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UTILIZATION OF RESPONSE SURFACE METHODOLOGY IN THE OPTIMIZATION OF ROSELLE ICE CREAM MAKING

[Penggunaan Response Surface Methodology dalam Optimisasi Pembuatan Es Krim Rosella]

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ABSTRACT

This research was carried out to develop a functional ice cream product with natural colorant derived from an optimum set of roselle calyces extract and citric acid concentrations. Although citric acid can improve red color stability of rosella, its addition is limited due to the acidic and bitter aftertaste it imparts. Response surface methodology (RSM) was employed to analyze the effect of roselle calyces extract and citric acid on physico-chemical characteristics and sensory acceptance of an ice cream. A central composite design consisting of two independent variables (roselle calyces extract and citric acid cocentrations) at five levels (-1.41421, -1, 0, +1, and +1.41421) with 13 runs (formulations) was prepared to establish the optimum set of variables. Higher concentration of roselle calyces extract significantly increased the total anthocyanin content and color acceptance, while decreased the °Hue and pH of the ice cream. Higher concentration of citric acid significantly increased the overrun and color acceptance, but decreased the viscosity, °Hue, pH, texture, taste acceptance, and overall acceptance of ice cream. The optimum scores of consumer sensory acceptance were met at 11.5% roselle calyces extract and 1.5% citric acid concentrations.

Keywords: citric acid, ice cream, optimization, response surface methodology, roselle calyces extract

ABSTRAK

Penelitian ini dilakukan untuk mengembangkan produk es krim fungsional dengan pewarna alami dari kelopak rosella pada konsentrasi ekstrak kelopak rosella dan asam sitrat optimum. Walau asam sitrat dapat meningkatkan stabilitas warna merah rosella, penambahannya dibatasi oleh aftertaste *rasa pahit yang dihasilkan.* Response surface methodology *(RSM) digunakan untuk menganalisa pengaruh konsentrasi ekstrak kelopak rosella dan asam sitrat terhadap sifat fisiko-kimia dan penerimaan es krim.* Central composite design *yang terdiri dari dua variabel independen (konsentrasi ekstrak bunga rosella dan asam sitrat) pada lima level (-1.41421, -1, 0, +1, and +1.41421) dalam 13 ulangan (formulasi) disiapkan untuk menetapkan titik optimum. Konsentrasi ekstrak kelopak rosella yang lebih tinggi secara signifikan meningkatkan kandungan antosianin dan penerimaan terhadap warna, namun mengurangi °Hue dan pH es krim. Konsentrasi asam sitrat yang semakin tinggi secara signifikan meningkatkan overrun dan penerimaan terhadap warna, namun mengurangi viskositas, °Hue, pH, tekstur, penerimaan terhadap rasa, dan penerimaan keseluruhan es krim. Skor optimum untuk penerimaan sensori ditetapkan pada konsentrasi ekstrak kelopak rosella 11.5% dan asam sitrat 1.5%.*

***Kata kunci**: asam sitrat, ekstrak kelopak rosella, es krim, optimisasi,* response surface methodology

INTRODUCTION

Color is an extremely important quality parameter and is directly related to the food acceptability. Since ice cream should have a delicate, attractive color that readily suggests to the consumer what the flavor is, most flavors of ice cream require the addition of at least a small amount of color (Marshall and Arbuckle, 2000; Durge *et al.* 2013). Carmoisine, ponceau 4R, and carmine are food colors which are most often used to impart red color in ice cream (Deshpande, 2002). However, due to their toxicity, they are banned in some countries (Saddleback, 2009; Amin *et al.* 2010). Consequently, there is an increase concern in research of colorants as their substitution in ice cream.

Roselle (*Hibiscus sabdariffa* L.) calyces contain anthocyanins, Dp-3-sambubioside and Cy-3-sambubioside (70.9:29.1), which are responsible for the brilliant red color of roselle calyces. In addition, anthocyanins possess antioxidant properties which may account for some of beneficial effects against diseases (Ilori and Odukoya, 2005). Due to their unique color and flavor, long history of safe usage, and no reported toxicity; the calyces are commonly used to make jellies, jams, sauces, and beverages (Tsai *et al.* 2002; Mohd-Esa *et al.* 2010). Roselle calyces possess a potential to be utilized as natural colorant and antioxidant source in ice cream (Ilori and Odukoya, 2005; Cisse *et al.* 2011). However, the anthocyanins are most strongly colored at low pH (around 3) by exhibiting their well-known purple-red color, while at alkaline-neutral pH values, they are inherently unstable and are converted to their corresponding carbinol bases, which are colorless (O'Connell and Fox, 2001).

The concentration of roselle calyces extract and citric acid which can lower the pH will affect the degree of coloration of the products and the concentration is limited due to the acidic and

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bitter aftertaste they imparted (Mounigan and Badrie, 2007; D'Heureux-Calix and Badrie, 2004; Durge *et al*. 2013). Hence, this research is carried out to observe the effect of different concentrations of roselle calyces extract and citric acid on the physicochemical characteristics and consumer sensory acceptance of ice cream and determine the optimum set of roselle calyces extract and citric acid levels added to produce the most sensory acceptable ice cream.

MATERIAL AND METHODS

Materials

The main materials used for the making of ice cream are sun-dried roselle (*Hibiscus sabdariffa* L.) calyces (m.c. of 15.83%) obtained from Bogor.

