Full Length Research Paper

Optimization of process variables to develop honey based extruded product

Praneeth Juvvi¹, Savita Sharma² and Vikas Nanda¹*

¹Department of Food Engineering and Technology, Sant Longowal Institute of Engineering and Technology, Longwal, Distt Sangrur-148106 Punjab, India.

²Department of Food Science and Technology, Punjab Agricultural University, Ludhiana, Punjab, India.

Accepted 9 March, 2012

In the present work, the processing variables were optimized to study their effect on functional properties of honey based ready-to-eat expanded snack food. The blends of rice flour, wheat flour and honey were extruded in a twin-screw extruder. Experimental design with temperature (115 to 135°C), screw speed (270 to 350 rpm) and feed proportion (5 to 15%) as independent variables which produced 20 different combinations, were studied using response surface methodology to investigate the effect of these variables on product responses (lateral expansion, bulk density, color, Water absorption and solubility index characteristics). Multiple regression equations were obtained to describe the effects of each variable on product responses. Results showed that increasing the barrel temperature resulted in extrudate with higher expansion, water absorption index, a* value and lower bulk density and L* and b* values. Increasing screw speed resulted in higher expansion, water solubility index, L value and lower bulk density whereas, increasing honey proportion of feed composition resulted in higher a* value, bulk density, water solubility index and lower expansion, water absorption index and L*value. The graphical optimization studies resulted in temperature (119 to 122°C), screw speed (301 to 321 rpm) and feed composition (9.68 to 10.35%) as optimum variables to produce acceptable honey based extrudates.

Key words: Twin screw extrusion, honey, response surface methodology, rice flour, wheat flour.

INTRODUCTION

Honey is considered a potential complete food, regarding nutritional standards, being a natural product, rich in simple, easy assimilable sugars (fructose, glucose), enzymes (invertase, glucose oxidase, catalase. phosphatase), amino and organic acids (proline, gluconic acid, acetic acid), vitamins (ascorbic acid niacin, riboflavin), volatile oils, phenolic acids and flavonoids, minerals and carotenoid like substances (Singh, and Bath 1996; Silici, 2010; Kahraman et al., 2010; Sudhanshu et al., 2010). Honey is one of the most complex mixtures of produced in carbohydrates nature. The maior carbohydrates in honey are glucose and fructose, which accounts for 65 to 75% of total soluble solids in honey and 85 to 95% of honey carbohydrates. The remaining

carbohydrates are mixture of at least 11 diasaccharides, 11 trisaccharides and several higher oligosaccharides (Cavia et al., 2002; Persano-Oddo and Piro, 2004; Kaskoniene et al., 2010). The various other chemical components of honey include: proteins that include a number of enzymes (diastase, invertase, glucose oxidase, catalase and others), eighteen free amino acids, of which the most abundant is proline and minerals potassium, calcium, sodium, phosphorous, magnesium, and iron (Hak-Gil et al., 1988; Rodriguez-Otero et al., 1992; Gomes et al., 2010; Ouchemoukh et al., 2007). Honey is largely used on a small scale as well as at an industrial level in beverages. baked products. confectionary, candy, marmalades, jams, spreads, etc, but there is no extrudate available, that contain honey as minor component, as it is well established that cereal grains are generally used as major raw material for development of extruded snack foods (Faubion and Hoseney, 1982; Kadan et al., 2003; Ding et al., 2006;

^{*}Corresponding author. E-mail: vik164@yahoo.co.in. Tel: +91-8815980054.

Altan et al., 2008). Extrusion cooking which is a continuous cooking, mixing and forming process has been used increasingly in the production of food ingredients and food such as breakfast cereals, baby foods, flat breads, snacks, meat and cheese analogues and modified starches etc (Anderson et al., 1969; Meuser and van Lengerich, 1992). Quality of extruded product varies depending on the extruder type, screw configuration, feed rate and die profile. Wheat flour, rice flour, maize grits, barley flour and their combinations along with byproducts of different industries like carrot and grape pomace, have been widely used in the extrusion industry and the effects of process variables on the physical and functional properties of extrudates have been studied (Yagci and Gogus, 2008; Altan et al., 2008; Ding et al., 2006; Ali et al., 1996; Tomschik et al., 1996; Aylin et al., 2008; Carvalho and Mitchell, 2001). Similarly, effect of sucrose and fructose incorporation has also been studied (Carvalho and Mitchell, 2001; Kalichevsky and Blanshard, 1993). Utilization of different cereals in combination with different non cereal ingredients demands empirical modifications in processing conditions to yield optimum extruded product with lower cost. Since no scientific studies are available on extrusion processing of cereals by addition of honey. So, the objective of this study was the incorporation of honey into ready-to-eat snacks based on rice and wheat flour, and to optimize the process variables (temperature, screw speed and feed proportion) to produce acceptable honey based extruded snack food by using a twin-screw extruder.

