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PROCESS OPTIMIZATION & QUALITY ASSESSMENT OF DATE JAM ENRICHED WITH ALMOND GUM POWDER

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Abstract

Date fruit (*Phoenix dactylifera*) is believed to be a "superfood"and has nutritional properties for human welfare. The production of a value-added product from the date is the prime objective of this study. The jam was formulated using the almond gum powder and RSM-CCRD was applied to optimize the processing conditions for jam. Almond gum powder (3-10 g), date pulp (200-300 g) and water (100-150 mL) were considered as the independent variables. At the same time, TPC, DPPH free radical scavenging activity, firmness and syneresis were decided as responses. The resulting optimized recipe had a formulation consisting of 10 g almond gum, 200 mL water and 289.45 g date pulp which gave the most desirable results having 6.34 mg GAE/g, 82.98% free radical scavenging activity, 16.83% of syneresis and 6.93 N of firmness at a desirability level of 0.89. The physicochemical analysis revealed that jam had TSS of 67.48°Brix, titratable acidity of 1.17%, pH of 4.28 and 33.59% moisture content. The findings of sensory properties concluded that the developed jam using almond gum powder exhibited greater acceptability. Textural properties including hardness, adhesiveness, gumminess and chewiness were 240.62, -11.19, 159.61 and 336.74, respectively indicating the acceptable texture.

***Keywords*:** Date pulp, Jam, Almond gum powder, Response surface methodology, DPPH free radical scavenging activity and Syneresis.

Introduction

Consumption of fruits and vegetable has always been associated with improved health and stronger immunity and is also strongly recommended by WHO. From the last two decades, due to an increased food consciousness and awareness, ample research has been performed concerning the health benefitting attributes of bioactive compounds commonly present in fruits and vegetables (Vayalil, 2012). Date palm (*Phoenix dactylifera*) is believed to be one of the oldest varieties of date palm trees and known to possess certain benefitting attributes apart from food including ornamental, economic and environmental (Barreveld, 1993). Dehydrated form of date fruits is available in the market with increased shelf life but a reduced nutritional value (Abdul-Hamid *et al*., 2015). It has been reported the nutritional composition of the date flesh to be 80% reducing sugars, rich in glucose, fructose, galactose and maltose along with good quality proteins, fats, calcium, potassium and zinc (Assirey, 2015; Khalid *et al*., 2017).

Jam is a gelatinized fruit product prepared by fruit pulp, sugar, acid and pectin and total soluble solids is considered a reliable factor to estimate the final concentration of the jam (Adriana, 2011). However, the quality of jam is influenced by many factors such as fruit quality, high pectin content, relatively low pH and high total soluble solids (TSS) (Ahmed, 2007). According to Pereira *et al*. (2011), the pulp to sugar ratio is an important determinant of jam quality since it has a great impact on the titratable acidity and water activity of jam. Because of their low water activity, they have a long shelf life and are thus used as a method to preserve surplus fruits (Lesschaeve *et al*., 1991; Touati *et al*., 2014). They are classified as intermediate moisture foods (IMF). As per FSSAI, the prepared fruit content in jams for most of the fruits shall be not less than 45%, and the total soluble solids content shall be not less than 65% by weight.

Gels are intermediate between a liquid and a solid. They have an interconnected molecular network consisting of polymeric cross-linked molecules, immersed in a liquid medium. The required stabilized structure in jams and marmalades is provided by hydrocolloids. Pectin is one of the most commonly used hydrocolloid stabilizers and is available based on their methoxy level. However, mechanical stress may cause damage to pectin gels as a result of expulsion of

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colloidal water which is also known as syneresis or weeping of the gel. Incorporating other stabilizers having thickening properties with pectin solves this problem (Kavaya *et al*., 2019). Apart from the existing stabilizers, the use of the new gum exudates with appreciable functional and phytochemical properties has seen remarkable growth in demand in recent times (Vilela and Ravetta, 2005). Almond gum (*Prunus dulcis*) is a natural polymer and utilized commercially, due to its significant properties such as good emulsification and matrix-forming. Previous studies have revealed that almond gum is a pool of minerals like Fe, Mg, Ca, Na and K (Rezaei *et al*., 2016). Almond gum is known to possess certain health-beneficial properties such as anti-cancerous and anti-inflammatory properties (Malsawmtluangi *et al*., 2014).

