Sustainable Food Technology

PAPER

Cite this: *Sustainable Food Technol.*, 2023, **1**, 750

Optimization of spray-drying conditions using response surface methodology, physico-chemical characterization and shelf-life estimation of pineapple powder

Ramesh Sharma,[a] Pinku Chandra Nath[a] and Dibyakanta Seth*[b]

Received 18th January 2023
Accepted 22nd July 2023

DOI: 10.1039/d3fb00008g

rsc.li/susfoodtech

Response surface methodology (RSM) was used in this study to optimize spray drying process conditions for pineapple juice powder (PJP) production. Box–Behnken design (BBD) with three factorials and three levels was used for experimental design. The inlet air temperature (X_1), feed rate (X_2) (10–20 ml min^{-1}), and maltodextrin (10DE) content (X_3) (30–50% of TSS of pineapple juice) were the independent factors. Multiple regression was used to build the model and statistical analysis of variance (ANOVA) was performed to establish the significance of the data. Moisture content and powder yield were the response variables employed for analysis. Moisture content and yield both had R^2 values of 0.92 and 0.90, respectively. The response model, which had a lower p-value (0.05), produced results that were statistically significant for the experimental data. The optimization conditions of an inlet air temperature of 171 °C, feed rate of 14.57 ml min^{-1}, and maltodextrin content of 40.83% were achieved and used to provide 68.20 ± 0.22 powder yield and 5.27 ± 0.12 moisture content (w.b). Adsorption isotherm curves of the powder revealed the characteristics of a type III shape. The Guggenheim–Anderson–de Boer (GAB) and Iglesias & Chirife models were the best-fitted models to demonstrate the sorption isotherm behaviour of dried PJP powder. The accelerated storage studies of PJP was performed at temperatures 40, 50, 60 & 70 °C where ascorbic acid loss was taken as the marker of thermal degradation of the product. At 40 °C, the shelf life of pineapple powder was obtained as 12.35 days, whereas on increasing the storage temperature the shelf life of the powder decreased. Similarly, the shelf life of pineapple powder at 50, 60 & 70 °C was obtained as 6.11, 2.78, and 0.8 days, respectively, and the calculated activation energy was 19.534 kJ mol^{-1}.

Sustainability spotlight

Pineapple is grown abundantly in the North-East region of India. Farmers are forced to sell it at a throw-away price at the time of harvest. Since, it is a highly perishable fruit, its conversion to various value added products will ensure a sustainable supply-chain in the fruit market. An attempt was made to develop spray dried pineapple powder so that it can be consumed at any time in the form of rehydrated pineapple juice. Value addition to pineapple fruit would be a step for sustainable production and supply-chains in North-East India.

1. Introduction

Ananas comosus, or pineapple is one of the crucial species in the Bromeliaceae family. After orange and apple juice, pineapple juice consumption ranks third with an annual consumption of 2000 tonnes.[1] A large new variety of pineapple is mostly grown for commercial purposes in India, along with other native varieties such as queen, Mauritius, Charlotte, Rothchild, Gallup, and Lakhat. The largest pineapple growing state in India is Meghalaya, which contributes 8% of the country's output on an area of 10 523 ha and produces 102 506 tonnes of pineapple annually (2008–2009).[2] Pineapple fruit is typically consumed in the form of candied pineapple, jams, juices, or by adding flavor to other processed foods. Due to its perishable nature, fruit juice with a greater ascorbic acid content presents one of the most difficult challenges for export or import. In order to increase its shelf life by turning it into powder, drying is considered a suitable alternative method. Fruit juice can be dried using spray drying that produces a powder with a fine texture and lower water activity, and it dries quickly while weighing less, making storage and transit easier.[3] The drying process is governed by a number of process variables, including temperature, airflow, and humidity.[4] The quick drying of materials and even distribution of particles make spray drying

[a]Department of Bio Engineering, National Institute of Technology Agartala, Jirania, 799046, India. E-mail: rameshdawari156@gmail.com; nathpinku005@gmail.com
[b]Department of Food Process Engineering, National Institute of Technology, Rourkela, 769008, India. E-mail: dibya05@gmail.com

the favored drying technique. The powdered pineapple juice's alleged extended shelf life and convenience of packaging and transportation are its main reported advantages.[5] The handling of powder in a clean environment, reduced operating costs, and less droplet contact in the drying chamber are some additional advantages of drying,[6] with the retention of nutrients and enzymes.[3] The dried goods are divided into sticky and non-sticky categories, and the sticky powder requires more drying time to produce. Sticky products are more difficult to dry, which results in decreased powder yield, equipment damage, and other drying issues related to stickiness in equipment.[7] The main cause of the stickiness is the presence of sugar, specifically glucose, sucrose, and fructose, which also contributes to the stickiness.[6] Due to stickiness issues brought on by the juice's sugar and acid content, the yield of powder is decreased throughout the drying process.[8] Low molecular weight substances, such as sugars like fructose and sucrose and organic acids like citric acid and malic acid, are what make up these components. Therefore, the value of the glass transition temperature (T_g) of powder materials can be used to forecast stickiness.[3] Maltodextrin (MD) can be used to make fruit juice less sticky.[9] These carrier agents are high molecular weight compounds that encase the juice particles to increase the mixture's T_g.[10] When it comes to orange juice powder, maltodextrin aids in enhancing product recovery and lightness.[11] Because of its connection to nutritional features that encourage the conversion of liquids to powder with better flow characteristics while preventing the loss of the taste, maltodextrin is utilized as a carrier agent.[12] The moisture sorption isotherm makes it simple to explain how water activity (a_w) and equilibrium moisture content are correlated. Each powder product has its own isotherm characteristics, making it possible to predict the lowest critical moisture content that will be present during the drying process or during storage.[13–15] Similar to this, the monolayer moisture content (m_o) denotes the acceptable moisture content for secure storage that preserves the quality of the dried product.[16] Due to physical moisture absorption from the porous structure of food ingredients, the isotherm curve changed its shape.[17] According to Brunauer's classification, type II and type III isotherms showed reduced water absorption as the temperature rose at a persistent a_w.[18,19] Food components such as starch undergo changes in their structure and physicochemical characteristics upon drying to become gelatinous,[20] and sugar undergoes crystallization at higher temperature. However, because pineapple juice tends to stick during drying processes, less research has been carried out on spray drying of pineapple juice. With an evaluation of sorption isotherm studies of an optimized powder sample, the current study sought to use response surface methodology (RSM) to quantify the effect on examined response variables, including the moisture content and yield of powder.

