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Optimization of Mulberry Jelly Making by Response Surface Methodology

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ABSTRACT Recently, though mulberry's superiority as a functional food has been proven, its use as a food material is limited. Therefore, in this study, to develop jelly using mulberry that is compatible with Korean tastes as health functional food by grafting the method to manufacture jelly consumed as a dessert or a snack in the west, according to the central composit design, mulberry jelly was produced by varying the content of citric acid (X_1), sucrose (X_2), and gelatin (X_3) at 5 levels. And by applying the response surface methodology, rheology and sensory preference experiment results were analyzed, the optimization of mulberry jelly manufacturing condition was carried out, and studies on the analysis of composition were performed. As the sensory preference of mulberry jelly, except the flavor, the remaining hardness, elasticity, sweetness, color, and the overall quality were found to be significant. And similarly, it was found to be influenced greatly by gelatin content generally. Based on the overlapped part of categories, in the range of factors that satisfy all the sensory categories, the value located in the middle was calculated, the optimization point was obtained, and it was found to be 6.2 g citric acid, 141.0 g sugar, and 13.5 g gelatin.

KEYWORDS: mulberry jelly, citric acid, sucrose, gelatin, response surface methodology

INTRODUCTION

According to old medical textbooks, mulberry was told to be sweet and cold without toxicity. So, it was described to be used to treat diabetes, beneficial to five viscera and liver to activate the vitality. Long term use of it relieved hunger, turned faded hair into black hair, and prevented aging (Kim et al 2003, Kim et al 1996). Also, the extract of mulberry has been known to have antihyperglycemic, antioxidant, antiflammation and antihypertensive physiological effects. The anthocyan colorant present in the flesh and peel of mulberry is highly expected to be used as the natural colorant, and it is also expected to have the effect of suppressing aging, diabetic retinopathy and improving eyesight improvement along with antioxidative physiological activity. So, it is spotlighted as a natural colorant unharmful to humans and as a functional food material (Kim et al 2003, Kim et al 1998, Kim et al 2001, Koh 1995). With the recent increase of consumer preference for products that use functional materials, like mulberry, along with upgrading and adding variety into one's diet, western dessert culture has rapidly spreaded in population amongst young people (Yu and C 1996). The consumption of jelly that includes the functionality and favoritism has been continued. As one of western desserts, jelly is a gel state food by containing about 20% of water and various textures could be assigned by using different gelling agents and manufacturing of various jelly products can be expected from different manufacturing processes (Shin 1998, Kim 2006).

Therefore, in this study, to develop jelly using mulberry that is compatible with Korean tastes as health functional food by grafting the method to manufacture jelly consumed as a dessert or a snack in the west, according the central composit design, mulberry jelly was produced by varying the content of citric acid (X_1), sucrose (X_2), and gelatin (X_3) at 5 levels. Applying the response surface methodology, rheology, and sensory preference experiments results were analyzed, the optimization of mulberry jelly manufacturing condition was carried out, and studies on the analysis of compositon were performed.

MATERIALS AND METHODS

Materials

Mulberry used in this experiment was harvested from Noksan Oddi Co., Ltd in 2006 (Jeonbok, Korea). The Mulberry was kept in a freezer and sucrose was white sugar (Baiksul, CJJailJedang, Inc., Korea), gelatin used in this

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Table 1. Basic formula for mulberry jelly preparation

Ingredient	Symbol	Increment	Coded-variables				
			-2	-1	0	1	2
Mulberry juice (g)	-	100	100	100	100	100	100
Water (g)	-	60	60	60	60	60	60
Citric acid (g)	X_1	±2	2	4	6	8	10
Sucrose (g)	X_2	±60	20	80	140	200	260
Gelatin (g)	X_3	±4	4	8	12	16	20

experiment was powder gelatin (Knox, a unit of Nabisco, Inc., USA). Water was distilled water.

Preparation of Mulberry jelly

The manufacturing of mulberry jelly was done by referring the precedent studies regard on fruit product manufactures and preliminary experiments have been conducted (Yu and C 1996, Choi et al 1994, Heo et al 2004, Jeong and Joo 2003, Paik et al 1996, Chun 1995, Park and Cho 1998, Lee et al 1986). The materials and amount used for the manufacture were summarized in Table 1. The basic mixing ratio was set to be 5 by including central value, and the 16 treatment groups were combined by using the fractional factorial design that includes 3 factors to manufacture 400 g of product. The manufacturing method is described as below.