Hence, the present study aimed to combine the appreciable bioactive properties of date fruit and almond gum powder to produce a value-added product. Almond gum powder was used along with pectin to reduce the rate of syneresis in jam. Response surface methodology (RSM) was utilized to design the processing condition considering the gum concentration, amount of water, and date pulp. The developed jam under optimized condition was evaluated for physicochemical properties, textural, surface morphological and sensory properties.

Materials and Methods

Materials

Fresh date (*Phoenix dactylifera*) fruit was obtained from INA market of New Delhi in packed condition. Almond gum (*Prunus dulcis*) nodule was collected from the local market of Jammu and Kashmir, India whereas pectin (Poly-D-Galacturonic Acid Methyl Ester) and citric acid monohydrate used in the study was of food-grade and procured from Sisco Research Laboratories Pvt Ltd., India. Analytical grade chemicals were used in the experiment which was obtained from Merck-Sigma, India.

Preparation of almond gum powder and characterization

The procured gum was processed as per Bashir *et al*. (2016) for powder production. The gum powder was analyzed for moisture content (AOAC 925.09), water activity according to Bashir *et al.* (2016) using water activity meter and colour properties using Colour flex (Hunter Associates Laboratory Inc., Reston, VA, USA) as per the procedure suggested by Das *et al*. (2020).

Preparation of Date-Almond gum powder jam

The procured fresh dates were cleaned with potable water to remove the extraneous dirt. The cleaned fruits were sliced into pieces and ground into a paste (Fig. 1). The gum concentration, amount of water and date pulp was selected as independent factors which varied from 3-10 g, 100-150 mL and 200-300 g, respectively. The jam was prepared according to the condition of the experimental run. In brief, sugar syrup was prepared, followed by the addition of date pulp. The mixture was allowed to boil with occasional stirring. Then, the pH was checked using Eutech pH 700 as per AOAC 942.15 and adjusted with citric acid to pH 3.2. The mixture was again cooked and pectin solution was added with continuous stirring (Kavaya *et al*., 2019). Different proportion of almond gum powder was added into it and the boiling was continued. The endpoint was confirmed by measuring the TSS using a handheld refractometer (AOAC 967.21). Then, the prepared jam was cooled at 37^0C followed by filling and bottling. The sealed bottles were stored at room temperature for further analysis.

Experimental design for development of Date-Almond gum powder jam

For modelling of jam variables, central composite rotatable design (CCRD) was used to design experiments that include 3 independent variables, 4-level CCRD and 20 tentative runs with six imitates at the center point. A second-order polynomial model was fitted to predict the optimal point of correlation between independent variables, including the amount of water, almond gum concentration and amount of date pulp while responses such as Total phenol content (TPC), DPPH, Syneresis, and Firmness. Eq. 1 presents the relationships between three factors:

$$Y_n = \beta_0 + \beta_1 A + \beta_2 B + \beta_3 AB + \beta_2 A^2 + \beta_2 A^3 \quad (1)$$

Where Y_n is one of the three responses, A and B are the independent variables, β_0 is a constant, β_1 and β_2 are the linear term, β_4 and β_5 are the quadratic-term and β_3 is interaction coefficients.

The obtained responses were Total phenol content (mg GAE/g), DPPH (% free radical scavenging activity), Syneresis (%) and Firmness (N). The actual variable designated to obtain second-order quadratic equation and was used to direct the individual response as a function of independent factors. The total error criteria were used to accomplish a 95% significance limit, and for analyzing the significance level, analysis of variance (ANOVA) was used for each response. The R^2 value was used to analyze the efficiency of the model, whereas the lack of fit test was helpful in the evaluation of accordance of models (Gupta *et al*., 2020a). The desirability function method helped in understanding the optimum levels of independent factors.