2. Materials and methods

2.1. Collection of raw materials

The Kew variety of pineapple was said to have a strong flavor and a yellowish color. The pineapple was also blended in a blender after being uniformly chosen based on its shape and level of ripeness. A medium-ripe pineapple was bought from the Tezpur market from Assam, India, and kept chilled for later usage. Maltodextrin with a dextrose equivalent (DE) of ten (10), a grade with a greater molecular weight, a glass transition temperature (T_g) of 188 °C, and a melting point of 240 °C was selected as the carrier agent. The chosen pineapple was peeled, and then the juice was extracted in a blender. To extract the pineapple juice, the gathered puree was forced through a muslin cloth to collect the juice. The extracted material was thrown away. The juice was kept in a refrigerator after analyzing the TSS and pH of the sample.

2.2. Preparation of juice

Prior to the addition of maltodextrin at concentrations of 30%, 40%, and 50% (wt/wt) of the total soluble solids (TSS) of pineapple juice, the juice was prepared in 600 ml batches for feeding into the drying chamber. Pineapple juice had an average TSS of 11.19 °Brix. Maltodextrin was added in amounts of 20.14 g, 26.85 g, and 33.57 g, respectively, to 600 ml of the extracted pineapple fruit juice to make juice samples with various concentrations of maltodextrin, *viz.* 30%, 40%, and 50%. The mixture was briskly agitated to ensure proper mixing while preventing the creation of lumps. The mixture was pre-heated to 50 °C for 10 minutes after adding maltodextrin before being fed to a spray dryer. Fig. 1 depicts the complete procedure for extracting and drying pineapple juice.

2.3. Spray drying

The extracted mixed juice containing maltodextrin was dried in a lab-scale spray drier (Model: SPD-P-111, Tezpur, Assam) equipped with an atomizing nozzle (1.2 mm-diameter) and a two-fluid spraying nozzle. The maltodextrin concentration was fixed at 30–50% and chosen based on the TSS of pineapple juice. The influence of drying parameters such as inlet drying air temperature (X_1) at 130 °C (−1), 155 °C (0), and 180 °C (+1), feed rate (X_2) at 10 (−1), 15 (0), and 20 ml min^{-1} (+1), and maltodextrin concentration (X_3) at 30% (−1), 40% (0), and 50% (+1) was evaluated using the Box–Behnken design (BBD). With respect to X_1, X_2, and X_3, the moisture content (Y_1) and yield (Y_2) are regarded as response variables. The analysis of variance (ANOVA) and RSM were used to forecast the main effect and interaction impact of X_1, X_2, and X_3. Using design expert software 13 (Tezpur university, Assam), the effects of X_1, X_2, and X_3 on yield and moisture content were assessed. Table 1 displays the experimental results for spray drying of PJP. The following empirical second-order polynomial equation was adopted.

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + = \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum \beta_{ij} x_i x_j$$

where y represents the response variable; β_0 is a constant term; X_i and X_j represent independent variables; and β_i, β_{ii}, and β_{ij} symbolize the linear, quadratic, and interaction coefficients, respectively. The optimizations of the response variable were performed through the design expert software. To generate the best response the desirability function was performed.

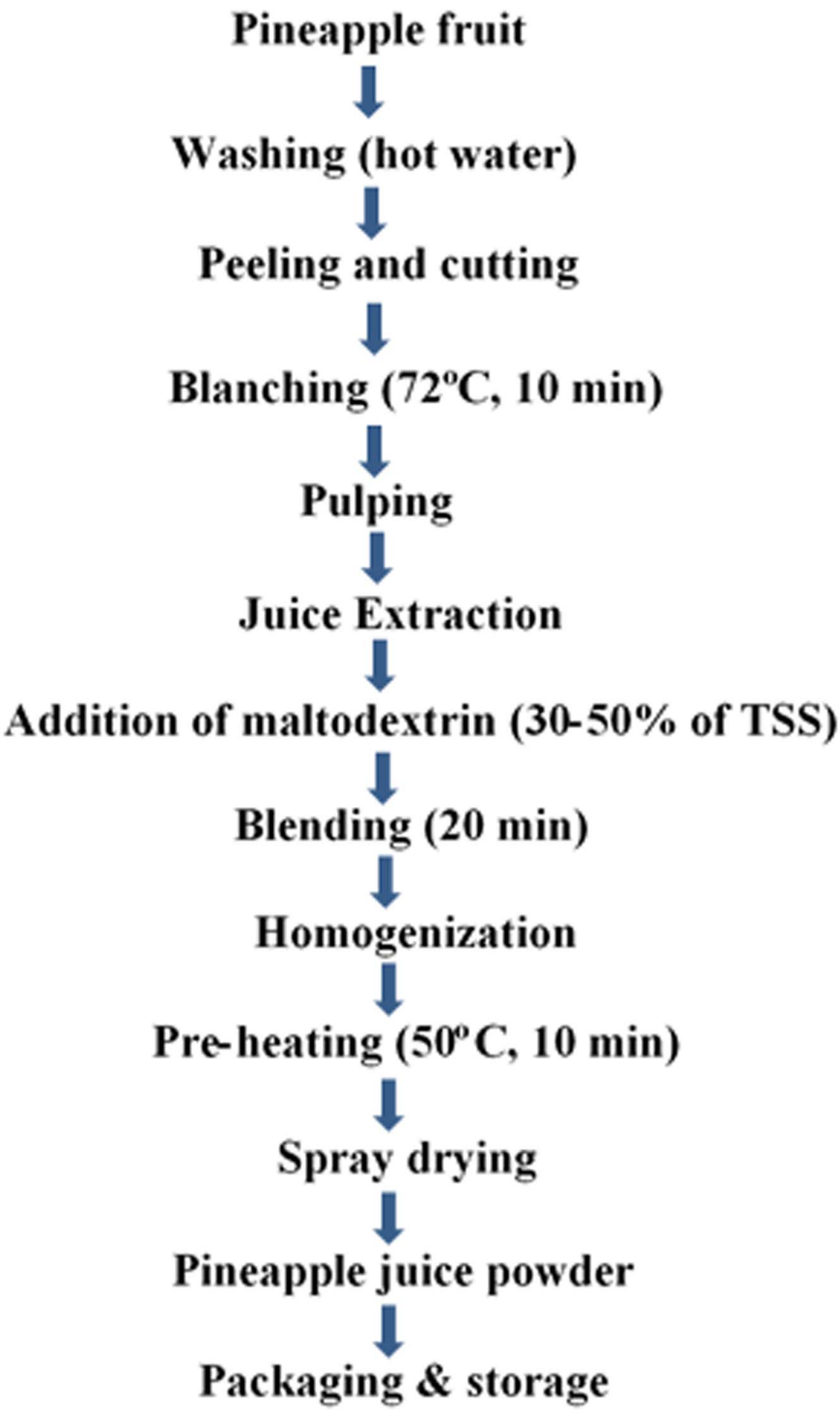

Fig. 1 Process flow chart for pineapple juice extraction and spray drying.