MATERIALS AND METHODS

Ingredients for snack food consisted of rice flour (prepared from the rice brokens (PR 14) procured from rice mill, Longowal, District.Sangrur, Punjab, India), wheat flour (prepared from the wheat (WH 17) procured from local market of Sangrur, Punjab, India), commercial honey (purchased from local market Sangrur, Punjab, India).

Sample preparation

Ingredient formulations for extrusion products are given in Table 1a and b. In the blend preparation rice flour (60%), wheat flour (40%) mixed with commercial honey at levels of 1.6, 5, 10, 15 and 18.4% were used. The moisture (18%) was adjusted by sprinkling the distilled water in all the dry ingredients. All the ingredients were weighed and then mixed in the Food Processor (Make: Maharaja Whiteline, Asiatic Engineers Pvt. Ltd., New Delhi 600W) with mixer attachment for 20 min. This mixture was then passed through a 2 mm sieve to reduce the lumps formed due to addition of moisture. After mixing, samples were stored in polyethylene bags at room temperature for 24 h (Stojceska et al., 2008).

Experimental design

Response surface methodology (RSM) was adopted in the design of experimental combinations (Altan et al., 2008; Ding et al., 2005; Montogomery, 2001). The main advantage of RSM is to reduce number of experimental runs needed to provide sufficient information for statistically acceptable results. A three variable (five levels of each variable) central composite rotatable experimental design was employed (Montogomery, 2001; Yagci and Gogus, 2008). The parameters (temperature and screw speed) and their levels were chosen based on the literature (Cheng and Friis, 2010). The ingredients used for the honey based ready-to-eat preparation were: rice flour, wheat flour and honey. The independent variables included the blend composition honey (5 to 15 %), temperature (115 to 135 rpm) and screw speed (270 to 350°C). Variables were response expansion index, bulk density, water absorption index, water solubility index and colour. The five levels of the process variables were coded as -1.682, -1, 0, +1, +1.682 (Montgomery, 2001) and design in coded (x) form and at the actual levels a X_1 , X_2 and X_3 are given Table 1a and b.

Extrudates preparation

Extrusion of samples performed using a co-rotating twin-screw extruder (Basic Technology Pvt. Ltd. Kolkata, India). The main drive is provided with 7.5 HP motor (400 V, 3 ph, 50 cycles). The output shaft of worm reduction gear was provided with a torque limiter coupling. The screw configuration that was a standard design for processing cereals and flour-based products was used. The barrel of the extruder received the feed from a co-rotating variable speed feeder. The barrel was provided with two electric band heaters and two water cooling jackets. A temperature sensor was fitted on the front die plate which was connected to temperature control placed on the panel board. The die plate of the die fixed by a screw nut was tightened by a special wrench provided. The automatic cutting knife is fixed on rotating shaft. The twin screw extruder was kept on for 30 min to stabilize the set temperatures and samples were then poured in to feed hopper and the feed rate was adjusted to 4 kg/h for easy and non-choking operation. The die diameter was selected at 4 mm as recommended by the manufacturer for such product and recommended by Stojceska et al. (2008). The product was collected at the die end and kept at 60 ± 0.5°C in an incubator (Orbital Incubator Macro Scientific works, New Delhi) at 60°C for 12 h duration to remove extra moisture from the product. The samples were packed in polythene bags for further analysis.

Evaluation for lateral expansion of extrudates

The ratio diameter of extrudates and the diameter of die were used to express the expansion of extrudates (Fan, 1996; Ainsworth et al., 2006). Six lengths of extrudate (approximately 120 mm) was selected at random during collection of each of the extruded samples, and allowed to cool to room temperature. The diameter of the extrudates was then measured, at 10 different positions along the length of each of the six samples, using a Vernier caliper. Lateral expansion (LE, %) was then calculated using the mean of the measured diameters:

Evaluation for bulk density of extrudates

Bulk density (BD, g/cm 3) was calculated according to the method of Stojceska et al. (2008):

$$BD = \frac{4 m}{\pi d^2 L}$$

where m is mass (g) of a length L (cm) of extrudate with diameter d (cm).

Evaluation for water absorption index (WAI) and water solubility index (WSI) of extrudates

WAI and WSI were determined according to the method developed for cereals (Stojceska et al., 2008). The ground extrudate was suspended in water at room temperature for 30 min, gently stirred during this period, and then centrifuged at 3000 g for 15 min. The supernatant was decanted into an evaporating dish of known weight. The WAI was the weight of gel obtained after removal of the supernatant per unit weight of original dry solids. The WSI was the weight of dry solids in the supernatant expressed as a percentage of the original weight of sample.

$$WAI (g/g) = \frac{Weight gain by gel}{Dry weight of extrudate}$$

$$WSI(\%) = \frac{Weight of dry solid in supernatent}{Dry weight of extrudate} \times 100$$

Evaluation for colour (L*, a*, b*) of extrudates

The colour of the extrudates were measured by using Colour Spectrophotometer (Hunter's colour lab, GretagMacbethTM, model – colour i.5, CH-8105 Regensdort, Switzerland) (Ranganna, 1997).