Physico-chemical properties of date jam

Physicochemical properties including titratable acidity, pH and TSS of jam were estimated according to AOAC (18^{th} edition) official methods. The prepared jam was analyzed for moisture content, water activity and colour properties similarly as discussed in section 2.2. Syneresis of the developed jam was estimated, according to the method suggested by Kavaya *et al*. (2019).

Textural properties of developed jam

The firmness of jam was conducted according to Banaś *et al*. (2018) using a texture analyzer (TA.HD, Stable Microsystems, UK). Hardness, resilience, adhesiveness, cohesiveness, chewiness, gumminess, springiness was recorded.

Surface micrograph and Sensory evaluation

Date jam prepared using almond gum powder was studied for morphological structure by a scanning electron microscope (JSM-6060 JEOL, Tokyo, Japan) as per the method suggested by Bashir and Haripriya (2016). The sensory evaluation of jam was performed using a hedonic scale of 1 to 9 where appearance, colour, texture, taste, aroma and overall acceptability were considered as key parameters and was performed by semi-trained panel of 20 members (Kavaya *et al*., 2019).

Spectrophotometric analysis of phytochemicals

Methanolic extract (solvent: water, 80:20, v/v) of jam was prepared according to Gupta *et al*. (2020a) for the determination of bioactive properties. The total phenol content of jam was assessed using Folin-Ciocalteu method (Gupta *et al*., 2020b). The DPPH free radical scavenging activity assay was adopted to assess the total antioxidant properties of jam and was determined as per Gupta *et al*. (2020b).

Statistical analysis

In the present study, Design-Expert software version 10.0.7.0 (Statease Inc., Minneapolis, USA) was used to optimize the processing conditions of Date-almond gum jam. IBM SPSS Statistics Version 20.0, Armonk, NY: IBM Corporation package was used for the statistical analysis of data and all the data represented as the mean ± standard deviation.

Result and Discussion

Characterization of almond gum

The procured almond gum powder was characterized and obtained results revealed that gum powder had a moisture content of 10.95% and water activity of 0.42. The colour properties analysed using hunter colour lab such as L*, a*, b* were found to be 80.56, 2.64 and 13.74, respectively. The similar observation was made by Bashir and Haripriya (2016) for almond gum powder.

Optimization of date jam

In the present study, RSM was used to optimize the processing condition where CCRD design based on the experimental run as per Table 1 and it was found optimum for significant regression with non-significant lack of fit. Also, the values of coefficients (R^2) for the different responses were obtained as 0.88, 0.88, 0.86 and 0.89 for TPC, DPPH, syneresis and firmness, respectively (Table 2). Table 3 gives the details of the desired goals for individual independent variables concerning each response.

Fig. 2 shows that 3D surface graphs for each response were established as a function of two independent factors [Fig. 2 (A-D)]. At the optimized parameters, the predicted values for TPC, DPPH, syneresis and firmness was found to be 6.34 mg GAE g^{-1}, 82.98%, 16.83% and 6.93 N, respectively, at the desirability level of 0.89. These predicted values were almost close to the experimental value such as 6.49 mg GAE g^{-1}, 81.62%, 17.25% and 7.09 N, respectively. These closed values represent the validation of the model as no significant difference was found in predicted values and experimental value. Furthermore, insignificant lack of fit and significant F-value confirmed the model's significance for optimizing processing conditions for jam.

Effect of process variables

1. Effect of process parameters on TPC

Independent factors such as almond gum and amount of water have shown a significant effect on TPC (Table 2). TPC of the developed jam was found to be 6.49 mg GAE/g under optimized condition. The regression coefficient, coefficient of variance (C.O.V) and adjusted R^2 for SI were recorded as 0.88, 18.36% and 0.77, respectively. These values demonstrate that model used for the optimization of TPC during jam preparation was best-fit. The effect of independent variables and quadratic terms (A^2 and C^2) are significant ($p<0.05$), while interaction among the variables was found insignificant (Fig. 2A). It was observed that as the gum concentration was increased, TPC was also increased. It has been reported that almond gum possesses an appreciable amount of TPC (Bashir *et al*., 2018). With an increase in the pulp concentration, the level of TPC was found to increase to a certain extent but was found to decrease after that. Fig. 2Ab showed a similar observation. Interaction of water level and date pulp was observed to decrease after an increase to certain extent.

