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Optimisation of stabiliser combinations in instant ice cream mix powder formulation via mixture design methodology

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Abstract

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Keywords

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The present work aimed at obtaining the optimum combination of stabilisers used in an instant ice cream formulation. Instant ice cream mixes were formulated using milk protein, fat, sucrose, stabilisers, emulsifiers and water. A basic formulation for ice cream mix was prepared according to the process flow of instant ice cream. Three different stabilisers mixtures, which were carboxymethyl cellulose (CMC), carrageenan, and sodium alginate used in 14 formulations were studied using mixture design methodology. Physical analyses on meltdown rate, hardness, melted ice cream viscosity, and overrun were performed. The results were compared with those of commercial instant ice cream to determine the acceptability and quality of the final products. From the optimisation study, the recommended combination of stabilisers was found to be 0.151% CMC and 0.149% sodium alginate. The optimised sample had a higher viscosity of 0.17 Pa.s than the commercial one (0.16 Pa.s), which corresponded to higher hardness thus indicating slower melting rate, which is a good texture for ice cream. The overrun of the optimised sample was 108.33%, which is considered high. The high amount of air, viscosity, and hardness resulted in a low melting rate of 0.22 g/min, which is desirable for ice cream consumption. Based on sensory evaluation, the optimised instant ice cream was the most preferred by panellists as compared to commercial sample.

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Introduction

Ice cream is a popular dairy frozen product. It is consumed by people from all ages. The major constituents in an ice cream include fat, sugar, non-fat milk solid, flavours and stabilisers (Haghighimanesh and Farahnaky, 2011). In ice cream manufacturing, mixing, pasteurisation, homogenisation, and freezing are the important steps required to produce a good final texture of ice cream. Instant ice cream is developed to introduce a new ice cream product that can indirectly assist consumers in making their own ice cream at home. Instant ice cream is developed as an option of ice cream product that still consists almost the same ingredients with conventional hard and soft ice creams available in the market. The preparation of ice cream made using the ice cream mix is simple and time effective as most of the conventional steps of making hard ice cream are eliminated, hence making it more convenient and economical for consumers.

Nevertheless, the biggest challenge is to get the right proportion of ingredients in producing the final formulation of the instant ice cream mix powder. The present work thus determined the most suitable combination of stabilisers to be incorporated in the formulation.

The texture and acceptability of ice cream may be affected by several formulations and processing factors. According to BahramParvar et al. (2015), stabiliser is an ingredient that despite its low level of consumption in formulation, delivers specific and important functions to the finished product. Stabilisers help to improve the body, texture, melting properties, and retard ice crystal growth (Bahramparvar and Mazaheri Tehrani, 2011). Although there are many studies available in this area, there is no consensus on the mechanisms in which the stabilisers influence the freezing properties or restrict the ice recrystallisation (Marshall et al., 2003).

There are many kinds of stabiliser used in food

products. In ice cream manufacturing, it is always difficult to get all the properties of ice cream using a single stabiliser. Syed and Ullah Shah (2016) used trial and error method in obtaining several synergistic mixtures of different stabilisers to achieve good quality products. The overall cost of a stabiliser system can be reduced due to the small quantities used (Bahramparvar and Mazaheri Tehrani, 2011). According to Haghighimanesh and Farahnaky (2011), stabilisers are usually applied in the combination of two to three gums in an ice cream formulation. The stabilisers bring some positive effects especially for the viscosity to avoid the formation of ice crystals during processing and storage as well as to preserve the structure by slowing down the melting rate of ice cream. As reported by Ashland (2014), carboxymethyl cellulose (CMC) is commonly used in ice cream production as the ingredient to produce economical and premium ice creams and frozen desserts. They also claimed that CMC can be used to control water mobility and texture of ice cream, which help to stabilise against heat shock, control the size of ice crystal, give excellent overrun, and provide good melting profile. Hence, CMC could improve the melting profile of ice cream. Carrageenan is also available in several types; the most common are kappa, iota, and lambda. For the composition of low fat and soft serve ice cream, Kappa carrageenan is usually used due to its gel forming functionality and reactivity with casein that prevents whey separation. It is important to add carrageenan if an ageing step exists in the manufacturing process (Naresh and Shailaja, 2006). Sodium alginate is a salt of a linear poly (uronic acid), which is alginic acid obtained from brown seaweeds, and the solutions of sodium alginate are highly viscous (Fennema, 1996). The calcium salt of alginates is insoluble due to the reaction of calcium ions and G-block regions in the chain where the gel depends on both. Alginates help to add body and texture to ice cream.