Statistical analysis of responses

The responses (lateral expansion, bulk density and colour) for different experimental combinations were related to the coded variables (x_i , i = 1, 2 and 3) by a second degree polynomial equation:

$$Y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{33} x_3^2 + \beta_{12} x_1 . x_2 + \beta_{13} x_1 . x_3 + \beta_{23} x_2 . x_3 + \varepsilon$$
(1)

Where x_1 , x_2 and x_3 are the coded values of temperature, screw speed and feed proportion, respectively. The coefficients of the polynomial were represented by β_0 (constant), β_1 , β_2 , β_3 (linear effects); β_{12} , β_{13} , β_{23} (interaction effects); β_{11} , β_{22} , β_{33} (quadratic effects); and ε (random errors). Data were modeled by multiple regression analysis and the statistical significance of the terms was examined by analysis of variance for each response. The statistical analysis of the data of three dimensional plotting was performed using Design expert software (Statease 6.0). The adequacy of the regression model was checked by R², Adequate Precision and Fischer's F-test (Montgomery, 2001). The regression coefficients were then used to make statistical calculation to generate three dimensional plots for the regression model.

RESULTS AND DISCUSSION

Variation of responses (lateral expansion, bulk density, colour, water absorption index and water solubility index) of extrudates with independent variables (temperature, screw speed and (rice flour + wheat flour) and honey

proportion) is shown in Table 1a and b. Complete second order model (Equation 1) was tested for its adequacy to decide the variation of responses with independent variables. To aid visualization of variation in responses with respect to processing variables series of three dimensional response surface (Figures 1 to 7) were drawn using design expert software (Statease 6.0).

Bulk density and lateral expansion

Lateral expansion of extrudates ranged from 110.5 to 184.7% with an average value of 110.76%. The maximum expansion at coded point (0, 0, -1.682) was about 1.7 times more than the minimum expansion at the coded point of -1, 1, 1 (Table 1a). The coefficients of the model and other statistics were given in Table 2a. The Model F value of 133.63 implies that model was significant (P < 0.05). R² and Adjusted R² values of the model were 0.9918 and 0.9843, respectively. The adequate precision value of 43.462 indicates that the model can be used to navigate the design space as it is greater than 4.0 (Montgomery, 2001). Considering these criteria, following response model was selected for representing the variation of lateral expansion for further analysis.

$$LE = 152.77 + 1.47x_1 + 0.37x_2 - 16.58x_3 - 12.08x_1^2 - 8.86x_2^2 + 0.95x_3^2 + 2.59x_1x_2 + 0.031x_1x_3 - 3.28x_2x_3$$
(2)

Where x_1 , x_2 and x_3 are the coded values temperature (°C), screw speed rpm and feed proportion [(rice flour + wheat flour): honey], respectively.

The following observations can be made from Equation 2. The coefficients of x_1 (temperature) and x_2 (screw speed) were positive, where x_3 (feed proportion) is negative. Therefore, increase in temperature and screw speed may increase the expansion of the product, whereas increase in honey proportion of feed proportion may reduce the expansion of the product. The coefficients of x_1^2 and x_2^2 were negative therefore they will show negative quadratic effect on lateral expansion ratio.

Analysis of variance of Equation 2 (Table 2a) showed that F-values for liner terms of temperature and feed proportion were 5.05 and 644.86 and p values 0.0485 (0.05) and <0.0001, showing that the x_1 and x_3 were significant terms. F-values for square terms of temperature (x_1^2) and screw speed (x_2^2) were 361.42 and 194.20 and p value <0.0001and <0.0001, showing that both terms were significant at 1%, respectively.

The maximum bulk density (0.442067 g/cc) of extrudates at coded point (-1, 1, 1) was about 2.11 times more than the minimum bulk density (0.209484 g/cc) at coded point (0, 0, -1.682) and the average value of bulk density was 0.3505201 g/cc (Table 1a). The coefficients of the model and other statistics were given in Table 2a.