By using a circulation pressure juice machine, the juice of mulberry was acquired by pressing a white bag filled with mulberry, and 100 g of mulberry juice from each treatment group was heated to 70°C and at the same pot and added with sugar and boiled it until the temperature turned to 100°C.

First, using a circulation pressure juice machine (HD-A110, Habdong-jeongmilKigong Inc., Korea), then, put into a pot, steamed at 70°C, and added sucrose, and steamed at 100°C. Then stirred gelatin powder and added citric acid and cool down for 30 min in a tool (15×15×4 cm), then Kept in a refrigerator for 3 hr. and cut the jelly as same size (2×2×3 cm).

pH

pH measurements were carried out with a pH meter (Model 34, Beckman Instrument Inc., Fullerton, CA, USA) on a 50 g sampled Mulberry jelly.

Brix

The entire experiment was repeated three times. The Brix values of Mulberry jelly were measured by a Salinity refractometer (Nippon optical works, Tokyo, Japan)

Color

The color values (L-,a- and b-value) of Mulberry jelly were measured using a colorimeter (CR-300; Minolta Co., Osaka, Japan). L value standard plate was 97.26, a values was -0.07 and b value was 1.86.

Table 2. Operating conditions of Rheometer

Sample Height	20 mm
Probe Diameter	9 mm
Clearance	30 mm
Chart Speed	120 mm/min
Table Speed	120 mm/min
Load Cell	2 kg
Repeat (Mastication)	0 sec

Sensory preference test

Sensory preference test was done according to the seven-point hedonic scale. Processed Mulberry jelly was evaluated for its sensory preference by a 21-student panel at Sookmyung university. The Mulberry jelly prepared for each test sample was corded with a random 4 digit number.

The panelists were asked to evaluate the preference of sweetness, color, flavor, hardness, elasticity and overall quality of the jelly by giving a score ranging from 1 (dislike extremely) to 7 (like extremely).

Statistical analysis

The experiments were repeated three times. To find the key characteristics affecting the sensory quality of Mulberry jelly, correlation analysis, and stepwise regression analysis were performed between the overall sensory quality and physicochemical characteristics of the mulberry jelly, using SAS package Version 8.12.

RESULT AND DISCUSSION

Physicochemical properties of mulberry jelly

Table 3 and 4 show the brix levels. The brix level of mulberry jelly could be varied by the amount of sugar added at the mulberry manufacture. The R^2 value of regression equation regard on the brix was 0.87 and its significance was found with a probability level of 0.05 (α=.05), which means the appropriateness of the response surface model that estimated it (Table 4). The amount of sugar addition was

Table 3. Effect of preparing condition of mulberry jelly on the sweetness, pH, color, texture and sensory evaluation different coded values of treatment

