
**UNIVERSITI PUTRA MALAYSIA**

# *EFFECTS OF OIL CONTENTS ON THE PHYSICAL AND MICROSTRUCTURAL CHARACTERISTICS OF ICE CREAM*

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# EFFECTS OF OIL CONTENTS ON THE PHYSICAL AND MICROSTRUCTURAL CHARACTERISTICS OF ICE CREAM

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## ABSTRACT

Observing the microstructure of ice cream is very crucial in order to understand the properties and behavior of ice cream. Recently, various technological devices have been introduced for the purpose of studying the microstructure which will eventually improve the quality of ice cream. However, most devices used in observing the microstructure of ice cream are very expensive and limited. This study was conducted to observe the effects of different percentage of oil contents on the physicals and microstructural characteristics of ice cream. The system was set up inside a freezer with surrounding temperature of -21 °C and direct observation of ice cream sample was done using Dino-Lite microscope at fixed magnification of 500x. Preparation of ice cream samples were manipulated by mixing different percentage of palm oil which were 0% (control sample), 4%, 5% and 6%. Melting rate, overrun and firmness of each hardened ice cream were measured and correlated with the structural attribute. Results showed that different percentages of oil content in ice cream affected the microstructure of ice cream i.e. the air cells, ice crystals and fat globules. It was observed that ice cream with higher percentage of oil content had smaller air cells and ice crystals sizes with fat globules were clearly visible. The melting rate decreased from 1.82g/min to 1.66g/min, while overrun decreased from 55.12% to 33.38% as the percentage of oil content increased. However, the firmness showed inconsistent trend where highest value recorded was 116.6N (control) and lowest value was 108.4N (5% oil). Overall, images of microstructure of ice cream were successfully obtained using the device developed and the physical properties were well correlated.



**Keyword:** ice cream fat content, microstructure, microscopy, dairy processing

## ABSTRAK

Pemerhatian terhadap struktur mikro ais krim adalah suatu perkara yang penting dalam kita memahami ciri-ciri dan tingkah laku ais krim. Kebelakangan ini, banyak peranti teknologi telah diperkenalkan untuk tujuan mengkaji struktur mikro yang akhirnya akan meningkatkan kualiti ais krim. Walaubagaimanapun, kebanyakan peranti yang digunakan untuk memerhati struktur mikro ais krim adalah sangat mahal dan terhad. Kajian ini telah dilakukan untuk memerhatikan kesan peratusan kandungan minyak yang berbeza terhadap ciri fizikal dan struktur mikro ais krim. Sistem ini dipasang di dalam peti sejuk dengan suhu sekitar -21°C dan pemerhatian secara langsung sampel ais krim telah dilakukan menggunakan mikroskop Dino-Lite pada pembesaran tetap 500x. Penyediaan sampel ais krim dimanipulasi dengan mencampurkan peratusan minyak sawit yang berbeza iaitu 0% (sampel kawalan), 4%, 5% dan 6%. Kadar lebur, overrun dan kepejalan setiap sampel ais krim diukur dan berkorelasi dengan atribut struktur. Hasil kajian menunjukkan bahawa peratusan kandungan minyak yang berbeza dalam ais krim mempengaruhi struktur mikro ais krim seperti sel udara, kristal ais dan globula lemak. Telah didapati bahawa ais krim dengan peratusan minyak yag lebih tinggi mempunyai sel udara dan ukuran kristal ais yang lebih kecil dengan globula lemak semakin jelas kelihatan. Kadar lebur menurun dari 1.82g/min menjadi 1.66g/min, sementara overrun menurun dari 55.12% menjadi 33.38% apabila peratusan kandungan minyak meningkat. Walaubagaimanapun, kepejalan menunjukkan trend yang tidak konsisten di mana nilai tertinggi dicatatkan ialah 116.6N (sampel kawalan) dan nilai terendah adalah 108.4N (5% minyak). Secara keseluruhan, gambar struktur mikro ais krim Berjaya diperoleh menggunakan alat yang dikembangkan dan sifat fizikalnya berkolerasi dengan baik.



**Kata kunci:** kandungan lemak ais krim, struktur mikro, mikroskopi, proses tenusu

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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 of ice cream

Ice cream is a special dessert that is popular in all ages, from young to old. Ice cream isn't just a treat for the elite anymore. Today, ice cream is found in nearly every restaurant or corner store and is known as the perfect summer treat worldwide. 1.6 billion gallons of ice cream and frozen desserts are made annually in the United States alone, with the average American consuming four gallons of ice cream every year (Stanpac, 2018).

Not only that, ice cream is also known as a complex food that consists of tiny air cells distributed into a continuous aqueous process that is partially frozen. It is important to choose the ingredient of ice cream such as milk , cream, sugar, stabiliser, and emulsifier mixture as it will affect the development of the form, texture, and palatability desired. Usually, milk fat, milk solid non-fat (MSNF), a sweetener that

typically combines sucrose and glucose, stabilisers, emulsifiers and water are the main ingredients for making an ice cream. In ice cream production, all of these ingredients have their own position, which will determine the quality of the ice cream itself.

The ingredients are not the only factor that can contribute to the physical properties of the ice cream in order to achieve an ideal taste, texture and flavour with good physical effects on the ice cream, but the processing of ice cream itself also plays a fundamental and significant role in creating the perfect ice cream with good physical properties. Mixing the food, pasteurising, homogenising, ageing, freezing, and hardening is the main technique for making the ice cream. The whole of this cycle has its own role and importance in the development of ice cream. Other than that, because of the high risk of heat shock with simultaneous loss of consistency, the consistency of ice cream products often depends on the storage and transportation of the ice cream from the factory through the shop and then to the consumer (Hanne K., 2011).

Approximately 30 percent water, 50 percent air, 5 percent fat and 15 percent matrix (sugar solution) by volume were standard ice creams (Clarke, 2004). There are four types of ice cream that are super-premium, premium standard, and economically available, depending on the percentage of each of the ingredients put into the ice cream. The greater the amount of fat, the greater the price. It is important to understand why certain ingredients are put in, why it takes time and certain procedures to produce a perfect ice cream.

## 1.2 Problem statement

To produce a high quality and super-premium ice cream, it is not only about the ingredients and the processes but it needs a study of the microstructure of the ice cream

itself. How the main components of the ice cream such as ice crystals, air cells and fat globules play their role in maintaining the best structure or the texture of the ice cream itself. What will change in the microstructure when we change the formula or the ingredients. Why certain component is important to be in a bulk density and why the other is not necessary and many other questions needs an answer.

In studying the microstructure of the ice cream, it demands a high quality and effective microscopy techniques so that the real data and information can be interpreted from the image analysis. Scanning Electron Microscopy (SEM) or Transmission Electron Microscopy (TEM) are some example of devices used in studying the microstructure of ice cream. However, the devices are very expensive and it not available for students in any institution to use it for experiment or for the purpose of study.

Another issue that can be stated is that we know ice cream is a frozen dessert where it needs a cold temperature below than -15°C to maintain the structure and the texture of a good ice cream. So, the process of observing the microstructure of ice cream needs to be inside a cold freezing room so the outcome of the images will be better. If we observe the ice cream in a room temperature of 25°C or in a chilled temperature only, the structure of ice cream will damage slowly. At some other point, in freezing condition, it is not good for the optical material as it can lead to humidity condensation or frost problems at the frozen sample surface or at the surface of the microscope oculars. Some action needs to be taken to avoid the situation.

Therefore, this study aimed to provide the solution for the problem where a portable device of microscopy will be developed where it can be handily placed inside a freezer with a new and fresh idea of engineering.

## 1.3 Objectives

The main objective of this study is to observe the effects of different percentage of oil contents on the physical and microstructural characteristics of ice cream. The microstructure of the ice cream samples was correlated with the physical properties of ice cream such as overrun, melting rate and firmness of ice cream.

# CHAPTER 2

# LITERATURE REVIEW

This chapter will be discussed about the literature review on ice cream in all view based on journals paper, review paper and website that can be referred.

## 2.1 Microstructure of ice cream

Multi-phase structures that include air , water, fat, and ice are frozen desserts (Figure 2.1). Some milk fat globules partially coalesce to form networks during the complex freezing process, while some remain as discrete globules dispersed in the unfrozen serum phase. Some water is turned into ice crystals in the mix, resulting in a serum process of freeze-concentrated containing sweeteners, dairy salts, water, and some MSNF. The primary components of frozen dessert structure include the serum phase, ice crystals, fat phase, air cells, and proteins and hydrocolloids.

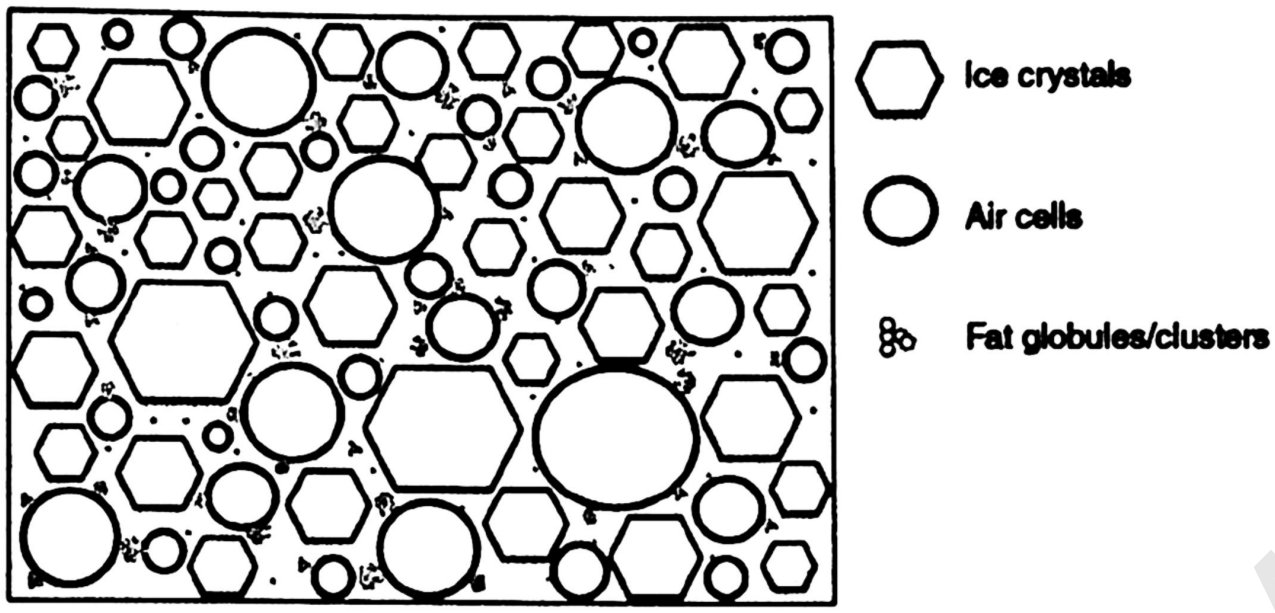



Figure 2.1: Ice cream microstructure (VanWees and Hartel, 2018).

### 2.1.1 Ice crystals

Ice crystals distinguish most dairy products from frozen dairy desserts. The hardness and scoopability of the component, the rate of melting and the smoothness during ingestion are influenced by the scale, number and distribution of ice crystals. Thus, during freezing, hardening, and storage, it is important to monitor the nucleation and growth of crystals. In the scrape-surface freezer (SSF), nucleation of ice crystals occurs only during dynamic freezing. During hardening, ice crystal growth occurs, as the volume of the process increases with decreasing temperature. Ice material fluctuates during storage and delivery as temperatures alter in an attempt to maintain phase equilibrium.