			Indepe	endent variables	6		Dependent variables				
S/N	Temperature (°C) X ₁		Screw speed (rpm) X ₂		Feed proportion (%) X ₃		LE (%)	BD (g/cc)	L*(g/g)	a*(%)	
	Coded	Uncoded	Coded x ₂	Uncoded	Coded x ₃	Uncoded	Y ₁	Y ₂	Y ₃	Y ₄	
1	-1	115	-1	270	-1	95:5	145.25	0.365266	64.46	3.61	
2	1	135	-1	270	-1	95:5	142.5	0.339454	62.94	3.82	
3	-1	115	1	350	-1	95:5	148	0.313406	60.00	4.28	
4	1	135	1	350	-1	95:5	158.75	0.256157	61.57	3.99	
5	-1	115	-1	270	1	85:15	117.75	0.436927	60.36	4.58	
6	1	135	-1	270	1	85:15	118.25	0.410509	56.82	5.47	
7	-1	115	1	350	1	85:15	110.5	0.442067	62.63	4.46	
8	1	135	1	350	1	85:15	118.25	0.403769	59.69	5.14	
9	-1.682	108	0	310	0	90:10	118	0.422816	62.69	4.26	
10	1.682	141	0	310	0	90:10	120.25	0.374153	64.97	3.88	
11	0	125	-1.682	242	0	90:10	130.25	0.392125	58.11	4.95	
12	0	125	1.682	377	0	90:10	126.25	0.360155	60.18	4.75	
13	0	125	0	310	-1.682	98.4:1.6	184.75	0.209484	63.90	3.03	
14	0	125	0	310	1.682	81.6:18.4	127.25	0.330898	59.69	4.67	
15	0	125	0	310	0	90:10	155.5	0.353676	58.21	4.68	
16	0	125	0	310	0	90:10	153.6	0.333706	59.89	4.63	
17	0	125	0	310	0	90:10	152.8	0.323706	58.46	4.50	
18	0	125	0	310	0	90:10	153.3	0.313456	58.83	4.53	
19	0	125	0	310	0	90:10	151.25	0.325606	58.08	4.93	
20	0	125	0	310	0	90:10	150	0.303066	57.88	4.56	

Table 1a. The CCRD (in coded and uncoded levels of three variables and five levels) employed for development of honey based extrudates and the responses (lateral expansion, bulk density, colour L* and a* value) of developed extrudates.

The Model F-value of 16.65 indicates that the model was significant (P < 0.05). R^2 (0.9375) and Adjusted R^2 (0.8812) values were near. The Adequate Precision (16.235) indicates that model can be used for prediction purposes. Considering these criteria, following response model was selected for representing the variation of bulk density for further analysis.

 $BD = 0.32 - 0.017x_1 - 0.014 x_2 + 0.046x_3 + 0.030x_1^2 + 0.022x_2^2 - 0.015x_3^2 - (5.415 \times 10^{-3})x_1x_2 + (2.293 \times 10^{-3})x_1x_3 + 0.017x_2 x_3$ (3)

It is evident from Equation 3 that coefficients of x_1 and x_2 were negative, but that of x_3 was positive. Therefore, increase in temperature and screw speed may reduce the bulk density, whereas increase in honey proportion of feed proportion may increase the bulk density of the product. The coefficients of x_1^2 and x_2^2 were positive therefore they will show positive quadratic effect on bulk density and the coefficients of x_3^2 was negative effect on bulk density.

It was observed from Table 2a and Equation 3 that F-values for liner terms of temperature, screw

			Independ	Dependent variables					
S/N	Temperature (°C) X ₁		Screw speed (rpm) X ₂		Feed proportion (%) X ₃		b*	WAI (g/g)	WSI (%)
	Coded x ₁	Uncoded	Coded x ₂	Uncoded	Coded x ₃	Uncoded	Y ₅	Y ₆	Y ₇
1	-1	115	-1	270	-1	95:5	17.58	5.195	4.5
2	1	135	-1	270	-1	95:5	18.42	5.340	4.0
3	-1	115	1	350	-1	95:5	17.97	5.395	6.0
4	1	135	1	350	-1	95:5	17.25	5.430	5.5
5	-1	115	-1	270	1	85:15	18.66	5.035	10
6	1	135	-1	270	1	85:15	18.85	5.105	8.5
7	-1	115	1	350	1	85:15	18.18	5.045	8.5
8	1	135	1	350	1	85:15	18.53	5.325	11
9	-1.682	108	0	310	0	90:10	18.66	4.945	5.5
10	1.682	141	0	310	0	90:10	17.13	5.130	5.5
11	0	125	-1.682	242	0	90:10	18.13	6.010	4.0
12	0	125	1.682	377	0	90:10	18.47	6.070	6.0
13	0	125	0	310	-1.682	98.4:1.6	16.48	5.630	8.5
14	0	125	0	310	1.682	81.6:18.4	18.3	5.400	11.5
15	0	125	0	310	0	90:10	17.08	6.200	6.0
16	0	125	0	310	0	90:10	16.68	6.380	7.5
17	0	125	0	310	0	90:10	17.38	6.150	7.0
18	0	125	0	310	0	90:10	17.30	6.350	6.5
19	0	125	0	310	0	90:10	1743	6.178	6.0
20	0	125	0	310	0	90:10	17.33	6.200	6.0

Table 1b. The CCRD (in coded and uncoded levels of three variables and five levels) employed for development of honey based extrudates and the responses [colour b* value, water absorption index (WAI), and water solubility index (WSI)] of developed extrudates.

speed and feed proportion were 9.35, 6.43 and 68.84 and p values 0.0121, 0.0295 (0.05) and <0.0001, showed that the x_1 , x_2 and x_3 were significant terms. F-values for square terms of temperature (x_1^2), screw speed (x_2^2) and feed proportion (x_3^2) were 32, 17.48 and 7.93 and p value 0.0002, 0.0019 and 0.0183, showing that three terms were significant at 1 and 5%, respectively.