No	Variable-level[1)]			Sweetness	pH	Color			Texture properties					Sensory evaluation				
	X_1	X_2	X_3			L	a	b	Y_1	Y_2	Y_3	Y_4	Y_5	Y_6	Y_7	Y_8	Y_9	Y_{10}
1	-1	-1	-1	43.8	3.22	11.12	2.05	0.69	152	87.2.077	94.57	476.50	3.82	3.03	3.99	4.01	3.48	3.24
2	-1	-1	+1	44.6	3.10	22.19	2.09	0.77	391	94.07	98.26	1231.32	4.01	3.71	3.94	4.27	3.57	3.52
3	-1	+1	-1	62.5	2.86	22.31	1.99	0.19	741	89.51	91.26	236.3	4.02	4.61	4.01	4.02	3.71	3.45
4	-1	+1	+1	61.0	2.97	14.21	1.87	0.58	285	90.24	94.23	872.62	4.23	3.51	4.02	3.98	2.95	4.58
5	+1	-1	-1	46.3	2.42	12.34	2.18	1.02	117	97.61	94.84	346.50	4.20	3.64	3.90	4.81	3.76	4.00
6	+1	-1	+1	47.1	2.62	10.33	2.72	1.26	315	93.11	95.68	927.38	4.01	4.34	3.94	4.63	3.48	4.69
7	+1	+1	-1	61.3	2.36	16.55	3.2	0.67	456	95.19	89.58	121.84	4.05	4.04	4.23	4.02	4.71	4.32
8	+1	+1	+1	45.7	2.65	12.38	1.72	0.66	225	91.57	96.26	751.76	4.36	3.01	4.22	4.69	5.14	4.88
9	0	0	0	54.5	2.30	13.00	1.06	0.57	211	91.74	94.97	677.00	4.86	4.67	4.62	4.90	4.19	5.86
10	0	0	0	52.8	2.22	12.72	1.06	0.60	215	88.96	95.14	690.29	4.92	3.84	4.38	4.95	3.62	5.95
11	-2	0	0	52.6	2.98	13.66	0.86	0.43	204	87.06	96.07	617.27	4.10	3.71	4.01	3.51	3.57	3.12
12	+2	0	0	54.0	1.96	13.16	1.70	0.72	162	89.37	95.86	546.94	4.18	3.71	3.32	4.53	3.48	3.43
13	0	-2	0	22.5	2.33	10.9	1.12	0.53	327	87.04	97.04	940.51	3.52	3.01	3.38	4.73	3.33	3.92
14	0	+2	0	64.3	2.55	13.26	0.95	0.49	770	85.82	91.56	198.24	3.92	3.48	3.41	4.71	3.67	4.12
15	0	0	-2	34.5	2.46	6.27	1.6	1.00	188	81.21	68.03	43.85	3.88	3.41	4.33	3.81	4.10	2.71
16	0	0	+2	38.2	2.63	12.54	0.96	0.58	258	99.13	97.65	846.53	3.91	3.38	4.35	3.3.4	4.43	2.89

Independent variables: X_1: Citric acid X_2: Sugar X_3: Gelatin
Instrumental texture parameters: Y_1: Hardness Y_2: Cohesiveness Y_3: Springiness Y4: Gumminess
Sensory preference: Y_5: Hardness Y_6: Elasticity Y_7: Sweetness Y_8: Color Y_9: Flavor Y_{10}: Overall quality

the most effective factor in the sweetness of mulberry jelly, followed by the amount of gelatin, and citric acid. The material content of mulberry jelly showing the optimum point of Brix was found to be 3.34 g of citric acid, 229.60 g of sugar, and 11.40 g of gelatin (Table 5).

The pH of mulberry jelly was highly affected by the amount of citric acid addition. The regression coefficient R^2 of the regression equation for pH was 0.93 and the significance was found at the probability level of 0.01 (α=0.01). The response surface of mulberry for pH did not largely affected by the amount of sugar or gelatin addition, but the pH was found to be lowered as the amount of citric acid addition was increased. A similar result was found from a study by Shim & Han (Shim et al 2006). The material content of mulberry jelly showing the optimum point of pH was found to be 2.15 g of citric acid, 114.41 g of sugar, and 13.29 g of gelatin. The L, a, b values of the color of mulberry did not show significance with a probability level of 0.05 (α=.05) when compared with the regression coefficient (R^2) of the regression equation for color.

Characteristics of rheology

As the results of physical property measurement of mulberry, the expected regression variation for the hardness and gumminess revealed a significant difference with a probability level of 0.01 (α=.01), but the cohesiveness and springness did not show significant differences with a probability level of 0.05 (α=.05).

The regression coefficient (R^2) of the regression equation for Hardness was 0.94 and its significance was found with the probability level of 0.01 (α=.01). Gelatin was found to have the most effect on the Hardness of mulberry jelly. In addition, the lower sugar concentration resulted in a lower effect on the Hardness.

The regression coefficient (R^2) of the regression equation for the Gumminess was 0.94 and the significance was found with a probability level of 0.01 (α=.01). Like Hardness, Gelatin was found to have the most effect on the Gumminess of mulberry jelly and the increase in viscosity was confirmed with the increase in gelatin amount.

Sensory preference test

As for sensory characteristics of mulberry jelly, besides flavor, the remaining hardness, elasticity, sweetness, color and the overall quality were found to be influenced greatly by gelatin generally.

