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OPTIMIZATION OF INGREDIENTS FOR THE MANUFACTURE OF INSTANT ICE CREAM BY RESPONSE SURFACE METHODOLOGY

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ABSTRACT

Instant ice cream mix powder was introduced to ease people to make the delicious dessert without using the ice cream machine. It can reduce time and convenience to make our own ice cream. This research aims to develop better ingredient composition for instant ice cream using economical standard ice cream. The ingredients formulated will be optimized by the Response Surface Methodology (RSM), Design Expert Software. The milkfat and milk solids-not-fat fat percentage were varied by the RSM. A design of experiment (DOE) was given by the RSM which suggested the percentages that will be used to make samples and analyze each of its their melting rate, hardness test and sensory analysis. The samples will be tested and compare with the commercial instant ice cream. Haan instant ice cream was selected as a controlled sample to be compared with the optimized ingredients. Three tests which were melting, hardness, and sensory analysis test were conducted. Sample 6 (16% Milkfat, 12% MSNF) has the highest melting rate, follow by the sample 1 (13% Milkfat, 9% MSNF), sample 5 (13% Milkfat, 12% MSNF), sample 9 (16% Milkfat, 9% MSNF), sample 7 (10% Milkfat, 9% MSNF), sample 3 (16% Milkfat, 10.5% MSNF), sample 8 (10% Milkfat, 12% MSNF), sample 2 (13% Milkfat, 10.5% MSNF), sample 4 (10% Milkfat, 10.5% MSNF), and sample 10 which is the commercial instant ice cream. . The high hardness value obtained is from sample 10 which is the Haan instant ice cream mix, follow by other formulation from this study. Sample 8 shows the high value of force, followed by sample 1, sample 2, sample 4, sample 9, sample 7, sample 3, sample 5 and lastly sample 6. Next, based on the result of the sensory analysis, the higher score in overall acceptability was obtained by the commercial instant ice cream which is sample 10 and follow by the other formulation sample 5, sample 7, sample 4, sample 9, sample 3, sample 6, sample 2,

sample 1, and sample 8.Therefore, 10% milkfat and 10.830% MSNF were chosen as an optimized value given by the RSM.

ABSTRAK

Serbuk campuran ais krim segera telah diperkenalkan untuk memudahkan orang ramai untuk membuat pencuci mulut yang lazat tanpa menggunakan mesin ais krim. Ia boleh mengurangkan masa untuk membuat ais krim kita sendiri. Projek ini dilakukan untuk menghasilkan komposisi bahan yang lebih baik untuk serbuk campuran ais krim segera menggunakan standard ais krim yang ekonomik. Bahan-bahan yang dihasilkan akan dioptimumkan oleh Response Surface Methodolgy (RSM), perisian Design Expert. Peratusan lemak susu dan susu pepejal tanpa lemak telah diubah oleh RSM. Satu Design of Experiment (DOE) telah diberikan oleh RSM yang mencadangkan peratusan yang akan digunakan untuk membuat sampel dan menganalisis setiap kadar pencairan, ujian kekerasan dan analisis deria. Sampel akan diuji dan dibandingkan dengan ais krim komersial . Ais krim Haan dipilih sebagai sampel terkawal untuk dibandingkan dengan bahan-bahan yang dioptimumkan. Tiga analisis iaitu kadar lebur, ujian kekerasan, dan analisis deria. Sample 6 (16% Milkfat, 12% MSNF) mempunyai kadar pencairan yang paling tinggi, diikuti dengan sampel 1 (13% Milkfat, 9% MSNF), sampel 5 (13% Milkfat, 12% MSNF), sampel 9 (16% Milkfat , 9% MSNF), sampel 7 (10% Milkfat, 9% MSNF), sampel 3 (16% Milkfat, 10.5% MSNF), sampel 8 (10% Milkfat, 12% MSNF), sampel 2 (13% Milkfat, 10.5 % MSNF), sampel 4 (10% Milkfat, 10.5% MSNF), dan sampel 10 ais krim segera komersial. . Nilai ujian kekerasan yang tinggi diperoleh dari sampel 10 yang merupakan campuran ais krim Haan segera, diikuti oleh perumusan dari penulis. Sampel 8 memperoleh nilai kekuatan tinggi, terus dengan sampel 1, sampel 2, sampel 4, sampel 9, sampel 7, sampel 3, sampel 5 dan terakhir sampel 6.

Oleh itu, 10% milkfat dan 10,830% MSNF dipilih sebagai nilai optimum dihasilkan oleh RSM.

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CHAPTER 1

INTRODUCTION

In this chapter, an overview of the project conducted which consist of the background, problem statement and objectives was described.

1.1 Background

Ice cream is one of the most famous desserts in the world. It is widely consumed and favorable by all ages ranging from child to adult. Therefore, the trend of consuming of ice cream is robust and the development of ice cream was overwhelming in either for small scale or large scale industries.

There are various types of ice creams, which have different types of textures, flavors, shapes and compositions in the ingredients. For instance, hard ice cream, French ice cream, reduced fat ice cream, light ice cream, no sugar added ice cream, lactose-free ice cream, gluten-free ice cream, organic ice cream and many more.

Additionally, ice creams can be categorized as super-premium, premium, standard or economy. Ice creams vary by fat content, total solids, overrun, flavors, packaging and cost (Tharp and Young 2013a, Goff 2010). As there are no legal guidelines of different ice cream categories, this is just a guideline and may differ between existing industries.

Rapid evolution of ice cream development in Malaysia is because people prefer creamery and scrumptious ice cream in the tropical weather. Therefore, food industries see, this as an opportunity for them to develop varieties of ice cream flavors and styles to be consumed by people.

Generally, to make ice cream, the basic components required are milkfat, milk solids-not-fat, sucrose, corn syrup solid, stabilizers, emulsifiers and water. All of these ingredients have their own functions in ice cream. The combination of these ingredients is important to make a delicious and creamier ice cream. The aspect ratio of each ingredient must be optimized to get the desired taste, texture and flavor.

Furthermore, not only ingredients contributed to tasty ice cream, but proper processing steps of making ice cream also must be taken into consideration. It starts with pre mixing of the ingredients, blending together wet and dry ingredients, followed by pasteurization, homogenization, cooling, ageing, freezing, packaging and hardening.

Proper control in every processing stage is important as every section contains its own criteria that need to be followed to avoid any destruction on the ingredients in the ice cream mix. The most important is to understand every process in the making of tasty and delicious ice cream.

Instant ice cream mix is common in other countries Indonesia, but in Malaysia it is still new and not popular. Not many ice cream companies in Malaysia produce instant ice cream mixes, but since other countries export their ice cream mix products to Malaysia, the popularity of instant ice cream among the consumer in Malaysia is getting higher. Therefore, commercializing instant ice cream in Malaysia will definitely give a good opportunity to ice cream companies in Malaysia.

Instant ice cream mix is an alternative for the consumer to enjoy ice cream. The consumer only needs to add cold water into the mix and blend it well, after that freeze in the freezer until it's hard. Then, the consumer can enjoy the ice cream with their favorite topping.

There will be issues between instant ice cream and any other ice cream that are ready-to-eat in store fridge. The reason is because you can add any other favorite food to it, for example, brownies, candies, nuts and others depending on your taste.

This study aimed to physical characteristic of newly formulated instant ice cream mix by varying the percentage of two main components which are milkfat and milk-solids-not-fat (MSNF). The selection of ingredients for these two components is important to create an economical type of instant ice cream.

After getting the formulation and the percentage range of the ice cream ingredients, the data will be used to optimize the result using the Response Surface Methodology (RSM). The software will generate list of formulation based on the percentage inserted.

1.2 Problem statements

For ready-to-market commercial instant ice cream mix that is being imported from the Indonesia, it used premium ingredients. Therefore, this study was conducted to produce an economical instant ice cream with acceptable taste and texture.

In this study the milkfat and milk-solids-not-fat (MSNF) components will be varied to produce the economical instant ice cream mix. A range of percentages will be decided to get an optimum formulation ingredient that is suitable to make an ice cream mix.

Milkfat or fat, including from nondairy fat content in ice cream may be based on the regulation, expected characteristics, price, and competition. Usually as the fat content of ice cream increases the MSNF must be decreased to avoid high viscosity and potential for "sandiness" which happens due to the crystallization of milk, sugar or lactose in the finished ice cream (Goff and Hartel, 2013)

Although altering the component, the percentage of the ingredient must be optimized to produce instant ice cream. Therefore, the aim of this study was to optimize the ingredients of the instant ice cream by using response surface methodology (RSM) to produce economical instant ice cream mix.

The texture, taste and also rheological properties should be accepted by the consumer when it is introduced into the market.

1.3 Objectives

The main objectives of this study were to study the physicochemical characteristic of formulated ice cream and optimize the ingredients for the manufacture of instant ice cream mix powder by using Response Surface Methodology (RSM). The specific objectives were:

i. to test the physicochemical characteristic of instant ice cream produces by the formulation.
ii. to conduct sensory evaluation by comparing instant ice cream produced and commercial instant ice cream.

In conclusion, chapter 1 generally discusses the instant ice cream mix, problem statements and objectives of the project conducted. It is important to focus the problem statements in order to achieve the objectives of the project.

CHAPTER 2

LITERATURE REVIEW

This section contains literature review based on journals and other resource related to ice cream in all view from composition until final product.

2.1 Overview

Ice cream gives palatability to the consumer with the creamier, sweet and tasty taste. It seems simple comestible but very complex and most intricate food to produces.

The composition of the ingredients was developed by considering the multiple, interacting and sometimes counterintuitive laws of chemistry and physics that apply to make an ice cream (Tharp and Young, 2006).

Additionally, to establish ice cream composition legal requirement, quality of product desired, raw materials, plant equipment and process, market demands, competition and cost. These considerations will affect the chosen of minimum, average or high components percentage and the selection of ingredients (Goff and Hartel, 2013).

Ice cream mixes generally consist of 7 components: fat (dairy or nondairy), milk solids-not-fat (the principle source of protein), sweeteners, stabilizers, emulsifiers, water and flavors. After ice cream was whipped and frozen, air will become another crucial component (Goff and Hartel, 2013).

Table 2.1 below show the general composition of the ice cream mix

Table 2.1: General composition of the ice cream mix

Ingredients	Percentage (%)
Milkfat	10 - 16
Milk solids-not-fat (MSNF)	9- 12
Sucrose	10 - 14
Corn syrup solids	4- 5
Stabilizers	0 - 0.4
Emulsifiers	0- 0.25
Water	55- 64

The MSNF contains, on average, dry wt. basis, 38% protein, 54% lactose, and 8% ash (including 1.38% Ca, 1.07% P, 1.22% K, 0.7% Na) (Goff, 2013).