Stabilisers improve air incorporation, air cell distribution, body or texture characteristics, storage stability, and melting properties. They also minimise the development of large ice crystals (Naresh and Shailaja, 2006). However, there is no comprehensive review available concerning various aspects of stabilisers in ice cream with their effects in ice cream that are unclear in practical situations.

The aim of the present work was to obtain the best combination of stabilisers to be used for producing instant ice cream. In general, D-optimal mixture design is used to determine the relationships between the proportions of different variables and responses (Chen *et al.*, 2010). Mixture design is a type of statistical experimental design that aids in obtaining the optimum formulation where the combination of ingredients is known. Hence, it reduces the trial experiments needed to evaluate the response and is a practical and effective tool for the optimisation of complex processes in industry. Thus, the effects of different stabilisers were studied to optimise these variables, which were viscosity, texture, and melting properties. Sensory evaluation to determine consumers' preference towards instant ice cream was also conducted.

Materials and methods

Materials

Milk powder, sugar, creamer, and vanilla flavouring were purchased from local retailers. Olein PK-10 (BIS Chemicals Sdn. Bhd., Selangor, Malaysia) was used as the emulsifier. Three types of stabiliser used were carboxymethyl cellulose (CMC) (Waris Nove Sdn. Bhd., Pahang, Malaysia), sodium alginate (BIS Chemicals Sdn. Bhd., Selangor, Malaysia) and carrageenan (BIS Chemicals Sdn. Bhd., Selangor, Malaysia).

Instant ice cream powder formulation and preparation

A preliminary study was conducted to obtain the final formulation of instant ice cream used in the present work. The ice cream formulations were based on 14.8% milk powder, 10.5% sucrose, 12.0% creamer, 0.4% emulsifier, 0.3% stabilisers, and 62% water. The combination of those ingredients was mixed with cold water and stirred at the highest speed for 5 min using hand-held mixer (Model BL1515, Khind, Malaysia) until the ice cream mix was whipped. After that, the samples were immediately stored in a freezer at -18°C for hardening process. Different types of stabiliser were used and chosen as variables in D-optimal mixture design as they may affect the responses. Each formulation was produced based on the mixture design (Table 1). The D-optimal mixture design method was used in the present work to determine the optimum proportion of stabiliser combination.

Melting characteristics

In order to study the melting behaviour of instant ice cream, the hardened ice cream was subjected to quantitative melting rate test. A 50 g sample was placed on a mesh grid (holes size of 1×1 mm). The sample was allowed to stand at ambient temperature for 30 min. The weight of melted ice cream was recorded at 5 min time interval.

Viscosity measurement

The measurement was conducted using a rheometer (AR-G2 TA Instruments, USA) coupled with a cone and plate system (d: 40 mm, α : 2°). The melted ice cream was rested for 5 min before measurement. At 25°C, shear stress from 0.5 to 200 s⁻¹ were run in 120 s and the flow curves were obtained (Pon *et al.*, 2015). The viscosity at shear rate 200 s⁻¹ was selected for comparison.

Texture analysis

Scoopability of ice cream is one of the most important criteria of a good-textured ice cream. Penetration (hardness) test resembles the force required during ice cream scooping. For the test, ice cream samples were initially tempered at 24°C for 5 min. The hardness test was conducted using a texture analyser (Model TA-XT2i, Stable Micro SystemsTM, England) equipped with Texture Exponent software version 2.0.7.0. For each sample, three measurements were performed using a 45° conical probe. The penetration depth and speed used were 10 mm and fixed at 2.00 mm s⁻¹ with compression mode. During penetration, the sample hardness was determined as the peak compression force (N) (Aime *et al.*, 2001; Soukoulis *et al.*, 2008).