2. Effect of process parameters on DPPH free radical scavenging activity

Independent factors such as date pulp and amount of water have shown a significant effect on DPPH free radical scavenging activity (Table 2). The antioxidant activity of developed jam samples was noticed to be 81.62%. The regression coefficient, coefficient of variance (C.O.V) and adjusted R^2 for the activity were 0.88, 4.51% and 0.78, respectively and they indicated that the model used for the optimization of antioxidant activity during jam preparation was best-fit. The effect of independent variables such as A and C while quadratic terms are found to be significant ($p<0.05$) and insignificant, respectively. The interaction among the variables was found significant (Fig. 2B). The dramatic effect

was observed on DPPH free radical scavenging activity. Interaction of almond gum and date pulp was found to be significant and increased proportionally, while DPPH and water was negatively correlated. On the contrary, the interaction of water level and almond gum (Fig. 2Bc) was slightly different where the proportional increase was noticed in DPPH with a rise in the concentration of variable.

3. Effect of process parameters on Syneresis

Syneresis of date jam was found to be 17.25% under optimized condition. The regression coefficient, C.O.V and adjusted R^2 for syneresis was found to be 0.86, 4.15% and 0.74 presented that model used for the process optimization of jam is the best-fitted model. The interactions (AB and AC) and quadratic models such as A^2 and C^2 had a negative effect on the syneresis of jam. Syneresis of jam increased with an increase in the amount of water; however, the decreasing pattern was seen with an increase in gum concentration (Fig. 2C). This change might be due to the rise in the absorption efficiency resulting in increased swelling and hydration capacity of granules. Also, increased concentration of gum has inhibited syneresis of starch due to the high-water holding capacity of gum (Kavaya *et al*., 2019).

4. Effect of process parameters on firmness

The firmness of developed jam using almond gum powder was observed to be 7.09 N. The regression coefficient, C.O.V and adjusted R^2 for firmness was found to be 0.89, 13.05% and 0.79 presented that model used for the optimization is the best-fitted model. The firmness of jam was negatively influenced by the interaction between A and C and A^2 and B^2. It was observed that, the firmness of date jam dropped with a substantial elevation in the concentration of almond gum and also with a rise in water level (Fig. 2D). Change in firmness might be due to the rise in the absorption efficiency due to increased concentration of gum and pulp produced the jam of low firmness (Gao *et al*., 2011; Guo *et al*., 2017).

Physicochemical analysis of date jam

The moisture content of date jam incorporated almond gum was 33.59% (Table 4). A rise in moisture content is could be due to the incorporation of gum in the jam, which has an impact on solute concentration resulting in absorption of moisture from granules. The water activity of jam was 0.86. Titratable acidity was analysed to affirm the changes happened during production and storage in the physicochemical properties (Kalra and Tandon, 1985). In the developed jam, acid content was 1.17% while pH was 4.28. It has been reported that, acidity of fruit products often associated with an increased shelf stability (Bafna and Manimehalai, 2013). TSS of jam was observed to be 67.48°Brix. Colour of the jam prepared under optimized condition with AG was evaluated where L*, a* and b* values were 20.83, 4.47 and 10.17, respectively.

Textural properties of jam

Table 4 presents the textural properties of jam where hardness, adhesiveness, chewiness, cohesiveness, gumminess and resilience were detailed. The strength of starch gel mainly depends on the influential factors such as the composition of gum powder and the interaction of water with molecules (Mir *et al*., 2014; Bashir *et al*., 2018). Cohesiveness and adhesiveness are the important factors which result due to the gel formation. The interaction with the protein, lipid and non-starch polysaccharide, have a positive or negative impact on the gel texture of the jam, resulting in an increase or decrease in hardness (Yu *et al*., 2012). Texture and consistency predominantly define the quality of jam and affects its sensory attributes.