Ice cream structure consists of four phases consists of ice crystals, air bubbles, fat droplets, and the unfrozen phase. Figure 2.1 shows the ice cream structures.

Figure 2.1: Ice crystals (blue - 'C'), air bubbles ('A'), fat droplets ('F'), and the unfrozen phase ('S' - yellow) (Goff, 2013).

2.2 Basic ingredients of ice cream

2.2.1 Milkfat

Milkfat is one of the major components in the ice cream ingredients and also known as Milkfat, or fat or butterfat. Milkfat consist of dairy milk or nondairy milk. According to the standard legal regulation, milkfat content should not be less than 10% and can be as high as 16% for premium ice cream (Goff, 2013).

The present of fat in the ice cream ingredients can increases the richness and flavor of ice cream produce. It also gives smooth texture by lubricating the palate, helps to give body structure, and contributes to its melting characteristics. Not only lubricate palate but it is also lubricating the freezer barrel during freezing process (Goff and Hartel, 2013).

However, using the milkfat has limitation. Excessive use of butterfat in mix will be costly to produce ice cream. It does also can hinder the whipping ability and the richness will be excessive which can retard consumption. Excessive fat also can lead to high caloric value which is not good for health (Goff, 2013).

2.2.2 Milk solid-not-fat

Serum solids or milk solids-not-fat (MSNF) contain lactose, caseins, whey proteins, and minerals (ash content) of the product from which they were derived. All ice cream formulations must include MSNF, either concentrated or dried milk sources, to reach sufficient levels in the mix (Goff and Hartel, 2013). Additionally, MSNF has significant function in ice cream and has an indirect effect flavor. According to Goff and Hartel (2013), proteins help give body and a smooth texture to the ice cream, through emulsification of the fat, foam formation and stability of the air bubbles, and viscosity enhancement in the unfrozen phase. Besides, excessive of MSNF can lead to bad flavors which may arise from some of the products, and an excess of lactose which can lead to the defect of sandiness dominant when the lactose crystallizes out of solution. Excessive concentrations of lactose in the serum phase may also lower the freezing point of the finished ice cream product to an unacceptable level (Goff, 2013).

2.2.3 Sweetener

Sweeteners used in ice cream include cane and beet sucrose ('sugar'), invert sugar, Corn Starch Hydrolysate Syrup (CSS), high maltose syrup, fructose or high fructose syrup, maltodextrin, dextrose, maple syrup or maple sugar, honey, brown sugar, and lactose. Sweeteners contribute metabolize energy in human diet; which are called 'nutritive' or 'caloric' sweeteners. The most common choice of nutritive sweetener is a combination of sucrose (10-12%) and CSS (3-5%) (Goff and Hartel, 2013). Basically, sweeteners provide sweetness and boost flavor to develop smooth and creamier texture; to make ice cream softer and easier to scoop. According to Goff & Hartel (2013) the desired sweetness of ice cream is equivalent to 13 to 16% sucrose in a 36 to 38% total solids mix. Consequently, of the adding sweetener can lower the freezing point of the mix, and this leads to an increased rate of melting. High levels of sweetener can also reduce whippability, especially important for batch freezer operation. The major considerations in blending sweeteners are relative sweetness, contribution to total solids, and freezing point depression of the mix.

2.2.4 Stabilizer

The primary purposes for using stabilizers in ice cream are to increase mix viscosity, to make the ice cream smooth and has creamy texture, to provide resistance to melting, to retard or reduce ice and lactose crystal growth during storage, to prevent wheying off, and to help prevent shrinkage during storage (Biasutti *et al.* , 2013). Many of these functions are attributed to enhanced viscosity of the ice cream mix. Besides, adding excessive of stabilizer can give excessive mix viscosity, contribution to a heavy, soggy body, and production of unwanted melting characteristics. Although stabilizers increase mix viscosity, they have little or no impact on freezing point depression.

2.2.5 Emulsifier

An emulsifier producing stable suspension between two liquids that are not able to mix naturally, for example oil and water. Emulsifiers and protein are surface active molecules that able to keep two liquids that do not mix naturally, in our case milkfat and water, from separating. The emulsifiers are used in ice cream because they produce an ice cream with smooth and creamy texture by promoting fat destabilization (Anonymous, 2014). If emulsifiers were not added, the air bubbles would not be properly stabilized and the ice cream would not have the same smooth texture (Goff and Hartel 2013). This beneficial fat destabilization is enhanced by the (Douglas Goff and Hartel, 2013). If an ice cream mix contains too much emulsifier, the formation of objectionable butter particles can happen. However, if there is too much protein, the emulsion may be too stable and not enough fat is destabilized. This produces unstable foam, and the ice cream is coarse and wet (Goff and Hartel 2013).

2.2.6 Water and air (overrun)

Water in the ice cream mix may come from dairy fluid product or by adding water in the ice cream recipe. Most of the water will be change into ice during the freezing and hardening process. Based on Goff & Hartel (2013) water is a major component in a mix formulation (100-total solids = water, so therefore typically 60–65% water in a 35–40% total solids mix). Air is one of the invisible ingredients in ice cream yet provides huge contribution in ice cream processing. Air or so called overrun was added during the freezing of ice cream mixture in freezing barrel. The amount of air incorporated during freezing, or overrun, affects the size of the ice crystals, with larger ice crystals observed at lower overrun (Arbuckle, 1977). According to Anonymous (2014) an ice cream with high overrun tends to be lighter, warmer and melt more slowly. Ice cream with low overrun is a denser, colder texture and melts more quickly. Manufacturers of premium brand ice cream have a lower overrun.

2.3 Ice cream manufacturing process

The basic steps in ice cream manufacturing are blending, pasteurization, homogenization, ageing, freezing and hardening. Continuous freezers dominate the medium- to large-scale processing industry, while batch freezers are limited to small-scale processors, retailers, including restaurants, and product development applications (Goff and Hartel, 2013). Figure 2.2 shows the flow chart of ice cream processing.

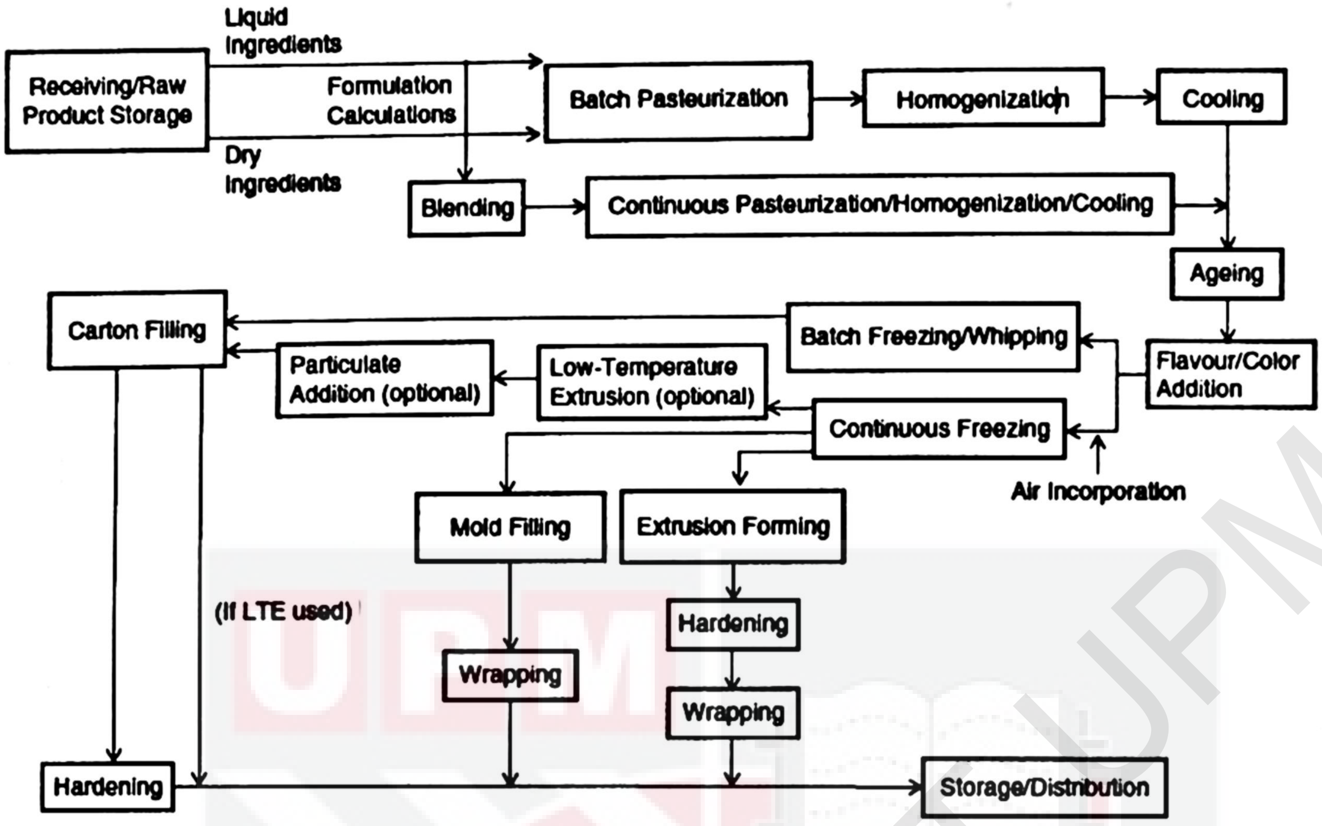


Figure 2.2: Schematic flow chart of ice cream processing, showing specific lines for batch or continuous pasteurization, batch or continuous freezing, and carton filling, molded novelties or extruded novelties (Goff and Hartel, 2013).

2.3.1 Blending

In blending process, the liquid and dry ingredients mix together and placed in the tank provided with agitator and where heating are started at once. Blending needs rapid agitation to incorporate powders and sometimes high speed blenders are used. This is because during blending of ingredients, there will be tendency of float or sink depending on their density. Referring to Morhan *et al.* (2013) it is important to be aware that during blending and subsequent storage before pasteurization, many of the ingredients are not dissolved in the liquid but are only dispersed. If insufficient agitation, for instance, they will separate at the top and bottom of the blend tanks.

2.3.2 Pasteurization

After blending, the ice cream mixture will be undergoing pasteurization process which is giving heat to the mixture. Pasteurization is necessary in the ice cream processing to destroy all pathogenic microorganisms. Pasteurization process also important to reduces the number of spoilage organism such psychrotrophs and helps to hydrate some of the components such as proteins and stabilizers (Goff and hartel, 2013). Both batch pasteurizers and continuous (HTST) methods are used. Based on the book written by Goff and Hartel (2013), proper pasteurization consists in rapidly heating to a definite minimal temperature, holding at that temperature for a minimal time, then rapidly cooling to below 5°C (below 40 °F). Pasteurization reduces the mix substantially free of vegetative microorganisms, killing all of the pathogens likely to be in the ingredients, make solids into solution, aids in blending by melting the fat and decreasing the viscosity, improves flavor of most mixes, keeping quality to a few weeks, and increases the uniformity of product.