It may be seen from Figure 1a and b that the

increase in honey proportion in feed composition resulted in decrease in expansion of extrudate and increase in bulk density of extrudate (Figure 2a). The opposite behavior of bulk density and lateral expansion with the change in process variables have also been reported by Ding et al. (2006).

Sugar effect on the extrusion of cereal source material has been studied by many scientists and

it was generally observed that an increase of monosaccharides and disaccharides concentration reduced the expansion of extrudates and increase the bulk density (Sopade and Le Grys, 1991; Hsieh et al., 1993; Ryu et al., 1993; Jin et al., 1994; Barret et al., 1995; Fan et al., 1996a, b). Moore et al. (1990) also reported that the presence of sugar tended to reduce the availability of water or water activity, which affect

Coofficients	Latera	Bulk density (g/cc)			Colour L* value				
Coefficients	Coeff. value	F	Р	Coeff. value	F	Р	Coeff. value	F	Р
X ₀	152.77***	133.63	< 0.0001	0.32***	16.65	<0.0001	58.75**	6.14	0.0045
X ₁	1.47**	5.05	0.0485	-0.017**	9.35	0.0121	-0.14 ^{ns}	0.17	0.6917
X ₂	0.37 ^{ns}	0.32	0.5857	-0.014**	6.43	0.0295	0.20 ^{ns}	0.35	0.5665
X ₃	-16.58***	644.86	<0.0001	0.046***	68.84	<0.0001	-1.21**	12.35	0.0056
X_{1}^{2}	-12.08***	361.42	< 0.0001	0.030***	32.00	0.0002	1.60***	22.73	0.0008
X_2^2	-8.86***	194.20	< 0.0001	0.022**	17.48	0.0019	0.015 ^{ns}	1.900 × 10 ⁻³	0.9661
X_{3}^{2}	0.95 ^{ns}	2.25	0.1647	-0.015**	7.93	0.0183	0.95**	8.04	0.0177
X ₁₂	2.59**	9.24	0.0125	5.415 × 10 ^{-3 ns}	0.57	0.4685	0.46 ^{ns}	1.05	0.3301
X ₁₃	0.031 ^{ns}	1.342 × 10 ⁻³	0.9715	2.293 × 10 ^{-3 ns}	0.10	0.7562	-0.82 ^{ns}	3.28	0.1001
X ₂₃	-3.28**	14.79	0.0032	0.017**	5.40	0.0425	1.37**	9.26	0.0124
R ²		0.9918		0	.9375			0.8467	
Adjusted R ²	0.9843		0.8812			0.7088			
Adeq. precision	43.462			16.235			7.545		
Lack of fit		2.16			1.73			3.02	

Table 2a. Regression coefficients of second order polynomial and their significance for lateral expansion, bulk density and colour L* value.

*Significant at P < 0.1, **Significant at P < 0.05, ***Significant at P < 0.001, ns- not significant.

the expansion and bulk density of extrudate. It may be observed from Figure 1a and b that the lateral expansion increased with the increase in the die temperature and screw speed and the bulk density decreased with the increase in the temperature and screw speed (Figure 2a and b). Fletcher et al. (1985), Lawton et al. (1985) and Mercier and Fillet (1975) reported that with an increase in the die temperature, there was increase in degree of superheating of water in the extruder encouraging bubble formation with decrease in melt viscosity, leading to increased expansion and decreased bulk density.

It is also evident from Figures 1b and 2b that the lateral expansion increased and bulk density decreased with the increase in the screw speed, which may be due to high mechanical shear resulting in higher expansion. Similar results have been reported by Ding et al. (2006).

Colour L*, a* and b* value

L* value of extrudates ranged from 56.82 to 64.9756 with an average value of 60.498. The maximum L* value at coded point (1.682, 0, 0) was about 1.14 times more than the minimum L* value at the coded point of 1, -1, 1 (Table 1a). The coefficients of the model and other statistics were given in Table 2a. The Model F value of 6.14 implies that model was significant (P < 0.05). R² and Adjusted R² values of the model were 0.8467 and 0.7088, respectively. The Adequate Precision value of 7.545 indicates that the model can be used for prediction purposes. Considering these criteria, the following response model was

selected for representing the variation of L* value for further analysis:

$$L^* = 58.75 - 0.14x_1 + 0.20 x_2 - 1.21x_3 + 1.60x_1^2 + 0.015x_2^2 + 0.95x_3^2 + 0.46x_1x_2 - n \ 0.82x_1x_3 + 1.37x_2x_3$$
 (4)

following observations can be made from Equation 4. The coefficients of x_1 (temperature) and x_3 (feed proportion) were negative, where x_2 (screw speed) is positive. Therefore, increase in temperature and feed proportion may decrease the L* value of the product, whereas increase in screw speed may increase the L* value of the product. The coefficients of x_1^2 and x_3^2 were negative, therefore they will show negative quadratic effect on L* value.