Analyses of roselle calyces extract

The analyses performed were color measurement using chromameter (Konica Minolta CR-400) (Madeira *et al*. 2003; Duangmal *et al*. 2008), determination of pH using pH meter (AOAC, 2005), total soluble solid using hand refractometer (ATAGO) (Nielsen, 2010), extraction yield using rotary vacuum evaporator (Buchi R-210) (Zhang *et al.* 2007), total anthocyanin content using pH differential method (Christian and Jackson, 2009), total phenolic content using Folin-Ciocalteu method (Slinkard and Singleton, 1977), antioxidant activity analysis using DPPH method (Amin and Lee, 2005).

Determination of roselle calyces extract concentration

The determination of roselle calyces extract concentration used for ice cream making was based on the sensory evaluation using Consumer Rejection Threshold (CRT) method (Prescott *et al*. 2005) to determine the level limit of roselle calyces in ice cream which cannot be accepted anymore by the consumers (CRT value). Thirty panelists were involved. Six concentration of roselle calyces extract were used in the making of ice cream, which are 0.9, 1.8, 3.6, 7.2, 14.4, and 28.8%. CRT was measured using series of six paired preference tests, one for each roselle calyces extract concentration level.

Response surface experimental design

The experimental design used in this research is Response Surface Methodology for optimization which was conducted according to Central Composite Design (CCD) with a quadratic model (Lee *et al*. 2006; Mendes *et al*. 2001) to study the combined effect of two independent variables, which were roselle calyces extract and citric acid levels. Each independent variable had five levels, which were −1.41, −1, 0, +1, and +1.41. The concentration level below the CRT value was determined as the highest level of independent variable of roselle calyces extract concentration for optimization (+1.41421), while the lowest level (-1.41421) was set to 5%. A total of 13 combinations (including five replicates at the center point with each value coded as 0) were carried out in random order according to a CCD configuration for the two chosen variables. RSM was done using Minitab 14 software.

The production of ice cream

The extraction of roselle calyces was performed according to Tsai *et al.* (2002) with modification. The dried calyces were extracted with water using vacuum in the ratio of 1:10 (w/v) at 60ºC for 2 hours, then were rapidly filtered through filter cloth to obtain the roselle calyces extract. The making of ice cream was performed according to Handayani *et al.* (2009) with modification, the procedure of which can be seen in Figure 1. The ice cream formulation is according to the low-fat ice cream formulation by Marshall and Arbuckle (2000) with modification. Formulation of ice cream per 1 kg mix used in this research is shown in Table 1.

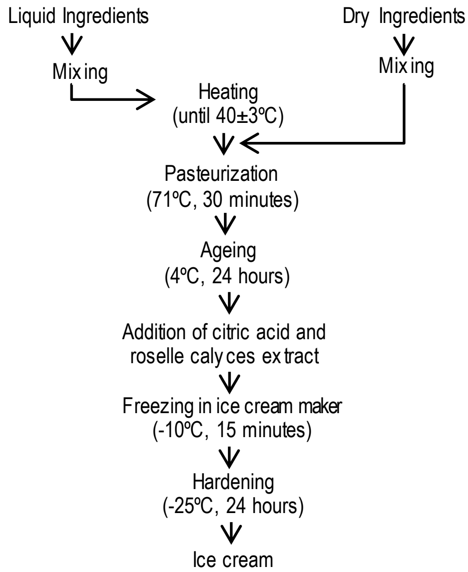

Figure 1. Flowchart of the making of ice cream
Source: Handayani *et al.* (2009) with modification

Table 1. Formulation of ice cream per 1 kg mix

Ingredient	Amount (g)
Cream	55.27
Skim milk powder	106.02
Sucrose	130.00
Maltodextrin 10.8 DE	50.00
CMC	3.50
Mono-diglyceride	3.50
Water	651.71
Roselle calyces extract	50 to 10 *X* (substitutes water in determined portion)
Citric acid	7.5 to 22.5

Note: *X* = highest level of independent variable of roselle calyces extract concentration; Source: Marshall and Arbuckle (2000) with modification

Physico-chemical characteristics and sensory analyses of ice cream

The physical analyses performed were determination of viscosity using Brookfield viscometer (Hwang *et al.* 2009), overrun (Marshall *et al.* 2003), meltdown test (Hwang *et al.* 2009), and color measurement using chromameter (Madeira *et al.* 2003; Duangmal *et al.* 2008). The chemical analyses performed were determination of pH using pH meter (AOAC, 2005; Hwang *et al.* 2009) and total anthocyanin content (Hwang *et al.* 2009). Sensory analysis was performed using scoring test on texture and melting characteristic in mouth; and hedonic test on color, aroma, taste, texture, melting characteristic in mouth, and overall.

Best formulation ice cream

The best formulation of ice cream was determined from the result of hedonic test, which was the one with optimum set of roselle calyces extract and citric acid levels. Its physical and chemical characteristics, as well as its consumer sensory acceptance were analyzed. The analyses performed were the same with the ones performed on ice cream in each design, in addition to the determination of total phenolic content using Folin-Ciocalteu method (Slinkard and Singleton, 1977; Hwang *et al.* 2009), antioxidant activity analysis using DPPH method (Hwang *et al.* 2009), chemical composition analysis using proximate analyses (AOAC, 2005).