Derringer's concept involves the conversion of each response (y_k) to the individual desirability (d_k). The scale of desirability ranged from 0 to 1; in the case of outside the range d_k is 0 and in the case of full desirability, d_k is considered to be 1. The value is considered as a quality indicator for spray-dried PJP with its significant effect on BBD and considering the best method.[21] The individual desirability was collected from the response, and overall desirability D (eqn (1)) was computed which is calculated by considering the geometric average of individuals' desirability values.

$$D = (d_1, d_2 \ldots d_k)^{1/k} \tag{1}$$

The higher values of D represented the best solution.[22]

Table 1 The predicted content of physicochemical properties of pineapple juice powder

Sl no.	(AIT) (°C) (X_1)	MTD (%) (X_3)	Feed rate (ml min^{-1}) (X_2)	Moisture content (% w.b) (Y_1)	Yield (%) (Y_2)	D values
1	180	50	15	8.50 ± 1.050	59.23 ± 2.170	0.884
2	130	50	15	11.1 ± 1.331	52.12 ± 2.070	0.801
3	155	50	20	12.1 ± 1.404	60.78 ± 1.970	0.833
4	155	40	15	11.5 ± 1.686	59.89 ± 2.370	0.829
5	180	40	15	11.68 ± 1.379	68.23 ± 2.270	0.932
6	180	40	10	6.34 ± 1.218	63.78 ± 2.470	0.920
7	130	40	10	13.25 ± 1.071	51.10 ± 1.890	0.792
8	155	50	10	9.22 ± 1.078	60.55 ± 1.970	0.857
9	155	40	15	10.65 ± 1.041	60.56 ± 2.570	0.852
10	130	30	15	14.6 ± 1.070	50.90 ± 2.178	0.788
11	155	30	20	13.67 ± 1.011	48.12 ± 2.070	0.825
12	130	40	20	13.3 ± 1.068	45.67 ± 2.150	0.760
13	130	40	15	12.75 ± 1.045	50.12 ± 1.707	0.762
14	155	40	15	12.67 ± 1.063	60.89 ± 1.670	0.850
15	180	30	15	7.98 ± 1.073	65.90 ± 1.630	0.901
16	180	40	20	10.24 ± 1.049	64.70 ± 1.460	0.887
17	155	30	10	9.94 ± 1.078	59.12 ± 1.870	0.832

2.4. Physicochemical properties of PJP

2.4.1. Total soluble solids, pH, water activity, and moisture content. The TSS of pineapple juice was measured by using a refractometer with a scale of 0–32 °Brix. A digital pH meter was used to record the pH values, and the moisture content of spray-dried powder was determined by the AOAC method.[23] A water activity meter was used to measure the water activity of the sample (HP23-AW-A, Tezpur, Assam) at 25 ± 1.0 °C. The collected powder was stored at 30 °C in a room and kept for further analysis. The optimized PJP was collected and used for the physicochemical analysis.

2.5. Yield

The yield of powder was estimated with eqn (2)

$$\text{Yield}(\%) = \frac{M_{\text{powder}}\left(1 - X_{\text{W,powder}}\right)}{M_{\text{juice}}\left(1 - X_{\text{juice}}\right)} \times 100 \tag{2}$$

where Y represents the yield of powder, M_{powder} is the collected powder after cyclone separation during the drying process, M_{juice} is the mass (g) of the sample fed to the spray dryer; $X_{\text{w,powder}}$ and $X_{\text{w,juice}}$ are the fractions representing the powder content and juice on a wet basis, respectively.

2.6. Sorption isotherms

The static gravimetric method was used for the sorption isotherm studies. Eight volumes were considered to make the saturated salt solutions including LiCl, $MgCl_2$, $Mg(NO_3)_2$, $NaNO_2$, KI, NaCl, KCl and $BaCl_2$ which provided a relative humidity of 11.30%, 32.80%, 52.90%, 65.40%, 68.90%, 75.30%, 84.30% and 90.30%, respectively.[24] The PJP sample of 1 g was weighed and placed in desiccators maintaining desired temperatures of 20 °C, 30 °C, 40 °C & 50 °C for the particular salt solution for the desired relative humidity. The temperatures were maintained in a controlled hot air oven (M: AI-7781, Tezpur, Assam). The weighing of the sample was performed at regular intervals and waited to achieve the equilibrium. The Guggenheim–Anderson–de Boer (GAB) model is represented by eqn (3), Brunauer–Emmett–Teller (BET) model by eqn (4), the Oswin model by eqn (5), and Iglesias Chirife model by eqn (6), which were fitted with sorption isotherm data of PJP.

$$M_{\text{w}} = \frac{M_{\text{o}}CKa_{\text{w}}}{(1 - Ka_{\text{w}})(1 - Ka_{\text{w}} + CKa_{\text{w}})} \tag{3}$$

$$M_{\text{w}} = \frac{M_{\text{o}}Ca_{\text{w}}}{(1 - a_{\text{w}}) + (C - 1)(1 - a_{\text{w}})a_{\text{w}}} \tag{4}$$

$$M_{\text{w}} = \left(\frac{a_{\text{w}}}{1 - a_{\text{w}}}\right)^n \tag{5}$$

$$\ln[M_{\text{w}} + M_{\text{W}}^2 + M_{\text{W},0.5}]^{1/2} = C_1 \times a_{\text{w}} + C_2 \tag{6}$$

where M_{w} signifies the moisture (d.b), a_{w} signifies the water activity, M_{o} represents the monolayer moisture content (d.b), and C, and K represent the model constant. These parameters were non-linearly analyzed using MATLAB software.