Analysis of variance of Equation 4 (Table 2a)



Figure 1a. Response surface plot for the lateral expansion as a function of feed proportion and temperature.



Figure 1b. Response surface plot for the lateral expansion of as a function of feed proportion and screw speed.

showed F-values for square terms of temperature (x_1^2) and feed proportion (x_3^2) were 22.73 and 8.04 and p value 0.0008 and 0.0177, showed that both terms were significant at 1 and 5%, respectively.

a* value of extrudates ranged from 3.03 to 5.47 with an

average value of 4.436. The maximum a* value at coded point (1, -1, 1) was about 1.8 times more than the minimum a* value at the coded point of 0, 0, -1.682 (Table 1a). The coefficients of the model and other statistics were given in Table 2b. The Model F value of



Figure 2a. Response surface plot for the bulk density as a function of feed proportion and temperature at screw speed.



Figure 2b. Response surface plot for the bulk density as a function of feed proportion and screw speed at temperature.

10.34 implies that model was significant (P < 0.05). R^2 and Adjusted R^2 values of the model were 0.9030 and 0.8157, respectively. The Adequate Precision value of

13.053 indicates that the model can be used for prediction purposes. Considering these criteria, the following response model was selected for representing

Coofficiento	Col	our a* value		Colour b* value			
Coefficients	Coeff. value	F	Р	Coeff. value	F	Р	
X ₀	4.63***	10.34	0.0005	17.19**	3.40	0.0349	
X ₁	0.062 ^{ns}	0.91	0.3614	-0.14 ^{ns}	1.14	0.3114	
X ₂	3.928 × 10 ^{-3 ns}	3.635 × 10 ⁻³	0.9531	-0.074 ^{ns}	0.32	0.5866	
X ₃	0.49***	56.85	<0.0001	0.44**	11.41	0.0070	
X_{1}^{2}	-0.16**	6.51	0.0288	0.30**	5.66	0.0386	
X_{2}^{2}	0.11 ^{ns}	3.23	0.1027	0.45**	12.25	0.0057	
X_3^2	-0.24**	14.28	0.0036	0.13 ^{ns}	0.97	0.3482	
X ₁₂	-0.089 ^{ns}	1.09	0.3217	-0.18 ^{ns}	1.04	0.3320	
X ₁₃	0.21**	5.87	0.0359	0.052 ^{ns}	0.094	0.7660	
X ₂₃	-0.16*	3.59	0.0874	-2.500 × 10 ^{-3 ns}	2.121 × 10 ⁻⁴	0.9887	
R ²		0.9030			0.7538		
Adjusted R ²		0.8157			0.5323		
Adeq. precision	13.053				5.529		
Lack of fit	3.68			4.94			

Table 2b. Regression coefficients of second order polynomial and their significance for colour a*value and b*value.

*Significant at P < 0.1, **Significant at P < 0.05, ***Significant at P < 0.001, ns- not significant.

the variation of a* value for further analysis:

$$a^{*} = 4.63 + 0.062x_{1} + (3.928 \times 10^{-3})x_{2} + 0.49x_{3} - 0.16x_{1}^{2} + 0.11x_{2}^{2} - 0.24x_{3}^{2} - 0.089x_{1}x_{2} + 0.21x_{1}x_{3} - 0.16x_{2}x_{3}$$
(5)

The following observations can be made from Equation 5. The coefficients of x_1 (temperature), x_2 (screw speed) and x_3 (feed proportion) were positive. Therefore, increase in temperature, screw speed and feed proportion may increase the a* value of the product. F-values for square term of temperature and feed composition (x_1^2 and x_3^2) were 6.51 and 14.28 and P values were <0.0288 and <0.0036, respectively, validating that these terms were significant. From the Table 2b, the coefficients of x_1^2 and x_3^2 were negative, therefore they will show negative quadratic effect on the a* value.

b* value of extrudates ranged from 16.48 to 18.85.with an average value of 17.79. The maximum b* value at coded point (1, -1, 1) was about 1.14 times more than the minimum b* value at the coded point of 0, 0, -1.682 (Table 1b). The coefficients of the model and other statistics were given in Table 2b. The Model F value of 3.40 implies that model was significant (P < 0.05). R² and Adjusted R² values of the model are 0.7538 and 0.5323, respectively. The Adequate Precision value of 5.529 indicates that the model can be used for prediction purposes. Considering these criteria, the following response model was selected for representing the variation of b* value for further analysis.

$$b^{*} = 17.19 - 0.14x_{1} - 0.074x_{2} + 0.44x_{3} + 0.30x_{1}^{2} + 0.45x_{2}^{2} + 0.13x_{3} - 0.18x_{1}x_{2} + 0.052x_{1}x_{3} - (2.500 \times 10^{-3})x_{2}x_{3}$$
(6)

The following observations can be made from Equation 6.