RESULTS AND DISCUSSION

Characteristics of analyses of roselle calyces extract

Some analyses were performed on roselle calyces extract to observe its general characteristics, in which the result is shown in Table 2.

Table 2. Analyses result of roselle calyces extract

Parameter	Amount
°Hue	27.95±0.10 (red)
pH	2.48±0.04
TSS (°Brix)	4.45±0.00
Extraction yield (%)	46.41±0.79
Drying yield (%)	13.16±0.10
Total anthocyanin content (mg/g extract)	2.51±0.57
Total phenolic content (mg GAE/g extract)	13.07±0.15
IC_{50} (ppm)	6.728.60±110.82

According to Cisse *et al.* (2011), the pH, total soluble solid, anthocyanin content, and phenolic content of roselle calyces extract are 2.30±0.05, 45±2 g/kg, 7.1±0.5 g/kg TSS, and 29.1±1.0 g/kg TSS, respectively. There is no significant difference between the result obtained and result from Cisseet *et al.* (2011) in terms of pH and TSS. However, the anthocyanin content and phenolic content are significantly lower, which may be caused by the different condition of plantation, drying, and storage of roselle. The pH of the roselle calyces extract is quite low due to the presence of various acid compounds, dominated by citric acid and hibiscus acid, which impart acidic taste (Mounigan and Badrie, 2007; D'Heureux-Calix and Badrie, 2004). The anthocyanins pigments, Dp-3-sambubioside and Cy-3-sambubioside, concentrated in the roselle calyces are responsible for the red color of roselle calyces extract (Ilori and Odukoya, 2005; Cisse *et al.* 2011). Hence, it can be utilized as natural colorant. The IC_{50} value shows that it possesses antioxidant properties and hence it can be considered as antioxidant source.

Concentration of roselle calyces extract

By counting the number of panelists preferring control sample in each pair of samples, the proportion can be obtained which then is plotted. By using 5% significance criterion (0.7) as a function of roselle calyces extract concentrations based on binomial distribution tables for paired preference tests (N = 30), the CRT value can be obtained (Prescott *et al.* 2005), as shown in Figure 2.

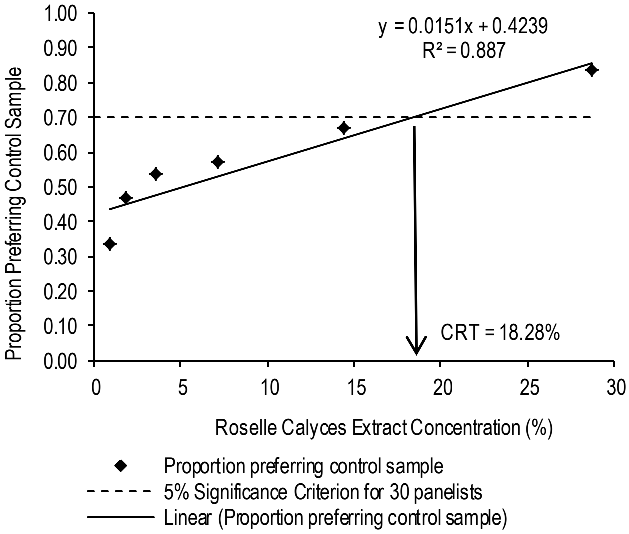

Figure 2. Proportion of panelists preferring control sample

The graph in Figure 2 shows that the CRT value of roselle calyces extract concentration in ice cream is 18.28%. Hence, for the making of ice cream, the roselle calyces extract concentration as the highest level of independent variable of roselle calyces extract concentration for optimization (+1.41421) is lower than 18.28%, which is 18%. The panelists or the consumers were not able to accept ice cream with the addition of roselle calyces extract more than that limit because roselle calyces has acidic and bitter aftertaste due to the presence of various acid compounds, which are dominated by citric acid and hibiscus acid (Mounigan and Badrie, 2007; D'Heureux-Calix and Badrie, 2004).

Response surface experimental design

The roselle calyces extract concentration obtained from CRT method, which is 18%, was set as the highest level of independent variable of roselle calyces extract concentration for optimization (+1.41421) in the response surface design, while the lowest level (-1.41421) is set to 5%. The variables and their levels are shown in Table 3, while the experimental design is shown in Table 4.

Table 3. Variables for optimization

Variables	Symbols	Levels (%)				
		-1.41421	-1	0	1	+1.41421
Roselle calyces extract concentration	X_1	5.00	6.90	11.50	16.10	18.00
Citric acid concentration	X_2	0.75	0.97	1.5	2.03	2.25

Table 4. Central composite experimental design for optimization

Design	Codified		Decodified	
	X_1	X_2	Roselle calyces Extract Concentration (%)	Citric Acid Concentration (%)
1	-1	-1	6.90	0.97
2	1	-1	16.10	0.97
3	-1	1	6.90	2.03
4	1	1	16.10	2.03
5	-1.41421	0	5.00	1.50
6	1.41421	0	18.00	1.50
7	0	-1.41421	11.50	0.75
8	0	1.41421	11.50	2.25
9	0	0	11.50	1.50
10	0	0	11.50	1.50
11	0	0	11.50	1.50
12	0	0	11.50	1.50
13	0	0	11.50	1.50

Optimized physical characteristics

Some physical characteristics optimized includes: viscosity, overrun, meltdown properties and color. Figure 3 (a) and (b) show the contour and surface plot of viscosity, respectively. Citric acid percentage significantly ($p \leq 0.05$) affects the viscosity. On the other hand, roselle percentage does not significantly ($p > 0.05$) influence the viscosity.