The hardness of mulberry resulted in higher sensory values as the content of gelatin and sugar got close to the centroid. Too small or excessive amount of gelatin or sugar resulted in a negative effect on the favorness for hardness (Table 3). The hardness of mulberry jelly resulted to show significant at the probability level of 0.001 (α=.001), and regression coefficient (R^2) value was found to be 0.96 (Table 4). The material content of mulberry jelly showing the optimum point of hardness was found to be 6.19 g of citric acid, 149.41 g of sugar, and 12.35 g of gelatin.

Table 4. Polynomial equations calculated by RSM program for processing of Mulberry jelly

Responses		Polynomial equation[1)]	R^2 [2)]	P value
Sweetness		Y_1=-64.763889+5.769792X_1+0.588368X_2+9.046354X_3-0.021875X_1^2-0.022396X_2X_1-0.000712X_2^2-0.220312X_3X_1-0.009740X_3X_2-0.270313X_3^2	0.87	0.0398*
pH		Y_1=2.959514-0.076771X_1+0.000830X_2+0.028073X_3-0.011250X_1^2+0.000135X_2X_1+0.000000347X_2^2-0.001406X_3X_1-0.000161X_3X_2-+0.000156X_3^2	0.93	0.0073**
Color	L	Y_3=-19.619792+0.257396X_1+0.144198X_2+3.854531X_3+0.034375X_1^2+0.003177X_2X_1-0.000054167X_2^2-0.142969X_3X_1-0.011109X_3X_2-0.053984X_3^2	0.53	0.6627
	a	Y_4=-0.202153+0.061875X_1+0.011611X_2+0.121146X_3+0.013750X_1^2+0.000312X_2X_1-0.000001736X_2^2-0.013438X_3X_1-0.001135X_3X_2+0.003438X_3^2	0.28	0.9656
	b	Y_5=0.443750+0.151667X_1+0.00917X_2-0.060938X_3-0.000625$_1^2$-0.000271X_2X_1-0.000005208X_2^2-0.003750X_3X_1+0.000031250X_3X_2+0.003203X_3^2	0.55	0.6232
Texture properties	Hardness	Y_6=124.151181+29.643958X_1-0.309701X_2+3.393646X_3-3.236563X_1^2+0.008146X_2X_1-0.000344X_2^2+0.329637X_3X_1+0.018083X_3X_2-0.121875X_3^2	0.94	0.0054**
	Cohesiveness	Y_7=48.302500+5.537813X_1+0.110781X_2+2.463906X_3-0.133438X_1^2-0.002469X_2X_1-0.000259X_2^2-0.244531X_3X_1-0.002703X_3X_2-0.002813X_3^2	0.52	0.6815
	Springiness	Y_8=67.544861-1.306042X_1-0.057882X_2+5.273854X_3+0.056875X_1^2+0.002771X_2X_1-0.000052431X_2^2+0.013437X_3X_1+0.002667X_3X_2-0.190859X_3^2	0.80	0.1270
	Gumminess	Y_9=-576.323958+55.692604X_1+-1.172406X_2+177.130990X_3-6.346250X_1^2+0.206885X_2X_1-0.007935X_2^2-2.817656X_3X_1-0.036182X_3X_2-3.725859X_3^2	0.94	0.0057**
Sensory evaluation	Hardness	Y_{10}=-1.165208+0.668958X_1+0.022354X_2+0.370521X_3-0.046875X_1^2-0.000229X_2X_1-0.000081250X_2^2-0.004375X_3X_1+0.000271X_3X_2-0.015547X_3^2	0.96	0.0016***
	Elasticity	Y_{11}=-5.312431+0.734063X_1+0.057462X_2+0.557344X_3-0.034063X_1^2-0.002406X_2X_1-0.000070139X_2^2+0.001406X_3X_1-0.001828X_3X_2-0.013438X_3^2	0.90	0.0235*
	Sweetness	Y_{12}=1.398056+0.505938X_1+0.019038X_2+0.053177X_3-0.052188X_1^2+0.000531X_2X_1-0.000076736X_2^2+0.001094X_3X_1+0.000005208X_3X_2-0.002500X_3^2	0.88	0.0346*
	Color	Y_{13}=-0.576528+0.815938X_1+0.002267X_2+0.437240X_3-0.056563X_1^2-0.000469X_2X_1-0.000014236X_2^2+0.004219X_3X_1+0.000286X_3X_2-0.021094X_3^2	0.90	0.0225*
	Flavor	Y_{14}=5.710208-0.206250X_1-0.006979X_2-0.199479X_3-0.023750X_1^2+0.003125X_2X_1-0.000028125X_2^2+0.012812X_3X_1+-0.000072917X_3X_2+0.005625X_3^2	0.68	0.3512
	Overall quality	Y_{15}=-11.189861+2.208646X_1+0.040392X_2+1.147656X_3-0.164375X_1^2-0.001073X_2X_1-0.000131X_2^2+0.002344X_3X_1+0.000391X_3X_2-0.048516X_3^2	0.93	0.0080**