Overrun

Overrun was measured by comparing the weight of liquid mix and ice cream in a fixed volume container (Özdemir *et al.*, 2003). The percentage of overrun was obtained according to the following equations:

$$O_n(\%) = (1 - \frac{\rho_t}{\rho_t}) \times 100$$
 (Eq. 1)

$$\rho_{\bar{v}} = \frac{M_{\bar{v}}}{V_{\bar{v}}} \tag{Eq. 2}$$

where O_n (%) = overrun percentage, ρ_{ic} (g/cm³) = density ice cream mix, M_{ic} (g) = weight of same volume of ice cream, and V_{ic} (cm³) = volume of ice cream.

Experimental design

The present work was aimed at developing an instant ice cream formulation and hence, the ideal parameters ranges for D-optimal mixture design had to be determined. This was done using the Design Expert Version 7.0 software (Stat-Ease, Minneapolis, MN). The factors studied were the amount of CMC, sodium alginate and carrageenan. Fourteen formulations were prepared according to the D-optimal mixture design and the optimum amount of ingredients used in each sample and the associated responses obtained are shown in Table 1. Statistical analysis was performed by analysis of variance (ANOVA) with the data shown in Table 2.

Comparison of optimised and commercial instant ice creams

Optimised instant ice cream (with 0.151% CMC and 0.149% sodium alginate) was then compared

Table 1. Stabilisers' combinations in instant ice cream samples and the associated responses.

Formulation code	CMC (%)	Sodium Alginate (%)	Carrageenan (%)	Melting rate (g/min)	Hardness (N)	Viscosity (Pa.s)	Overrun (%)
F1	0.300	0.000	0.000	0.186	2.92	0.10	129.89
F2	0.000	0.300	0.000	0.383	2.51	0.12	123.71
F3	0.150	0.000	0.150	0.219	1.85	0.13	132.56
F4	0.000	0.150	0.150	0.412	2.11	0.10	94.17
F5	0.193	0.053	0.053	0.079	1.23	0.09	98.01
F6	0.000	0.106	0.194	0.348	1.28	0.16	94.18
F7	0.048	0.252	0.000	0.083	3.60	0.22	103.25
F8	0.053	0.194	0.053	0.255	3.25	0.15	105.34
F9	0.000	0.300	0.000	0.383	2.51	0.12	123.71
F10	0.000	0.000	0.300	0.068	0.83	0.12	86.57
F11	0.000	0.000	0.300	0.068	0.83	0.12	86.57
F12	0.150	0.150	0.000	0.023	1.73	0.16	98.02
F13	0.053	0.053	0.194	0.279	1.54	0.19	164.55
F14	0.300	0.000	0.000	0.186	2.92	0.10	129.89

Regression coefficient	Melting rate	Viscosity	Hardness	Overrun
b1	0.19	0.098	2.92	129.92
b_2	0.38	0.12	2.52	123.94
bз	0.069	0.12	0.83	86.51
<i>b</i> 1 <i>b</i> 2	-1.06	0.22	-3.86 0.0075 1.66	-113.92 99.20 -51.28
<i>b1b3</i>	0.34	0.067		
<i>b2b3</i>	0.85	-0.088		
<i>b</i> 1 <i>b</i> 2 <i>b</i> 3	-0.90	0.79	5.00	732.86
<i>b</i> 1 <i>b</i> 2(<i>b</i> 1- <i>b</i> 2)	1.15	-0.86	-15.87	81.37
<i>b1b3(b1-b3)</i>	-1.83	-0.29	-3.05	-746.10
<i>b2b2(b2-b3)</i>	0.72	-0.78	6.33	-117.21
R^2	0.9717	0.9915	0.9960	0.9983
$R^2(adj)$	0.9717	0.9915	0.9869	0.9946

Table 2. Regression coefficient (R²) and coded factors for four different responses.

in terms of melting rate, viscosity, overrun, and hardness. Preliminary studies were conducted to determine the best commercial instant ice cream mix powder available in the market in Malaysia in terms of sensory attributes. As previously explained, there were four different commercial brands of instant ice creams tested for the comparison. Only one was selected as the most acceptable sample. The panels were asked to evaluate all the ice creams produced from different brands for nine attributes namely appearance, aroma, firmness, sweetness, mouth coating, iciness, acceptability, sandiness, overall liking, and overall texture. The best commercial instant ice cream mix was used as comparison with the optimised formulation produced.

