**UNIVERSITI PUTRA MALAYSIA**

# *EFFECTS OF USING NANOBUBBLE LIQUID ON THE PHYSICAL PROPERTIES OF HARD ICE CREAM*

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# EFFECTS OF USING NANOBUBBLE LIQUID ON THE PHYSICAL PROPERTIES OF HARD ICE CREAM

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**ABSTRACT**

Nanobubbles are bubbles that exist in a system in which of nanometer size to micrometer size and are dispersed in liquid. In recent years, the nanobubbles technologies has been used in an extensive range of application such as in drinking water, agriculture, fishery, wastewater treatment and food. In ice cream, there were air cells that will improve the smoothness and the texture of ice cream. In this study, effects of nanobubbles liquid on ice cream was compared with effect of normal water on ice cream. The sample was prepared and the analysis was conducted to check the physical properties of the ice cream. Based on the result, the particle size distribution for the ice cream mixture were 338nm for nanobubbles liquid and 97.9 µm for normal water. The viscosity for ice cream mixture for nanobubbles liquid and normal water were 0.211 Pa.s and 0.149 Pa.s respectively. Next, nanobubbles liquid ice cream had higher density and firmness which were 0.769 g/mL and 34.80g respectively while normal water ice cream had lower density and firmness which were 0.742 g/mL and 29.93g respectively. The overrun of nanobubbles ice cream were slightly lower which was 46% while normal water ice cream was 54%. Not only that, for melting rate, nanobubbles ice cream melted slightly later than normal water ice cream. As a conclusion, ice cream with lower overrun is denser and has rich texture. The smaller air bubbles size holds the body better and cause the ice cream to melt a bit later. Nanobubbles liquid ice cream has higher firmness value which indicates its ability to retain its shape better and has a better texture.

# ABSTRAK

Buih nano adalah buih yang wujud dalam sistem di mana ukuran nanometer untuk saiz mikrometer tersebar dalam cecair. Dalam beberapa tahun kebelakangan ini, teknologi buih nano telah digunakan dalam pelbagai aplikasi seperti air minuman, pertanian, perikanan, rawatan air sisa dan makanan. Dalam ais krim, terdapat sel udara yang akan meningkatkan kelicinan dan tekstur aiskrim. Dalam kajian ini, kesan cecair buih nano pada aiskrim dibandingkan dengan kesan air biasa pada aiskrim. Sampel disediakan dan analisis dilakukan untuk memeriksa ciri-ciri fizikal aiskrim. Berdasarkan hasilnya, pengedaran ukuran zarah untuk campuran aiskrim adalah 338nm untuk cecair buih nano dan 97.9 µm untuk air biasa. Kelikatan campuran aiskrim untuk cecair buih nano dan air biasa ialah masing-masing 0.211 Pa.s dan 0.149 Pa.s. Seterusnya, aiskrim cecair buih nano mempunyai ketumpatan dan kepejalan yang lebih tinggi iaitu masing-masing 0.769 g/mL dan 34.80g manakala air biasa mempunyai kepadatan dan kepejalan yang lebih rendah iaitu masing-masing 0.742 g/mL dan 29.93g. "Overrun" aiskrim cecair buih nano sedikit rendah iaitu 46% manakala aiskrim air biasa adalah 54%. Bukan itu sahaja, untuk kadar lebur, aiskrim cecair buih nano lebih lambat daripada aiskrim air biasa. Untuk kadar kecairan, aiskrim cecair buih nano cair sedikit lebih lambat daripada air biasa. Kesimpulannya, aiskrim dengan "overrun" yang lebih rendah adalah lebih padat dan mempunyai tekstur yang kaya. Saiz buih udara yang lebih kecil memegang badan lebih baik dan menyebabkan aiskrim cair sedikit lambat. Aiskrim cecair buih nano mempunyai nilai kepejalan yang lebih tinggi yang menunjukkan kemampuannya mengekalkan bentuknya dengan lebih baik dan mempunyai tekstur yang lebih baik.