[1)] X_1 is Citric acid content, X_2 is Sugar content, X_3 is Gelatin content and Y_1-Y_{15} are intensity score of the attributes.
[2)] R^2 is coefficient of determination.
*significant at p<.05 level, **significant at p<.01 level, ***significant at p<.005 level.

Table 5. Predicted level of optimum preparation for the maximized sensory properties of mulberry jelly by ridge analysis and superimposing of their response surfaces

Preparation condition	Level for maximum responses														
	Sweetness	pH	Color			Texture properties				Sensory evaluation					
			L	a	b	Y_1	Y_2	Y_3	Y_4	Y_5	Y_6	Y_7	Y_8	Y_9	Y_{10}
X_1	3.34	2.15	3.30	8.30	8.54	2.63	0.98	1.85	2.17	1.13	2.91	2.59	2.30	2.38	0.83
X_2	229.26	114.41	83.03	189.65	87.73	362.60	374.95	395.63	394.47	318.34	334.94	325.09	379.69	389.37	323.80
X_3	11.44	13.29	16.52	6.35	6.89	3.06	5.99	6.29	5.76	8.32	6.32	8.00	2.91	5.43	7.35
Morphology	Sad[1)]	Sad	Sad	Sad	Sad	Max	Sad	Sad	Max[1)]	Max	Sad	Max	Max	Max	Max

[1)] Max.: Maximum [2)] S.P.: Saddle point [3)] Min.: Minimum
X_1: Citric acid X_2: Sugar X_3: Gelatin
Y_1: Hardness Y_2: Cohesiveness Y_3: Springiness Y_4: Gumminess Y_5: Hardness Y_6: Elasticity Y_7: Sweetness Y_8: Color Y_9: Flavor Y_{10}: Overall quality

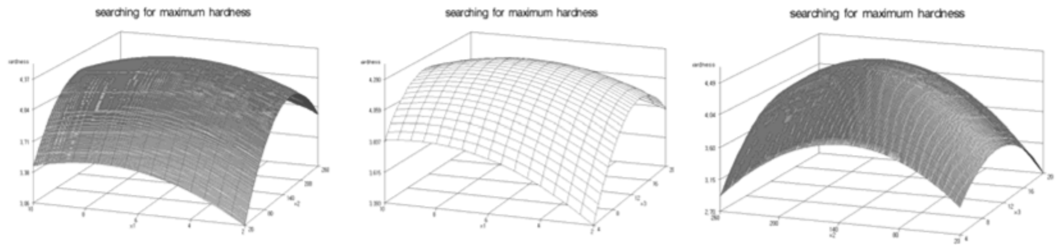

Fig. 1. Response surface for hardness of mulberry jelly.

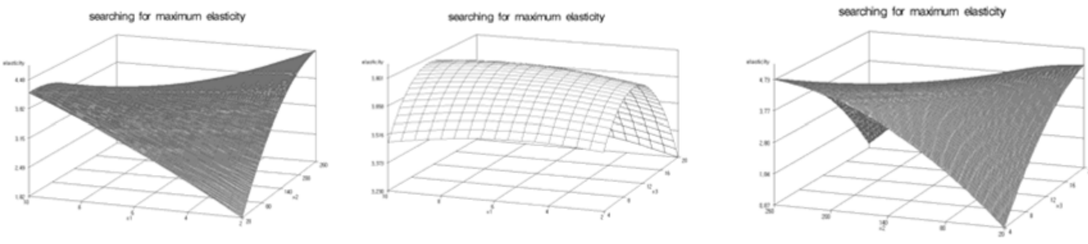

Fig. 2. Response surface for elasticity of mulberry jelly.