2.7. Accelerated storage studies

Temperatures of 40, 50, 60 & 70 °C were used for storage studies. The powder samples for each batch were kept at a particular temperature in a hot air oven (AI: 7781, Tezpur Assam). The powder samples were taken at particular time intervals of 7, 14, 21, 28, 35 & 42 days for all sets of temperatures for measuring the physicochemical quality, including the ascorbic acid. The ascorbic acid content of pineapple powder was measured by dissolving it in distilled water following the following methodology.[25] The logarithmic ascorbic acid content (mg g^{-1}) was calculated from all sets of experiments and linearly plotted concerning time intervals of 7, 14, 21, 28, 35 & 42 days, and the rate constant (d^{-1}) was calculated from each temperature as shown in Fig. (2). The Arrhenius equation correlates the occurrence of the chemical reaction rate over

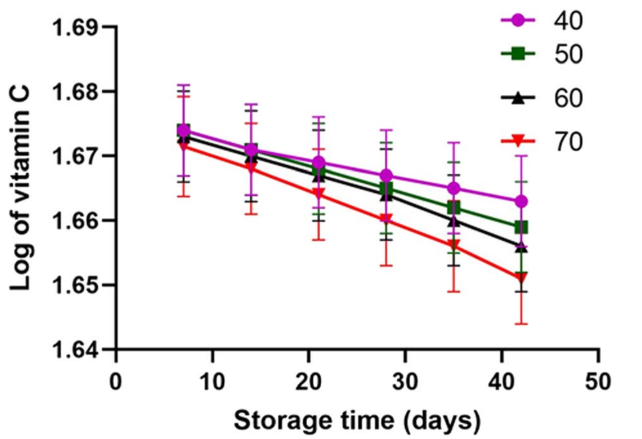

Fig. 2 Graph representing log of vitamin C *versus* storage time at 40, 50, 60 & 70 °C.

the change in temperature of the system. The prediction of shelf life and establishment of a relation between spoilage conditions and the rate of degradation of products is shown in eqn (9)

$$k = A\mathrm{e}^{\frac{-E_a}{RT}} \tag{7}$$

Taking ln on both the sides the linearized eqn (7) is transformed into eqn (8)

$$\ln(k) = \ln(A) - \frac{E_a}{RT} \tag{8}$$

where the graph was generated for ln(k) *vs.* 1/T and the activation energy was computed as shown in Fig. (3).where A signifies the frequency factor, R is the universal gas constant (J mol^{-1} K^{-1}), T is the absolute temperature, E_a represents the activation energy in J mol^{-1} K^{-1} and k is the rate constant in d^{-1}. The correlation of the rate constant (d^{-1}) *versus* the shelf life of the product was generated with eqn (9).

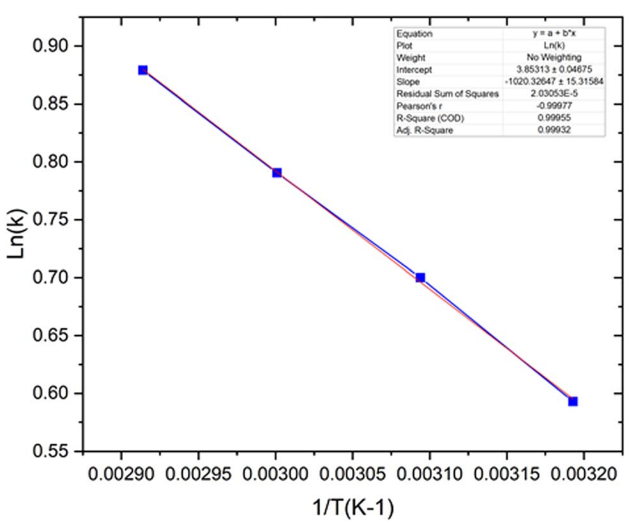

Fig. 3 Plotting of ln(k) *vs.* 1/T($K - 1$) for estimation of activation energy.

$$k = \frac{2.303}{t}\log\frac{C_o}{C} \tag{9}$$

where t (days) is the shelf life of pineapple powder in days, C_o is the concentration of ascorbic acid (mg g^{-1}) under the initial conditions, and C is the concentration of ascorbic acid after time interval t in days.

2.8. Statistical analysis

Tukey's test was applied to examine the means at a $p < 0.05$. The results were presented as mean ± standard deviation (SD). For the experimental design, Design Expert was applied. Regression analysis of the experimental data, and 3D response surface were executed using Design Expert Version 13.11.

3. Results and discussion

3.1. Characterization of the PJP

The TSS of pineapple juice was in the range of 11.19 ± 0.32 °Brix and the titratable acidity was 1.07 ± 0.05%. The pH value of pineapple juice lies in the range of 2.78 ± 0.05 to 3.07 ± 0.0.08. The estimated physico-chemical characteristics of spray-dried PJP are presented in Table 1. The optimized PJP was collected and used for physicochemical analysis and the obtained physicochemical properties were 9.78 ± 0.27 °Brix of TSS, 1.23 ± 0.15% (titratable acidity) and 2.90 ± 0.10 to 3.34 ± 0.09 of pH. The water activity of the PJP ranged from 0.31 ± 0.52 to 0.35 ± 0.48.

3.2. Fitted models and response surfaces

Seventeen trials were carried out in accordance with the BBD design using RSM. Maltodextrin was not initially added when the pineapple juice was spray-dried, and this resulted in an issue with stickiness inside the drying chamber, which prevented the powder from being formed. The formation of free-flowing powder by spray drying is hampered by the greater levels of acids and sugar in pineapple juice. After several failed attempts, maltodextrin was finally chosen as the carrier agent, which helped to improve the powder's flow properties while reducing stickiness. The range of maltodextrin concentration chosen was 30 to 50% of the TSS of pineapple juice, and higher maltodextrin concentrations also increased the stickiness of powder. At an air inlet temperature of 180 °C, the spray-dried powder had a more dark-colored appearance. The ideal air temperature for drying was 171 °C, at which point the powder quality was good with a low moisture content and increased yield. A maltodextrin concentration of 40% produced the best powder flow characteristics, a low moisture content, and a higher yield value, while concentrations of 30% and 50% gave good and bad powder features. After a number of trials, a feed rate concentration of 10–20 ml per minute was established. As the feed rate increased, the retention period of the droplets inside the chamber also grew, and more time was needed for the droplets to evaporate the moisture. In comparison to other feed rates, 14.57 ml per minute produced the powder with the best overall attributes. The best optimized conditions include an air inlet temperature of 171 °C, maltodextrin concentration of 41%, and feed rate of 14.57 ml min^{-1}, giving the predicted response values of 7.10 ± 0.783 (moisture content) and 69.23 ± 1.230 (yield) (Table 5). The spray-dried PJP was stable at room temperature for one week. Physico-chemical parameters of PJP under various drying conditions are shown in Table 1. The optimization parameters including the coefficient of determination (R^2), coefficients of regression (β) of linear and quadratic polynomial equations, the interaction effect on response variables, and the lack-of-fit test and probability test are reported in Table 2. The ANOVA of the two response variables is presented in Table 3. After analysis, it is revealed that the obtained quadratic model adequately correlated with experimental data with a predicted R^2 of 0.92 & 0.90, for moisture content and yield as the response variables (Table 2). As per the suggestion it is recommended to have an R^2 higher than 80%. In this case R^2 is closer to unity for an obtained empirical model which revealed the good fitting with actual data. In contrast, the lower value of R^2 is an indication of an inappropriate model fitting for selected variables.[26] de Oliveira *et al.*[27] reported that an R^2 value more than 0.80 is considered acceptable while drying cashew apple in a spray dryer and Loh *et al.*[28] reported that R^2 values greater than 0.75 are suitable for prediction purposes. The adj-R^2 with higher values is indicative of the removal of non-significant terms from the model equations. The coefficient of variation (CV) of yield was 5.76% but the CV value of moisture content was 3.12%, indicating that the experiments were more precise and reliable for estimation of moisture content. The lack-of-fit measured the model's fitness, and the results of the *P*-value were showing a significant effect on both moisture content and yield suggesting the accurate prediction of response variables. The obtained predicted value was compared with the experimental value. Considering these values as constraints, the desirability of each response was evaluated and finally, these values were combined to calculate overall desirability by using eqn (2), and the maximum *D* value was selected as the optimum condition for each response with significance influenced by RSM. Thirteen experimental results were obtained with desirability (*D* value greater than 0.80) as shown in Table 1. The results concluded that the optimum drying conditions were obtained at an inlet drying air temperature of 171 °C, feed flow rate of 14.57 ml min^{-1}, maltodextrin concentration of 40.83%, and moisture content of 10.23% giving the actual value of 9.23% under the optimum conditions. The results concluded that the models generated from the study were fruitful in identifying the optimum drying operating conditions while spray drying pineapple fruit juice.