Sensory evaluation

Sensory evaluation was conducted to measure human responses on the optimised and commercial instant ice creams. A graphical method was used to evaluate the differences in several specific sensory attributes for the appraisal and review of product comparison (https://www.fusioncharts.com/ resources/chart-primers/radar-chart). These attributes include appearance, aroma, sweetness, firmness, mouth coating, iciness, and sandiness for both samples. Overall acceptability of both samples was also determined. Twenty qualified panellists among the staff and students of the university were selected and asked to compare the coded samples based on the selected attributes. All the samples were evaluated using the 9-point Hedonic Scale (Score 1 = dislikeextremely; Score 9 = like extremely). However, for overall acceptance, the scale (1 = dislike extremely; 5)= like extremely) was used to facilitate the panellists in evaluating the products.

Results and discussion

Statistical analysis

Fourteen instant ice cream formulations were prepared based on three different stabilisers, and the associated responses are presented in Table 1. Table 2 illustrates the polynomial model describing the correlation between responses and variables. The estimated regression models and ANOVA of all the terms described the effects of stabilisers' combination on the quality properties of instant ice cream where b_1 , b_2 and b_3 are linear coefficients; b_1b_2 , b_1b_3 and b_2b_3 are quadratic coefficients; and $b_1b_2b_3$ are cubic coefficients. The responses measured were melting rate, viscosity, hardness and overrun of the samples. In general, a positive sign for the coefficient in the fitted model indicates the ability of the variable to increase the response, whereas a negative sign shows the ability of a variable to decrease the response to describe the correlation between variables and responses. Coefficient of determination (R^2) and the adjusted R^2 values indicated that the selected model had a good fit with the data (Table 2). The closer the value of R^2 approaches unity, the better the empirical model fits the actual data. The P-values were used as a tool for checking the significance of each coefficient, which indicated the interaction patterns between the variables. Only significant P-value (< 0.05) was included in the regression model. Analysis of variance (ANOVA) results for the responses were summarised. The R^2 values obtained were 0.9717 for melting, 0.9960 for hardness, 0.9915 for viscosity and 0.9946 for overrun. The closer the R^2 value to unity, the better the empirical model fits the actual data supported by the lack of fit test.



Figure 1. Contour plots for the effects of different combinations of stabiliser on the properties of instant ice cream.

Melting rate

Slow meltdown, slow serum drainage, good shape retention, and slower foam collapse are among the desired important quality parameters of ice cream (Marshall *et al.*, 2003). When ice cream is in the form of a cone or stick novelty, melting rate is one of the greatest importance to the consumers. Figure 1(a) shows the influence of formulation variables on the meltdown of instant ice creams. Quantitatively, the melting rate (g/min) of each ice cream was different for each stabiliser.

As shown in Table 1, F_{12} consisting two types of stabiliser namely CMC and sodium alginate showed the slowest melting rate of 0.023 g/min, while F_4 (sodium alginate + carrageenan) showed the highest melting rate (0.412 g/min). The addition of 0.15% CMC in F_{12} decreased the melting rate of the instant

ice cream. As shown in Figure 1(a), the melting rate decreased when sodium alginate and CMC were used, and increased when sodium alginate was mixed with carrageenan. The effects of stabilisers on melting rates were significant (P < 0.05) as melting rates increased with the addition of carrageenan. Melting rate showed a negative effect on the cubic term of the stabilisers' combination of CMC, sodium alginate and carrageenan as shown in Figure 1(a). It can be seen that melting rate decreased when CMC and sodium alginate was mixed. Higher content of CMC in ice cream as compared to sodium alginate and carrageenan slowed down the melting rate due to the good capacity of CMC in holding water. Thus, CMC was considered as a very good ice cream stabiliser (Djali et al., 2017) and helped to control the melt profile. According to Tharp et al. (2008),

lower melting rates are related to the sustainability of the body of ice cream, which is usually considered as a good quality ice cream. As reported by Sakurai *et al.* (1996), Alamprese *et al.* (2002), and Hartel *et al.* (2003), ice cream with low overrun melted faster than those with higher overrun. This might be due to the higher amount of air cells that decelerate the heat transfer across the ice cream, therefore slowing the melting rate (Flores and Goff, 1999).