Heterogeneous nucleation occurs on the walls of the SSF in frozen dessert processing, where the temperature gradient between the refrigerant and the bulk solution is highest (Cook and Hartel, 2010). The coolant in the SSF must effectively extract heat from the dasher friction and release latent fusion heat from ice crystallisation while maintaining a proper temperature gradient to induce nucleation. A condensed slush containing several small dendritic-shaped crystals is the ice that forms on the wall, which is then scraped off the walls by the continuous rotating of

the scraper blades on the dasher and dispersed into the freezing barrel 's inner bulk. Most of the crystal nuclei melt during the initial stages of freezing due to the comparatively high temperature of the bulk. The crystal nuclei ripen into disc-shaped crystals as freezing persists and the bulk temperature drops (Cook and Hartel, 2010). The temperature at the centre of the freezer barrel decreases as more ice is produced along the length of the freezer, and the ice process at typical draw temperatures of-6 to-8 ° C is approximately at phase volume equilibrium.

Ice crystal size is critical for product quality; an example of ice crystals in ice cream is shown in Figure 2.2. Large ice crystals greater than 50 μm in diameter are easily detected by the consumer, and cause a textural defect called coarseness or iciness (Arbuckle, 1966). According to a recent survey of U.S. commercial products, the mean size of ice crystals in hardened ice cream ranged from 26–67 μm (Warren and Hartel, 2014). This variation is caused by differences in formulation and process, but also through differences in storage and distribution.


Figure 2.2: Ice crystals in hardened ice cream (Donhowe et al. 1991).

Ice crystal size also contributes to the product's many sensory properties. Increased ice crystal size and number also correlates with a more pronounced product

coldness since large crystals do not melt as easily and thus spend more time on the tongue (Roland et al., 1999; Wildmoser et al., 2004; Warren and Hartel, 2014; Amador et al., 2017). The scale of large ice crystals has also been associated with increased frozen product hardness (Muse and Hartel, 2004). In literature, the mean size of ice crystals is often mentioned, but the sensory quality of ice cream is often more specifically linked to the distribution of crystals. Items of high quality are smooth and creamy with a fine distribution of several tiny ice crystals, while the user senses ice crystals greater than 50 μm, resulting in a coarse or icy texture.

### 2.1.2 Fat globules

The fat globules are one of the most important components in ice creams and frozen desserts. Milkfat is used mainly in the formulation of most frozen dairy desserts, although non-dairy fats can be used in some applications. The mixture of fats and oils in products that use non-dairy fats should be balanced to imitate the action of milk fat during processing and consumption. During the processing of frozen desserts, while the composition of the fat does not change, the structure and functionality of the fat step changes drastically.

During freezing, some fat globules are destabilized and aggregate to form a large, complex, three-dimensional fat network within the frozen product. This action is called partial coalescence, or arrested, which explains how fat globules come together in the emulsion to share a membrane but do not coalesce completely. During the whipping and freezing process, the shear induced by the dasher drives fat globules into contact, which initiates coalescence. Fat globule coalescence activity is a function of many globule properties, including solid fat content, interfacial energy and membrane

properties (Goff, 1997a; Thiel et al., 2016). As the droplets merge, the free energy of two coalescing droplets is decreased; however, crystalline fat present in the globules prevents complete coalescence of the droplets (Figure 2.3). To avoid coalescence, the solid fat content of the globules must be large enough, but small enough to maintain some liquid-like character in the droplet, otherwise droplets would be completely stable to coalescence (Thiel et al., 2016). If too little fat is crystalline, the droplets will coalesce completely to minimize surface free energy.

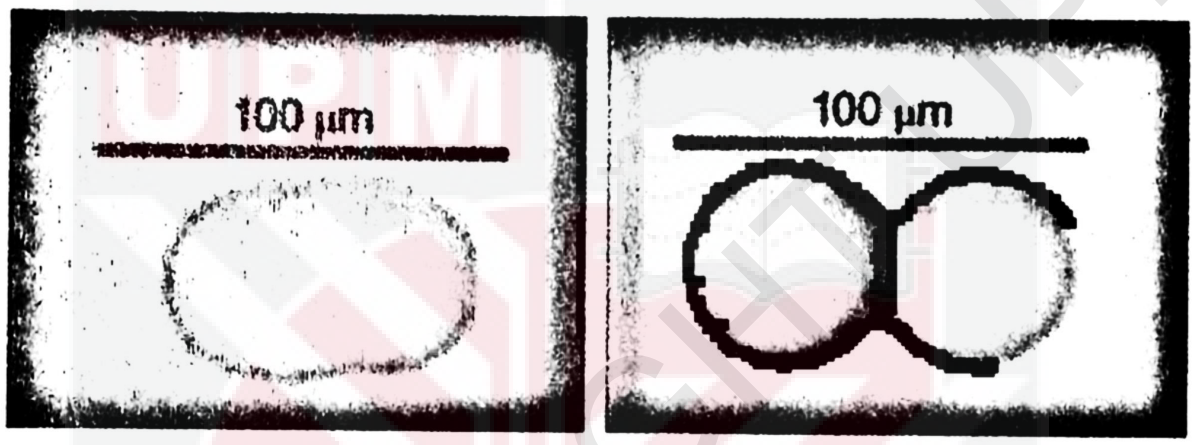



Figure 2.3: Different degrees of coalescence behavior of emulsions (VanWees and Hartel, 2018).

In frozen milk desserts, fat increases characteristic creaminess and smoothness by lubricating the palate and masking the coarse characteristics of ice crystals (Amador et al., 2017). Many flavour compounds are also solubilized by fat, enhancing product consistency and flavour. However, large clusters of partially-coalesced fat may detach from the emulsion and form undesirable butter grains if high-fat products are over-churned (Goff, 1997a).

### 2.1.3 Air cells

Air is a primary component of frozen desserts, often comprising a majority of the product by volume. During the complex freezing process, air cells are integrated and

are stabilised by protein and emulsifier adsorption at the interface, destabilised fat networks, and hydrocolloid stabilisers (Euston, 2008). In general, the initial size and distribution of the air cells is highly dependent on the whipping and freezing step processing parameters, and the final distribution depends on the conditions of formulation and storage. Ice cream air content is determined by its percentage overrun, which is expressed as a percentage rise in product volume higher than the sum of mix. For instance, the product has an overrun of 100 percent, and is 50 percent air by volume, if 200 L of product is generated from 100 L of mix. Goff and Hartel (2013) previously mentioned detailed overrun calculations for ice creams and frozen desserts).

To achieve the desired physical properties of the product, but also to manage production costs and meet regulatory requirements, strict control of overruns is necessary. Low-overrun goods produce more mixed ingredients per unit quantity, resulting in higher manufacturing costs and a higher quality product in general. U.S. federal identification requirements for product container weight cannot be met by goods with an overrun of more than 100 percent for a 35-40 percent solid ice cream.

The combination is aerated during freezing by the rotation of the dasher and the introduction of ambient or compressed air. Shear forces from the dasher cause air from the headspace to be incorporated during batch freezing operations. During batch freezing, overrun cannot be measured; instead, overrun is created by the relative proportions of air in the headspace and mix applied to the freezer. Batch freezers allow small-scale producers to manufacture a variety of products and flavours over a short period of time on a single model. In batch operations, ice creams, sherbets, and other frozen desserts that will be hardened are readily frozen. Batch equipment almost exclusively manufactures soft-serve ice creams, frozen yoghurt, frozen custard, gelato

and other items that are enjoyed directly after freezing. These products also generally have lower overrun (50–60%) than hardened products (80-100%).

During hardening and storage, the distribution of air cells shifts by three main phenomena: drainage, coalescence, and disproportionation. During hardening, coalescence or disproportionation leads to a decrease in the number of small air cells and a higher mean size of 20-60 μm of air cells (Warren and Hartel, 2014). The broad variety of air cell sizes and distributions found in commercial products is affected by variations in formulation, packaging, hardening and other parameters. Figure 2.4 shows typical air bubbles in ice cream observed by optical microscopy (Chang and Hartel, 2002b). Due to the low temperatures reached during extrusion, products made by LTE processes have been shown to have a fine distribution of small air cells (Wildmoser et al., 2004).

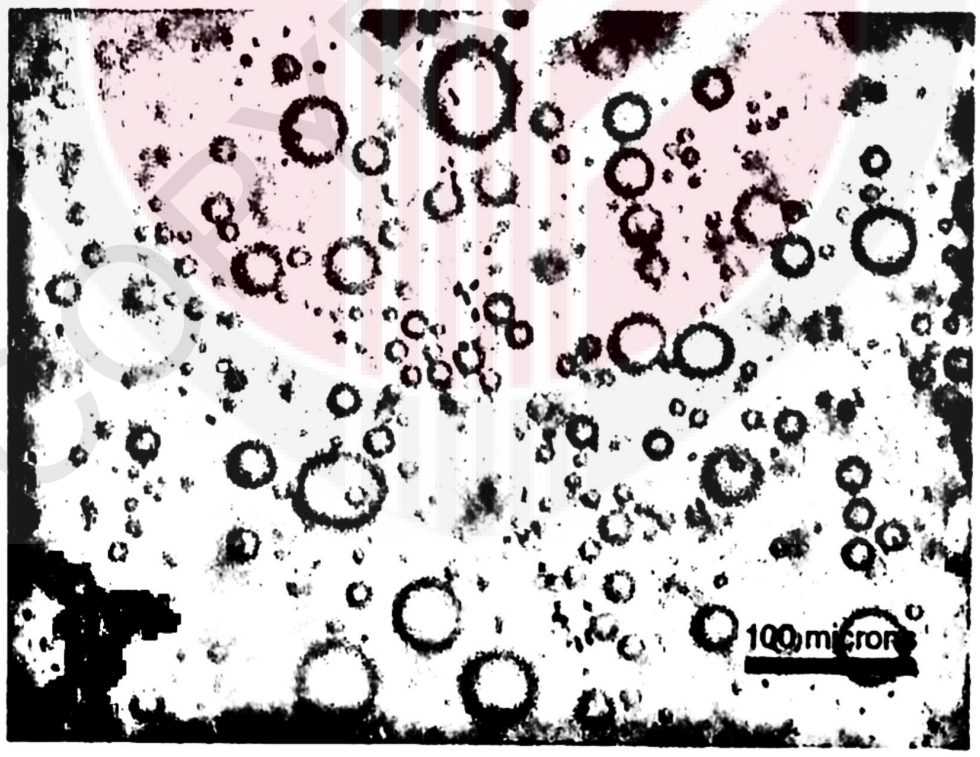



Figure 2.4: Air cells in commercial ice cream (Chang and Hartel, 2002b).

The stability of air cells may be influenced by other variables, such as fat content, fat destabilisation, and stabiliser addition. Sung and Goff (2010) found that increasing the degree of partial coalescence increased the stability of air cells but did not decrease

the size of air cells. Caldwell et al . ( 1992) found that ice creams had a higher mean air cell size without added stabilisers than stabilised items, making them more vulnerable during storage to shrinkage. Due to the high viscosity created during the low temperature process, frozen desserts developed by low temperature extrusion have very fine dispersions of small air cells. Changes in the structure of air cells as a result of hardening are not observed, as these materials seldom undergo additional hardening steps, but the instability of air cells during prolonged storage can still be observed.

## 2.2 Microscopy Techniques for Dairy Products

The three-dimensional structure of its structural elements and their interactions are highly influenced by the textural properties of a particular dairy product (Heertje, 1993). It is therefore not enough to know the chemical composition and bulk physical properties to fully understand the behaviour of dairy products, but how they interact and influence the spatial structure or organisation of the food constituents on the nano- and micro-length scales. Therefore, food microstructure studies provide a correlation between the physio-chemical properties, the behaviour of the process and the organoleptic properties of a specific dairy product (Figure 2.5). In particular , it is important to link microscopy to rheological and sensory techniques for a fuller understanding of food behaviour, requiring a multivariate approach to experimental design.