Table 2 Main effects and interactions of the response surface models with their *P* values in spray drying of pineapple juice[a]

Coefficients	Moisture content (Y_1)	*P* value (Y_1)	Yield (Y_2)	*P* value (Y_2)
Model		0.0001[d]		0.0001[d]
β_0	11.45		64.60	
$\beta_{1(X1)}$	−2.65	0.0001[d]	−5	0.0001[d]
$\beta_{2(X2)}$	0.70	0.0001[d]	2.63	0.0001[d]
$\beta_{3(X3)}$	−0.036	0.0001[d]	−0.62	0.0001[d]
$\beta_{12(X1,X2)}$	0.21	0.0410[c]	−1	0.0459[c]
$\beta_{23(X2,X3)}$	0.29	0.0648[b]	−4.25	0.0729[b]
$\beta_{13(X1,X3)}$	−0.25	0.0837[b]	1	0.0315
$\beta_{11(X1^2)}$	+1.18	0.0520[b]	−6.43	0.0907[b]
$\beta_{22(X2^2)}$	0.26	0.0412[c]	−5.17	0.0725[b]
$\beta_{33(X3^2)}$	0.020	0.0947[b]	−6.19	0.0309[c]
Regression(*p*-value)	<0.01		<0.01	
Lack of fit (*p*-value)	0.01		0.04	
Coefficient of variation (%)	3.12		5.76	
Adjusted R^2	0.99		0.98	
Predicted R^2	0.92		0.90	

[a] β_0, constant; β_1, linear coefficient of inlet temperature; β_2, linear coefficient of maltodextrin concentration, β_3, linear coefficient of feed rate, β_{12}, β_{23} & β_{13} interaction, and β_{11}, β_{22} & β_{33}, quadratic coefficient of inlet temperature, maltodextrin concentration and feed rate. [b] Not significant. [c] Significant at <0.05. [d] Significant at <0.0001.

Table 3 Analysis of variance of quadratic models developed from experimental trials

	Sum of square	Degree of freedom	Means of square	*F*-Values	*P* value > *F*
Moisture content					
Regression	190.78	6	42.12	472.17	<0.0001
Residual	0.70	8	0.70		
Total	191.48	14			
Yield					
Regression	600.10	6	132.20	2018.07	<0.0001
Residual	5.38	8	0.98		
Total	605.48	14			

3.3. Moisture content

The powder's moisture content lies in the range of 6.34 ± 1.218% to 14.6 ± 1.070%. The highest moisture content of PJP was found at 130 °C, 30% maltodextrin and 15 ml min^{-1} feed rate and the lowest moisture content of PJP was obtained with a value of 6.34 ± 1.218% at 180 °C (air inlet temperature), 40% maltodextrin and 10 ml min^{-1} feed rate (Table 1). The analysis by RSM of the obtained data in Table 2 revealed that the moisture content correlated with independent variables in the quadratic form with a positive regression coefficient. Eqn (10) describes the relations of moisture content with independent variables including temperature (X_1), feed rate (X_2), and maltodextrin concentration (X_3).

$$Y_1 = 11.45 - 2.65 \times X_1 + 0.70 \times X_2 - 0.036 \times X_3 + 0.21 \times X_1 \times X_2 + 0.26 \times X_2^{\,2} \tag{10}$$

Fig. 4a and b show the plot after response surface analysis showing the effect of temperature, feed rate and maltodextrin concentration on the moisture content. Temperature showed a negative linear effect ($p < 0.0001$) and a positive quadratic effect ($p < 0.10$) on the moisture content as shown in Table 2. The feed rate showed a positive linear effect on the response ($p < 0.0001$). Higher feed rates resulted in having a positive effect on moisture content, as clearly visible from the response surfaces

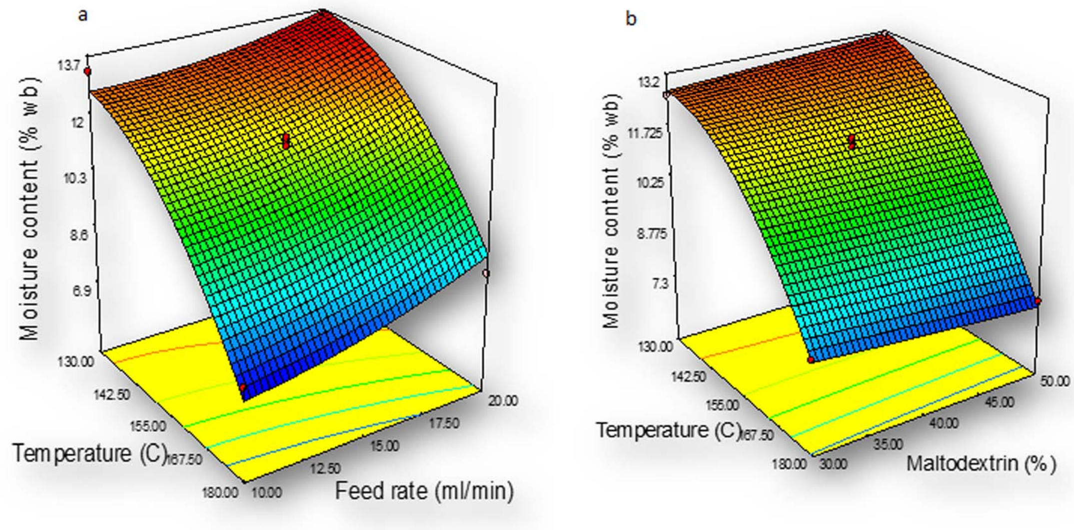

Fig. 4 (a) & (b) Effect of air inlet temperature, feed rate and maltodextrin on moisture.