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## NOMENCLATURES

| | |
|---|---|
| CMC | Carboxymethyl cellulose |
| CSS | Corn Starch Hydrolysate Syrup |
| $CO_2$. | Carbon dioxide |
| $H_2O_2$ | Hydrogen Peroxide |
| HTST | High Temperature Short Time |
| MSNF | Milk Solid Non Fat |
| OTE | Oxygen Transfer Efficiency |
| Pa.s | Pascal-second |
| nm | Nanometer |
| µm | Micrometer |
| g/min | Grams per minutes |
| g/mL | Grams per millilitres |

# 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 and Nanobubbles liquid

Ice cream is a special dessert that popular among all ages from young to aged people. Ice cream is no longer, just a treat for the elite. Today ice cream is found in almost any restaurant, or corner store, and is recognized globally as the perfect summer treat. In the United States alone 1.6 billion gallons of ice cream and frozen treats are produced annually, with the average American eating four gallons of ice cream each year (Stanpac, 2018).

Not only that, ice cream is also known as a complex food consists of small air cell dispersed in a partially frozen, continuous aqueous phase. It is important to choose the ingredient of the ice cream such as mixture of milk, cream, sugar, stabilizer and emulsifier as it will affect the development of the desired structure, texture and palatability. Generally, the main ingredients to make an ice cream is milk fat, milk solid non-fat (MSNF), sweetener that usually a combination of sucrose and glucose, stabilizers, emulsifier and water. All of this ingredients have its own function in production of ice cream. The combination of this ingredients can make a perfect taste, texture and flavour on the ice cream depends on its composition.

To get an ideal taste, texture and flavour with good physical properties effects on the ice cream, the ingredients is not the only factor that can contribute on the physical properties of the ice cream, the manufacturing of ice cream itself also additional fundamental and big role to get the perfect ice cream with good physical properties. The main process to produce the ice cream is blending the ingredient, pasteurization, homogenization, aging, freezing and hardening. All of this process have its own function and important-ness in the manufacturing of the ice cream.

Typical ice cream comprised of approximately 30% water, 50% air, 5% fat and 15% matrix (sugar solution) by volume (Clarke, 2004). There are four categories of ice cream that are commercially available which are super-premium, premium, standard and economy. The higher the fat content, the more expensive the price. It is essential to know the reason why certain ingredients being put, why the time consuming and certain processes are needed in the making ice cream.

Nowadays, there is various type of ice cream that have been produced to satisfy the customer taste. Rapid development in ice cream have produced various different type of ice cream. The type of ice cream that have in the world is hard ice cream, French ice cream, soft ice cream, light ice cream, reduced fat ice cream, fat free frozen dairy dessert, no sugar added ice cream or frozen dairy dessert and so on. All of this ice cream was prepared with different composition of ingredient that suit to everyone taste. In Malaysia, the type of ice cream that popular among the people is soft ice cream and hard ice cream.

Nowadays, the advancement of nanotechnology has led to the development of nanobubbles which is drawing a lot of attention from variety of fields especially food industry (AzoNano, 2018). Nanobubbles play a vital role in many sectors that hat include scientific research, medical and food sectors. Generally, Nanobubbles are nanoscopic gaseous typically air cavities in aqueous solutions that have the ability to change the normal characteristics of water. Ordinary bubbles (>1nm diameter) quickly rise to the surface and burst but the smaller nanobubbles (<100 nm diameter) have a lower buoyancy and will remain suspended in liquids for an extended period of time. (Soutter, 2013).