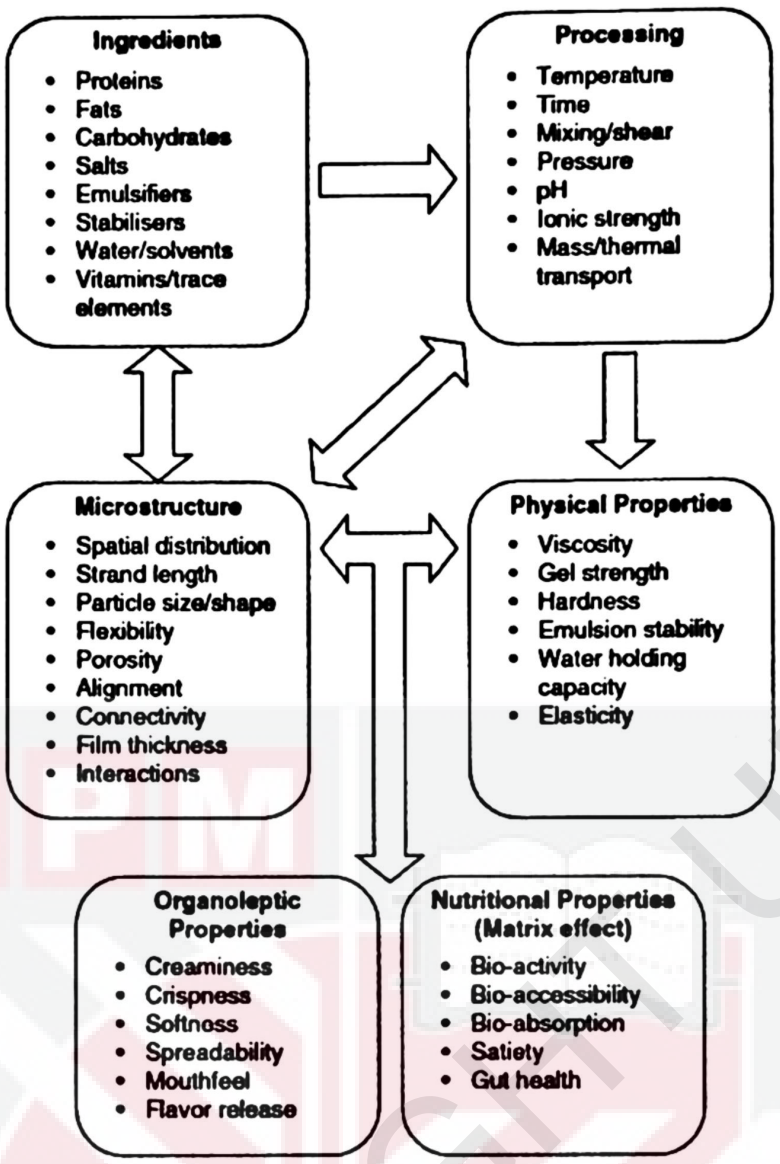



Figure 2.5: Diagram showing inter-relationships between microstructure and functionality of dairy products (Auty, 2018).

Many of the common techniques used to study food microstructure have been adapted from specimen preparation procedures for biological tissue. There are, however, clear concerns relating to the preparation of food items for microscopic analysis that the investigator should be aware of. Many foods have high moisture, fat or sugar levels and it may be difficult to preserve the original microstructure of such materials , particularly for electron microscopic studies that may require low moisture, conductive specimens. Dried ingredients with a relatively small particle size (< 100 μm) such as spray-dried powders, crystalline sugars, starches, etc. can be examined in their natural state and require little sample preparation. However, highly refractile or opaque solid and semi-solid food materials must be rendered sufficiently thin to transmit light, and this is normally accomplished either by compression or sectioning.

It is possible to compress or smear soft materials around a microscope slide. Chemically fixed solid food materials and cellular tissues may be dehydrated, then trapped in paraffin wax or plastic resin prior to microtomy sectioning. Alternatively, in a cryostat, frozen parts, approximately 5-20 μm thick, can be cut. It is then possible to observe sectioned material using either of the optical or chemical contrast techniques listed below. For example, powdered materials, such as spray-dried milk powder particles, should be installed in a transparent, immiscible liquid, such as sunflower oil, which should be sufficiently viscous to limit the particles' movement in Brown. The key techniques of microscopy used to study dairy products are described in the following table;

Table 2.1: Main microscopy techniques used in food microscopy (Auty, 2018).

| Technique | Radiation type | Incident radiation wavelength | Approximate resolution | Application |
|---|---|---|---|---|
| Stereo-microscopy | Photons | 400–750 nm | 5 μm | Overview of microstructure |
| Light microscopy | Photons | 400–750 nm | 200 nm | Ingredient localization |
| Fluorescence microscopy | Photons | 350–400 nm (u.v.)<br>400–750 nm (visible) | 200 nm | Ingredient localization |
| Confocal microscopy | Photons | 350–400 nm (u.v.)<br>400–750 nm (visible) | 200 nm | Ingredient localization, 3D information |
| SEM | Electrons | 0.001–0.01 nm | 4 nm | Large depth of field-simulated 3D view |
| TEM | Electrons | 0.01–0.01 nm | 1 nm | Fine structural detail, macromolecular interactions |
| AFM | N/A (physical cantilever) | N/A | | Surface topology, nano-mechanical behavior |

### 2.2.1 Scanning Electron Microscopy (SEM)

As shown in Figure 2.6, the scanning electron microscopy technique is used to form the image by scanning a focused electron beam across the sample. The first

electrons enter the solid sample and are enveloped by a broad variety of processes of elastic dispersion. Diverse signals are produced as long as the electrons collide. The sample surface is then analysed or an image is created. Secondary electrons are the energy of a few tens of eV, high-energy electrons back-scattered from the primary beam, and characteristic X-rays (Bogner et al., 2007).

As a combination of light microscopy and transmission electron microscopy, SEM has been clarified (Aguilera and Stanley, 1999). SEM has many benefits, such as easy preparation of samples, a lot of magnification, high field depth and the fact that the image is a representation of electronic data that enables image processing and quantification to be carried out. It has some drawbacks, however, such as the analysis of insulating samples and the difficulty of examining hydrated samples without in some way altering their condition (either drying or freezing). Objects can therefore be added by these pretreatments (Harker et al., 2006; James and Fonseca, 2006).

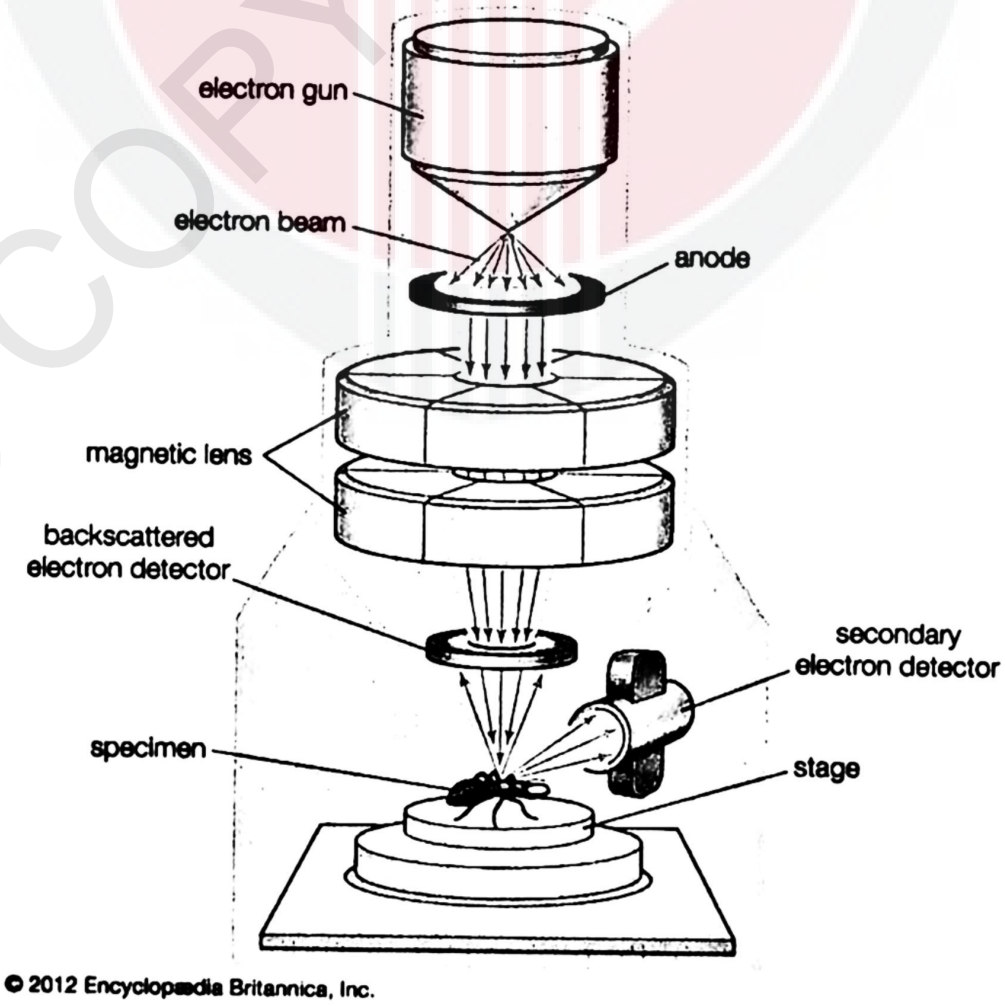



Figure 2.6: Scanning Electron Microscopy (Bradburry, 2009).

### 2.2.2 Cryo-SEM

Cryo-SEM (cryo-SEM) scanning electron microscopy is a straightforward way of examining without resolving the ice crystals of frozen samples. In addition, for improved conductance of the electrons, the sample is coated with metal (Preetz et al., 2010). The frozen sample used in cryo-electron microscopy can be moved within the microscope immediately to a low temperature level and viewed directly (Belkoura et al., 2004). Rapid physical detection is provided in cryo-SEM, and the possibility of introducing chemical detection artefacts, structural failure or shrinkage is avoided. Thus, a sample's triphasic structure is maintained in this technique with the distribution of solid, liquid and gas in a state closer to its natural state (James and Fonseca, 2006). However, like SEM, this technique is considerably expensive, and it is one of the main drawbacks.

### 2.2.3 Transmission Electron Microscopy

Transmission electron microscopy involves moving a small beam of electrons at accelerating voltages in the 40-120 kV range through a thin specimen. Either as a negatively stained dispersion or in the form of a thin section or a metallic copy, the sample may be prepared. A relatively fast technique where the sample is dissolved in a solution of heavy metal salt such as uranyl acetate is negative staining. The sample appears transparent when dried, but allows internal or surface structures to be observed. For dilute protein dispersions, this approach is useful and was used to study casein micelles and their interaction with whey proteins (Creamer et al., 1978).

In TEM, the picture is created by the interactions between the sample and electrons as shown in Figure 2.7. The resolution is in proportion to the electrons'

acceleration voltage. Although the voltage rises, because of the short wavelength of the electrons, the resolution is greater. Increased acceleration voltage, however, results in poorer contrast as the higher velocity reduces electron scattering. TEM may have voltages ranging from 40 to 120 kV and microscopes ranging from 200 to 400 kV. Moreover, when colloidal systems are used, voltages between 80 and 200 kV are usually employed (Kuntsche et al., 2011; Klang et al., 2012).