in Fig. 4b and also in Table 2. This phenomenon is described by the fact that at a higher feed rate the feed extract received a lower time of contact with the drying chamber which caused slow evaporation and resulted in higher moisture content in the powder.[29] The moisture content increased when the feed rate increased, which revealed that a higher feed rate resulted in obtaining a higher moisture content. According to Zimik *et al.*, (2014) increasing the feed rate reduced the water availability in the dryer and therefore increased the moisture in PJP,[30] and similar results were observed in our study. At a higher temperature, the moisture content also decreased when the feed rate is the lowest. As the temperature increases the moisture content decreases, which may be due to the temperature effect on the powder obtained. Maltodextrin showed a negative linear effect ($p < 0.0001$) and a positive quadratic effect ($p < 0.10$) on moisture content (Table 2). The incorporation of maltodextrin into the juice sample could increase the solid content, thereby reducing the free water availability for evaporation. Similar results were observed on spray drying of tomato juice.[30] The interaction effect of temperature (X_1) and feed rate (X_2) showed a significant effect ($p < 0.05$) on moisture constant and feed rate showed a positive quadratic effect ($p < 0.05$) as shown in Table 2. Earlier studies reported that the elevation of temperature increased the rate of drying, and finally the moisture content was lowered in the final product.[18,19,31] These results are consistent with other findings.[32,33]

3.4. Yield of powder

The performance of the drying process is determined by the powder yield. In our study, the yield of PJP ranged from 45.67 ± 2.150 to 46.90 to 68.23 ± 2.270 (Table 1). The highest yield of PJP was obtained at 180 °C air inlet temperature, 40% maltodextrin and 15 ml min^{-1} feed rate and the lowest yield% was obtained at 180 °C air inlet temperature, 40% maltodextrin and 20 ml min^{-1} feed rate (Table 1). Fig. 5a and b show that the factors including inlet air temperature, feed rate, and maltodextrin concentration had influence on powder yield. The coefficients of regression from eqn (11) justify that temperature, feed rate, and maltodextrin concentration showed a linear negative, positive and positive effect on powder yield giving significant results ($p < 0.0001$). Similarly, the interaction effect of temperature (X_1) and maltodextrin (X_3) had significant ($p < 0.05$) influence on the yield (Table 2). Maltodextrin showed a significant quadratic effect ($p < 0.05$) on the yield of powder (Table 2). The yield of powder linearly increases with increasing the maltodextrin concentration to a certain extent. The addition of maltodextrin encapsulates the sugar and organic acid which are present in pineapple, thereby increasing the flow properties of powder inside the drying chamber while reducing the stickiness. The earlier results found maltodextrin concentration to be in the range of 65–75% w.b while drying concentrated orange juice with the incorporation of protein in a spray dryer,[34] and 53.0–63.3% w.b using vacuum spray drying, and in our study, a higher yield percentage was achieved. In our study, the obtained powder yield was higher by 20–30% than the previously reported yield,[9] and another report claimed 40–85% w.b of yield in spray drying using maltodextrin as the carrier agent.[6] The other obtained range of yield was 31.2–65.6% w.b in the case of skim milk.[35] The higher content of organic acid and sugar resulted in stickiness with poor powder recovery.[36] Many research studies were carried out to study the drying behavior of sticky products including blackberry and sour cherry juices and reported a powder yield of more than 50%.[37,38] In correlation with the previously reported studies,[39] the temperature had a negative linear effect on yield.

$$Y_2 = +64.60 - 5.00 \times X_1 + 2.63 \times X_2 + 0.62 \times X_3 + 1.00 \times X_1 \times X_3 - 6.17 \times X_3^2 \quad (11)$$

3.5. Optimization of spray drying conditions

The spray drying conditions would be optimum if the moisture content of pineapple powder is minimum with maximum powder yield. The response values were obtained under the mentioned operating conditions and converted to desirability functions. The ranges of desirability values were 0 with the minimum and 1 with the maximum values. The values of the desirability function were maximum under optimum conditions of operation.[40] The optimal spray drying conditions with maltodextrin as the binding material were an inlet air temperature of 171 °C, feed rate of 14.57 ml min^{-1}, and maltodextrin content of 40.83%.

3.6. Verification of the predicted independent variables

The model equation was used to generate the optimum response values and was verified using the optimum drying conditions. The optimum conditions obtained after the optimization of experimental data by running experimental sets (Table 1) were an inlet air temperature of 171 °C, feed rate of 14.57 ml min^{-1}, and maltodextrin content of 40.83%. Under the optimum conditions, spray drying was performed and the obtained response variables under predicted conditions were 5.27 ± 0.12%, and 69.23 ± 1.230% of moisture content and yield, respectively. Under actual conditions the response variable obtained was 5.27 ± 0.12% and 68.20 ± 0.22% moisture and yield respectively (Table 4).

3.7. Moisture sorption isotherms

Fig. 6 presents the adsorption isotherm characteristics of the PJP for (a) GAB, (b) BET, (c) Oswin & (d) Carrie models at 20 °C, 30 °C, 40 °C & 50 °C. As per the data from adsorption, the product will retain the microbial stability at a_w less than 0.6.[41] From the BET equations, the monolayer moisture content for adsorption is recorded to be 4.3 g per 100 g d.b and 7.6 g per 100 g for the desorption isotherm. The lower value of monolayer moisture content shows the higher stability of powder. The sorption isotherm characteristics featuring type III behaviour are normally obtained for sugar-rich products as per the characterization by Brunauer *et al.*[42] A similar isotherm behavior was seen when making syrup powder by the atomization process[43] of strawberry powder[44] and orange juice powder,[45] using maltodextrin as a carrier agent. Ghorab *et al.*,[46] reported the isotherm characteristics of amorphous materials including maltodextrin and showed the type II or type III isotherm characteristics. The adsorption isotherm behaviour is characterized by a linear increase up to 0.5 a_w, and after that point, an exponential plot is obtained with an increment of moisture content level as shown in Fig. 6. When a product is in the exponential phase, it needs to be handled carefully because it is susceptible to humidification and flavour loss when the RH is above 50%. Hence, to restrict the gain of moisture it is required to use impermeable packaging or low water permeable packaging materials. Additionally, the product has featured the presence of a bioactive compound like vitamin C (around 47.34 mg per 100 g) and other phenolic compounds which accelerate the oxidation process. Hence, it is strongly recommended to use good packaging materials with lower permeability to light. Some of the listed packaging materials including PET with aluminium foil and LDPE are considered ideal for packaging of processed products.[47]