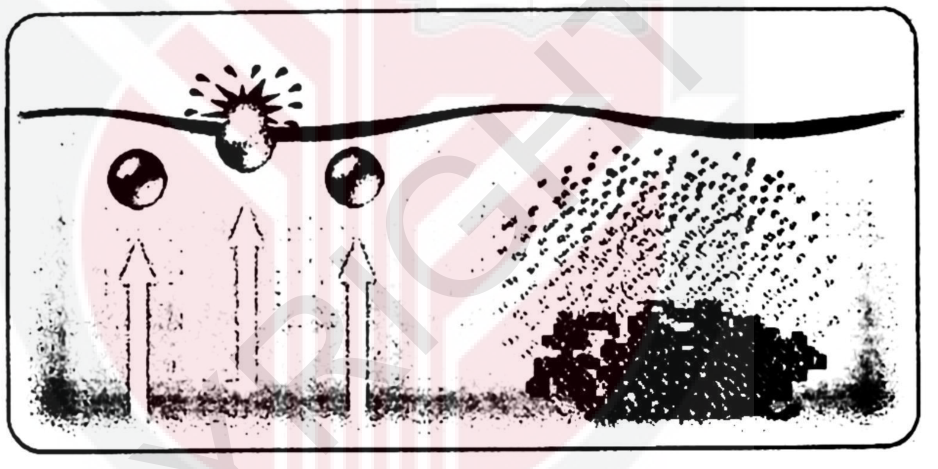


Figure 1-1: Bubbles with Micrometer and Nanometer size

This study aimed to determine the effect of nanobubbles water on the physical properties of the ice cream such as melting rate, overrun, hardness, texture and viscosity of the ice cream. This study is important because there were no study on effects of nanobubbles water on ice cream has been conducted. After getting the desired result, the nanobubbles water will be compare with the normal water that used in the ingredients of the ice cream and the effectiveness of nanobubbles water on ice cream can be determined.

## 1.2 Problem Statements

Nowadays, many researches have been done study on effects of ingredients such as type of milk, stabilizer and emulsifier on the physical properties of ice cream. Most ice cream is made from 55%-64% of water, more than 28% of milk and cream, 10%-14% of sucrose or sugar, flavouring and additives to help maintain the stability of the frozen structure (Brown, 2003). Ice cream is a food that have a complex multi-phase system and very sensitive to temperature.

Usually, the quality of the ice cream is effected by the amount of fat globules and type of emulsifier and stabilizer that was used. There are three main structural components of ice cream which is air cell, ice crystal and fat globules that distributed throughout continuous phase of unfrozen solution (Muse & Hartel, 2004). It is the relative amount of these phases and the interactions between them that determines the properties of the ice cream whether soft, whippy or hard (Brown, 2003).

This study was conducted to determine either air bubbles with smaller particle size by using nanobubbles liquid can affects the viscosity, melting resistance, overrun and firmness of the ice cream since there is no study about nanobubbles liquid has conducted yet. It is assumed that by using nanobubbles, the physical properties of ice cream can be improved and increase the quality of ice cream.

## 1.3 Objectives

The main objective of this study is to determine the effectiveness of nanobubbles water on ice cream. To be more specific, the main objectives of this study were:

i. To determine the effects of shear on the viscosity and particles size of ice cream mixture.

ii. To determine the effects of using nanobubble liquid on the physical properties of ice cream i.e. firmness, overrun, melting rate and density.

# 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 Overview of ice cream

Ice cream can be defined as a smooth, sweet, cold dessert food prepared from a frozen mixture of milk product and flavouring. The ice cream mix is pasteurized, homogenized aged to improve the physical properties before freezing process. Ice cream is a frozen foam that consists of air cells dispersed in an aqueous matrix (Marshall et al., 2003).

## 2.2 Ice Cream Mix Ingredient

Ice cream mix consists of milk fat, milk solids not-fat (MSNF), sweeteners, stabilizers, flavourings, emulsifiers and water. Dairy and other ingredients used to supply these components are chosen on the basis of their cost, availability, and quality of the finished product. Goff and Hartel (2013) found that usually ice cream has following composition which is greater than 10% milk fat by legal definition, and usually between 10% and as high as 16% fat in some premium ice creams, 9 to 12% milk solids not-fat (MSNF) which is in this component, also known as the serum solids, contains the proteins (caseins and whey proteins) and carbohydrates (lactose) found in milk, 12 to 16% sweeteners that usually a combination of sucrose and glucose-based corn syrup sweeteners 0.2 to 0.5% stabilizers and emulsifiers and 55% to 64% water which comes from the milk or other ingredients. These percentages are by weight, either in the mix or in the frozen ice cream. For hard ice cream, there is formulation suggestion as the figure below.

Table 2-1: Formulation suggestion for hard ice cream

| | Percent (%) | | | | | | |
|---|---|---|---|---|---|---|---|
| Milk fat | 10 | 11 | 12 | 13 | 14 | 15 | 16 |
| Milk Solid non Fat | 11 | 11 | 10.5 | 10.5 | 10 | 10 | 9.5 |
| Sucrose | 10 | 10 | 12 | 14 | 14 | 15 | 15 |
| Corn Syrup Solids | 5 | 5 | 4 | 3 | 3 | - | - |
| Stabilizer | 0.35 | 0.35 | 0.3 | 0.3 | 0.25 | 0.2 | 0.15 |
| Emulsifier | 0.15 | 0.15 | 0.15 | 0.14 | 0.13 | 0.12 | 0.1 |
| Total Solids | 36.5 | 37.5 | 38.95 | 40.94 | 41.38 | 40.32 | 40.75 |

### 2.2.1 Milk fat

Milk fat is important as the ingredient of ice cream as it increases the richness of flavour in ice cream, helps to give body to the ice cream and in good melting properties due to its role in fat destabilization. Not only that, milk fat also helps in lubricating the freezer barrel during manufacturing and produced a characteristic smooth texture.