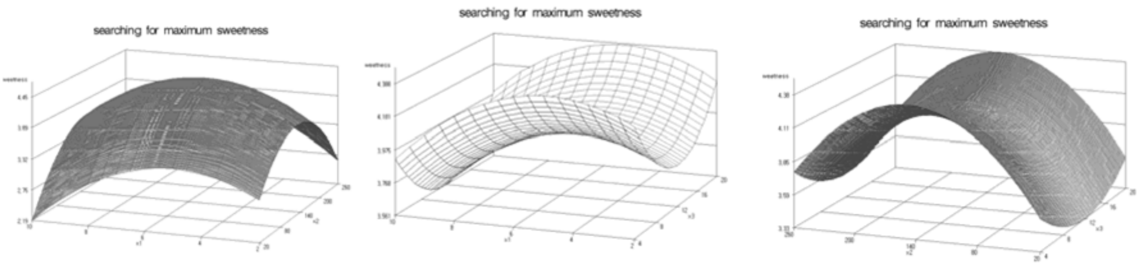

Fig. 3. Response surface for sweetness of mulberry jelly.

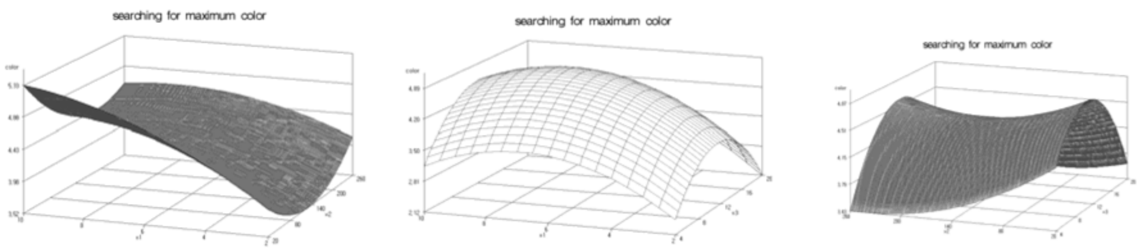

Fig. 4. Response surface for color of mulberry jelly.

Figure 2 represents the elasticity of mulberry at the sensory test for mulberry with different mixing ratios. The 3-dimentional graph shows the interaction of citric acid, sugar, and gelatin content by seeing the recovering ability of mulberry by pressing mulberries with a finger. The elasticity of mulberry revealed the significant differences with a probability level of 0.05 (α=.05) with a regression coefficient (R^2) of 0.90 (Table 4). Sugar was found to have the most significant effect on the elasticity, which was followed by gelatin, and citric acid. The material content of mulberry jelly showing the optimum point of elasticity was found to be 4.10 g of citric acid, 220.41 g of sugar, and 7.43 g of gelatin.

Figure 3 represents the favorability of sweetness in the sensory test of mulberries produced with different mixing ratios. The peak point was found to be the maximum point at the citric acid$\times$sugar, and the peak points of the citric acid$\times$gelatin and sugar$\times$gelatin are seen to be saddle point. The favorability of mulberry sweetness resulted to show significant difference with a probability level of 0.05 (α=.05) with the regression coefficient (R^2) of 0.88 (Table 4). Sugar was found to have the most significant effect on the sweetness, which was followed by gelatin, and citric acid. The material content of mulberry jelly showing the optimum point of sweetness was found to be 5.70 g of citric acid, 114.21 g of sugar, and 12.03 g of gelatin.

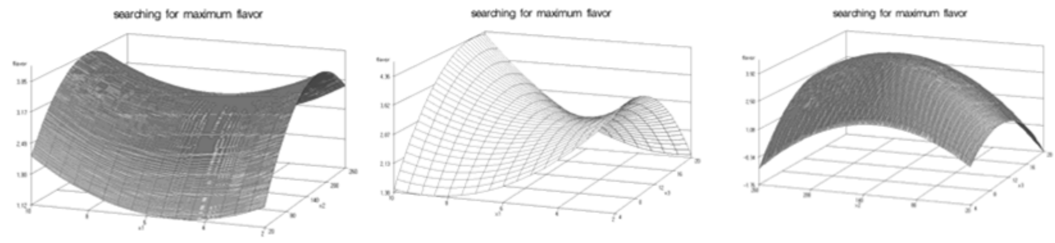

Fig. 5. Response surface for flavor of mulberry jelly.