3.7.1. Moisture sorption isotherm modelling. The coefficient of determination (R^2), standard error of estimate (SEE), root mean square error (RMSE%), and chi-square (χ^2) including the model parameters were fitted to the sorption experimental data in the selected model at different temperatures as presented in Table 5. As per the results, Peleg and Iglesias & Chirife models predicted the goodness of parameters of the fitted

Table 4 Experimental verification of predicted independent variables

Responses	Predicted value	Actual values
Moisture content	5.10 ± 0.783	5.27 ± 0.12
Yield (%)	69.23 ± 1.230	68.20 ± 0.22

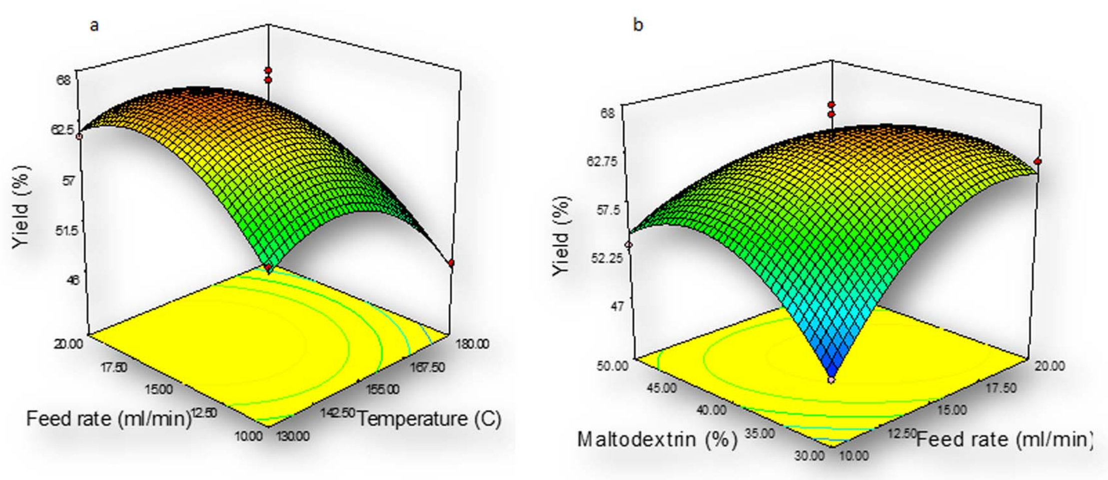

Fig. 5 (a) & (b) Effect of air inlet temperature, feed rate and maltodextrin on yield.

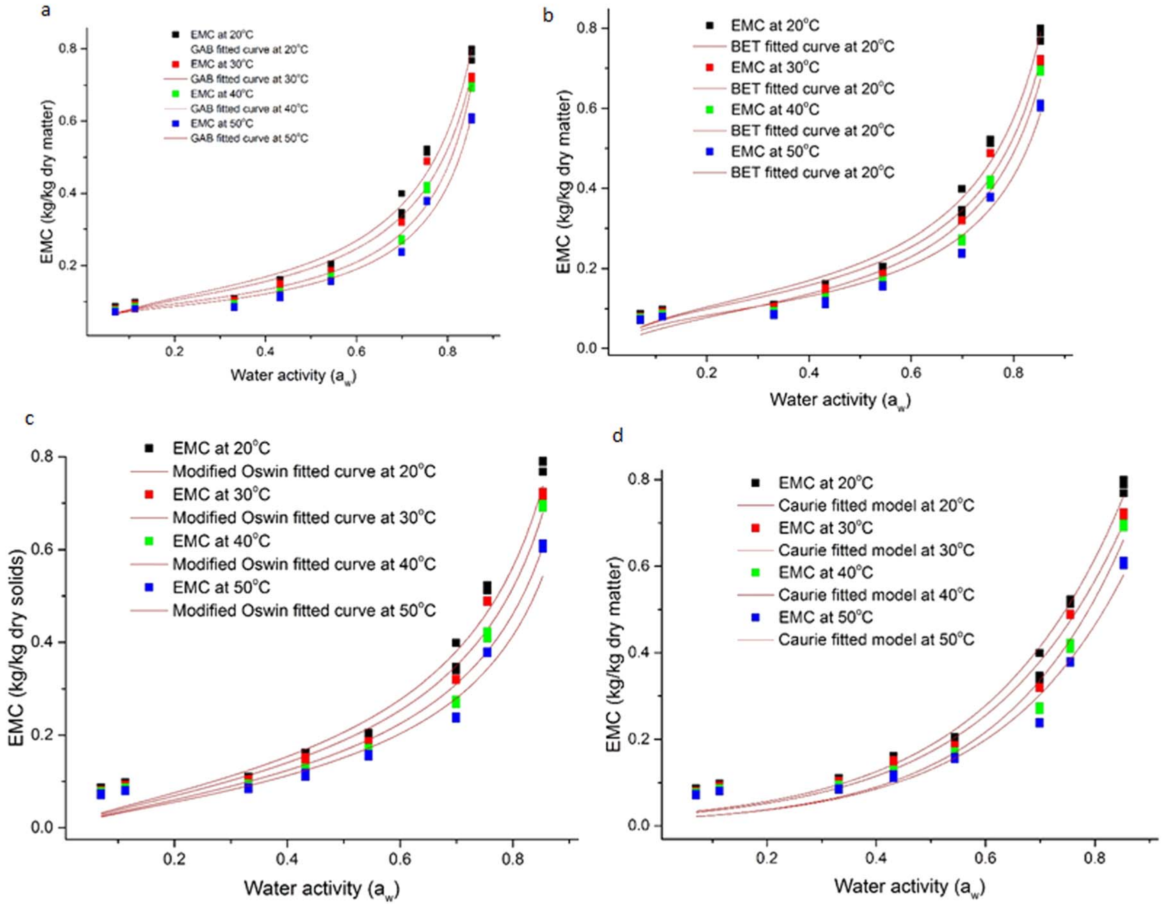

Fig. 6 Sorption isotherm studies of pineapple juice powder using (a) GAB, (b) BET, (c) Oswin & (d) Caurie models.