2.3.3 Homogenization

The function of homogenization is to forms the fat emulsion found in milk or cream by breaking down or reducing the size of the fat globules to less than 1 μ m. Usually homogenize milk involve two stages. Goff and Hartel (2013), stated that if a two stage homogenizer is used, a pressure of 2000 to 2500 psi on the first stage and 500 to 1000 psi on the second stage should be acceptable under most conditions. Two stage uses in homogenization are usually preferred for ice cream mix. Clumping or clustering of the fat is reduced thereby producing a thinner, more rapidly whipped mix. Melt-down is also improved. Homogenization is also required for any mix containing a fat or oil that is not in a relatively stable emulsion. Homogenization of the mix should take place at the pasteurizing temperature. High temperature produces more efficient breaking up of the fat globules at any given pressure and also reduces fat clumping and the tendency to thick, heavy bodied mixes.

2.3.4 Ageing

After the ice cream mixture completely homogenized the milk is not ready to freeze at that point in the process but it will transfer to aging process which the milk will be placed in aging tank. The milk will be aging for a minimum of 4 hours or maximum of 24 hours. The function of the milk to be age is because crystallization of the fat in the emulsified state occurs more slowly than when it is in the bulk state (not emulsified). Fat crystallization rate dependable on the type of fat and emulsifier used (Adleman and Hartel, 2001), but generally requires up to 4 hours for complete crystallization (Inoue *et al.*, 2009). Mixture temperature inside the tank should be maintained as low as possible without freezing, at 5 °C or lower.

2.3.5 Freezing

In freezing step, the mixture of ice cream enters the cylindrical freezer barrel and is chilled with liquid refrigerant. The mixture will be whipped with dasher, a sharp scraper blade inside the freezer barrel, which contacts the surface of freezing cylindrical smoothly.

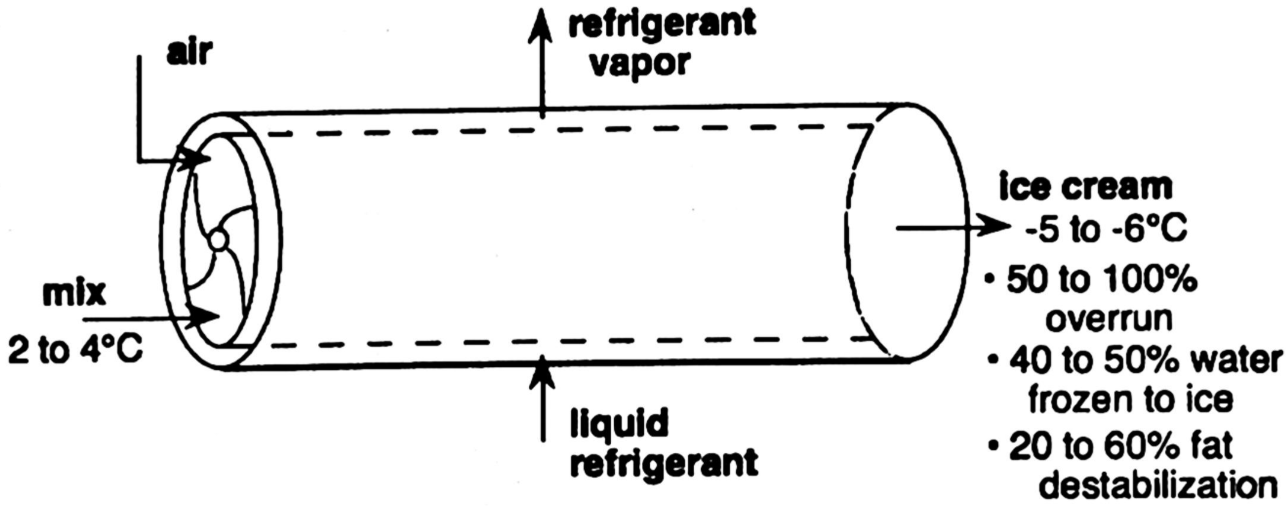


Figure 2.3: Schematic drawing of scraped-surface freezer used for ice cream manufacture (Goff and Hartel, 2013).

Based on the Figure 2.3, there are rotating blades inside the barrel that keep the ice scraped off the surface of the freezer and also the dashers inside the machine which help to whip the mix and incorporate air. Ice cream contains a quantity of air, up to half of its volume. This gives the product its characteristic of lightness. Without air, ice cream would be similar to a frozen ice cube (Goff, 2013).

2.3.6 Hardening

Ice cream produced from freezing process will be packaged and is placed into a blast freezer at -30°C to -40°C. This process is to further frozen remaining water inside the ice cream and also harden the ice cream mixture. The ice cream produce at freezing still contain half unfrozen water and the texture also no firm to hold in shape. Therefore, further freezing process is necessary to harden the ice cream. Approximately, below -25° C, ice cream is stable for indefinite periods without danger of ice crystal growth, however, above this temperature, ice crystal growth is possible and the rate of crystal growth is dependent upon the temperature of storage and this limits the shelf life of ice cream (Goff, 2013).

2.4 Response Surface Methodology (RSM)

Response Surface Methodology (RSM) is a collection of mathematical and statistical techniques useful for the modeling and analysis of problems in which a response of interest is influenced by several variables and the objective is to optimize this response (Pintor *et al.*, 2014). RSM are designs and models for working with continuous treatments when finding the optimum or describing the goal of the response (Oehlert, 2000). The first goal of RSM is to find the optimum response. When there is more than one response then it is important to find the compromise optimum that does not optimize only one response (Oehlert, 2000). Therefore, to optimize the instant ice cream ingredients, the RSM is suitable to be used.

2.5 Physicochemical properties

The physical properties of ice cream consist of testing the ice cream hardness and the melting rate. Hardness is a measure of the resistance to plastic deformation. During hardness test, an indenter is penetrating into the material. Force is recorded as a function of the depth of penetration. A typical measurement procedure consist of inserting cylindrical probe a few millimeters in diameter into a block if ice cream at -10°C to a depth of about 1 cm (Pintor, A. *et al.*, 2017). The hardness test was conducted using Texture analyzer to see the deformation of the sample in controlled manner while recording the applied force with time. Hardness of stabilized ice cream was found to be higher than that of unstabilized ice cream, indicating that there are a larger number of large crystals in ice cream with stabilizers. Therefore, ice cream containing high stabilizer will have higher hardness.

Next, melt test for ice cream is used to determine the effect of stabilizers on the structural behavior of ice cream (Of *et al*., 1974). However, melting rate of ice cream is influenced by various factors including its composition, additives used, amount of overrun, nature of ice crystals and network of fat globules formed during freezing (Moeenfard and Teharni, 2008). Based on Goff and Hartel (2013), stabilizers also influenced the melt resistance for ice cream. According to Guinard *et al.* (1977), "Perceived melting rate was affected more by fat content than by sugar content". During melting, the flow of solution initially over the exterior of the ice cream, but when sufficient heat penetration, it will causes melting of the ice crystals in the interior (Muse and Hartel, 2004a).

In conclusion, chapter 2 discussed on the basic ingredients in ice cream, process involved in ice cream manufacturing and also some analysis which involved in determining the properties of ice cream

CHAPTER 3

METHODOLOGY

In chapter 3, methodology was discussed and described steps involve to optimize the ingredients of the ice cream using RSM.

3.1 Stage 1: Creating ice cream formulation

Before comparing to commercial ice cream, an ice cream formulation must be created beforehand. The ingredients was finalized for the milkfat and milk solid-not-fat based on the range desired. The composition will be optimized using Response Surface methodology (RSM). The ice cream formulation was developed by using the result given by the RSM.

According to Goff (1997), a typical composition range for economy ice cream are; milkfat (10-16 %), milk solids-not-fat (9-12%), sucrose (9-12%), corn syrup solids (4-6%), stabilizers/emulsifiers (0-0.5%), total solids (36-45%) and water (55-64%). Using the reference above, a formulation was created by the author. The percentages of ingredients are listed in Table 3.1.

Table 3.1 : Composition of ingredients for instant ice cream

Ingredients	Percentage of composition (%)
Full cream milk powder	10
Skim milk powder	9
Sucrose	12.8
Salt	0.6
Stabilizer	0.4
Emulsifier	0.3
Vanilla flavor powder	2.9
Water	64

Furthermore, to develop instant ice cream mix, dry ingredients was chosen. Therefore, to prepare ice cream from the powder, consumer only adding water or milk depends on the instruction given.

The percentage of two basis ingredients which are milkfat and milk solid-not-fatare varied. Both ingredients provide significant function in producing good ice cream texture and taste.

The percentage of composition of ice cream was chosen wisely to avoid any destruction on the ice cream texture. The function of milkfat in ice cream is to increases the richness of flavor and lubricates the mouth. Besides, milkfat also have their limitation in ice cream composition such as using milkfat to which is too costly and affect the smoothness of ice cream texture. Furthermore, milkfat also can hinders whipping and limit consumption due to high calories and not good for health.

Additionally, for milk solid-not-fat (MSNF), it can improve body and texture of ice cream but high amount of MSNF can cause cooked or salty flavor and also potential for sandiness. Therefore, it is very important to carefully choose the composition of these two constituents.

After that, Design Expert software was used by inserting the range of milkfat and MSNF provided to generate suggested composition for both constituents. Using the suggested composition, sample was prepared and tested to identify the physicochemical properties. Sensory analysis was also made to compare the prepared formulation for this research with the commercial instant ice cream.

3.2 Stage 2: Preparing the sample

Nine samples were prepared based on the data given by the RSM software.

Table 3.2: Percentage composition sample generated by the RSM software

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8	Sample 9
Full cream milk powder (%)	13	13	16	10	13	16	10	10	16
Skim milk powder (%)	9	10.5	10.5	10.5	12	12	9	12	9
Sucrose (%)	12.8	12.8	12.8	12.8	12.8	12.8	12.8	12.8	12.8
Stabilizer (%)	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4
Emulsifier (%)	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
Vanilla powder (%)	2.9	2.9	2.9	2.9	2.9	2.9	2.9	2.9	2.9
Salt (%)	0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6
Water (%)	61	59.5	56.5	62.5	58	55	64	61	58
Total solid (%)	39	40.5	43.5	37.5	42	45	36	39	42

3.2.1 Weighing ingredients

The percentages of the ingredients were calculated into gram to make it easier for weighing process. The weight basis was 500 g. Figure 3.1 shows the weighing balance used to weight the ingredients. After the plastic container was put, tare it to have net weight of the ingredients.

