department of PHPFE, College of Technology, GBPUAT Pantnagar, to give us an opportunity to pursue this research.

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Received: August 9, 2021
Accepted: September 1, 2021