The coefficients of x_1 (temperature) and x_2 (screw speed) were negative, where x_2 (feed proportion) was positive. Therefore, increase in temperature and screw speed may decrease the b* value of the product, whereas increase in feed proportion may increase the b* value of the product. The coefficients of x_1^2 and x_2^2 were positive; therefore they will show positive quadratic effect on b value.

Analysis of variance of Equation 6 (Table 2b) showed that F-values for linear term of feed proportion was 11.41 and p value 0.0070 (0.05), showed that the x_3 was significant term. F-values for square terms of temperature (x_1^2) and screw speed (x_2^2) were 5.66 and 12.25 and p value 0.0386 and 0.0057, showed that both terms were significant at 5%, respectively.

It may be seen from Figure 3a, 4a and 5a that the increase in honey proportion in feed composition resulted in decrease in L* value of extrudate and increase in a* and b* value of extrudate. This was due to occurrence of browning reaction, which may be due to caramellization of sugars present in honey (Maga and Kim, 1989).

Figures 3a, 4a and 5a show that the L* and a* value increased and b* decreased with the increase in the screw speed. The reason for the increasing L* value with increasing screw speed, there was reduction in the residence time and, hence, reduction in the extrudate browning (Zin et al., 1994).

Water absorption index

The water absorption index of extrudates varied in the range of 4.945 to 6.38 g/g with an average value of 5.62 g/g. The maximum water absorption index at coded point (-1.682, 0, 0) was about 1.3 times more than the minimum water expansion index at the coded point of 0,



Figure 3. Response surface plot for the colour L* value as a function of feed proportion and screw speed.



Figure 4. Response surface plot for the colour a* value as a function of screw speed and feed proportion.



Figure 5. Response surface plot for the colour b* value as a function of feed proportion and screw speed.

Osstisiente	Water al	osorption index	(g/g)	Water solubility index (%)			
Coefficients	Coeff. value	F	Р	Coeff. value	F	Р	
X ₀	6.25***	18.25	<0.0001	6.49	8.11	0.0015	
X ₁	0.062 ^{ns}	1.86	0.2021	0.000 ^{ns}	0.000	1.0000	
X ₂	0.045 ^{ns}	1.02	0.3374	0.54*	3.73	0.0822	
X ₃	-0.091*	4.03	0.0725	1.69***	36.54	0.0001	
X_1^2	-0.48***	119.33	<0.0001	-0.27 ^{ns}	0.99	0.3441	
X_2^2	-0.13**	8.15	0.0171	-0.45 ^{ns}	2.70	0.1313	
X_3^2	-0.31***	50.13	<0.0001	1.32***	23.63	0.0007	
X ₁₂	0.013 ^{ns}	0.045	0.8363	0.50 ^{ns}	1.88	0.2004	
X ₁₃	0.021 ^{ns}	0.13	0.7260	0.25 ^{ns}	0.47	0.5087	
X ₂₃	-7.500×10 ^{-3 ns}	0.016	0.9013	-0.25 ^{ns}	0.47	0.5087	
R ²		0.9426			0.8795		
Adjusted R ²		0.8910		0.7710			
Adeq. precision		12.389		12.607			
Lack of fit		4.94		4.32			

Table 2c. Regression coefficients of second order polynomial and their significance for water absorption index and water solubility index.

*Significant at P < 0.1, **Significant at P < 0.05, ***Significant at P < 0.001, ns- not significant.

0, 0 (Table 1b). The coefficients of the model and other statistics were given in Table 2c. The Model F-value of 18.25 signifies that the model was significant (P < 0.05). R^2 (0.9426) and Adjusted R^2 (0.8910) were in reasonable agreement. The Adequate Precision value of 12.389 implies that the model can be used for prediction purposes. Considering these criteria, the following response model was selected for representing the

variation of water absorption index for further analysis:

 $WAI = 6.25 + 0.062x_1 + 0.045x_2 - 0.091x_3 - 0.48x_1^2 - 0.13x_2^2 - 0.31x_3^2 + 0.013x_1x_2 + 0.021x_1x_3 - (7.500 \times 10^{-3}) x_2x_3$ (7)

It can be seen from Equation 7 that the coefficients of x_1 and x_2 were positive, but that of x_3 is negative; therefore



Figure 6a. Response surface plot for the water absorption index (WAI) as a function of screw speed and temperature.



Figure 6b. Response surface plot for the water absorption index (WAI) as a function of screw speed and feed proportion.

increase in temperature and screw speed may increase the water absorption index, whereas increase in feed proportion may decrease the water absorption index of the product. The coefficients of x_1^2 , x_2^2 and x_3^2 were negative therefore they will show negative quadratic effect on water absorption index.

Analysis of variance of Equation 7 (Table 2c) showed that F-values for square terms of temperature (x_1^2) , screw speed (x_2^2) and feed proportion (x_3^2) were 119.33, 8.15

and 50.13 and p value <0.0001, 0.0171 and <0.0001, showed that three terms were significant at 1 and 5%, respectively.