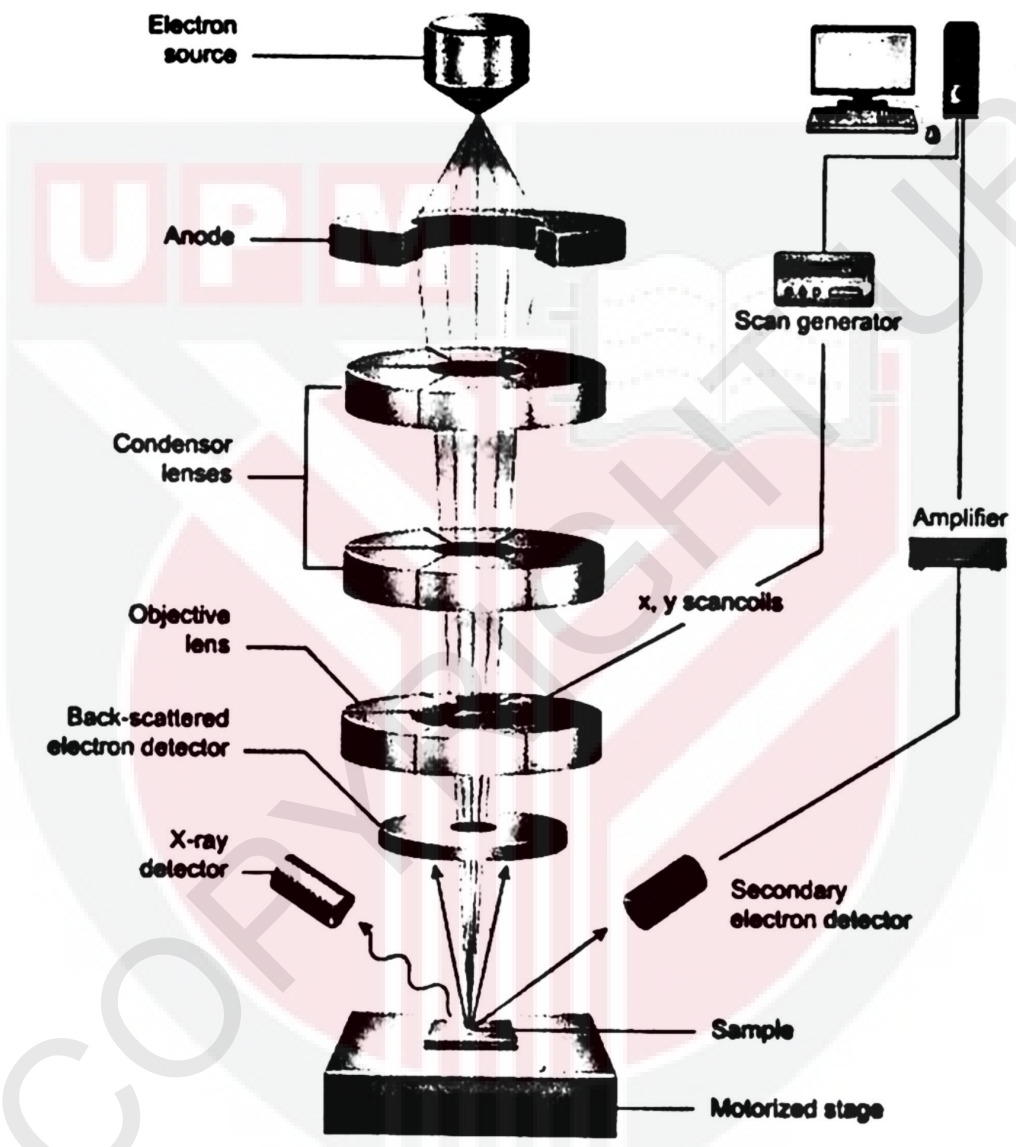



Figure 2.7: Transmission Electron Microscopy (Inkson, 2016).

## 2.3 Dino-Lite

Dino-Lite (Figure 2.8) is a portable digital microscope and eyepiece camera that can provide a portable and a high-quality microscopy images and videos up to 900x magnification and 5 Megapixel resolution interfacing to PC and MAC. The Dino-Lite digital handheld microscope series and Dino-Eye microscope eyepiece series have

been developed and extended over the years to better serve customers, offering a wide variety of digital microscope solutions for all applications, including industrial, electronics, quality control, scientific, study, health care, forensics, education and many others.


Figure 2.8: Dino-Lite

Nowadays, for thousands of businesses and practitioners worldwide, the Dino-Lite optical microscope is an irreplaceable instrument. The Dino-Lite range includes various networking options with over 150 models: USB, TV or VGA, as well as advanced lighting, such as ultraviolet or infrared, and numerous levels of magnification. The product line-up is completed by a wide variety of stands and accessories and ensures that the Dino-Lite variety provides solutions to meet the needs of the home consumer through to the most demanding professional. While it is tiny in size, it is full of features.

# CHAPTER 3

# METHODOLOGY

In this chapter, the method of the experiment will be explained. This experiment will consist of four stages which are preparation of device, preparation of the ice cream mix, production of the ice cream mix and last stage will be analysis of ice cream mix.

## 3.1 Preparation of device

Direct observation approach by optical microscopy with episcopic coaxial lighting was originally developed by the physicists studying the polar ice structures (Arnaud et al., 1998), and Faydi et al. (2001) adapted this process to the ice cream to describe the frozen ice cream mix. This approach bases basically on the light flux that the sample surface represents. For example, surfaces that are present in commercial ice creams containing overrun at the air bubbles. (Caillet et al., 2003), (Goff, 1997). A thin slide (1 mm thick) of fresh ice cream that has just completed the freezing process is placed in a −18 ° C freezer for 24 hours until the ice cream has matured.

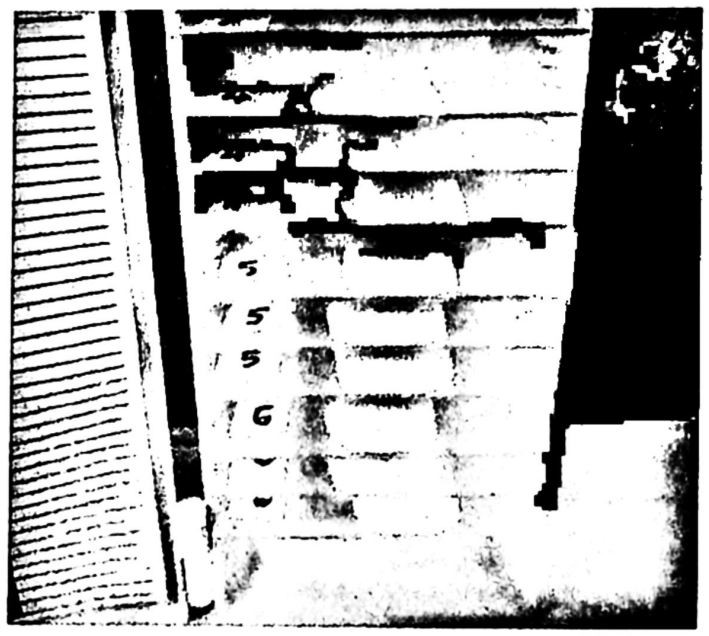


Figure 3.1: Ice sample was prepared on a slide for 24hr

After obtaining a suitable surface quality, the ice cream sample was directly observed inside a freezer with a digital stereomicroscope (Dino-Lite), equipped with a digital video camera and a LED light source providing the episcopic coaxial lighting. The freezer was initially set to −40 °C which is very cold. However, the experiment was conducted with the freezer door opens. Thus, the ambient temperature between the freezer temperature and the surrounding temperature was −21.5 °C which is an optimal temperature for the experiment to be conducted. The video microscope was placed on top of the box provided in the freezer and the images were stored with a laptop located outside the freezer where it is connected via USB.

## 3.2 Preparation of ice cream mix

Preparation of ice cream samples were conducted in the Food processing Quality Lab, Faculty of Engineering, University Putra Malaysia (UPM). Ice cream mix was prepared based on the formulation that was given. The detailed of the ingredients will be show on table below.

### 3.2.1 Ice cream mix formulation

The ice cream samples were prepared based on the ingredient as shown on the table below:

Table 3.1: Formulation was used to prepare hard ice cream (Parid et al, 2018)

| Ingredients | Weight Composition (%) |
|---|---|
| Water | 60.9 |
| Skimmed Milk Powder | 18.1 |
| Sugar | 16.3 |
| Creamer | 3.6 |
| Emulsifier | 0.4 |
| Stabilizer | 0.3 |
| Flavoring | 0.4 |

For this experiment, 4 different samples were prepared by manipulating the oil content which is 4%, 5%, 6% oil and also a control sample (0% oil). Each percent of the oil replaced the percentage of water. The ingredient as table below:

Table 3.2: Formulation was used to prepared hard ice cream with added oil.

| Ingredients | Weight Composition (%) |
|---|---|
| Water | 56.9 |
| | 55.9 |
| | 54.9 |
| Oil | 4 |
| | 5 |

| | |
|---|---|
| | 6 |
| Skimmed Milk Powder | 18.1 |
| Sugar | 16.3 |
| Creamer | 3.6 |
| Emulsifier | 0.4 |
| Stabilizer | 0.3 |
| Flavoring | 0.4 |

## 3.3 Production of ice cream

After formulation of ice cream has been finalized, process of production of ice cream took place. The process of making ice cream includes weighing the ingredients, mixing, pasteurization, homogenization, ageing, froze and hardening.

### 3.3.1 Weighed Ingredients

Dry and wet ingredients such as sugar, skimmed milk powder, creamer, emulsifier, stabilizer, flavoring and oil were weighed using a weighing balance before putting them together in a mixing bowl. The weight of each ingredient was referred to the mass that have been calculated.

Figure 3.2: The ingredients was placed together inside a beaker.

### 3.3.2 Mixed

The sample formulation of ice cream mix was mixed together using the mixer (Model 5K5SS, KitchenAid, St Michigan, USA) as shown in Figure 3.3. The ice cream mix was mixed for 5 minutes so that all the ingredients which is wet and dry ingredients is mixed well with each other.

Figure 3.3: Mixing process

### 3.3.3 Pasteurization

After mixing, the sample was pasteurized. The mixture was pasteurized on the stove with slow fire until the temperature of the mix reached of 80°C and was held for 15 seconds. The temperature of mix was detected by using thermometer. Make sure that the mixture is always stirred to prevent crust from take place.

Figure 3.4: Pasteurization process on the stove

### 3.3.4 Homogenization

After pasteurization, the ice cream mix was immediately transfer into a jug to undergo homogenization process. The mixture was homogenized by using the homogenizer (Success Technic Industries, Model WT500, Malaysia) at 12 000rpm for first stage for 120 seconds and then was reduced to 10 000rpm at second stages for another 120 seconds. Figure 3.5 shows how the homogenization process of ice cream mix.

Figure 3.5: Homogenization process


Figure 3.6: Lab scale Homogenizer

### 3.3.5 Ageing process

After homogenization, ice cream mix was transferred into container for ageing process. The mix was rapidly cooled at 4°C and aged for overnight to prevent contamination and to improve the whipping qualities of mix and texture of the ice cream mix. Minimum time required for ageing process is 4 hours and usually overnight. Ageing process was shown in Figure 3.7.

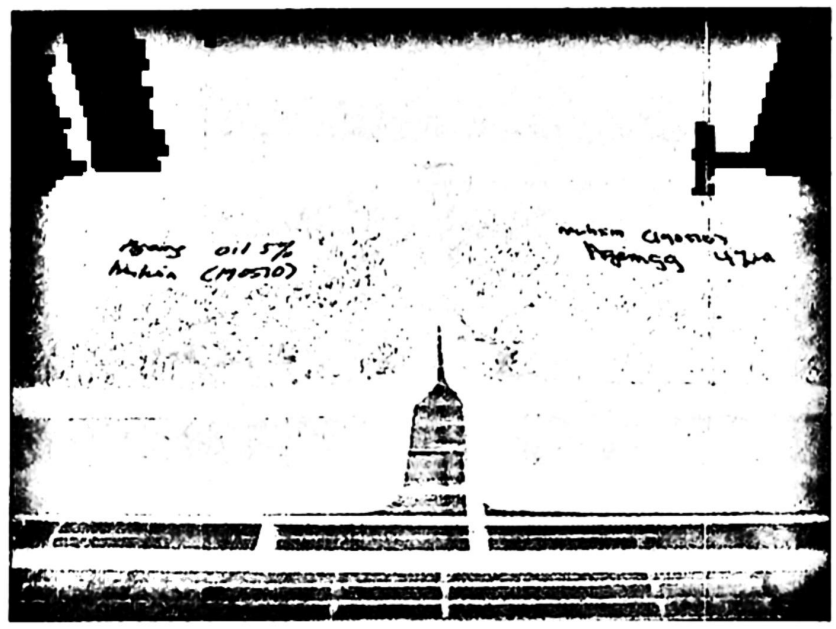


Figure 3.7: Ice cream mix undergo ageing process

### 3.3.6 Freezing process

After ageing the ice cream mix for overnight, the aged ice cream mix was whipped and frozen in the batch ice cream freezer (Breville, Model BC1600, Australia). The freezer was set to -20°C. The freezing process for ice cream mix was shown in Figure 3.8 and Figure 3.9.