Figure 4 (a) and (b) show the contour and surface plot of overrun, respectively. Citric acid percentage significantly ($p \leq 0.05$) increases the overrun, which is coherent with the result of viscosity determination in which there is decreasing of viscosity along with the increasing of citric acid concentration used. According to Marshall and Arbuckle (2000), as the viscosity of the mix decreases, the rate of whipping increases. It is due to the decrease of mix surface tension which causes the air to be easier to go through the surface of the mix to be incorporated into the mix during aeration process, and therefore the overrun is higher. On the other hand, roselle percentage does not significantly ($p > 0.05$) influence the overrun.

Figure 5 (a) and (b) show the contour and surface plot of melting rate, respectively. Roselle percentage significantly ($p \leq 0.05$) affects the melting rate. On the other hand, citric acid percentage does not significantly ($p > 0.05$) influence the melting rate.

The °Hue of ice cream in various designs ranged from 13.02 to 51.40°. The °Hue in the range of 342-18° represents the red purple color, 18-54° represents red color, and 54-90° represents yellow red color (Hutchings, 1999). Hence, the color of ice cream samples is ranging from red purple to red, in which the lower °Hue represents darker red color which shows higher intensity of red color. Figure 6 (a) and (b) show the contour and surface plot of °Hue, respectively.

Roselle percentage significantly ($p \leq 0.05$) decreases the °Hue, which is coherent with the result of total anthocyanin content and pH determination in which there is increasing of total anthocyanin content and decreasing of pH of ice cream along with the increasing of roselle calyces extract concentration used. Anthocyanins pigments which are concentrated in roselle calyces, are responsible for the brilliant red color of roselle calyces (Ilori and Odukoya, 2005). Hence, higher concentration of roselle calyces extract added to the ice cream gives higher pigment concentration, and therefore the intensity of red color in the ice cream increases as the increasing of roselle calyces extract concentration, shown by the lower °Hue value. Higher concentration of roselle calyces extract added to the ice cream imparts more acid compounds, and therefore the acidity of the ice cream increases (decreasing of pH) as the increasing of roselle calyces extract concentration. Acidic pH (low pH) favours the appearance of the colored forms, the flavyliumcation AH^+ which is red (Selim *et al.* 2008; D'Heureux-Calix and Badrie, 2004). Hence, higher concentration of roselle calyces extract added to the ice cream increased the red color exhibition of roselle anthocyanins, and therefore the intensity of red color in the ice cream increases as the increasing of roselle calyces extract concentration, shown by the lower °Hue value.

Citric acid percentage significantly ($p \leq 0.05$) decreases the °Hue, which is coherent with the result of pH determination in which there is decreasing of pH of the ice cream along with the increasing of citric acid concentration used. Citric acid has the ability to lower the pH, and hence the pH of ice cream decreases as the increasing of citric acid concentration added (Smith and Hong-Shun, 2003; Durge *et al.* 2013). Acidic pH (low pH) favors the appearance of the colored forms, the flavyliumcation AH^+ which is red (Selim *et al.* 2008; D'Heureux-Calix and Badrie, 2004). Hence, higher concentration of citric acid added to the ice cream increased the red color exhibition of roselle anthocyanins, and therefore the intensity of red color in the ice cream increases as the increasing of citric acid concentration, shown by the lower °Hue value.

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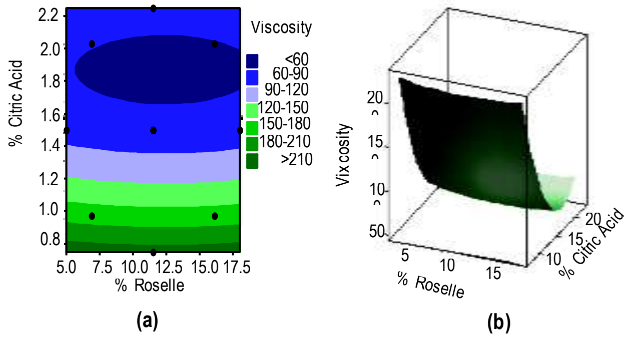

Note: The dots show the points of design in CCD

Figure 3. (a) Contour plot and (b) Surface plot of viscosity vs citric acid (%), roselle (%)