The effect of barrel temperature on WAI observed in this experiment was not completely in agreement with Ding et al. (2006), who found that WAI decreased with increasing the die temperature and feed moisture. As shown in Figure 6a and b, initially, there was increase in WAI with increase in temperature and feed proportion,



Figure 7a. Response surface plot for the water solubility index (WSI) as a function of screw speed and temperature.

but after obtaining the maximum value, there was decrease in WAI with further increase in temperature and feed proportion. Such kind of variation may be due to keeping the feed moisture constant throughout the experiment because feed moisture also exerts the effect on extrudate by promoting gelatinization. At the temperature of about 125°C, gelatinization was dominating over the starch melting or dextrinization and further increase in temperature results in more dextrinization in comparison to gelatinization. The binding of water due to addition of honey might have also limited the availability of water, which could be reason of greater dextrinization.

Water solubility index

Water solubility index was used as an indicator of degradation of molecular components. It measures the amount of soluble polysaccharide released from the starch component after extrusion (Ding et al., 2005). Water solubility index of extrudates ranged from 4.0 to 11.5 % with an average value of 6.9%. The maximum water solubility index at coded point (0, 0, 1.682) was about 2.9 times more than the minimum water solubility index at the coded point of 1, -1, -1 and 0, -1.682, 0 (Table 1b). The coefficients of the model and other statistics were given in Table 2c. The Model F-value of 8.11 indicates that the model was significant (P < 0.05). R^2 (0.8795) and Adjusted R^2 (0.7710) were in reasonable agreement. Moreover, the adequate precision (12.607) is greater than 4, indicating a good fit of experimental data and the acceptability of the model for prediction

purposes. Considering these criteria, the following response model was selected for representing the variation of lateral expansion for further analysis.

$$WSI = 6.49 + 0.000x_1 + 0.54x_2 + 1.69x_3 - 0.27x_1^2 \quad 0.45x_2^2 + 1.32x_3^2 + 0.50x_1x_2 + 0.25x_1x_3 - 0.25x_2x_3 \tag{8}$$

The following observations can be made from Equation 8. The coefficients of x_1 (temperature), x_2 (screw speed) and x_3 (feed proportion) were positive. Therefore, increase in temperature, screw speed and feed proportion may increase the water solubility index of the product. The coefficients of x_3^2 were positive therefore it will show positive quadratic effect on water solubility index.

Analysis of variance of Equation 8 (Table 2c) showed that F-value for square term of feed proportion (x_3^2) was 23.63, and p value 0.0007 showed that the term was significant at 1%, respectively.

It is clear from Figure 7a that there was slight decrease in water solubility index with increase in screw speed. Ding et al. (2006) reported that there was no significant effect of screw speed on the WSI. However, results of effect of temperature on WSI (figure 7a) were in agreement with the Ding et al. (2006), which showed increase in WSI with increase in die temperature. Results of WSI were also in agreement with Mercier and Feillet (1975), who found that WSI increased with increase in temperature at the feed moisture of 18.2%. In the present work, the feed moisture was maintained at 18% throughout the experiment. With the increase in the feed proportion of honey, there was increase in WSI (Figure 7b). The increase in WSI with increase in honey



Figure 7b. Response surface plot for the water solubility index (WSI) as a function of temperature and feed proportion.

proportion was due to restriction on starch gelatinization by limited water availability so it promoted more dextrinization in comparison to gelatinization.

Superimposition of contour plots of responses

A graphical multi-response optimization technique was adopted to determine the workable optimum conditions for the extrudate. The contour plots for all responses were superimposed and regions that best satisfy all the constraints were selected as optimum conditions. The main criterion for constrained optimization was maximum possible lateral expansion, L* value and water absorption index while lower bulk density, water solubility index, a* and b* values of the extrudates, keeping other responses within the acceptable range. These constrains resulted in "feasible zone" of the optimum solutions (shaded area in the superimposed contour plots). Superimposed contour plots having common superimposed area for all responses for honey based extrudates are shown in Figure 8a and b.

The graphical optimization, the process variables were optimized as, temperature (119 to122°C), screw speed (301 to 321 rpm) and feed composition (9.68 to 10.35%).

Conclusion

The optimum conditions obtained for development of honey based extrudates indicated that honey could be incorporated into ready-to-eat expanded products up to the level of 10.35% and therefore could be considered a source of nutrients present in honey. The degree of starch gelatinization and extrudate expansion was reduced as the amount of honey in feed proportion increased beyond this level. The reduction in water availability due to addition of honey also resulted in more dextrinization in comparison to gelatinization. Increasing the concentration of honey in the feed proportion had



Feed proportion (%)

Figure 8a. Superimposed contours for product responses affected by feed proportion and temperature at screw speed 310 rpm.



Screw speed (rpm)

Figure 8b. Superimposed contours for product responses affected by screw speed and temperature at feed proportion 10%.

similar effects as increasing the feed moisture.

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