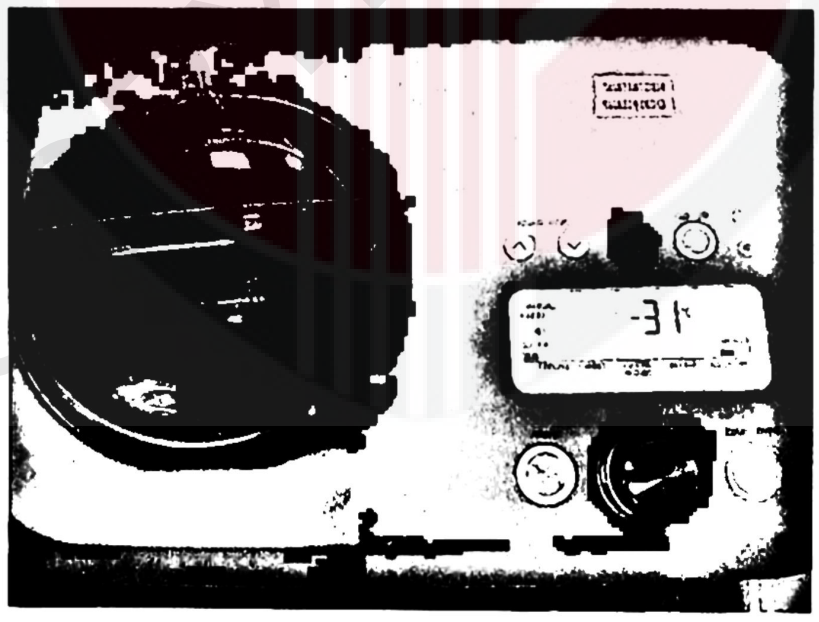


Figure 3.8: Batch ice cream freezer

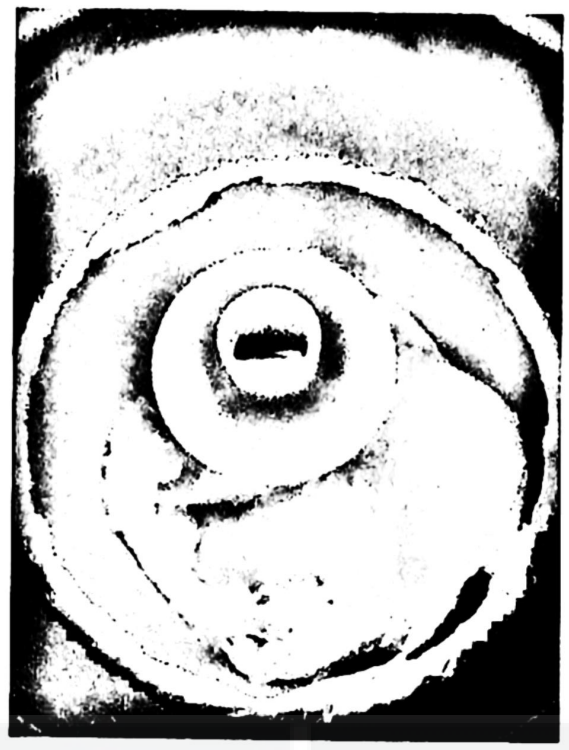


Figure 3.9: Ice cream mix after undergo freezing process

### 3.3.7 Hardened

After the freeze-churning process, the ice cream was filled into the air-tight container and was hardened in the freezer at -18°C to -25°C for minimum 24 hours. Figures 3.10 shows the ice cream sample after hardening process in the batch-freezer.

Figure 3.10: Ice cream after hardening process

## 3.4 Analysis of physical characteristics of ice cream

Four analyses were conducted to determine the physical characteristics of the ice cream sample. The ingredient of each of the sample was manipulated by the content

of cooking oil which is 4%, 5%, 6% and also a blank sample (0% cooking oil). The analysis involved are the melting rate, firmness, overrun and microstructure analysis.

### 3.4.1 Analysis of microstructural characterization of ice cream

Microstructural analysis is one of the important analyses in this experiment. This was to observe the structure of ice cream such as fat globules, air cells and ice crystals. The ice cream sample was directly observed inside a freezer with a digital stereomicroscope (Dino-Lite), equipped with a digital video camera and a LED light source providing the episcopic coaxial lighting. The freezer was initially set to −40 °C.. The temperature surrounding the setup was −21.5 °C as the freezer door was opened during the experiment, which is an optimal temperature for the experiment to be conducted. The video microscope was placed on top of the box provided in the freezer and the images were stored with a laptop located outside the freezer where it is connected via USB.

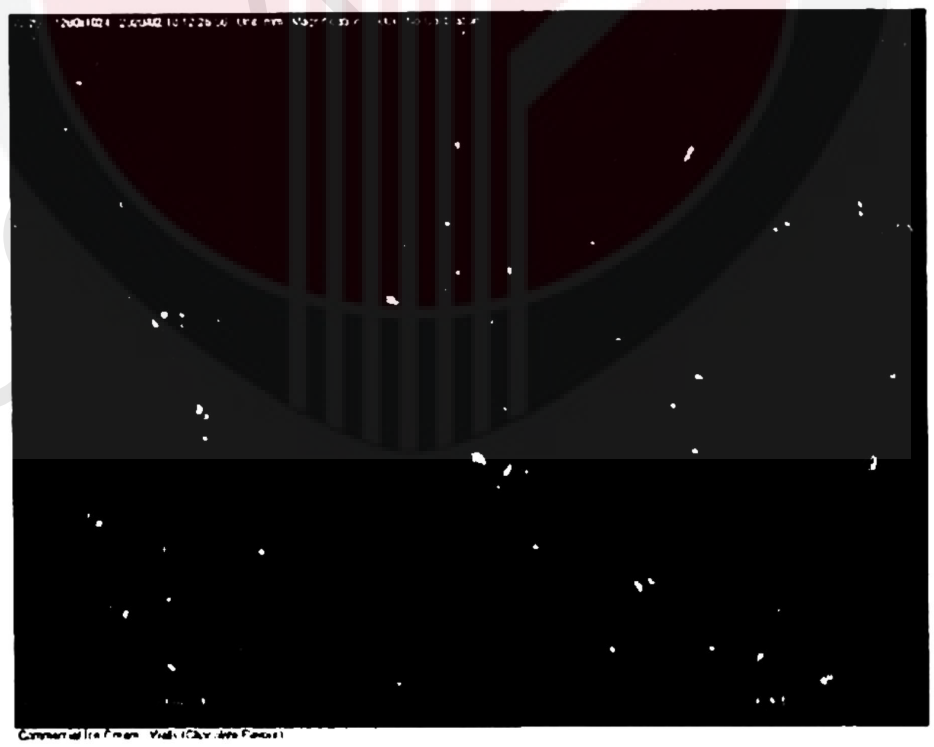
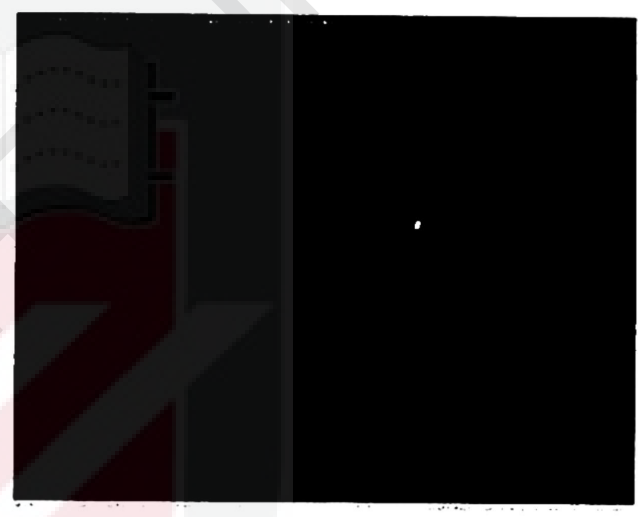
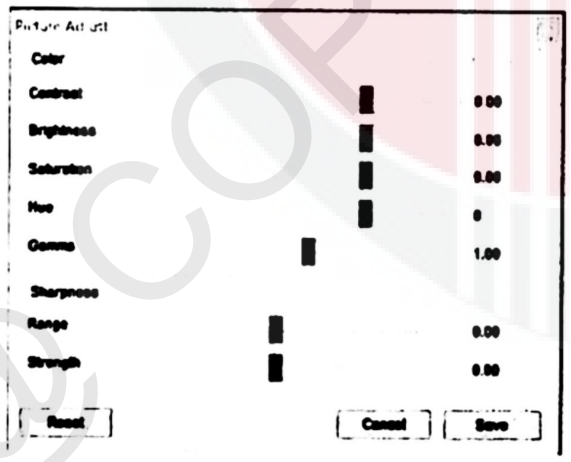
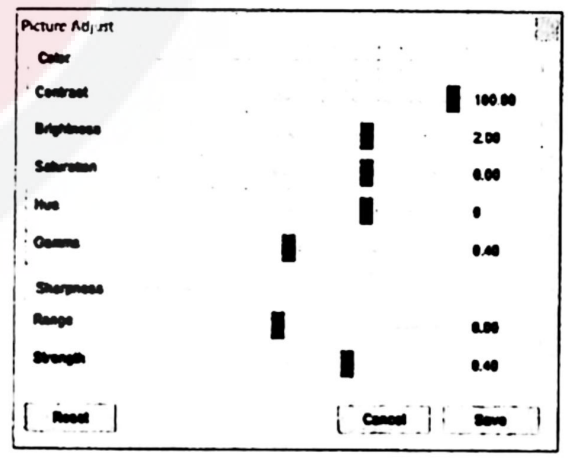


Figure 3.11: Sample image from commercial ice cream

*Image Adjustment*

Some images obtained needed to be adjusted to have a better view on the microstructure. So, when we have a better observation, it is much easier for us to identify the structures such as air cells, fat globules and ice crystals. Below are some adjustments that the author made from Dino-Lite software which is DinoCapture 2.0.

Table 3.3: Image adjustment

| | Before | After |
|---|---|---|
| Image Captured |  |  |
| Image Setting |   |   |

This setting is not a fixed setting but it is flexible based on the image we obtained. The important part in adjusting the image setting is to get the clearest image of the structure without changing important aspect such as color, saturation and structure.

### 3.4.2 Analysis of melting rate of ice cream

Melting rate was measured for each sample. In this melting test, wire gauze, stopwatch, beaker and weighing balance were used. 200g of hardened ice cream samples were taken. Each sample was placed above the wire gauze and a beaker was placed on the weighing balance and the weight was recorded. The duration of the experiments depends on the samples, considering the time needed for the total loss of the structure. The reading was taken and recorded for every 5 minutes. The weight of the melted ice cream was recorded and tabulated with time taken. Setup for this test is shown in Figure 3.12.

Figure 3.12: Before melting started

Figure 3.13: After melting ended

### 3.4.2 Analysis of firmness of ice cream

Texture analysis is an important test in determining the textural properties of food. The texture of any food is multi-faceted and connects with the consumer sensory attributes. Figure 3.14 shows the test conducted using texture analyzer (TA.TX.plus, Stable Microsystem, England). The results were automatically generated by the

system. For each sample, three measurements were carried out using 45° Perspex cone probe with test speed of 1.0 mm/sec. The ice cream was penetrated by a probe to a distance of 20 mm. The test must be done as quickly as possible because to prevent the samples from melt down thus will affect the hardness of the ice cream. The texture analyzer was used for this test by giving controlled force to the ice cream and respond taken is force versus time.

Figure 3.14: Ice cream sample placed on a texture analyzer (TA.TX.plus, Stable Microsystem, England).