Sensory analysis and Surface micrograph of jam

Fig. 3a illustrates the image of jam prepared from gum and date pulp. It was observed that the appearance and aroma of jam scored highest (Table 4). Bafna and Manimehalai (2013) reported similar observation for the jam prepared from Kokum fruit using optimized condition. Surface micrograms of jam samples were analyzed using SEM at 300x, which showed the compact and highly interspersed structure of jam due to the formation of associative interaction between the AG and pectin, as shown in Fig. 3b.

Conclusion

The process had been developed for the preparation of jam from a highly nutritious fruit (*Phoenix dactylifera*) using almond gum powder. With the help of RSM, date jam was successfully prepared using almond gum along with pectin. A number of physicochemical, textural, sensory and surface parameters were studied. Independent factors such as almond gum and amount of water showed a significant effect on TPC. With an increase in pulp concentration, the level of TPC was found to increase to a certain extent but was found to decrease after that. Similarly, independent factors such as date pulp and the amount of water have shown a profound effect on DPPH. Syneresis of jam increased with an increase in the amount of water. However, the decreasing pattern was seen with a rise in the level of gum. The firmness of jam dropped with an elevation in concentration of gum and also with an increase in water level. Textural properties of jam such as hardness, adhesiveness, chewiness, cohesiveness, gumminess and resilience were evaluated. Sensory analysis revealed that the appearance and aroma of jam scored highest. Surface micrograms of jam samples showed the compact and highly interspersed structure of jam due to the formation of associative interaction between the AG and pectin. Based on the outcome of present study, it can be suggested that the almond gum powder incorporated date jam was successfully developed with good overall acceptability and other desirable characteristics.

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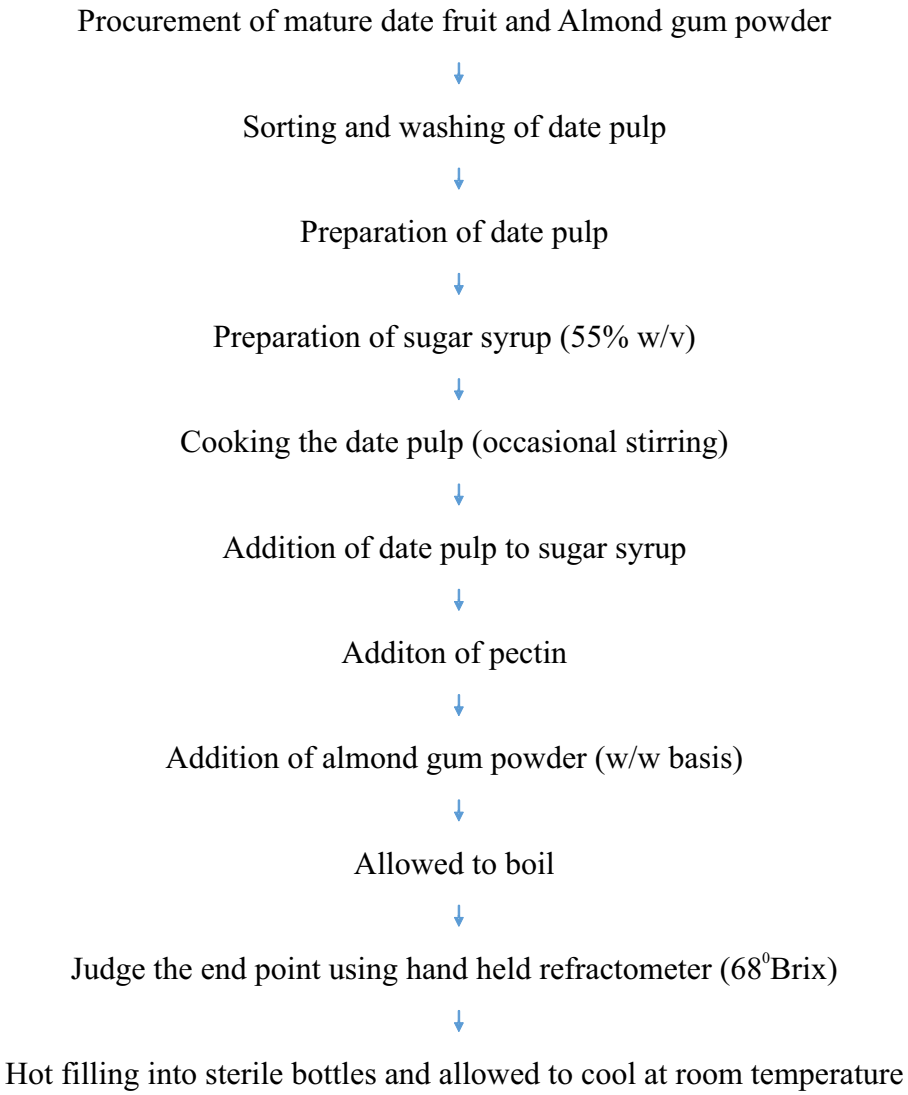