Viscosity

Figure 1(b) shows the combination of CMC and high proportion of sodium alginate that increased the viscosity of the samples. Giving a cubic model effect, CMC and sodium alginate showed a positive relationship with the viscosity measurement of the samples. The combination of CMC and sodium alginate could improve the viscosity, which corresponded well to the slow melting rate (Table 1). The combination of CMC, sodium alginate, and carrageenan produced ice cream with different viscosities depending on the percentage of each stabiliser used. Combination with the highest percentage of carrageenan produced ice cream with slightly high viscosity. Hence, the addition of carrageenan in ice cream brought the reinforcement that this stabiliser is more suitable for soft serve ice cream (Naresh and Shailaja, 2006). The viscosity of ice cream is considered a key attribute where it affects the body and texture of the finished product (Stanley et al., 1996). All the 14 melted ice cream samples showed a shear thinning behaviour where the viscosity decreased as shear rate increased. This rheological behaviour of melted ice cream has been previously observed by several authors (Hartel et al., 2003; BahramParvar et al., 2015; Pon et al., 2015). Sample F₅ (0.193% CMC, 0.053% sodium alginate, and 0.053% carrageenan) showed the lowest viscosity value among all samples. The formulation with 0.048% CMC and 0.252% sodium alginate (F₇) as stabilisers produced ice cream with the highest viscosity.

According to Hartel et al. (2003), the rheological properties of mix have the largest effect on the hardness of the ice cream. The ice cream becomes harder when the viscosity increases. Figure 2 shows a shear thinning behaviour resulted from ice cream formulation for shear stress between 0.5 to 200 s⁻¹ in 120 s. The behaviour of melted ice cream was seen similar as most of ice cream mix. F7 had that maximum viscosity as compared to other formulas due to the effects of the combined CMC and sodium alginate. Viscosity values for ice cream mixes are usually ranged from 0.1 Pa.s to 0.8 Pa.s at 4°C (Goff and Hartel, 2013). Bahramparvar and Mazaheri Tehrani (2011) reported that the decreased viscosity of all mixes with increased shear rate is associated with increased molecular alignment system constituents. The aggregation of fat globules in ice cream has been recommended to partly be responsible for the shear thinning behaviour (Aime et al., 2001). Several researchers have also observed shear thinning behaviour performed in all ice cream mixes (Hagiwara and Hartel, 1996; Prindiville et al., 1999; Aime et al., 2001; Kaya and Tekin, 2001; Alvarez et al., 2005; Soukoulis et al., 2008; BahramParvar et al., 2010).

Texture: hardness of the product

The combination of stabilisers resulted in a negative effect on hardness in all samples. Carrageenan alone was not suitable for ice cream since it gave the lowest hardness value that made



Figure 2. Viscosity against shear rate curves obtained for 14 different samples of instant ice creams with different stabilisers' combinations.

the ice cream less viscous and melted fast. The percentage of carrageenan should be lower to ensure good physical quality of ice cream. Melting point, total solids, overrun, and amount or type of stabiliser are among the factors that can affect the hardness of ice cream (Marshall et al., 2003). From the data obtained, it was found that the hardness of ice cream had significantly decreased with the increase of carrageenan in the sample (P < 0.05). The role of cryo-protective of carrageenan may be attributed to the decrease of hardness. Hydrocolloid is a gelling agent improved by milk proteins to control the recrystallisation (Soukoulis et al., 2008). Figure 1(c) depicts that higher sodium alginate content produced ice cream with higher hardness. However, the addition of carrageenan significantly increased the resistance of the sample to deformation, thus contributing to lower hardness measurement.

Overrun

The overrun of ice cream affects its hardness. Wilbey et al. (1998) stated that ice cream is softer when the overrun is higher. Meanwhile, Ozdemir et al. (2008) mentioned that the value of overrun could be 80-100% based on general literature. A potential reason for obtaining lesser overrun value is due to the inconsistency during the whipping process (Chang and Hartel, 2002), which is caused by limitation of equipment. Ice cream and related products are generally aerated and characterised as frozen foams. From the results obtained, overrun values were found to range between 86% - 164% for all samples (Table 1). Muse and Hartel (2004) reported that the overrun of ice cream produced by a batch freezer is about 40 to 70%. From the data obtained, it was found that there was no significant difference in overrun in all samples. The response surface for overrun of instant ice cream as a function of different proportions of stabilisers is presented in Figure 1(d). It can be seen that the combination of 0.053% CMC, 0.053% sodium alginate, and 0.194% carrageenan (F13) produced ice cream with the highest overrun among the samples. It can be observed in Figure 1(d) that high proportion of carrageenan helped to increase the overrun with the addition of CMC and sodium alginate, therefore producing the maximum overrun as compared to others. In contrast, high value of carrageenan caused the overrun to decrease. However, sodium alginate did not show any significant effect towards the response. It was evident that using a mixture of gums gave a higher viscosity to the system than a single gum (Haghighimanesh and Farahnaky, 2011). Higher viscosity of ice cream promotes lesser overrun. Bahramparvar and Mazaheri Tehrani (2011) stated