Figure 3.1: Weighing balance

3.2.2 Mixing ingredients

All the dry ingredients that have been weighed will be mixed using a hand mixer with added of water as shown in the Figure 3.2. The process of mixing took about 5 minutes to complete. The 5 minutes duration was to ensure that the ingredients mix well with the water. Hand mixer was used instead of blender because hand mixer can whip the ice cream mix with more air. Mixing step is very important to have a smooth texture.

Figure 3.2: Mixing process of instant ice cream

3.2.3 Ice cream maker machine

Next, the well mixed ice cream solution will be used ice cream maker machine (Figure 3.3) to make hard ice cream. The machine was set to turn the ice cream mixture into a hard ice cream texture. The machine was set in pre-cool to cool down to -30°C which gives the ice cream a head start. After the mixture of ice cream was totally turned into ice cream texture, the finish product was transferred to a small container and was kept in the freezer for hardening process.

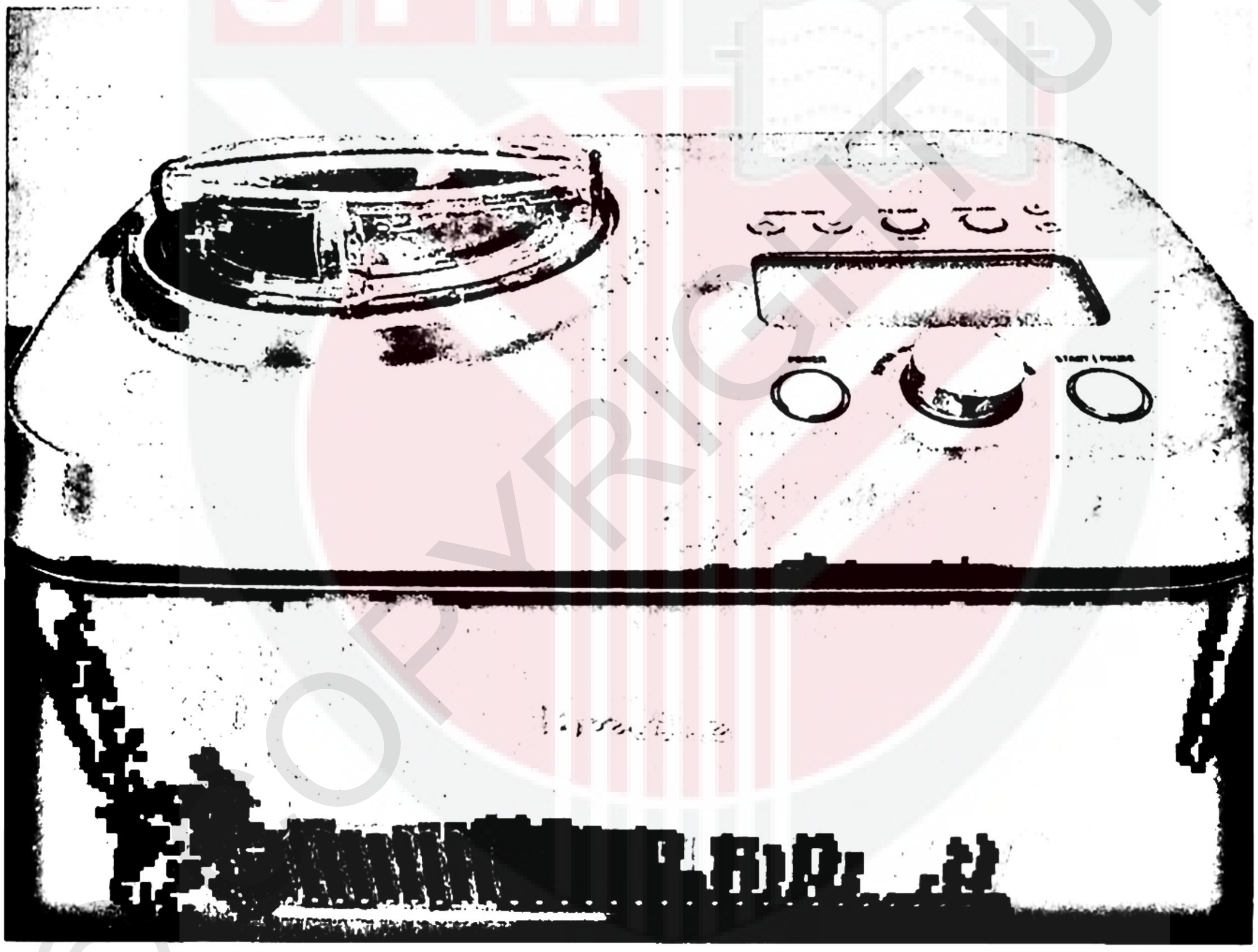


Figure 3.3: Ice cream maker machine

3.2.4 Hardening process of instant ice cream

The ice cream was then stored in the freezer to harden as shown in Figure 3.4. The temperature of the freezer is -18°C. The ice cream was stored in the freezer atleast 8 hours or more. This is to ensure the ice cream completely becomes hard and can be scope easily.

Figure 3.4: Freezer used to harden ice cream

3.3 Stage 3: Determination of physicochemical characteristic of formulating instant ice cream

Analysis was conducted to determine the physical characteristic of the instant ice cream produced such as hardness, melting and sensory.

3.3.1 Analysis of hardness of instant ice cream

Texture analysis is a popular test in determining the textural properties of food. The texture of any food is multi-faceted and connects with the consumers' sensory attributes. Figure 3.5 and 3.6 shows the test conducted using texture analyzer (TA.TX.plus, Stable Microsystem, England). A 2 mm diameter stainless steel cylindrical probe and set up to record the desired forces for penetrating distance = 15 mm, force = 5 g, probe speed during penetration = 3.3 mm/s, probe speed pre-and postpenetration = 3 mm/s (Prindiville, Marshall, and Heymann, 1999). Before testing, samples were cooled down to -10°C and was measured using digital temperature which can detect negative temperature. The texture analyzer was run for this test by giving controlled force to the ice cream and the respond taken is force versus time.

Figure 3.5: Texture analyzer for hardness test of instant ice cream (TA.XT plus)

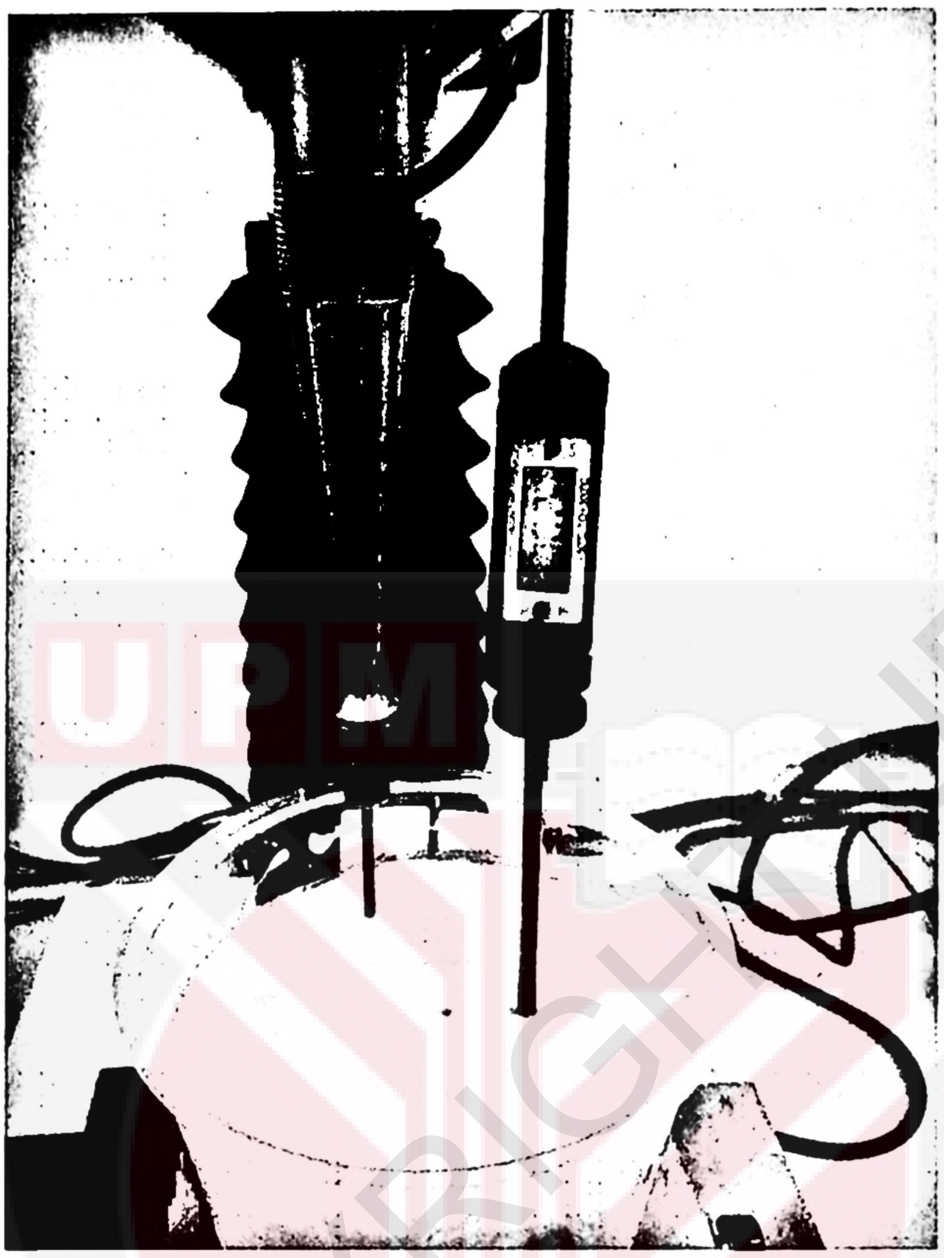


Figure 3.6: Testing the sample when temperature reach -10°C

3.3.2 Analysis of melting rate

Melting rate was measured for each sample. In this melting test, wire gauze, stopwatch and electronic balance were used. The ice cream will be placed above the wire gauze and the duration of the experiment was 60 minutes, considering time needed for the total loss of the structure. Every 5 minutes reading was taken and recorded. The weight of the melt ice cream was recorded and will be tabulated with time taken. The melting rate of the sample was produced based on Equation 4.1 (Boonterm *et al.*, 2012). Make sure the weighing was tare to have net weigh of the melted ice cream. Setup for this test is shown in Figure 3.7.