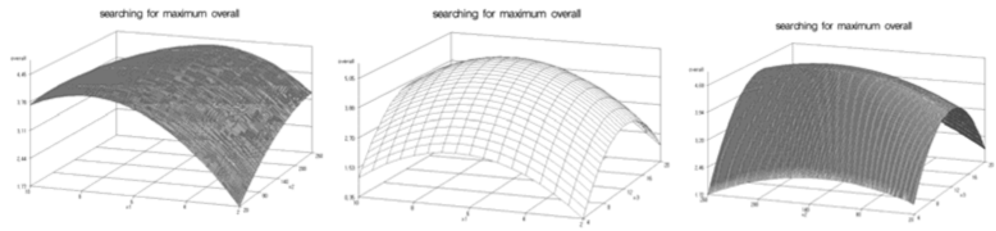

Fig. 6. Response surface for overall quality of mulberry jelly.

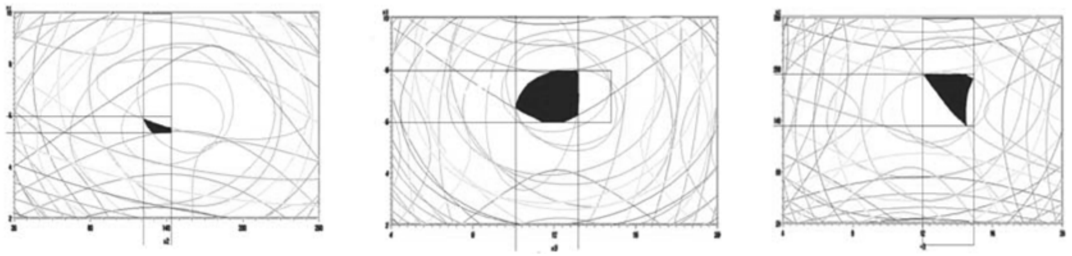

Fig. 7. Optimal condition of mulberry jelly in sensory quality.

The peak point was found to be the maximum point corresponding to citric acid×gelatin, and the peak points of sugar×gelatin and citric acid×sugar were found to be the saddle point. The favorability of mulberry color resulted to show significant difference with a probability level of 0.05 (α=.05) with the regression coefficient (R^2) of 0.90 (Table 4). Gelatin was found to have the most significant effect on the Color, which was followed by citric acid, and sugar. A similar result was found from a study by Park & Joo (Park and Joo 2006) that reported an increase in gelatin content contributed to an increase the sensory score for the optimization of noni jelly manufacture by using the response surface analysis method. The material content of mulberry jelly showing the optimum point of color was found to be 7.33 g of citric acid, 75.75 g of sugar, and 11.61 g of gelatin.

Flavor was found to have a higher sensory score as it is close to the centroid. The results of conducting the regression analysis by using an assumed regression model for the results on flavor revealed no significance at the probability level of 0.05 (α=.05) with a regression coefficient (R^2) of 0.68 by showing low confidence suggesting the necessity to conduct the multiple regression analysis (Table 4).

Figure 6 represents the overall quality with different mixing ratios of mulberries in a 3-dimentional graph. At the citric acid×sugar, citric acid×gelatin and sugar×gelatin, the peak points were found to be the maximum points, and the maximum mixing ratio for overall quality could be acquired from the peak point coordinates. The overall quality of mulberry showed significant differences with a probability level of 0.01 (α=.01) with a regression coefficient (R^2) of 0.93 (Table 4). Gelatin was found to have the most significant effect on the overall quality, which was followed by citric acid, and sugar. The material content of mulberry jelly showing the optimum point for overall quality was found to be 6.33 g of citric acid, 147.11 g of sugar, and 12.57 g of gelatin.

Table 6. Optimum process conditions for maximum response of hardness, elasticity, sweetness, color and overall quality by superimposition their contour maps

Process conditions	Range of optimum condition	Optimum condition
Citric acid (g)	6.1-6.3	6.2
Sucrose (g)	132-150	141.0
Gelatin (g)	12.2-14.7	13.5

Based on the overlapped part of categories (Fig. 7), in the range of factors that satisfy all sensory categories, the value located in the middle was calculated, and the optimization point was obtained which was found to be 6.2 g citric acid, 141.0 g sugar, and 13.5 g gelatin (Table 6).

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