Goff & Hartel (2013) found that there were five factors of great interest in selection of fat source which is the crystal structure of the fat, the rate of the fat crystallizes during dynamic temperature conditions, the temperature-dependent melting profile of the fat, especially at chilled and freezer temperatures, the content of high melting triglycerides which can produce a waxy, greasy mouth feel and the flavour and purity of the oil.

### 2.2.2 Milk Solid Non Fat

Milk solids not-fat (MSNF) or can be called serum solid contain the lactose, caseins, whey proteins, and minerals (ash content) of the product from which they were derived. This ingredients is important as MSNF can improve the texture of ice cream, due to the protein functionality, help to give body and chew resistance to the finished product. Not only that, MNSF capable of allowing a higher overrun without the characteristic snowy or flaky textures associated with high overrun because of the protein functionality and can be cheap source of total solids, especially whey powder.

There are limitation for this ingredients because of the flavour may arise from the product and an excess of lactose which can lead to the defect of sandiness prevalent when the lactose crystallizes out of solution. Also, excessive concentrations of lactose in the serum phase may also lower the freezing point of the finished product to an unacceptable level (Goff & Hartel, 2013)

### 2.2.3 Sweeteners

Ice cream usually sweet in taste which is desired by the consumer. The main function of sweeteners is to increase the acceptance of ice cream by making it sweet and by enhancing the pleasing creamy flavour. Lack of sweetness produces a flat taste where too much sweetness will tends to mask desirable flavours (Goff & Hartel, 2013). There are many types of sweeteners that can be used such cane and beet sucrose sugar or known as 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. The most common choice of nutritive sweetener is a combination of sucrose (10-12%) and CSS (3-5%) (Goff & Hartel, 2013).

Besides enhancing sweetness and flavour, nutritive sweeteners also determine textural creaminess and mouth feel (Stampanoni, 1993; Guinard et al., 1997). In general, increasing the sweetener level increases creaminess as a result of the reduction in the size of ice crystals. Smooth and creamy ice cream requires the majority of ice crystals to be small. If many crystals are large, the ice cream will be perceived as being coarse or icy.

### 2.2.4 Stabilizers

The stabilizers are a group of compounds, usually polysaccharide food gums that are responsible for adding viscosity to the mix and the unfrozen phase of the ice cream. The are many types of stabilizer that was used in industry such as guar gum, Carboxymethyl cellulose (CMC), Xanthan gum, Sodium alginate and Carrageenan. The function of stabilizer is usually to increase viscosity so that the ice cream will have a smooth and creamy texture. Also, stabilizers can provide resistance to melting as it will affect the melting quality of ice cream through their viscosity-enhancing properties.

Next, stabilizer will retards or reduce ice and lactose crystal growth during storage as ice crystal is critical factor in the development of smooth and creamy ice cream. Based on Adapa et al (2000), guar gum and locust bean gum is widely used as stabilizer to inhibit ice crystal growth and recrystallization. Also, stabilizer was used to prevent wheying off which refers to the leaking of a clear watery serum layer during the melting of ice cream, which has an undesirable appearance. Lastly, stabilizers help prevent shrinkage which is defined as the loss of volume in ice cream before any part of the product has been removed from the container (Dubey & White,1997).

### 2.2.5 Emulsifiers

Emulsifiers and protein are surface active molecules that act to keep two liquids that do not mix naturally which is milk fat and water, from separating. Emulsifiers are used in ice cream because they contribute greatly to smooth and creamy texture by promoting fat destabilisation. Emulsifier also incorporate more and smaller air bubbles and form thinner lamellae between air bubbles (Marshall et al, 2003) Each molecule of an emulsifier contains a hydrophilic portion and a hydrophobic portion, they reside at the interface between fat and water (Goff & Hartel, 2013). As a result they act to reduce the interfacial tension or the force which exists between the two phases of the emulsion.