Figure 3.7: Melting test of instant ice cream

3.3.3 Sensory analysis

A total of 13 panels were gathering to evaluate the formulation including the commercial instant ice cream. The criteria that have been evaluated by the panels were smell, texture, and overall acceptance. Refer appendices Figure A.1 for the sensory analysis form. The sensory analysis was conducted during daylight and after lunch hours. Each untrained panel was explaining on criteria that have to be evaluated and give score from 1(Dislike extremely) to 9 (Like extremely). The panel was told for every sample testing they should have to rinse the mouth to avoid any effect on the next sample testing.

3.4 Summary of methodology

Chapter 3 discussed on the methodology used in this study to produce an instant ice cream formulation started with varying the percentage of milkfat and MSNF. Then, the test required for analysis for the physical properties including hardness test, melting test and sensory evaluation. Figure 3.8 shows the flow chart of methodology

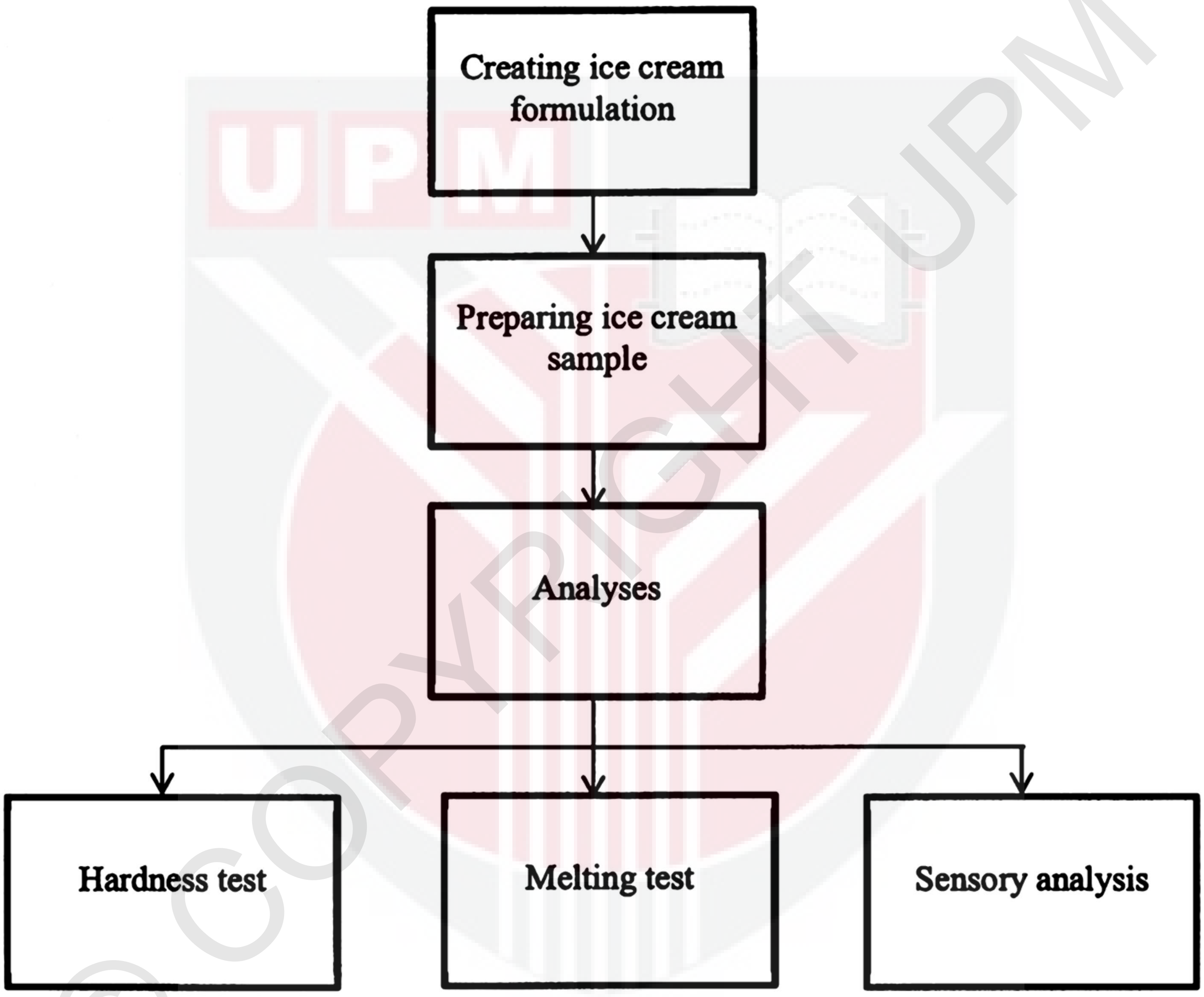


Figure 3.8: Flow chart summary of methodology

CHAPTER 4

RESULTS AND DISCUSSION

This chapter consists of the results obtained from the experiments, which are hardness test, melting test, and sensory analysis. Comparison of the results was done for different percentages of milkfat and MSNF.

4.1 Melting test

The melting test was carried out based on the methodology discussed in the previous chapter. A graph of mass of melted ice cream over time for every sample was plotted as shown in Figure 4.1.

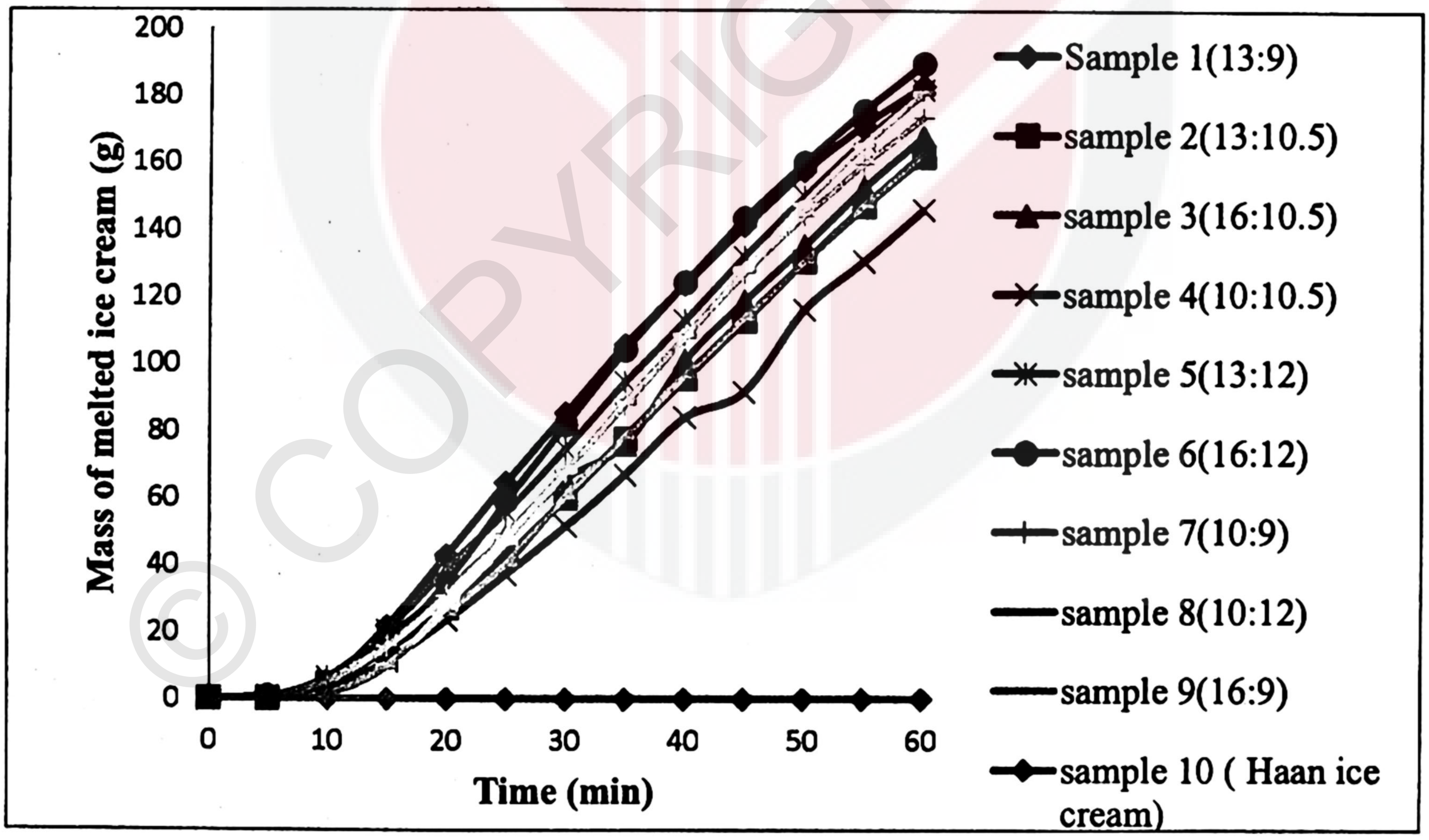


Figure 4.1: Graph of mass of melted ice cream over time. Each sample was quoted with the ratio milkfat: MSNF

Melting rate is used to assess the viability of ice cream at maintaining its structure at room temperature. The melting rate of ice cream is very important to the consumer to consume the product without loss of its structure for longer time. If the ice cream melts too fast, consumer will have difficulty during eating the product. Therefore, a slow melting rate is more preferable, especially in the tropical countries.

From Figure 4.1 shows the graph of mass of melted ice cream over time. All nine samples were compared with the commercial instant ice cream (sample 10). It was found that as time increases the mass of the melted ice cream also increases, but for commercial instant ice cream the ice cream did not melt during the testing time.