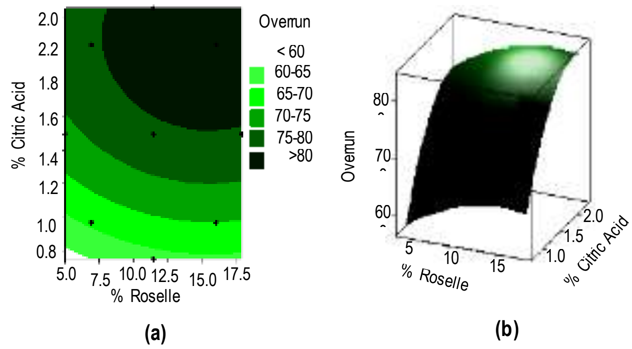

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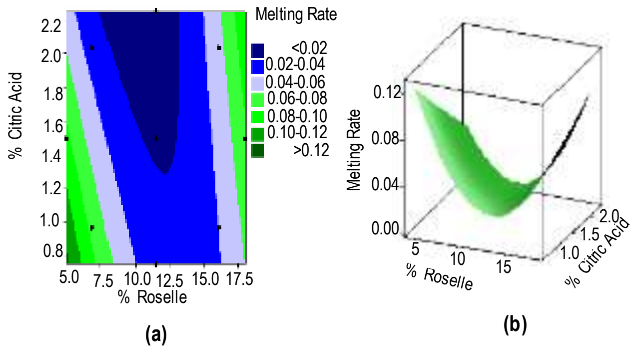
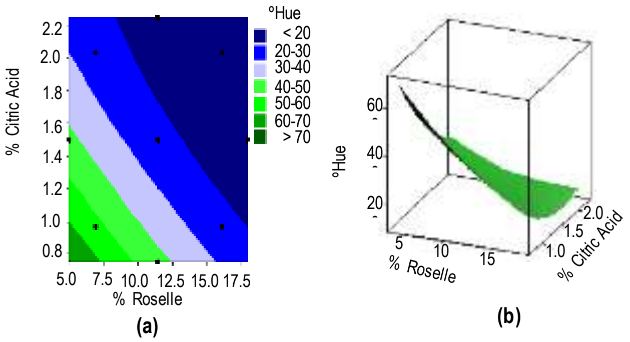
Figure 4. (a) Contour plot and (b) Surface plot of overrun vs citric acid (%), roselle (%)



Note: The dots show the points of design in CCD

Figure 5. (a) Contour plot and (b) Surface plot of melting rate vs citric acid (%), roselle (%)



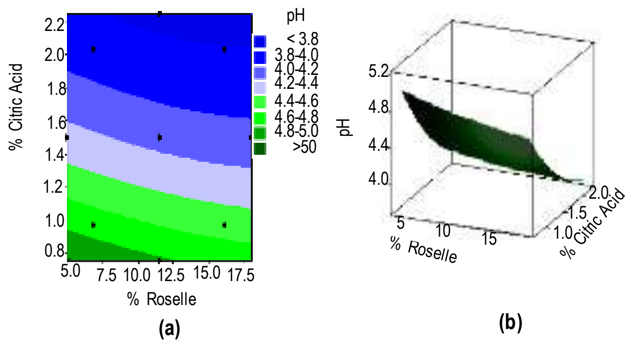
Note: The dots show the points of design in CCD

Figure 6. (a) Contour plot and (b) Surface plot of °Hue vs citric acid (%), roselle (%)

Optimized chemical characteristics

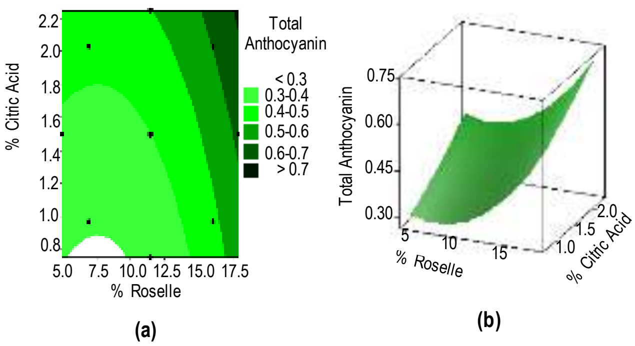
Figure 7 (a) and (b) show the contour and surface plot of pH, respectively. Roselle percentage significantly (*p*≤0.05) decreases the pH. The result of pH determination of roselle calyces extract shows that the pH of roselle calyces extract is quite low, which is 2.48. It is due to the presence of various acid compounds, which are dominated by citric acid and hibiscus acid, in roselle calyces. Malic acid and tartaric acid are also present in roselle calyces (Mounigan and Badrie, 2007; D'Heureux-Calix and Badrie, 2004). Hence, higher concentration of roselle calyces extract added to the ice cream imparts more acid compounds, shown by the lower value of pH of ice cream. Citric acid percentage significantly (*p*≤0.05) decreases the pH. Citric acid as the pH control agents has the ability to lower the pH (Smith and Hong-Shun, 2003; Durge *et al.* 2013).

Figure 8 (a) and (b) show the contour and surface plot of total anthocyanin content, respectively. Roselle percentage significantly (*p*≤0.05) affects the total anthocyanin content. According to Duangmal *et al.* (2008), per 100 grams dry weight of roselle calyces extract, there is about 1.5 g total anthocyanins, which consists of Dp-3-sambubioside and Cy-3-sambubioside (70.9:29.1), and also small amount of Dp-3-glucoside and Cy-3-glucoside. Hence, higher concentration of roselle calyces extract added to the ice cream imparts more anthocyanins, shown by the higher total anthocyanin content of ice cream. Citric acid percentage does not significantly (*p*>0.05) influences the total anthocyanin content.



Note: The dots show the points of design in CCD

Figure 7. (a) Contour plot and (b) Surface plot of pH vs citric acid (%), roselle (%)



Note: The dots show the points of design in CCD

Figure 8. (a) Contour plot and (b) Surface plot of total anthocyanin content vs citric acid (%), roselle (%)

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Optimized sensory properties

Texture and melting characteristics in mouth are analyzed using panel to obtain the optimal ice cream. Figure 9 (a) and (b) show the contour and surface plot of texture (1 (extremely coarse) – 5 (extremely smooth)), respectively. Citric acid percentage significantly ($p \le 0.05$) decreases the texture of ice cream. On the other hand, roselle percentage does not significantly ($p > 0.05$) influence the texture.