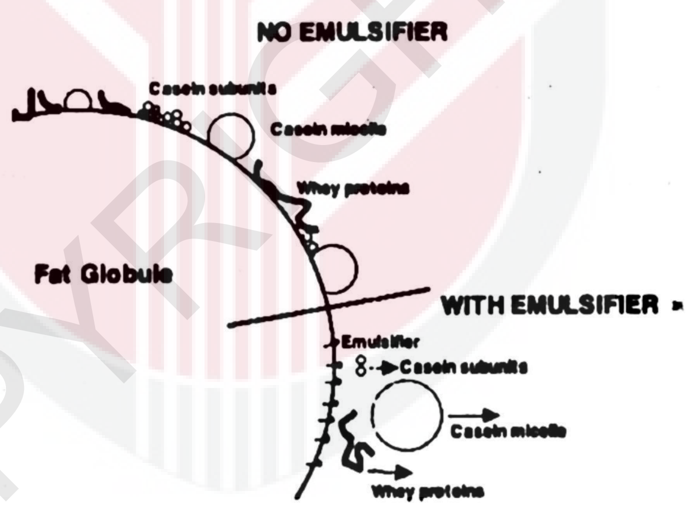



Figure 2-1: Fat globule with emulsifier and without emulsifier

## 2.3 Ice Cream Manufacturing

Ice cream manufacturing is a process to produce ice cream from mixing, pasteurized, homogenized, aging and freezing. All of this process is important to produces a good texture of ice cream.

### 2.3.1 Mixing

First the ingredients are selected based on the desired formulation and the calculation of the recipe from the formulation and the ingredients chosen. Then the ingredients are weighed and blended together to produce what is known as the ice cream mix. Generally speaking, all dry ingredients are weighed, whereas liquid ingredients can be either weighed or proportioned by volumetric meters. In plants with small capacities and small total volumes, dry ingredients are generally weighed and supplied to the mix tanks by hand.

These tanks are designed for indirect heating and equipped with efficient agitators. Large-scale producers use automatic batching systems, which are often custom-built to the user's specifications. Mixing requires rapid agitation to incorporate powders and often high speed blenders are used. It is important to ensure that the dried ingredients are properly suspended in the mixing tank to avoid lumpiness of the mix. Proper suspension can be achieved by mixing dry ingredients thoroughly with part of the sugar before slowly adding the remaining sugar. Also, sifting and slowly adding these dry ingredients into the liquid while slowly agitating the entire mix.

### 2.3.2 Pasteurization

After mix all the ingredient, the ice cream mix need to be pasteurized. In ice cream production, pasteurization is the biological control point in the system where the objective of the process is to kill pathogenic bacteria. Pasteurization also reduces the number of spoilage organisms such as psychrotrophs and helps to hydrate some of the components such as proteins and stabilizers. Psychrotrophs are cold-tolerant bacteria that have the ability to grow at low temperatures but have optimal and maximal growth temperatures above 15°C and 20°C, respectively. Not only that, there is two type that can be used which is batch pasteurization and continuous (HTST) for pasteurization process (Goff, 2013). Batch pasteurizers lead to more whey protein denaturation, which some people feel gives a better body to the ice cream.

In a batch pasteurization system, mixing of the proper ingredient amounts is made in large jacketed vats equipped with some means of heating, usually steam or hot water. Product is then heated in the vat to at least 69°C and held for 30 minutes to satisfy legal requirements for pasteurization, necessary for the destruction of pathogenic bacteria. Various time temperature combinations can be used. The heat treatment must be severe enough to ensure destruction of pathogens and to reduce the bacterial count to a maximum of 100,000 per gram (Goff, 2013).