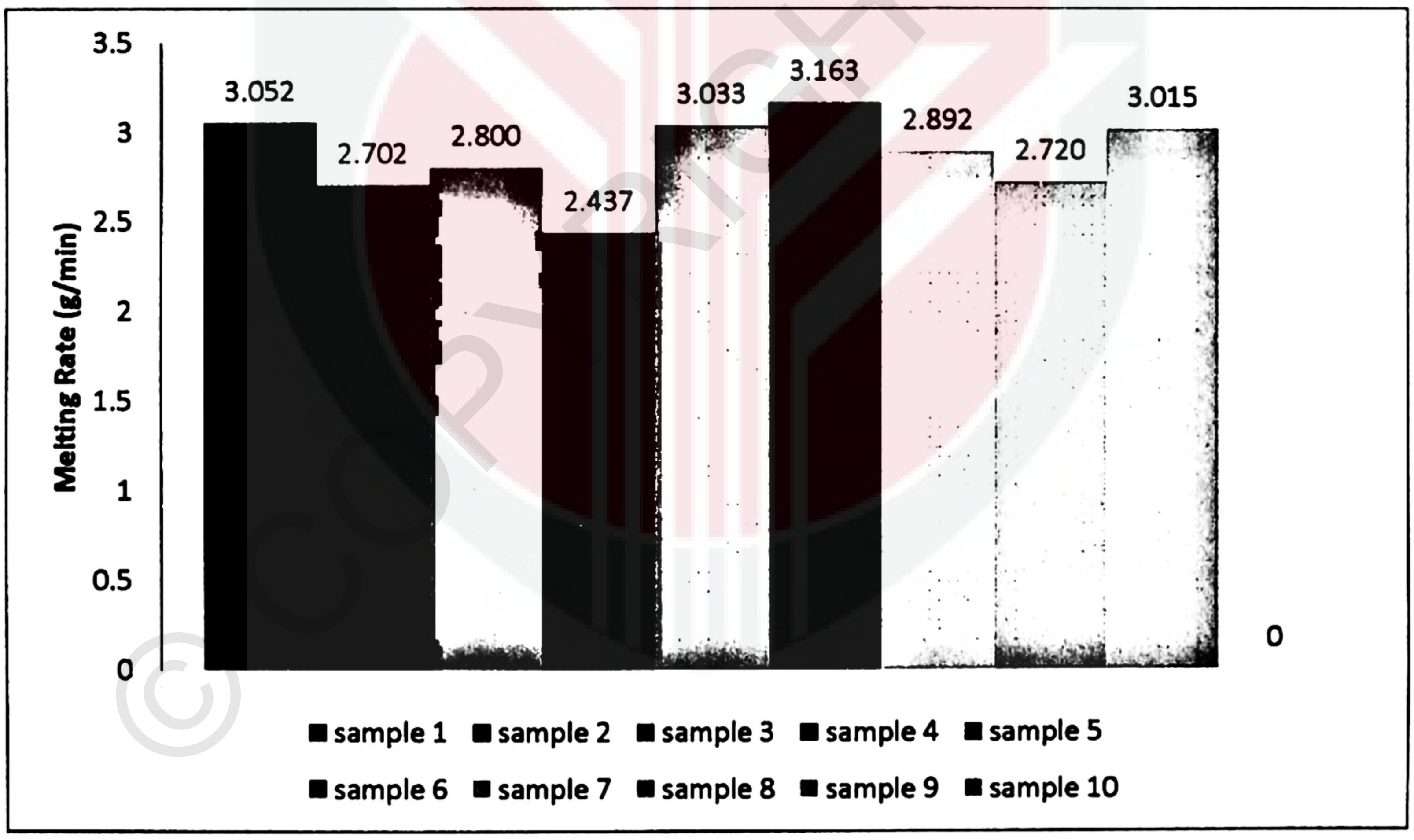


Figure 4.2: Chart of melting rate of each type of ice cream sample

Figure 4.2 shows a graph of melting rate for each sample tested. The melting rate of the sample was produced based on Equation 4.1 (Boonterm *et al.*, 2012):

$$\text{Melting rate (g/min)} = \frac{\text{weight of melted ice cream within 60 min (g)}}{\text{60 (min)}} \quad \text{(eq 4.1)}$$

Sample 6 has the highest melting rate, follow by the sample 1, sample 5, sample 9, sample 7, sample 3, sample 8, sample 2, sample 4, and sample 10.

According to Hartel *et al.* (2003), structural attributes include properties of the air phase (overrun and air cell size distribution), fat phase (total fat content, fat globule size distribution and extent of fat destabilization), ice phase (ice phase volume and ice crystal size distribution), and the continuous phase (viscosity) that can affect the melting rate of ice cream.

Fat destabilization has the largest effect on the melting rate of ice cream. Destabilized fat in ice cream takes the form of clumps of fat globules that cover and support the air cells and also the chain of fat globules that build a fat network in the ice cream (Ohmes *et al.*, 1998). Milkfat aids in good melting properties, due to its role in fat destabilization.

By varying the percentage of the milkfat in the formulation, the higher value of milkfat will have fastest melting rate. Besides, the percentage's composition of MSNF also increases as meltdown rate increases. This is the factor that resulted in an unusual trend of the melting rate. When protein content (MSNF) was increased, the melt rates also increase significantly, which is due to the ice creams had little structure contribute from partially coalesced fat to hold the ice cream (Daw and Hartel, 2015). Furthermore, overrun can affect the melting resistance which higher overrun can increase the melting resistance (Dertli *et al.*, 2016).

Therefore, based on the data obtained from the melting test, the temperature of the surrounding also may have some effect on the melting of the ice cream. Maintaining the temperature during the experiment can minimize error of the result obtained. Both, milkfat and MSNF have their own effect on the melting rate properties.

4.2 Hardness test

Hardness test for ice cream is very important since it directly affects the scoop-ability. If consumer trying to scoop the ice cream directly taken from very cold freezer, the hardness of the ice cream is very high. Therefore, customers should leave the ice cream at room temperature to warm the ice cream. Sufficient ice has to be melted so that the hardness of ice cream can be reduced to allow easier scooping. The cold temperature indicates high phase volume, which can disturb penetration of the spoon into the product.

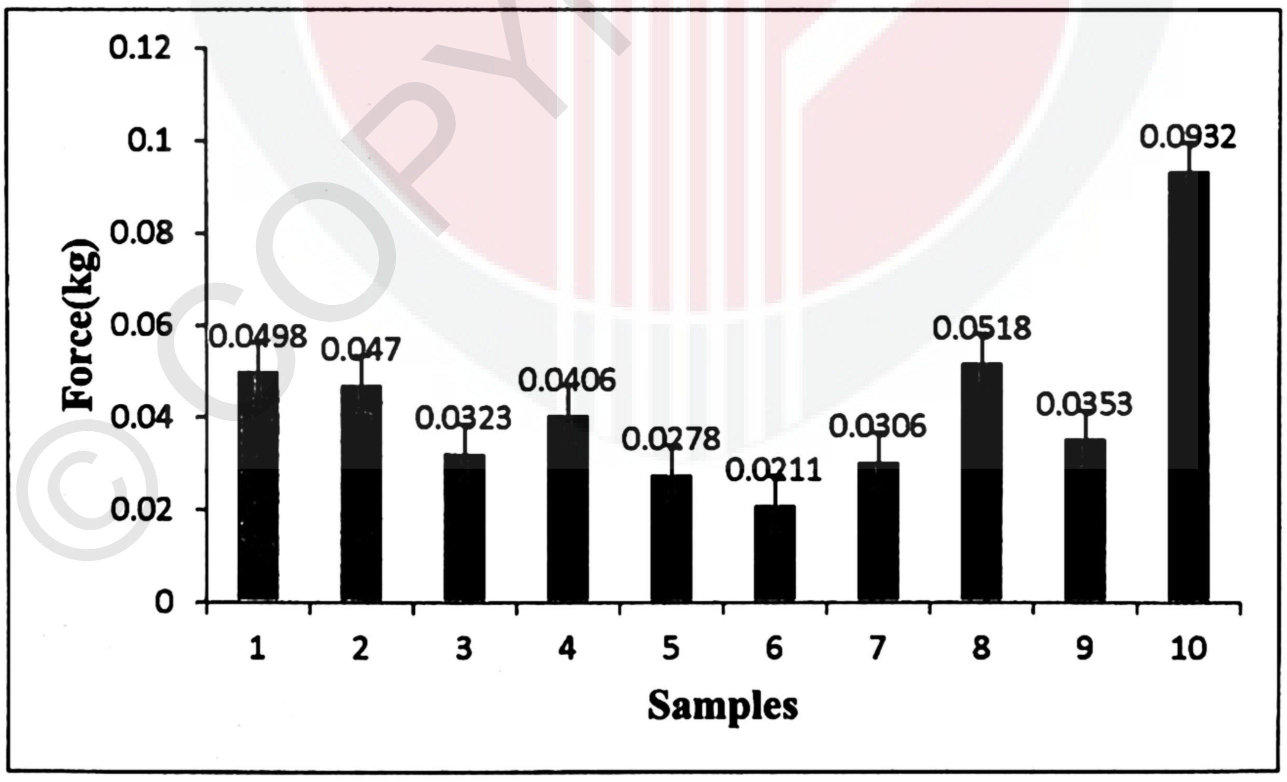


Figure 4.3: Hardness of ice cream.

From figure 4.3, the graph shows the hardness of ice cream samples and commercial instant ice cream. The high hardness value recorded is from sample 10 which is the Haan instant ice cream mix, follow by other formulation. Sample 8 obtains a high value of force, continues with the sample 1, sample 2, sample 4, sample 9, sample 7, sample 3, sample 5 and lastly sample 6.

According to Hartel and Muse (2004), hardness was influenced by the ice phase volume, ice crystal size, overrun, fat destabilization, and the rheological properties of the mix. The level of fat destabilization had an effect on the hardness of ice cream, which destabilize fat provides a network between the air cells in the ice cream and can thereby increase the hardness of ice cream (Muse and Hartel, 2004b).

Based on the experiment conducted by the Prindiville, *et al.* (1999), when ice cream contains high amount of milkfat, the hardness of ice cream also will be increased. In addition to that, sucrose concentration also affects the hardness of ice cream. This is why this study's ice cream formulation is has lower hardness compared to the Haan instant ice cream.

In addition, the effect of total solid may affect the hardness of the ice cream. Sample 8 has the lowest total solid and therefore the hardness value is much higher. According to Kurultay, *et al.* (2009), when overrun rate is fixed, the hardness was increased by increasing total solid level.

4.3 Sensory analysis

Sensory analysis was conducted to the samples obtained from the study with commercial instant ice cream. The sensory analysis was done to know the acceptability for the samples.