Figure 10 (a) and (b) show the contour and surface plot of melting characteristic in mouth (1 (extremely quickly melt) – 5 (extremely slowly melt)), respectively. Roselle percentage significantly ($p \le 0.05$) affects, meanwhile citric acid percentage did not affect the melting characteristic in mouth.

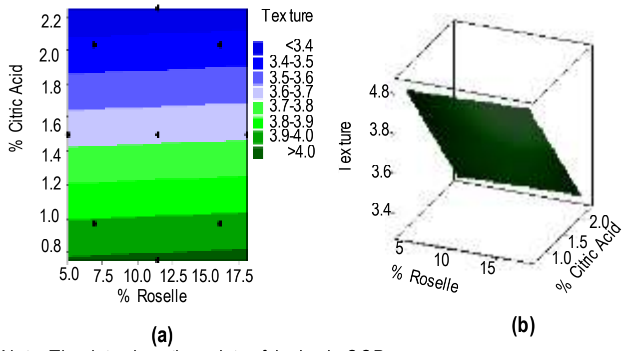

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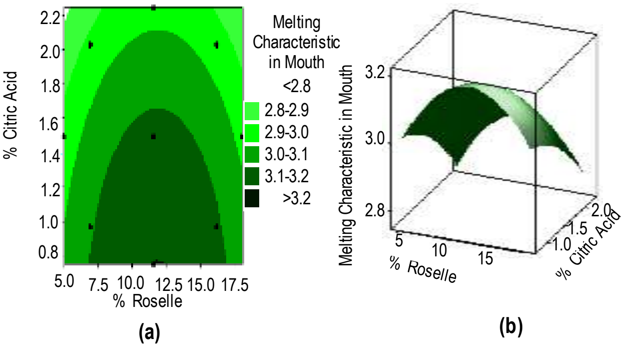
Figure 9. (a) Contour plot and (b) Surface plot of texture vs citric acid (%), roselle (%)

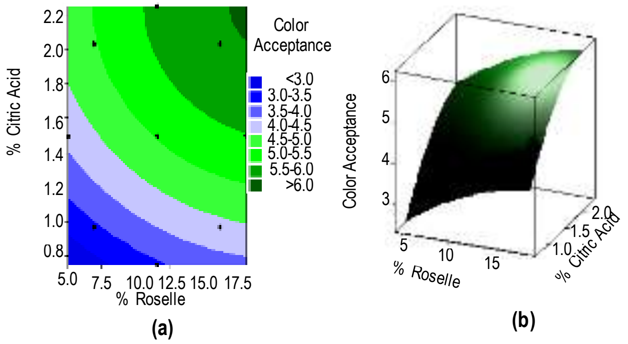


Note: The dots show the points of design in CCD

Figure 10. (a) Contour plot and (b) Surface plot of melting characteristic in mouth vs citric acid (%), roselle (%)

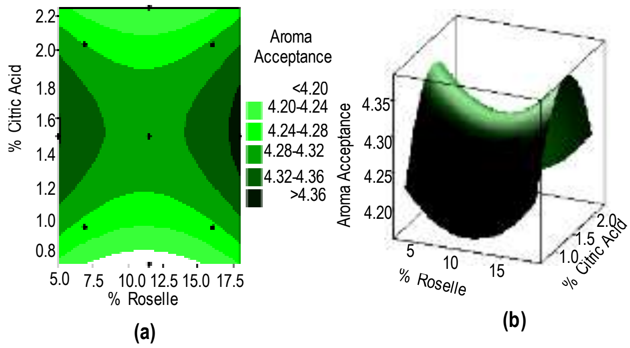
Optimized sensory acceptance

The acceptance of panelists was optimized in terms of color, aroma, taste, texture, and overall parameters. Figure 11 (a) and (b) show the contour and surface plot of color acceptance, respectively. Both roselle and citric acid percentage significantly ($p \le 0.05$) increases the color acceptance, coherent with the result of color measurement in which there is decreasing of °Hue (increasing of red color intensity) of the ice cream along with the increasing of concentration of roselle calyces extract and citric acid used. Hence, the consumers prefer the ice cream to be in redder color. It is supported by the research done by Duangmal *et al.* (2008) which revealed that the observers preferred a roselle-colored drink to be in a shade of vivid deep red to pale dull red. Food colors are added to improve the acceptance of food products (Mudambi *et al.* 2006). In this research, the addition of roselle calyces extract is proven to improve the color acceptance of ice cream.