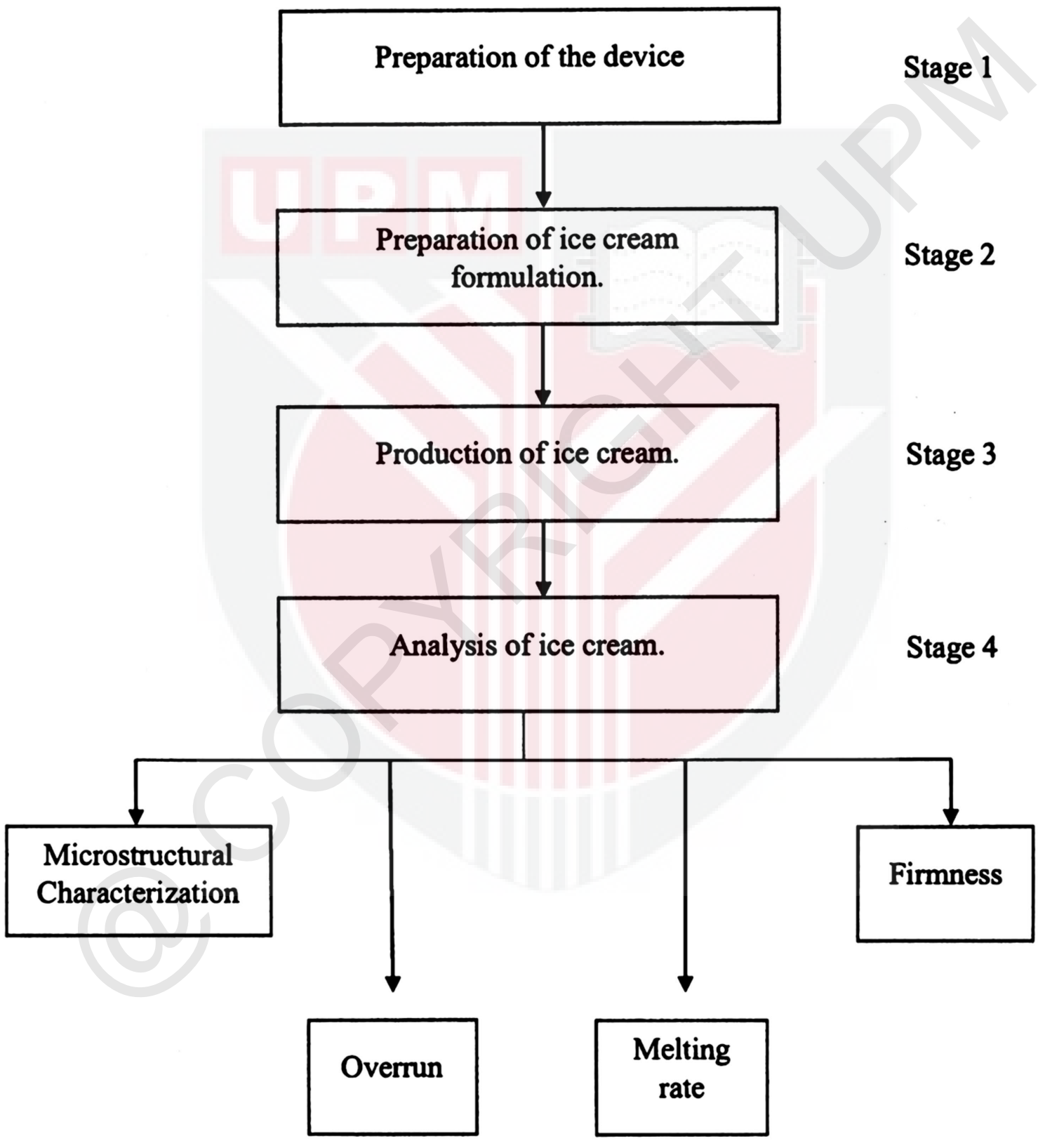
### 3.4.3 Analysis on overrun of the ice cream

Overrun is the amount of ice cream in the product that is air added during the freezing process (Slices Concession, 2017). Overrun can be calculated by comparing the weight of ice cream before (ice cream mix) and after (ice cream) undergo freezing process using the same volume of container. The value was then plugged into the formula below:

$$\text{Overrun (\%)} = \frac{\text{weight of the ice cream mix} - \text{weight of the ice cream}}{\text{weight of the ice cream mix}}$$

## 3.5 Summary of methodology

Chapter 3 discussed on the methodology used in this study to prepare the device and also the preparation and the production of the ice cream sample throughout the process. After that, the tests required for analyses such as melting rate, firmness and overrun were explained.



# CHAPTER 4

# RESULT AND DISCUSSION

This chapter consist of the results obtained from the experiments which are microstructural characterization, melting rate, overrun and hardness analyses.

## 4.1 Analysis on microstructural characterization of ice cream.

The result of this analysis was obtained by comparing the image between all ice cream samples available. By using digital stereomicroscope (Dino-Lite, AM4113 Series, Taiwan) with a fixed 500x magnification, main structure of ice cream such as ice crystals, air cells and fat globules was identified. The images obtained as below:


Figure 4.1: 0% Oil


Figure 4.2: 4% Oil

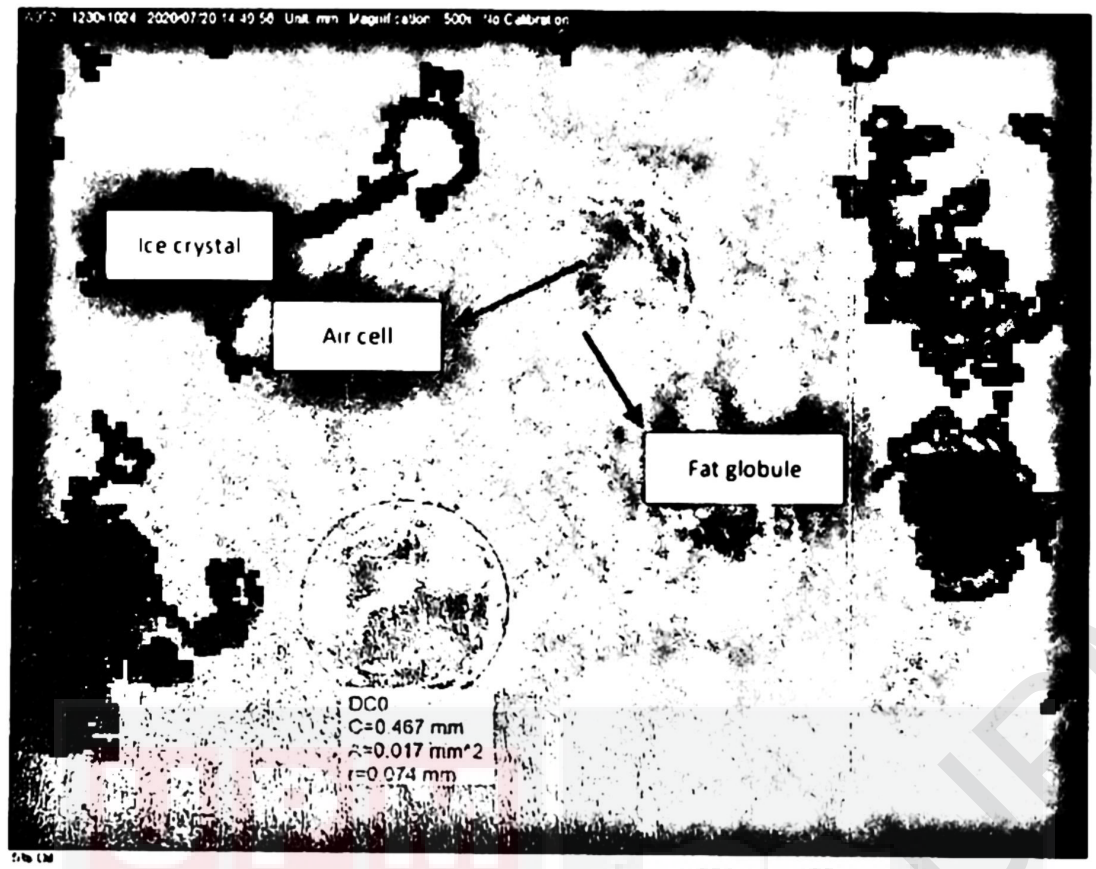



Figure 4.3: 5% Oil


Figure 4.4: 6% Oil

From images obtained, we can observe a few structures of ice cream that was successfully captured using Dino-Lite with 500x magnification. We can see that when the percentage of fat increase, yellowish colour of the fat was increasingly visible on

the surface of the ice cream. From all the images, we can observe that structure such as ice crystals, fat globules and air cells in Figure 4.1, Figure 4.2, Figure 4.3 and Figure 4.4 visible clearly. Usually, fat globule will attach to the air cells during ageing process in occurrence of fat crystallization. The milkfat exists in tiny globules that have been formed by the homogenizer. The emulsifiers were added to ice cream to actually reduce the stability of this fat emulsion by replacing proteins on the fat surface. If emulsifiers were not added, the fat globules would have so much ability to resist this coalescing. Due to the proteins being adsorbed to the fat globule, the air bubbles would not be properly stabilized and the ice cream would not have the same smooth texture.

From the images, we can see that the size of air cells decreases and the shape is more even as the percentage of oil increases. The radius of the air cell from ice cream sample of 0% oil is 0.089mm and from ice cream sample of 6% oil is 0.056mm. Higher fat content will produce higher viscosity of ice cream. Mixture with high viscosity holds air structure better than lower viscosity mixture. Ice crystals also shows increment in size as the percentage of oil increase which promotes higher firmness (Muse & Hartel, 2004).

## 4.2 Analysis on melting rate of ice cream.

The melting rate of ice cream was conducted as stated on the research methodology. It is important to determine the melting rate of the ice cream for the consumer to consume the product without loss of its structure for a longer time. Figure 4.5 below shows the result of melting rate for all ice cream samples.