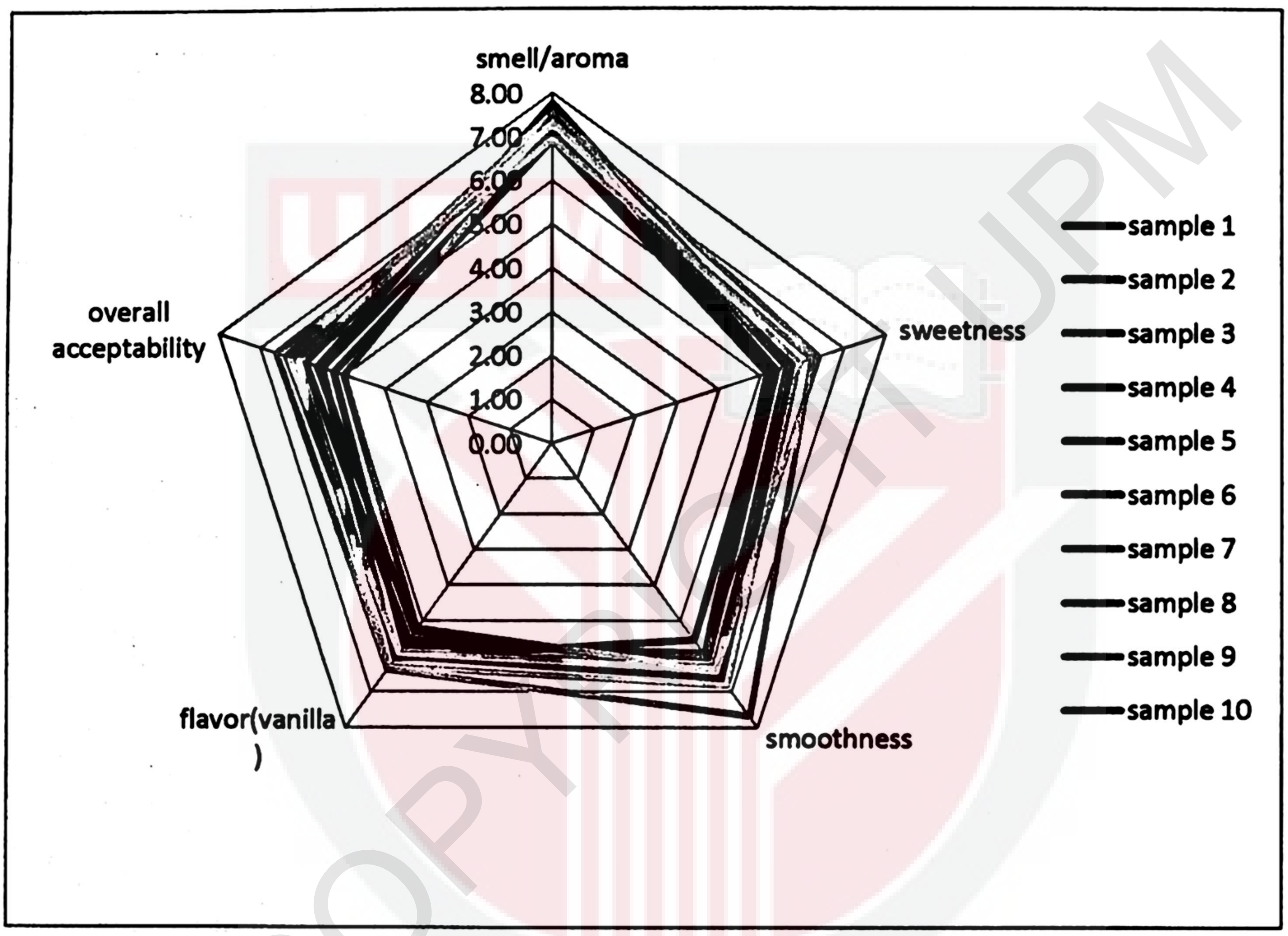


Figure 4.4: Sensory Analysis of chart for all samples

Table 4.1: Sensory analysis data for all samples

Attributes	Samples									
	1 (13:9)	2 (13:10.5)	3 (16:10.5)	4 (10:10.5)	5 (13:12)	6 (16:12)	7 (10:9)	8 (10:12)	9 (16:9)	10
Smell/ aroma	6.92	7.08	7.46	7.38	7.08	7.77	7.54	7.46	7.31	6.92
Sweetness	5.15	5.31	5.38	5.77	5.92	5.77	6.38	5.62	5.92	6.23
Smoothness	5.92	5.54	6.08	6.62	6.31	6.38	6.38	6.38	6.85	7.69
Flavor (vanilla)	5.69	6.08	5.54	6	5.92	5.31	5.92	5.23	6.31	6.38
overall acceptability	5.23	5.54	5.92	6.23	6.38	5.62	6.31	5	6.08	6.62

Sensory analysis was conducted on untrained 13 panelists in order to obtain the result. From the result obtained, sample 10 has the highest value overall acceptability in comparison to this study's formulation except for the smell/aroma and sweetness. This is because the commercial has a light smell compared to this study's formulation where the smell come from vanilla and milk powder used. The sweetness of Haan instant ice cream also was found not too sweet and was mild, compared to this study's formulation where it was found too sweet.

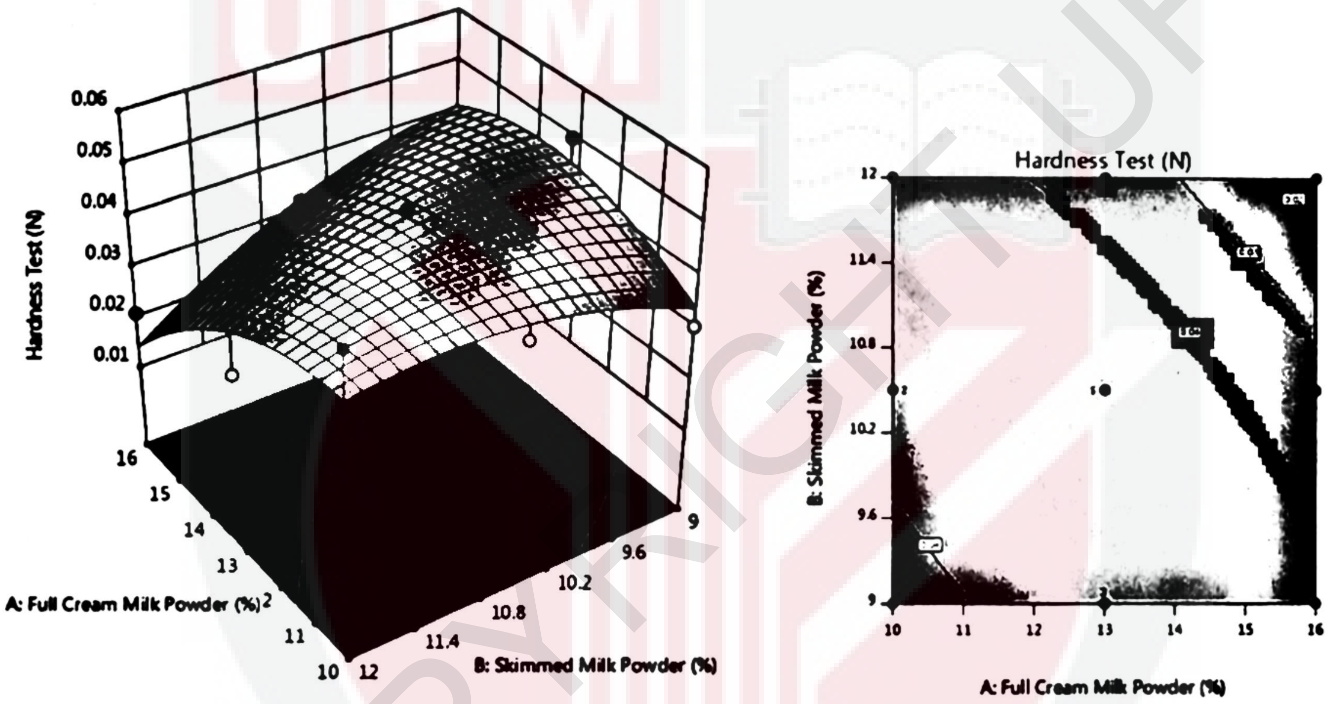
Between this study's formulations, sample 6 is leading in smell acceptance compare to others. For the sweetness acceptance sample 7 has the highest value and the least value was obtained for sample 1.

Next, the smoothness attribute in ice cream was found the highest sample 9, this is because the milkfat content is high. Milkfat functioned to give richness in ice cream and lubricates and insulate the mouth. The increase in fat content have been shown to reduce ice crystal size and affect sensory evaluation by causing lubrication in the mouth (Abdel-

Haleem and Awad, 2015). According to Prindiville *et al.* (1999), experimental conducted by varying the fat content and found that the higher fat content the higher the score for smoothness.

4.4 Response surface methodology (RSM) analysis

A RSM was used to study the effect of Milkfat and MSNF to the ice cream physicochemical characteristic and to determine the optimum blend ratio.



(a) (b)

Figure 4.5: Response surface showing the of the effect of milkfat and MSNF percentage to hardness of ice cream in (a) 3D surface and (b) contour.

Table 4.2: ANOVA for response surface quadratic model hardness test score

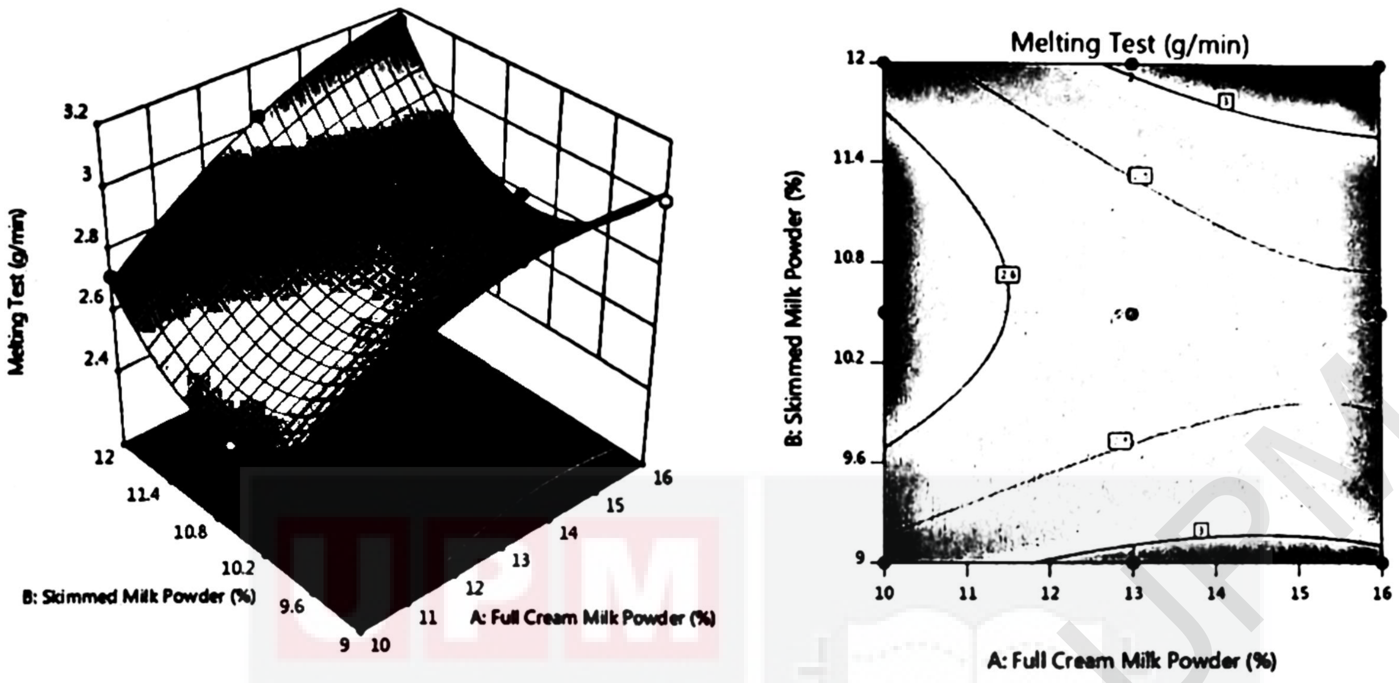
Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	0.0011	5	0.0002	6.25	0.0055	significant
A-Full Cream Milk Powder	0.0002	1	0.0002	6.57	0.0264	
B-Skimmed Milk Powder	0.0002	1	0.0002	4.95	0.0479	
AB	0.0003	1	0.0003	9.07	0.0118	
A^2	0.0002	1	0.0002	6.87	0.0238	
B^2	0.0001	1	0.0001	3.24	0.0995	
Residual	0.0004	11	0.0000			
Lack of Fit	0.0004	3	0.0001			significant
Pure Error	0.0000	8	0.0000			
Cor Total	0.0015	16				

R^2 =0.7396

The Model F-value of 6.25 implies the model is significant. There is only a 0.55% chance that an F-value this large could occur due to noise.