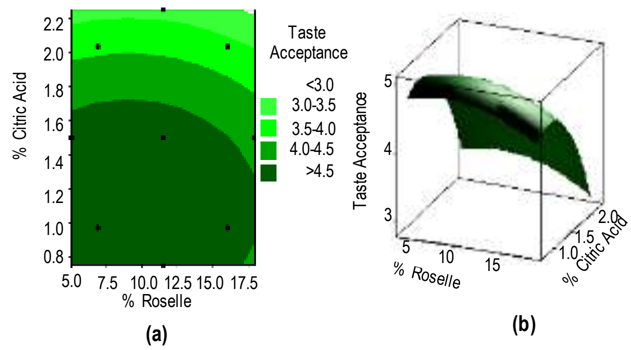
Note: The dots show the points of design in CCD

Figure 11. (a) Contour plot and (b) Surface plot of color acceptance vs citric acid (%), roselle (%)



Note: The dots show the points of design in CCD

Figure 12. (a) Contour plot and (b) Surface plot of aroma acceptance vs citric acid (%), roselle (%)



Note: The dots show the points of design in CCD

Figure 13. (a) Contour plot and (b) Surface plot of taste acceptance vs citric acid (%), roselle (%)

Figure 12 (a) and (b) show the contour and surface plot of aroma acceptance, respectively. Neither roselle nor citric acid percentage significantly ($p \le 0.05$) affects the ice cream aroma acceptance. Figure 13 (a) and (b) show the contour and surface plot of taste acceptance, respectively. Citric acid percentage significantly ($p \le 0.05$) decreases the taste acceptance, coherent

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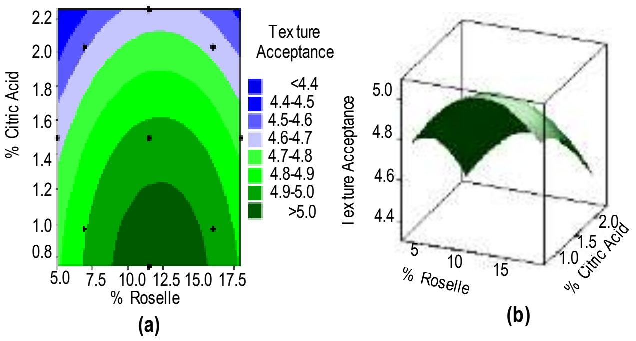
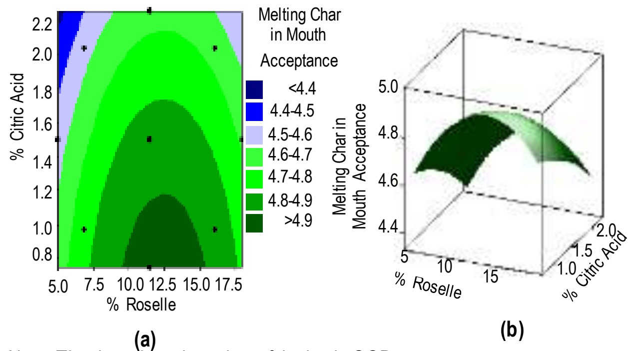
with the result of pH determination in which there is decreasing of ice cream pH along with the increasing of citric acid concentration. Hence, the consumers prefer the ice cream to be in less acid taste. Roselle percentage does not significantly (p>0.05) influence the ice cream taste acceptance.



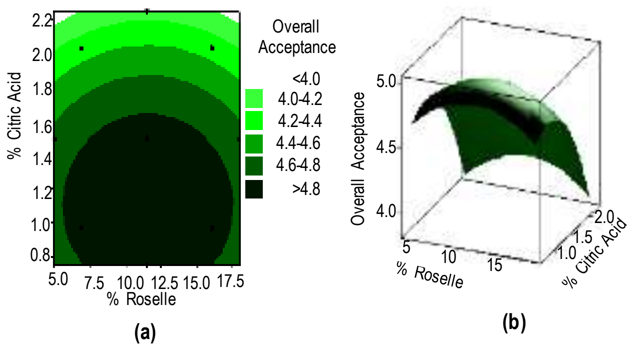
Note: The dots show the points of design in CCD

Figure 14. (a) Contour plot and (b) Surface plot of texture acceptance vs citric acid (%), roselle (%)



Note: The dots show the points of design in CCD

Figure 15. (a) Contour plot and (b) Surface plot of melting characteristic acceptance vs citric acid (%), roselle (%)



Note: The dots show the points of design in CCD

Figure 16. (a) Contour plot and (b) Surface plot of overall acceptance vs citric acid (%), roselle (%)

Figure 14 (a) and (b) show the contour and surface plot of texture acceptance, respectively. Neither roselle nor citric acid percentage significantly (p≤0.05) affects the ice cream texture acceptance. Figure 15 (a) and (b) show the contour and surface plot of melting characteristic in mouth acceptance, respectively. Roselle percentage significantly (p≤0.05) affects the melting characteristic in mouth acceptance in the trend that is coherent with the result of scoring test on melting characteristic in mouth in which it increases (slower melting) up to certain point, then decreases (quicker melting), along with the increasing of roselle calyces extract level. Hence, the consumers prefer the ice cream to be melted slower in mouth. Citric acid percentage does not significantly (p>0.05) influence the melting characteristic in mouth acceptance. Figure 16 (a) and (b) show the contour and surface plot of overall acceptance, respectively. Citric acid percentage significantly (p≤0.05) affects the overall acceptance. On the other hand, roselle percentage does not significantly (p>0.05) influence the ice cream overall acceptance.

Best formulation ice cream

The best formulation ice cream is determined by finding the best combination of independent variables (percentage of roselle and citric acid) to maximize the response functions of ice cream consumer sensory acceptance (color, taste, melting characteristic in mouth, and overall acceptance). These criteria are selected since the acceptability information is extremely useful in determining the optimal design of food products since consumer is the ultimate judge, therefore these characteristics need to be assessed in relation to the acceptability of the product to the consumer (Lawless and Heymann, 2010). The response functions of aroma acceptance and texture acceptance are not included in the determination of best combination because different concentrations of roselle calyces extract and citric acid have no significant effect on these two parameters.