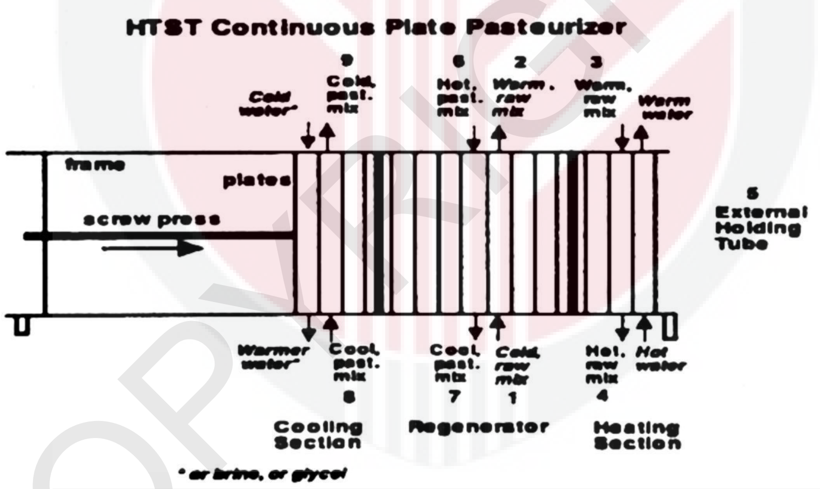



Figure 2-2: HTST Continuous Plate Pasteurizer

### 2.3.3 Homogenization

After pasteurization, the ice cream mix is homogenized by using homogenizer. The function for this homogenize process is to forms the fat emulsion by breaking down or reducing the size of fat globules found in milk or cream to less than 1µm (Goff, 2013). By breaking down the fat molecules into smaller particle, the size of the fat globules can be reduces, thus surface area can be increase, form membranes that makes possible the use of butter and frozen cream. By forming the fat structure, the texture of ice cream more smooth, there is better air stability, increases resistance to melting and give rich flavour and palatability to the ice cream.

Two stage homogenization is usually preferred for ice cream mix so that clumping or clustering of the fat is reduced thereby can produce a thinner and rapidly whipped mix. The melt-down time also can be improved. Homogenization of the mix should take place at the pasteurizing temperature. The 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. The higher the fat and total solids in the mix, the lower the pressure should be. If a two stage homogenizer is used, a pressure of 2000 - 2500 psi on the first stage and 500 - 1000 psi on the second stage should be satisfactory under most conditions.

### 2.3.4 Aging

After homogenization process, the mix need to be aged at least four hours and usually overnight. This allows time for the fat to cool down and crystallize, and for the proteins and polysaccharides to fully hydrate. The function of aging process is to improve the whipping qualities of mix and body and texture of the ice cream (Goff, 2013). This impact can be done by providing time for fat crystallization, so the fat can partially coalesce. In this stage also, it can allowed time for full protein and stabilizer hydration so that the viscosity will increase. Not only that, there is time that allow membrane rearrangement and protein or emulsifier interaction, as emulsifiers displace proteins from the fat globule surface, which allows for a reduction in stabilization of the fat globules and enhanced partial coalescence. Aging usually performed in insulated or refrigerated storage tank such as silo at 2°C to 5°C. Aging also can be performed in single walled tanks in chilled room, where valve and pipeline can be also kept cold.

### 2.3.5 Freezing

After aged the mix, the mix will go through freezing process. The process involves the freezing the mix and incorporating air. Ice cream mix can be frozen in batch or continuous freezers and the conditions used will depend on the type of freezer. Batch freezers are commonly used by small ice cream units that make ice cream on the premises (Deosarkar et al, 2015). Batch freezers consist of a rotating barrel that is usually filled one-third to one-half full with ice cream mix. As the barrel turns, the air in the barrel is incorporated into the ice cream mix. Ice cream freezers designed for home use are batch freezers.

Continuous freezers are commonly used in larger ice cream manufacturing plants where more than 500 gal (1875 L) of ice cream per day may be manufactured. These freezers have larger capacities and can be operated continuously, ingredients can be added in-line, and packaging can be also automated. Continuous freezers consist of a fixed barrel that has a blade inside that constantly scrapes the surface of freezing barrel. The ice cream mix is pumped from a bulk tank to the freezing barrel and the air is incorporated with another pump just before it enters the freezing barrel. The continuous freezing process is much faster than the batch freezing process.

The ice cream from a continuous freezer is smoother and creamier than a product from a batch freezer. This is because the ice crystals formed in a continuous freezer are smaller and the air cells may also be more uniform. The ice cream exiting a continuous freezer is also generally colder than that coming out of a batch freezer. The addition of air is called overrun and contributes to the lightness or denseness of ice cream. Up to 50% of the volume of the finished ice cream (100% overrun) can be air that is incorporated during freezing. The overrun level can be set as desired to adjust the denseness of the finished product. Premium ice creams have less overrun (approximately 80%) and are denser than regular ice cream (Milk Facts, nd).