Fig. 1: Flowchart for Date-Almond gum powder jam preparation

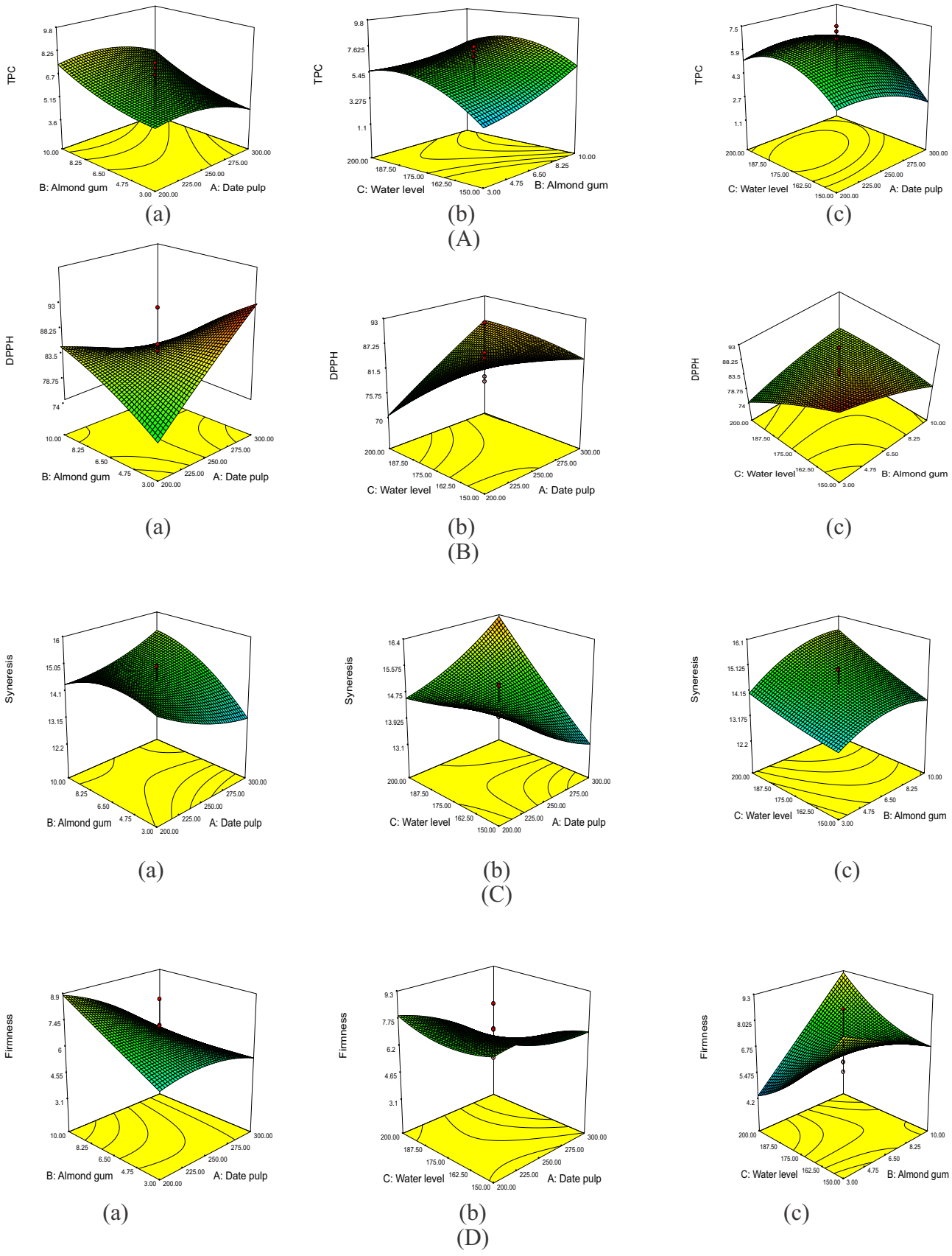

Fig. 2: 3D plots for process optimization of date jam using almond gum powder

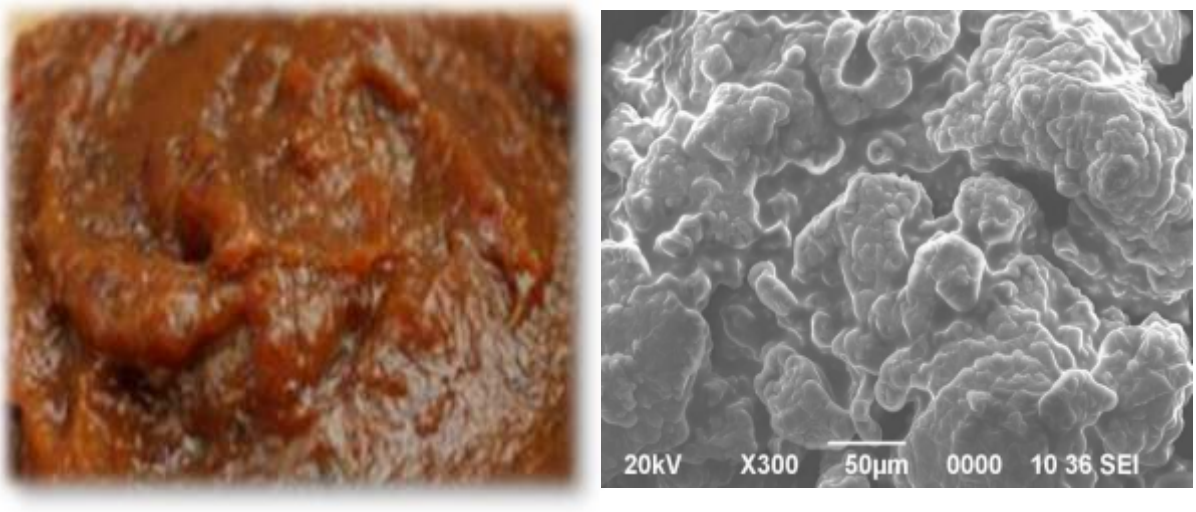

Fig. 3: (a) Date-almond gum powder jam produced under optimized conditions (b) Surface micrograph of jam

Table 1: Experimental run for process optimization for date jam using almond gum powder

Runs	Date pulp (g)	Gum concentration (g)	Amount of water (mL)	TPC (mg GAE/g)	DPPH (% free radical scavenging activity)	Syneresis (%)	Firmness (N)
1	200.00	3.00	150.00	3.43	88.60	15.04	7.51
2	300.00	3.00	150.00	1.08	92.90	12.01	9.42
3	200.00	10.00	150.00	5.39	88.30	14.00	7.53
4	300.00	10.00	150.00	4.23	71.95	13.12	5.04
5	200.00	3.00	200.00	5.05	58.30	14.00	5.04
6	300.00	3.00	200.00	5.19	89.10	14.73	3.21
7	200.00	10.00	200.00	5.99	82.10	13.71	10.90
8	300.00	10.00	200.00	5.63	82.80	17.00	6.78
9	165.91	6.50	175.00	5.49	77.30	16.00	7.32
10	334.09	6.50	175.00	3.67	88.08	15.00	3.18
11	250.00	0.61	175.00	5.23	84.60	12.29	5.25
12	250.00	12.39	175.00	9.71	77.30	15.00	7.98
13	250.00	6.50	132.96	1.12	88.60	14.11	9.24
14	250.00	6.50	217.04	4.07	74.10	16.01	7.04
15	250.00	6.50	175.00	6.03	79.80	14.00	6.04
16	250.00	6.50	175.00	6.67	83.08	15.00	6.04
17	250.00	6.50	175.00	5.51	92.07	14.00	8.61
18	250.00	6.50	175.00	7.14	78.65	14.21	5.55
19	250.00	6.50	175.00	7.49	85.30	15.00	7.21
20	250.00	6.50	175.00	4.21	84.07	14.96	7.16