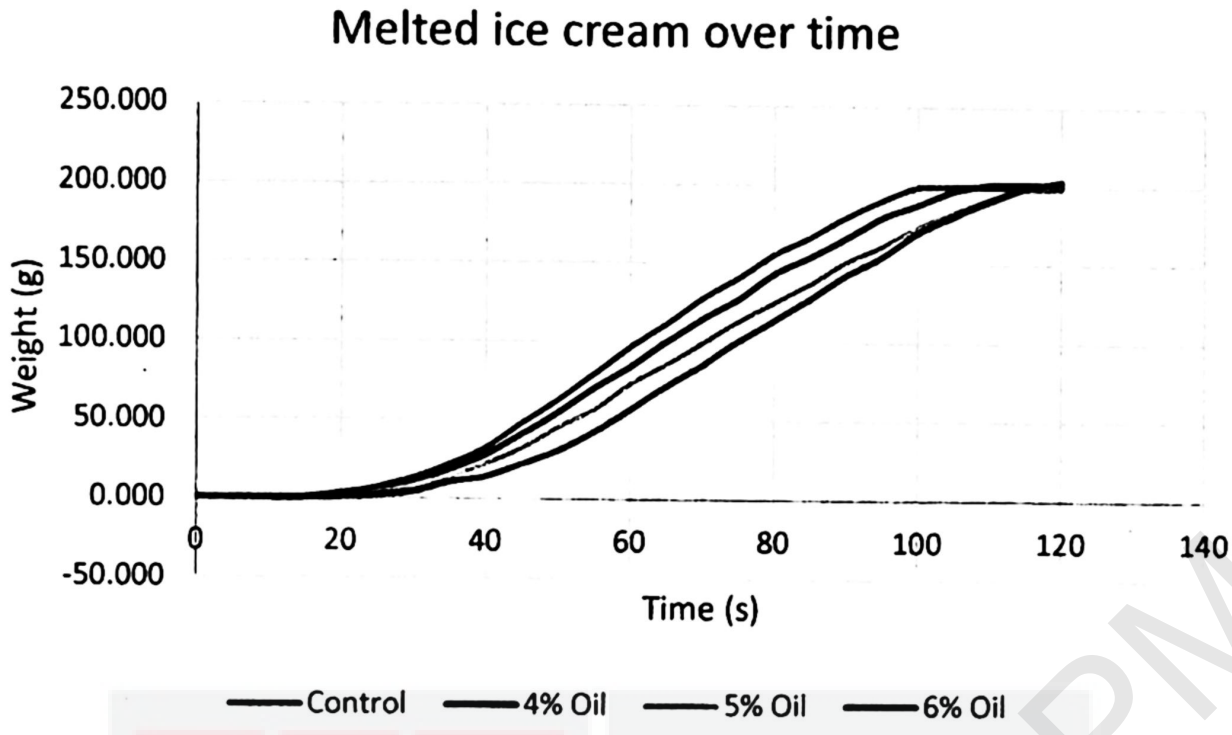



Figure 4.5: Graph of melted ice cream over time

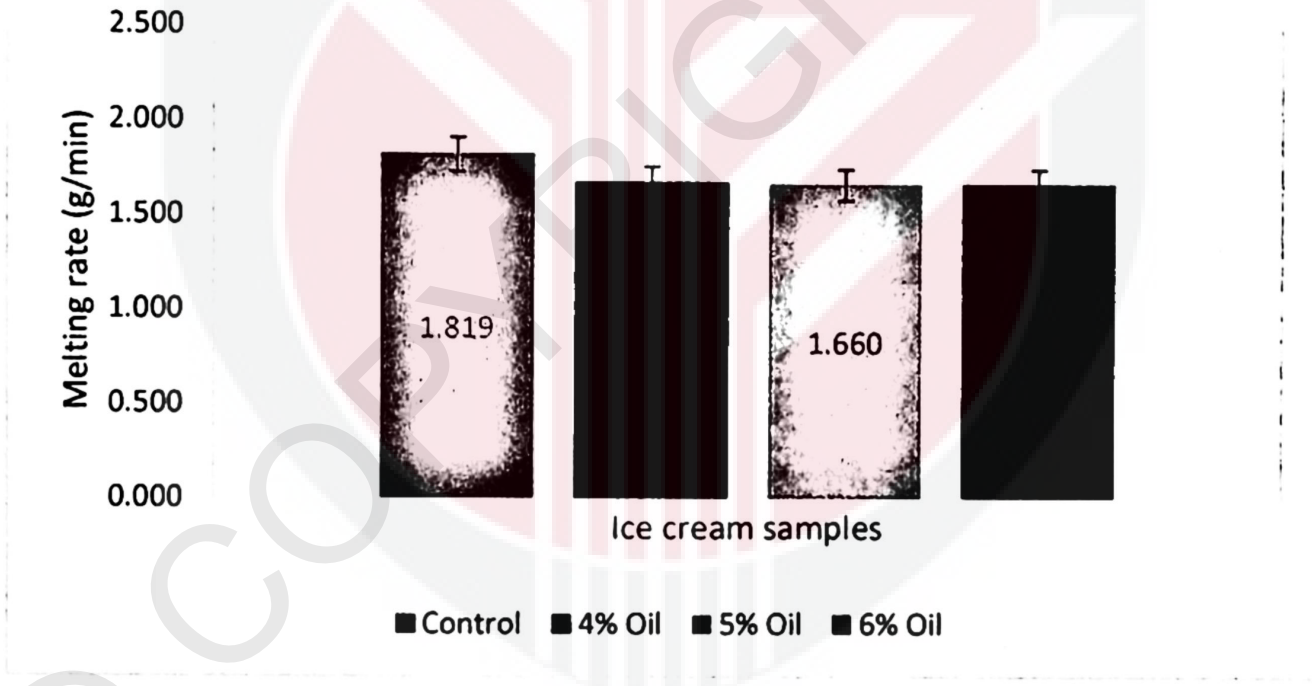



Figure 4.6: Graph of melting rate of ice cream samples

Figure 4.5 shows the data on mass of melted ice cream over time for all the samples. It was found out that as time increases, the mass of melted ice cream increases too. We can observe from the graph that as the percentage of oil increase in the ice cream mix, it will take longer time for the ice cream to melt down. At the end of the first 60 minutes, mass of melted ice cream was 56.923 g for 6% oil, 73.158 g for 5% oil, 83.987 g for 4% oil and 96.687 g for controlled sample. It was found that almost

each sample of ice cream begins to melt in the 15th minute and have a constant melt down after 45th minute until 100th minute. All of the samples have the same trend for melt down. From Figure 4.6, the melting rate for 4% oil is 1.673 g/min, for 5% oil is 1.660 g/min, for 6% oil is 1.659 g/min and 1.819 g/min for controlled sample. This result shows that 6% oil have a slower melting rate and 0% oil (control) have the highest melting rate. The melting rate of the samples was calculated based on the equation below (Boonterm et al., 2012):

$$\text{Melting rate (g/min)} = \frac{\text{Weight of melted ice cream within total minutes (g)}}{\text{Total minutes (min)}}$$

Ice cream that has a high fat content tends to melt more slowly. Rolandet al. (1999) analyzed ice cream formulated with various fat percentages and found that high fat content reduces the melting rate. This has been verified by Alamprese et al. (2002) who reported a softer ice cream with higher fat content and a slower melting rate. Hyvonn et al. (2003) found similar results and reported that a slightly retarded melting of ice cream in the mouth was due to the increase in fat content.

During dynamic freezing, fat undergoes partial coalescence, or destabilisation. Partial coalescence occurs when a protruding fat crystal from one fat globule pierces another globule's interfacial film, creating a largely irreversible connection between the inner phases of the globules (Walstra, 2003). These partly coalesced globules are essential to the production of smooth texture and melt down resistance (Goff, 1997): melt down levels typically decrease as fat destabilization grows (Nielsen, 1976; Muse & Hartel, 2004; Goff & Hartel, 2013).

Study by Campbell and Pelan (1998) found that melt down of ice cream was also influenced by draw temperature (temperature at which ice cream is removed from the

ice cream machine). As the draw temperature decreases, the resistance toward melt down increases due to increase in overrun and fat destabilisation, although ice crystals may also have influenced the melt-down rate. However, Goff & Hartel (2013) states that there are many other structural factors that can be considered as the cause of ice cream melt down. No direct association has always been observed between partial coalescence and meltdown rate.

In recent years, the impact of ice cream microstructure on meltdown has been debated. Changing the structure by changing recipes for ice cream offers various meltdown behaviours. The level of fat destabilization, mix viscosity, and overrun was found to affect meltdown (Amador et al., 2017).

## 4.3 Analysis on overrun of ice cream.

Overrun is an important aspect in ice cream production. Air in ice cream influenced the melting rate and firmness of ice cream. Figure 4.7 shows the overrun data for all ice cream samples.

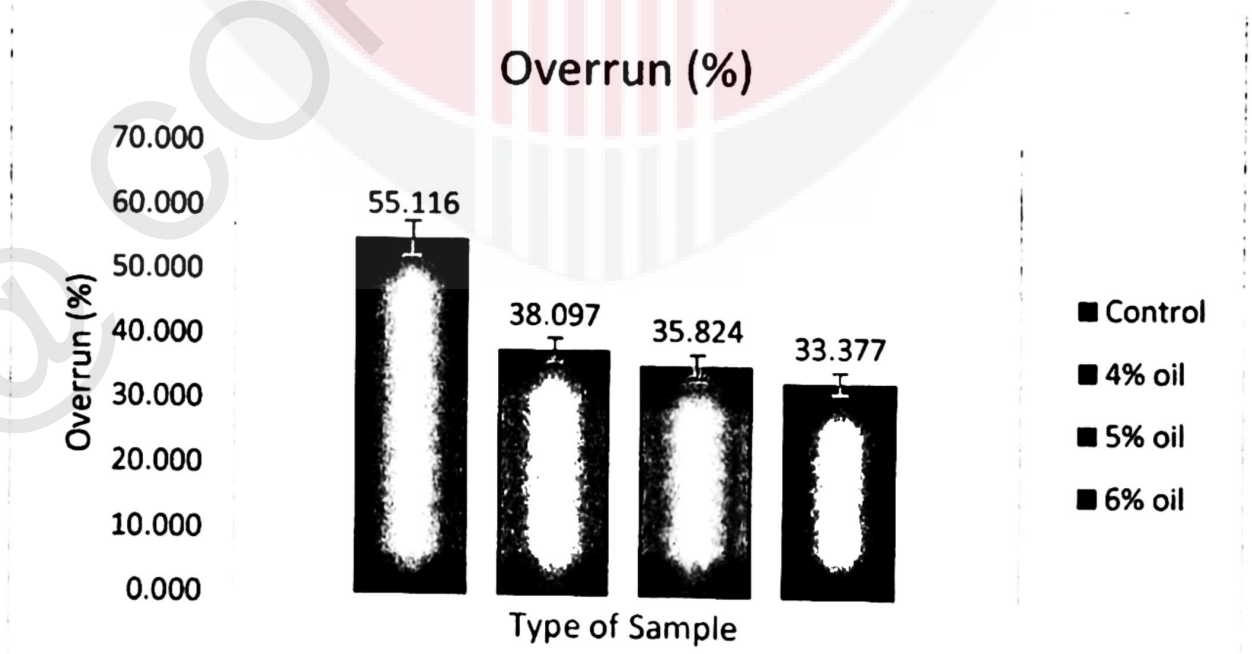



Figure 4.7: Overrun of ice cream samples

Figure 4.7 above shows the overrun values of 4 different samples of ice cream. Overrun was known as the amount of the air added in ice cream by comparing the

weight of a fixed volume of ice cream mix before and after the ice cream undergo freezing process. From the bar graph, we can see that controlled sample has the highest overrun value which was 55.116% compared to three other samples. The other samples, 4%, 5% and 6% do not have so much different from each other which are 38.097%, 35.824% and 33.377% respectively. We can conclude from the graph overrun value decreases as percentage of fat increases.

According to Adapa et al. (2000), ice cream mix with high fat content will produce higher viscous component especially in a freezing temperature. High viscous component could have prevented air incorporation. According to them, high viscous systems do not favour foaming capacity but do favour foam stability. We can observe from Figure 4.1 until Figure 4.4 that the microstructure of air cells getting smaller and more even as the fat content increases proved that the stability of the foam was increased. Apart from that, previous research by Stanley et al. (1996) has shown that the uses of stabilizers in ice cream mix led to increases viscosities which in turn resulted in lower overruns but more stable foam.

## 4.4 Analysis on firmness of ice cream.

The firmness of the ice cream had been carried out as the methodology that had been explained. The figure below shown the graph that has been plotted based on the results obtained from the experiment.