### 2.3.6 Hardening

After the particulates have been added, the ice cream is packaged and is placed into a blast freezer at -30° to -40° C where most of the remainder of the water is frozen. Below about -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. This limits the shelf life of ice cream. The rate of heat transfer in a freezing process is affected by the temperature difference, the surface area exposed and the heat transfer coefficient. Thus, the factors affecting hardening are those affecting this rate of heat transfer. Regarding the temperature of blast freezer, the colder the temperature, the faster the hardening, the smoother the product. The colder the ice cream at draw when placed in the hardening freezer, the faster the hardening. The size of container also can affect the hardening time.

## 2.4 Nanobubble Liquid

Industrial application of nanobubbles has exponentially increased over the past two decades due to their reactivity and stability compared to macrobubbles and microbubbles. Due to the size, nanobubbles have high specific surfaces areas and high stagnation time which will increases mass transport efficiencies, physical absorption and chemical reactions at the gas-liquid interfaces. (Scoutter, 2017). Not only that, nanobubbles have long residence time in solutions and electrically charged surfaces (IDEC Corporation, 2017). Malvern Instrument LTD (2017) stated that because of the nanobubbles properties many industrial applications such as manufacturing of functional materials, soil and sediment decontamination, pharmaceutical delivery and disinfection of food products.

Nanobubbles as the name implies are extremely small gas bubbles in liquid – so small in fact that millions of nanobubbles can fit inside a one micron (micro) bubble. Nanobubbles have several unique physical properties that make them very different from a normal bubble. Most notable of which is their long stable life span in liquid due to their very low buoyancy and negatively charged surface (zeta potential). The concept of nanobubbles is not new. The application and utilization of nanobubbles in various industries is vast and proven to be highly beneficial. Years of studies have already documented their physical attributes and effects in a broad range of applications.