Table 2: Analysis of variance (ANOVA) as a function of coded regression showing the variables as surface quadratic and interaction terms on each response of the jam process optimization

Source	Degree of freedom (df)	TPC (mg GAE/g)		DPPH (% free radical scavenging activity)		Syneresis (%)		Firmness (N)	
		Sum of square	p>f	Sum of square	p>f	Sum of square	p>f	Sum of square	p>f
Model	9	66.57	0.0013	1103.08	0.0010	23.54	0.0024	65.38	0.0009
A-Date pulp	1	3.38	0.0790	103.41	0.0209	0.18	0.4955	13.33	0.0021
B-Gum concentration	1	14.40	0.0024	18.81	0.2701	3.20	0.0140	6.83	0.0147
C-Amount of water	1	11.79	0.0044	212.22	0.0029	5.25	0.0034	3.87	0.0511
AB	1	0.06	0.8004	321.95	0.0007	2.77	0.0198	5.59	0.0238
AC	1	1.35	0.2440	237.08	0.0020	7.86	0.0009	3.60	0.0582
BC	1	1.74	0.1907	187.70	0.0042	0.46	0.2876	23.77	0.0003
A^2	1	5.69	0.0295	1.82	0.7242	0.75	0.1801	3.33	0.0670
B^2	1	2.23	0.1428	13.57	0.3447	2.63	0.0223	0.00	0.9933
C^2	1	25.49	0.0003	9.90	0.4167	0.08	0.6561	4.22	0.0433
Lack of Fit	5	1.59	0.938	24.21	0.942	2.32	0.267	1.60	0.920
R^2	-	-	0.88	-	0.88	-	0.86	-	0.89
Adj R^2	-	-	0.77	-	0.78	-	0.74	-	0.79
Pred R^2	-	-	0.69	-	0.70	-	0.28	-	0.69
Mean	-	-	5.11	-	82.35	-	14.45	-	6.80
CV%	-	-	18.36	-	4.51	-	4.15	-	13.05

Table 3: Process parameters and responses attained for jam process optimization using date pulp and almond gum powder

Constraints	Goal	Lower limit	Upper Limit	Predicted value	Actual value
Date pulp (g)	Maximize	200	300	289.45	-
Gum concentration (g)	is in range	3	10	10	-
Amount of water (mL)	is in range	150	200	200	-
TPC (mg GAE/g)	Maximize	1.08	9.71	6.34	6.49
DPPH (free radical scavenging activity)	Maximize	58.3	92.9	82.98	81.62
Syneresis (%)	Minimize	12.01	17	16.83	17.25
Firmness (N)	Minimize	3.18	10.9	6.93	7.09

Table 4: Physicochemical, Sensorial and textural properties of developed jam

Parameter	Date-almond gum powder jam	Sensory parameters	Date-almond gum powder jam	Parameter	Date-almond gum powder jam
Moisture content (%)	_	Appearance	7.95±0.85	Hardness	240.62±31.19
Water activity ($a_{w)}$	0.86±0.00	Aroma	7.90±0.58	Adhesiveness	-11.19±1.02
TSS (°Brix)	67.48±0.86	Taste	7.45±0.35	Springiness	4.93±0.21
pH	4.28±0.78	Texture	7.80±0.34	Cohesiveness	0.58±0.02
Titratable acidity (% citric acid)	1.17±0.28	Color	7.60±0.38	Gumminess	159.61±9.46
L*	20.83±0.31	Overall acceptability	7.34±0.36	Chewiness	336.74±11.41
a*	4.47±0.04	-	-	Resilience	0.25±0.01
b*	10.17±0.13	-	-	-	-