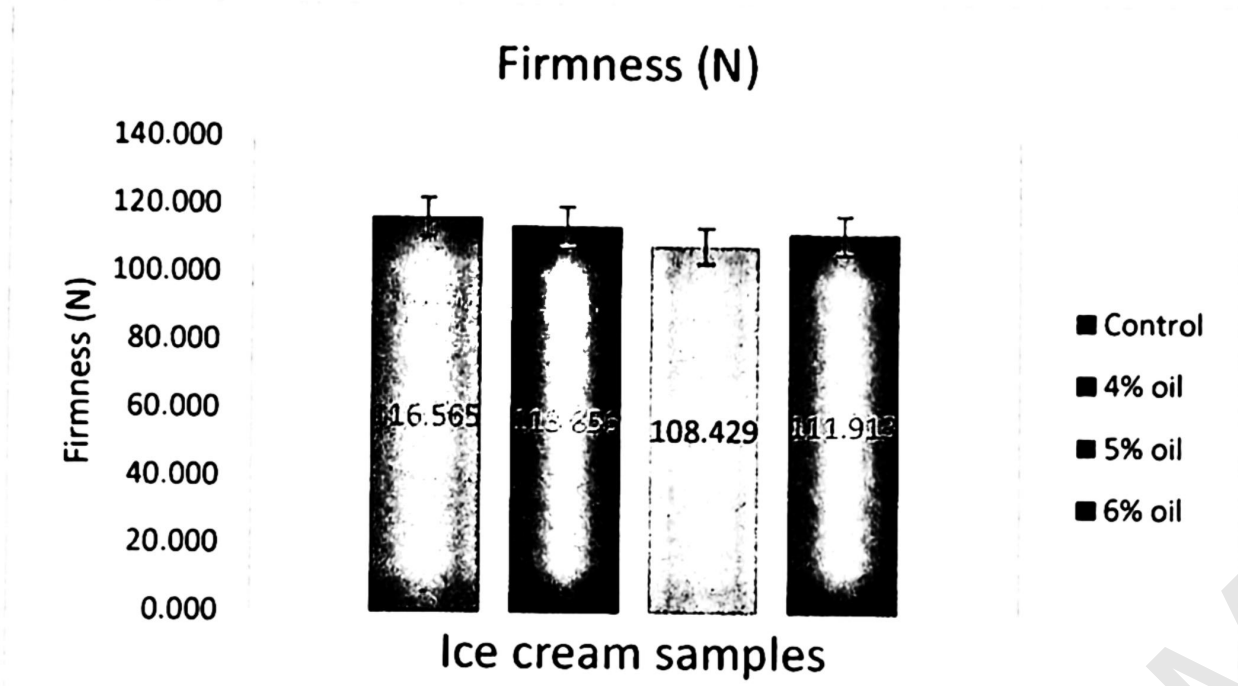



Figure 4.8: Firmness of ice cream samples

Figure 4.8 above shows the firmness value of all ice cream samples. The value for 0% oil (control), 4% oil, 5% oil and 6% oil are 116.565 N, 113.856 N, 108.429 N, 111.913 N respectively. Controlled sample has the highest value of firmness which is 116.565 N while 5% oil sample has the lowest which is 108.429 N. The firmness is compared at the same distance of penetration which is 20mm. This graph shows that percentage of oil does not affect so much to the firmness of ice cream. According to Muse & Hartel (2004), firmness of an ice cream can be affected by overrun, ice crystal size, ice phase volume and extent of fat destabilization. So, supposedly fat will play some role in improving the texture of an ice cream.

Ice crystal size also have a strong correlation with firmness of an ice cream. According to Muse and Hartel (2004), they found that larger ice crystals will increase the firmness of an ice cream. As compared to ice cream with smaller ice crystals, larger ice crystal can provide more resistance against the probe penetration and deformation. Muse (2003) also related this relationship to larger ice crystals coming into contact when compressed and forming a network, providing greater resistance from an applied force compared to smaller ice crystals, which are less likely to come into contact with each

other. As we can see in Figure 4.1 to Figure 4.4, ice crystals show increment in size as the percentage of oil increase which promotes higher firmness (Muse & Hartel, 2004).

# CHAPTER 5

# CONCLUSION AND RECOMMENDATION

This chapter summarizes the project that I have done. The main objective of this project was to observe the effects of different percentage of oil contents on the physicals and microstructural characteristics of ice cream. Four different samples were prepared which were controlled, 4% oil, 5% oil and 6% oil samples to facilitate the process of differentiation of the microstructure. Different tests and analyses were also conducted to study the differences between all the samples i.e. microstructure characterization, melting rate, overrun and also firmness analyses. The results from the analyses were discussed and some conclusions have been made. The recommendations for future studies are also included in this chapter.

## 5.1 Conclusion

Observing the microstructure of an ice cream requires an efficient equipment with strict rules and conditions because ice cream itself is a sensitive food. Without the right conditions, it is hardly to tell that we have obtained the correct results. From the

experiments, I managed to obtain the microstructure of ice creams from 4 different samples which were the controlled, 4% oil sample, 5% oil and 6% oil samples. In the experiment, I manipulated the percentage of oil added into the ice cream mix. To test the rate of successfulness of the microstructure observation technique, changes in the microstructure of ice cream of different samples were needed to be observed. From the results it was found out that there are slight changes in colour, size and behaviour of the structure of ice crystals, fat globules and air cells.

Different analyses were made to complete the mission of this project and to correlate between the microstructure and the results of analyses. For melting rate analysis, it was found out that as the percentage of oil increase, the melting rate was decreased. Ice cream with higher fat content melts slower because more partial coalescence between fat globules occurred in which creates softer texture and extra melt down resistance.

For overrun analysis, result shown that controlled sample has the highest overrun compared to other samples with added oil. In freezing condition, ice cream with higher fat percentage will tend to have higher viscous system. High viscous component could have prevented air incorporation. The system do not favour foaming capacity but do favour foam stability.

For firmness analysis, there are no significant changes in value between all four samples. The results showed that controlled sample has the highest value of firmness while 5% oil sample ranked last. Firmness is strongly related to building up shield or resistance toward forces.

To summarize, there are many improvements that need to be done to this experiment but with limited time and resources. I am proud to have completed this

project and learned new things especially the science behind ice cream. Microstructure of ice cream is an interesting topic to be explored but it requires sophisticated equipment to get good results.

## 5.2 Recommendation

The study in ice cream is very wide but, in our country, Malaysia there is still less research and studies about ice cream especially in observing the microstructure of ice cream. As mention earlier, studying the microstructure of ice cream requires correct and sophisticated equipment to obtain good and discussable results. One of the objectives of this experiment was to introduce a new method for observing the microstructure of ice cream that is inexpensive and easy to own by various parties. The rate of successfulness of the device depends on the budget and time provided and most important aspect is the knowledge on the study to complete the project.

In production of ice cream, there are many factors that can affect the structure, taste and other properties of ice cream. Inconsistencies in the process of ice cream production will affect the results that will be obtained later and will give confusion in concluding the experiment. For this experiment, a part of varying the percentage of oil in the ice cream mix, we also need to vary the ingredient which assist or cooperate with oil in maintaining the properties of ice cream such as stabiliser and emulsifier. Both ingredients play an important role such as to reduce the stability of fat emulsion by replacing proteins on the fat surface and also increase air cell stability to make the ice cream more prone to shrinkage during storage.

As for the device, I would recommend a microscope or stereomicroscope that are able to adjust the magnification rate, in order to have better understanding on the structure of ice cream. The device that I used in this experiment was fixed at 500x

magnification rate. So, it was challenging to tell the overall structure because I cannot zoom out or zoom in the images obtained using the device.

# REFERENCE


Aguilera, J.M., Stanley, D.W. 1999. Examining Food Microstructure. Microstructural Principles of Food Processing and Engineering, 2nd edn. Aspen Publishers, Gaithersburg, MD, pp. 1–70.

Amador, J., Hartel, R. W. and Rankin, S. A. (2017) The effects of fat structures and mix viscosity on physical and sensory properties of ice cream. Journal of Food Science, 82(8), 1851–1860.

Arbuckle, W. S. (1966) Relation of freezing and hardening to the body and texture of ice cream. Ice Cream Field and Trade Journal, 48(6), 34–47.

Belkoura, L., Stubenrauch, C., Strey, R. 2004. Freeze fracture direct imaging: a new freeze fracture method for specimen preparation in cryo-transmission electron microscopy. Langmuir, 20, 4391–4399.

Bogner, A., Jouneau, P.H., Thollet, G., Basset, D., Gauthier, C. 2007. A history of scanning electron microscopy developments: Towards "wet-STEM" imaging, Micron, 38, 390–401.

Caillet, A., Cogne, C., Andrieu, J., Laurent, P., Rivoire, A., 2003. Characterization office cream structure by direct optical microscopy. Influence of freezing parameters. Lebensm. -Wiss. U. -Technol. 36, 743–749

Caldwell, K. B., Goff, H. D. and Stanley, D. W. (1992) A low-temperature scanning electron microscopy study of ice cream. II. Influence of selected ingredients and processes. Food Structure, 11(1), 11–23.

Chang, Y. and Hartel, R. W. (2002b) Measurement of air cell distributions in dairy foams. International Dairy Journal, 12(5), 463–472. doi: 10.1016/S0958-6946(01)00171-6.

Clarke, C. (2004). Making ice Cream in the factory. In The Science of Ice Cream ed. Clarke C. pp 72-75, UK: The Royal Society of Chemistry.

Cook, K. L. and Hartel, R. W. (2010) Mechanisms of ice crystallization in ice cream production. Comprehensive Reviews in Food Science and Food Safety, 9(2), 213–222. doi: 10.1111/j.1541-4337.2009.00101.x.

Creamer, L.K., Berry, G.P. and Matheson, A.R. (1978). The effect of pH on protein aggregation in skim milk. New Zealand Dairy Science and Technology 13, 9–15.

Degner, B.M., Olson, K.M., Rose, D., Schlegel, V., Hutkins, R., McClements, D.J., 2013. Influence of freezing rate variation on the microstructure and physicochemical properties of food emulsions. J. Food Eng. 119, 244–253.

Euston, S. R. (2008) Emulsifiers in dairy products and dairy substitutes. In: Food Emulsifiers and Their Applications, Second Edition. New York, New York: Springer US, 195–232.

Faydi, E., Andrieu, J., Laurent, P., 2001. Experimental study and modelling of the ice crystal morphology of model standard ice cream. Part I: Direct characterization method and experimental data. J. Food Eng. 48, 283–291.

Goff, H. D. (1997a) Colloidal aspects of ice cream – A review. International Dairy Journal, 7(6–7), 363–373. doi: 10.1016/S0958-6946(97)00040-X.

Goff, H. D. and Hartel, R. W. (2013) Ice Cream, 7th Edition. New York, New York: Springer US.

Harker, F.R., White, A., Gunson, F.A., Hallett, I.C., De-Silva, H.N. 2006. Instrumental measurement of apple texture: a comparison of the single-edge notched bend test and the penetrometer. Post-Harvest Biology and Technology, 39, 185–192.

Heertje, I. (1993). Structure and Function in Food Products – A Review. Food Structure 12, 343–364.

James, B.J., Fonseca, C.A. 2006. Texture studies and compression behavior of apple flesh. International Journal of Modern Physics, 20, 3993–3998.

Klang, V., Matsko, N.B., Valenta, C., Hofer, F. 2012. Electron microscopy of nanoemulsions: An essential tool for characterisation and stability assessment. Micron, 43, 85–103.

Kuntsche, J., Horst, J.C., Bunjes, H. 2011. Cryogenic transmission electron microscopy (cryo-TEM) for studying the morphology of colloidal drug delivery systems. International Journal of Pharmaceutics, 417, 120–137.

Muse, M. R. and Hartel, R. W. (2004) Ice cream structural elements that affect melting rate and hardness. Journal of Dairy Science, 87(1), 1–10.

Preetz, C., Hauser, A., Hause, G., Kramer, A., Mader, K. 2010. Application of atomic force microscopy and ultrasonic resonator technology on nanoscale: distinction of nanoemulsions from nanocapsules. European Journal of Pharmaceutical Science, 39, 141–151.

Roland, A. M., Phillips, L. G. and Boor, K. J. (1999) Effects of fat replacers on the sensory properties, color, melting, and hardness of ice cream. Journal of Dairy Science, 82(10), 2094–2100. doi: 10.3168/jds.S0022-0302(99)75451-2.

Stanpac. 2018. Retrived from http://www.stanpacnet.com/who-invented-ice-cream/.

Sung, K. K. and Goff, H. D. (2010) Effect of solid fat content on structure in ice creams containing palm kernel oil and high-oleic sunflower oil. Journal of Food Science, 75(3), C274–C279. doi: 10.1111/j.1750-3841.2010.01539.x.

Thiel, A. E., Hartel, R. W., Spicer, P. T. and Hendrickson, K. J. (2016) Coalescence behavior of pure and natural fat droplets characterized via micromanipulation. Journal of the American Oil Chemists' Society, 93(11), 1467–1477. doi: 10.1007/s11746-016-2896-4.

Warren, M. M. and Hartel, R. W. (2014) Structural, compositional, and sensorial properties of United States commercial ice cream products. Journal of Food Science, 79(10), E2005–2013. doi: 10.1111/1750-3841.12592.

Wildmoser, H., Scheiwiller, J. and Windhab, E. J. (2004) Impact of disperse microstructure on rheology and quality aspects of ice cream. LWT – Food Science and Technology, 37(8), 881–891. doi: 10.1016/j.lwt.2004.04.006.