Table 5 Estimated parameters and performance criteria of the models fitting to the moisture sorption data of pineapple juice powder at different temperatures

Model	T (°C)	Goodness of fit parameters				Model coefficients		
—	—	SSE	% RMSE	χ^2	R_{adj}^2	A	B	C
GAB	20	0.0251	0.03457	12.0×10^{-5}	0.9993	11.02, m_o	15.3717, C	1.012, K
	30	0.0249	0.03444	11.9×10^{-5}	0.9955	10.00, m_o	18.8565, C	1.013, K
	40	0.0107	0.02253	5.07×10^{-5}	0.9982	7.99, m_o	52.7214, C	1.039, k
	50	0.0112	0.02314	5.35×10^{-5}	0.9937	7.27, m_o	72.1722, C	1.034, K
BET	20	0.0262	0.0545	11.9×10^{-3}	0.9793	11.82, m_o	9.5518, C	—
	30	0.0462	0.0445	11.9×10^{-3}	0.9754	10.80, m_o	10.9810, C	—
	40	0.0522	0.0318	10.1×10^{-3}	0.9765	10.18, m_o	6.1679, C	—
	50	0.0388	0.0692	8.54×10^{-3}	0.9740	8.80, m_o	12.2120, C	—
Modified Oswin	20	0.0752	0.0572	3.27×10^{-3}	0.9433	−9.4264	0.4787	0.995, N
	30	0.0309	0.0483	1.47×10^{-3}	0.9696	−9.4066	0.4797	0.768, N
	40	0.0576	0.0363	1.32×10^{-3}	0.9693	−9.4222	0.4789	0.842, N
	50	0.0457	0.0896	1.22×10^{-3}	0.9627	−9.4283	0.4786	0.814, N
Caurie	20	0.05222	0.03827	14.6×10^{-4}	0.9746	−3.6719	3.9877	
	30	0.06138	0.03777	14.3×10^{-3}	0.9705	−3.7570	3.9864	
	40	0.08992	0.04260	18.1×10^{-3}	0.9577	−4.1759	4.4133	
	50	0.06387	0.03924	15.4×10^{-3}	0.9531	−4.1577	4.2376	
Iglesias & Chirife	20	0.01778	0.02843	8.08×10^{-4}	0.9860	0.0683	0.05482	
	30	0.01826	0.02881	8.30×10^{-4}	0.9828	0.0639	0.04915	
	40	0.00885	0.02006	4.02×10^{-4}	0.9906	0.0505	0.04441	
	50	0.00937	0.02064	4.26×10^{-4}	0.9870	0.0497	0.04297	

model (Table 5), since the R^2 was maximum and SEE, RMSE, & χ^2 were minimum, representing the best adsorption & desorption data of the dried powder. Similarly, the other results obtained were also comparable to this work. Similar results were reported by Farahnaky *et al.*[43] for the powder syrup. Other studies reported the higher efficiency of the GAB model in estimating the isotherm characteristics of powdered fruits including for the drying of cupuassu powder in the absence of a carrier agent,[48] processed strawberry powder,[44] processed mango powder,[49] processed yellow mombin powder[49] and processed tamarind powder.[50] Previously few reports were available for the implementation of the Chirife model in the isotherm prediction of powdered fruits. As per the report by Silva *et al.*,[51] the Peleg model predicted the better fitting of evaluated data of umbu-caja powder.

3.8. Accelerated storage studies

The activation energy was estimated using eqn (8) and the calculated value was 19.534 J mol^{-1} K^{-1}. The change in the concentration of ascorbic acid over the storage period at different temperatures was reported. The initial content of ascorbic acid was reported to be 47.34 mg/100 g. Ascorbic acid was assumed to degrade by more than 10% from its initial concentration when estimating the shelf life of the PJP powder. At 40 °C, the shelf life of pineapple powder was obtained as 12.35 days which was calculated using eqn (9), whereas on increasing the storage temperature, the shelf life of the powder decreased. Similarly, the shelf life of pineapple powder at 50, 60 & 70 °C was obtained as 6.11, 2.78, and 0.8 days, respectively.

4. Conclusions

The optimized values after RSM and desirability function strategies were 171 °C (inlet temperature), 14.57 ml min^{-1} (feed flow rate), and 40.83% (maltodextrin concentration to the TSS of pineapple juice), and the obtained experimental data with a higher value of desirability were concluded from the experiment. With these optimum conditions, the obtained powder yield and moisture content of PJP were 68.20 ± 0.22% and 5.27 ± 0.12% (w.b), respectively. The actual results were obtained after the predicted values verified the experiment. The PJP containing 40.83% of maltodextrin to the TSS of pineapple juice presents type III sorption isotherms. The predicted sorption isotherm data revealed that the product should be stored at less than 50% RH where the moisture of powder remains low to achieve the microbiological stability of PJP. The obtained value of monolayer moisture content was 7.27%, signifying greater powder stability by the GAB model. The GAB and Iglesias & Chirife models could predict accurately the adsorption and desorption isotherm characteristics of the studied powdered product. The shelf life of pineapple powder was 48.35 days at 40 °C which was followed by 32.11 d, 23.78 d, and 11.18 d at 50, 60 & 70 °C respectively. Finally it is concluded that with the optimum spray drying conditions, a stable powder can be formed and with proper packaging material the shelf life can be enhanced. This study will help commercialize pineapple powder with better retention of ascorbic acid.

Abbreviations

ANOVA	Analysis of variance
BBD	Box–Behnken design
BET	Brunauer–Emmett–Teller
CV	Coefficient of variation
GAB	Guggenheim–Anderson–de Boer equilibrium
EMC	moisture content
MD	Maltodextrin
PJP	Pineapple juice powder
RSM	Response surface methodology
RMSE	Root mean square error
SEE	Standard error of estimate
TSS	Total soluble solids

Author Contributions

Ramesh Sharma: written original draft including formatting, referencing *etc.*, corrected the manuscript according to reviewer comments. Pinku Chandra Nath: helped Ramesh in writing the manuscript, helped in statistical data analysis. Dibyakanta Seth: edited the manuscript, reviewed the corrected mc, complied to reviewer's comments, guided in mc content.

Conflicts of interest

The authors have no conflicts of interest to declare.

Acknowledgements

The authors acknowledge the help of Tezpur University, Assam, India, for providing laboratory equipment and other facilities to carry out this work.

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