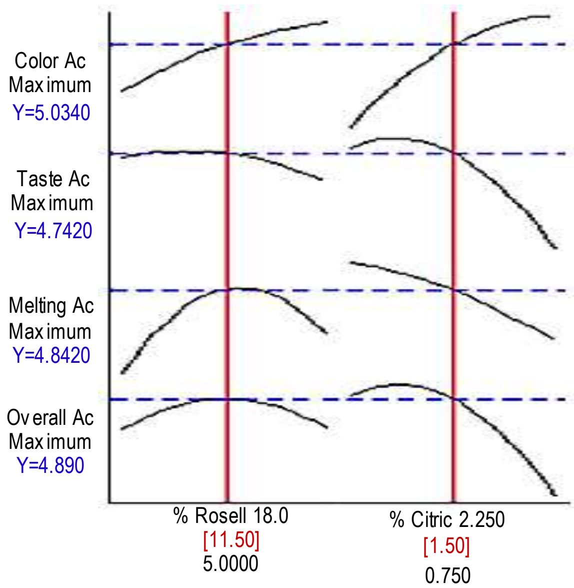


Figure 17. Response optimization

From the optimization plot in Figure 17, 11.5% roselle and 1.5% citric acid are obtained as the optimal solution for the variable combination in which the composite desirability is 0.86353. So, the ice cream which is made with combinations of 11.5% roselle calyces extract and 1.5% citric acid will have the highest consumer sensory acceptance among ice cream with other formulation.

Table 5 shows the result of analyses performed on the best formulation ice cream. The viscosity and overrun of the best formulation ice cream is in the range for typical ice cream. The melting rate is a little bit too small and undesirable, but this melting characteristic is acceptable to the consumers. The pH is lower than normal ice cream due to the presence of acid compounds imparted by the addition of roselle calyces extract and citric acid. In this low pH, the roselle anthocyanin is more stable and expressed red color in the ice cream which is acceptable to the consumers. Although the low pH imparts acid taste to the ice cream, this taste is in the level of sensory acceptable to the consumers. The best formulation ice cream is also acceptable to the consumers in terms of aroma, texture, and overall.

Table 5. Result of analyses of best formulation ice cream

	Parameter	Amount
Physical	Viscosity (cPs)	72.12±0.48
	% Overrun	78.80±0.65
	Melting rate (g/min)	0.02±0.005
	ºHue	23.41±0.50 (red)
Chemical/Nutrition	pH	4.13±0.01
	Total anthocyanin content (mg/g ice cream)	0.40±0.01
	Total phenolic content (mg GAE/g ice cream)	2.04±0.10
	% Scavenging effect	54.62±1.45
	Moisture content (%)	69.90±0.01
	Ash content (%)	0.39±0.01
	Protein content (%)	2.46±0.15
	Fat content (%)	3.00±0.15
	Carbohydrate content (%)	24.25±0.30
Sensory	Texture*	3.51±0.28
	Melting characteristic in mouth*	3.12±0.05
	Color acceptance**	5.03±0.18
	Aroma acceptance**	4.31±0.09
	Taste acceptance**	4.74±0.23
	Texture acceptability**	4.93±0.20
	Melting characteristic in mouth acceptance**	4.84±0.13
	Overall acceptance**	4.89±0.17

Note:
* Texture : 1 (extremely coarse) – 5 (extremely smooth)
Melting characteristic in mouth : 1 (extremely quickly melt) – 5 (extremely slowly melt)
** 1 (extremely dislike) – 7 (extremely like)

The best formulation ice cream contains phenolic compounds and possesses antioxidant properties, shown by the scavenging effect. The fat content is a bit higher than the formulation which may be due to the fat content imparted by the roselle calyces. However, it is still in the range of fat content for low-fat ice cream, hence the ice cream is still categorized as low-fat ice cream. The moisture content is also a bit higher than the formulation. The ash content is imparted by the mineral content in skim milk powder and roselle calyces. The protein content is a bit lower than the average values of protein content in low-fat ice cream. The carbohydrate content is higher than the average values of carbohydrate in low-fat ice cream which may be due to the high carbohydrate content in roselle calyces.

CONCLUSIONS

Higher concentration of roselle calyces extract significantly increased the total anthocyanin content and color acceptance of ice cream. In contrast, it significantly decreased the ºHue and pH of ice cream. Different concentrations of roselle calyces extract significantly affected the melting rate, melting characteristic in mouth, and melting characteristic in mouth acceptance of ice cream, but the trends were unclear.

Higher concentration of citric acid significantly increased the overrun and color acceptance of ice cream. However, it significantly decreased the viscosity, ºHue, pH, texture, taste acceptance, and overall acceptance of ice cream. The optimum set of roselle calyces extract and citric acid levels for making of ice cream were 11.5% roselle and 1.5% citric acid.

The color stability of roselle anthocyanins in ice cream during storage is needed to be observed, hence further research can be done by conducting the stability test of the pigments on ice cream. Moreover, the characteristics of the ice cream can be compared with the commercial ones. The antioxidant activity of roselle calyces in ice cream and factors affecting it can also be studied further.

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