P-values less than 0.0500 indicate model terms are significant. In this case A, B, AB, A^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve author model.

The model for the hardness of the formulated ice cream had a significant (P=0.0055) effect and a high correlation (R^2=0.7396). The ANOVA in Table 4.2 showed that only linear term for full cream milk powder (A) presented a highly significant ($P<0.01$) effect on hardness of ice cream, whereas skimmed milk (B) linear terms, full cream milk powder×skimmed milk powder (AB) interaction and quadratic term (A^2) had a significant effect ($P>0.05$) on this parameter. Only quadratic term (B^2) is not significant.



(a) (b)

Figure 4.6: Response surface showing the of the effect of milkfat and MSNF percentage to melting rate of ice cream in (a) 3D surface and (b) contour.

Table 4.3: ANOVA for response surface quadratic model of melting rate score

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	0.7309	5	0.1462	475.99	< 0.0001	significant
A-Full Cream Milk Powder	0.2096	1	0.2096	682.56	< 0.0001	
B-Skimmed Milk Powder	0.0005	1	0.0005	1.58	0.2341	
AB	0.0256	1	0.0256	83.30	< 0.0001	
A^2	0.0335	1	0.0335	108.99	< 0.0001	
B^2	0.4742	1	0.4742	1544.11	< 0.0001	
Residual	0.0034	11	0.0003			
Lack of Fit	0.0034	3	0.0011			significant
Pure Error	0.0000	8	0.0000			
Cor Total	0.7343	16				

R^2 =0.9954

The Model F-value of 475.99 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise.

P-values less than 0.0500 indicate model terms are significant. In this case A, AB, A^2, B^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve author model.

The model for the melting of the formulated ice cream had a significant ($P< 0.0001$) effect and a high correlation (R^2=0.9954). The ANOVA in Table 4.3 showed that only linear term for full cream milk powder (A), quadratic term (B^2) ,full cream milk powder×skimmed milk powder (AB) interaction and quadratic term (A^2) presented a highly significant ($P<0.01$) effect on melting of ice cream. Only skimmed milk (B) linear terms is not significant.

4.4.1 Optimize ingredients by Response Surface Methodology

Table 4.4: Constraint

Name	Goal	Lower Limit	Upper Limit
A:Full Cream Milk Powder	is in range	10	16
B:Skimmed Milk Powder	is in range	9	12
Hardness Test	maximize	0.0211	0.0518
Melting Test	minimize	2.43655	3.16335

Table 4.5: Table shown given by RSM is a solution based on the constraint (Table 4.5)

Number	Full Cream Milk Powder	Skimmed Milk Powder	Hardness Test	Melting Test	Desirability	
1	**10.000**	**10.830**	**0.044**	**2.450**	**0.858**	**Selected**
2	10.000	10.820	0.044	2.450	0.858	
3	10.000	10.852	0.044	2.451	0.858	
4	10.000	10.810	0.044	2.450	0.858	
5	10.000	10.887	0.044	2.453	0.858	
6	10.000	10.748	0.044	2.448	0.857	
7	10.000	10.725	0.044	2.448	0.857	
8	10.000	11.025	0.044	2.464	0.853	
9	10.000	10.598	0.044	2.449	0.851	

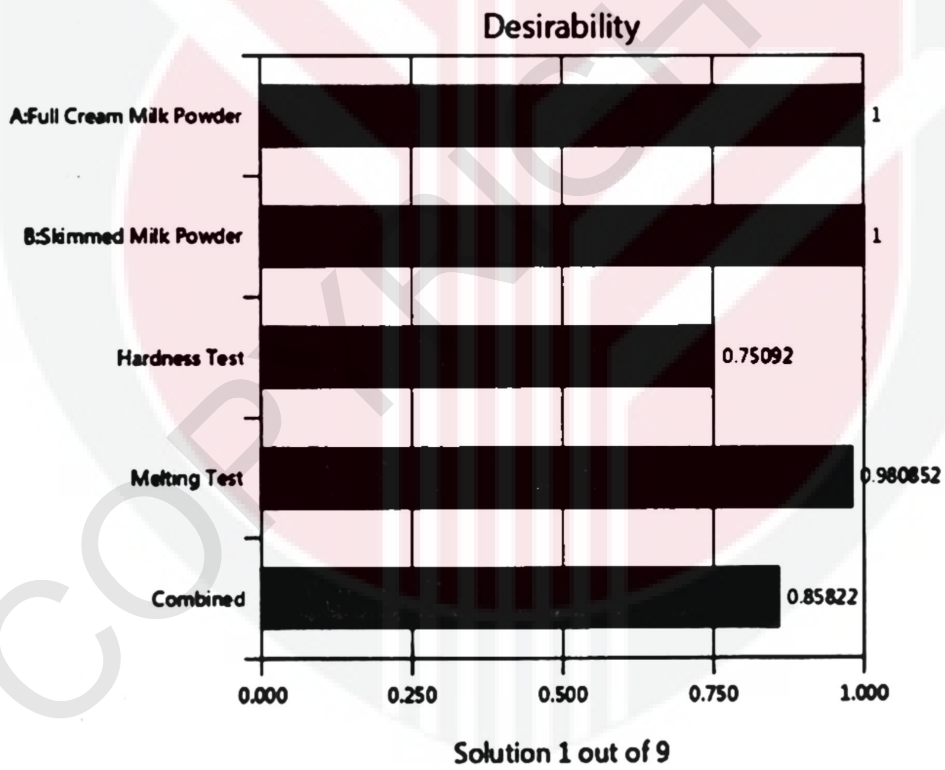


Figure 4.7: Bar chart of the desirability for solution 1

Solution Number 1 was chosen as it as good desirability. Ramp functions from 0 to 1 will project the output variables to a value between 0 and 1, with 0 being very bad and 1 being ideal. Depending on the type of optimization, the ramp function will try to maximize, minimize or hit a target value.

Therefore, 10% milkfat and 10.830% MSNF were chosen as an optimized value obtain by the RSM. In general optimized value obtained from RSM is different from the data calculated in Table 3.2. This is because the optimization has been carried out by the RSM and the variables in the range have been selected to obtain the optimum response.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

This chapter summarizes the project work done. The main objectives were to optimize of ingredients for the manufacturing of instant ice cream by response surface methodology. The ingredients that were verified were milkfat and MSNF. Different tests were done to optimize the ingredients include melting rate, hardness test and sensory analysis. The recommendation is also included in this chapter.

5.1 Conclusions

The variation of milkfat and MSNF in the ingredient composition of ice cream gave impact to the quality of the ice cream. Milkfat and MSNF can affects the ice cream melting rate, hardness and sensory. The optimization was done to obtain the best formulation to produce better quality of ice cream.

Type of milkfat and MSNF chosen is full cream milk powder and skim milk powder respectively. Percentage of Milkfat and MSNF was varied and to be optimized using RSM. A list of suggested formulation generated by the RSM and the experiment was conducted and analyzed for the hardness, melting and sensory.

For melting test, it was found that as time increases the mass of the melted ice cream also increases, but for commercial instant ice cream the ice cream did not melt during the testing time. Furthermore, for hardness test, the high hardness value recorded is from sample 8 with ratio milkfat: MSNF (10:12). Next, for the sensory analysis commercial instant ice cream has highest score for overall acceptance and for the this study's formulation sample 5 with ratio (13: 12) has higher overall acceptability.

To summarize, using RSM, 10% milkfat and 10.830% MSNF were chosen as an optimized value with desirability of 0.858. The percentage chosen is based on the hardness test, meltdown rate and sensory analysis. Both percentage of milkfat and MSNF give high desirability on the hardness and melting rate.

5.2 Recommendation

This project can be improved by varying other ingredients such as the sucrose, stabilizer, emulsifier and even total solid. This is because to produce better ice cream many factors should take into consideration.

Based on this formulation, it still can't compete with the commercial instant ice cream. Therefore, other alteration should be made. The hardness of ice cream affected by many factors such as the ice phase volume, ice crystal size, overrun, fat destabilization, and the rheological properties of the mix. As for melting rate, factors that can affect are air phase (overrun and air cell size distribution), fat phase (total fat content, fat globule size distribution and extent of fat destabilization), ice phase (ice phase volume and ice crystal size distribution), and the continuous phase (viscosity).

Using different type of milkfat and MSNF also can show different result on the hardness, melting and sensory analysis. Therefore, by varying the type of milkfat and MSNF also can be considered.

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APPENDICES

SCORE SHEET 1.1

QUESTIONNAIRE FOR HEDONIC SCALE

PANEL NO./ NAME : ____________________DATE: _______________

PRODUCT : Vanilla Ice Cream

Instruction:

As representative of the consuming population, quantify the degree of liking or disliking of the products one by one separately. Evaluate each given attribute one by one separately. Fill in the scale at the space provided. Don't forget to rinse your mouth with water in between the samples.

Scale	Description
9	Like extremely
8	Like very much
7	Like moderately
6	Like slightly
5	Neither like or dislike
4	Dislike slightly
3	Dislike moderately
2	Dislike very much
1	Dislike extremely

Sample Attributes	**Sample Code**									
	101	102	103	104	105	106	107	108	109	110
Smell/aroma										
Sweetness										
Smoothness										
Flavour (Vanilla)										
Overall acceptability										

Comment:

____

Thank you.

Figure A. 1: Sensory evaluation form