that when viscosity increases, the rate of whipping decreases. The cause of higher viscosity is related to the high-water binding capacity of stabilisers. Thus, least amount of air is incorporated. Hardness might increase as a result of larger ice crystals due to lower overrun. Hardness was found to be inversely proportional to overrun. Thus, it can be stated that the softness of ice cream is increased by air (Kurultay *et al.*, 2010).

Optimisation

In ice cream production, the stabilisers' combination must be properly prepared using suitable stabilisers in the correct proportion to ensure the quality of the final product. Therefore, by considering the importance of getting the most suitable stabilisers with the correct proportion in producing instant ice cream mix, the best combination of response functions (melting rate, viscosity, hardness, and overrun) were identified. The optimum types and proportion of stabilisers required were determined by superimposing all the contour plots of all responses. The final result would be considered optimum if the melting rate is minimum while the overrun, viscosity, and hardness of the sample are maximum. From the numerical response analysis, the optimum combined stabilisers were found to be 0.151% CMC and 0.149% sodium alginate. The predicted responses for melting rate, viscosity, hardness, and overrun obtained using the software were 0.02 g/min, 0.16 Pa.s, 1.73 N, and 98.62%, respectively.

Comparison between optimized and commercial instant ice creams

Optimised and commercial instant ice creams were compared following preparation as previously mentioned. From the results obtained, the viscosity of the optimised sample (0.17 Pa.s) was higher than the commercial ones (0.16 Pa.s). This corresponds to the higher hardness (1.51 N) of the optimised sample as compared to the commercial sample (1.03 N). The overrun of the optimised sample was 108.33%, which was considered high. The high amount of air, high viscosity, and hardness resulted in the low melting rate of 0.22 g/min, which is desirable during ice cream consumption. The commercial instant ice cream had a very high overrun of 148.67%, which indicates a very high amount of air bubbles trapped in the sample. During the melting rate test, it was observed that the melted commercial sample formed a foamy structure with barely any drainage collected.

Based on the sensory evaluation conducted by 20 panellists, the optimised instant ice cream had comparable sensory attributes (appearance, aroma,



Figure 3. Sensory evaluation obtained for optimised instant ice cream and commercial instant ice cream obtained from (a) spider diagram of the sensory attributes obtained from average scores, and (b) comparison of overall acceptability obtained from average scores.

firmness, sweetness, mouth coating, iciness, and sandiness) to the commercial ones (Figure 3a). The average score obtained for all properties namely appearance, aroma, firmness, sweetness, mouth coating, iciness, and sandiness were 6.65, 6.40, 6.30, 6.30, 5.65, 4.10, and 3.55, respectively. Meanwhile, the results obtained for the commercial instant ice cream were 5.95, 5.45, 6.20, 6.10, 5.95, 4.35, and 4.25, which suggest a significant difference for those properties. In general, high ratings for appearance, aroma, firmness, sweetness, and mouth coating are considered as positive effects that would be preferred by consumers, whereas the decrease in iciness and sandiness indicate favourable ice cream texture. Thus, this indicates that the instant ice cream mix powder produced was of high quality, which influenced the perception of taste. For the overall acceptability (Figure 3b), the optimised instant ice cream was the most preferred by panellists as compared to commercial sample.

Conclusion

From the mixture design analysis, it was found that the optimum types and proportion of stabilisers to produce instant ice cream mix powder were 0.151% CMC and 0.049% sodium alginate. The content and proportion of both stabilisers in instant ice cream were significant on the quality attributes. The instant ice cream produced using the optimised formulation was comparable to the commercial ones. The present work has successfully produced a better-quality instant ice cream mix powder than that available in the market in Malaysia. Optimised ingredients proportions could be successfully obtained by varying the types of the stabilisers used to produce high quality product.

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