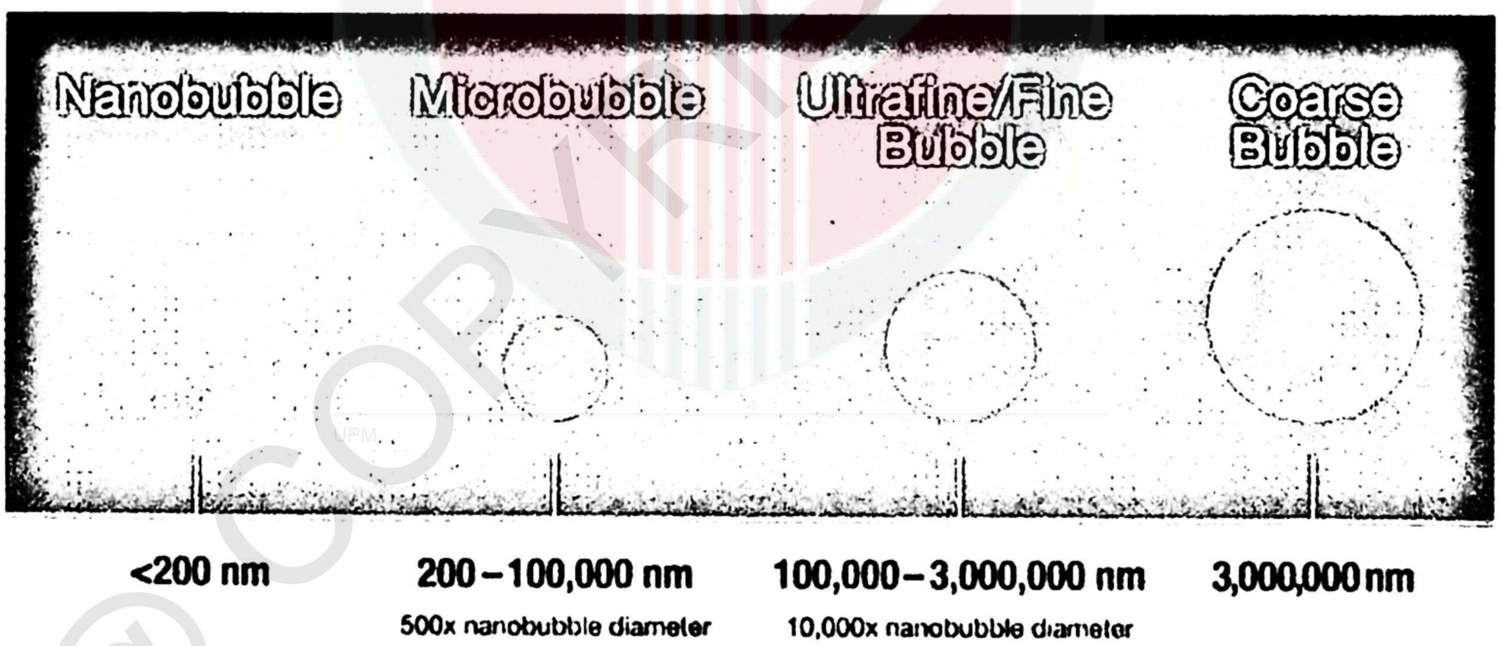



Figure 2-3: Differences sizes of bubbles

### 2.4.1 Properties of Nanobubbles Liquid

Firstly, nanobubbles is neutral buoyancy. A singular nanobubble has little to no buoyancy. Rather than rising vertically they can remain suspended in liquid for months at a time until they dissolve, travelling randomly throughout the body of water and efficiently aerating the entire water column. Secondly, nanobubbles have a wider surface area. The surface area is 400 times of a typical microbubbles which measured at 40 micrometers in diameter. The larger surface area allows for increased mass transfer, ensure any gas is effectively delivered to water.

Thirdly, nanobubbles liquid has higher oxygen transfer efficiency. Aeration application is depending on the oxygen transfer onto the body of the water. Generally, aeration system transfer only 1% to 3% of oxygen per foot of water due their size and corresponding rapid rise rate. With the unique properties of nanobubbles liquid, it delivers an industry leading oxygen transfer efficiency (OTE) over 85% per foot of water. The nanobubbles' longevity in water, together with their high surface area per volume, makes them the most efficient aeration method on the market today and allow operators to significantly reduce operating costs (Molear, 2019).

Next, nanobubbles naturally poses a surface charge. The smaller the bubble, the stronger the surface charge. Nanobubbles have high zeta potential which is the electro kinetic potential in colloidal dispersion (Molear, 2019). Strong negative charge of nanobubbles limit their coalescence that makes the integrity of the bubbles is preserved at any depth for extended periods of time.

Not only that, AzoNano (2018) stated that in saturated liquids, these nanobubbles are extremely stable due to the absorption of ions on their surface. The gas molecules inside the nanobubbles do not come in contact with the bulk liquid, allowing the nanobubbles to last for a much longer time. Larger bubbles have air that is above atmospheric pressure, but nanobubbles contain internal pressures of tens or even hundreds of atmospheres. Under such high pressures, it has been proven that the liquid absorbs the gas inside the Nanobubbles.

### 2.3.2 The Application of Nanobubble Liquid

Nanobubbles have an extensive range of applications such as in drinking water, agriculture, fishery and wastewater treatment, including decontamination of groundwater; decontamination of sediments and soils, biomedical engineering and food. One of the best uses of nanobubbles is the treatment of wastewater and drinking water that have been recently developed due to their ability to generate highly reactive free radicals (Agarwal et al., 2011). Hu and Xia (2018) showed the feasibility of remediating groundwater using ozone micro-and nanobubbles.

Meegoda (2017) have proposed a new technology to decontaminate sediments using ultrasound with ozone nanobubbles. In this technology, they uses three innovative technologies, which is ultrasound, ozone, and nanobubbles, to provide a cost effective and environmentally sustainable onsite treatment of sediments with lower total cost over a shorter time span. It also has minimal adverse impact on the environment and the socioeconomic growth of the region. The ultrasound energy provides agitation and sediment decontamination. The ozone reacts with desorbed contaminants for removal from water. The nanobubbles help the dissolution of ozone gas in water (Meegoda and Batagoda, 2016; Meegoda et al., 2017). The use of nanobubbles was motivated by their air sparging experiment (Hu et al., 2010, 2011, 2014).

There are many biomedical applications of nanobubbles. One of them is the delivery of cancer drugs, where nanobubbles are placed in the body and are given the ability to identify tumor cells. The bubbles are blown up when they approach tumor cells, destroying the cancer (NHI, 2017). Nanobubbles have also been used in emergency procedure, where nano oxygen bubbles are injected directly into the bloodstream allowing people who are suffocating an extra 15min during transportation to hospitals. While this is not a long time but it does allow for higher survival rate (Narayan, 2017).

There are many industrial applications of nanobubbles. Nanobubbles have shown the ability to create reactive oxygen species which contribute to seed germination. This increase in reactive oxygen species has the same effect as adding $H_2O_2$ resulting in higher germination rates (Liu et al., 2015). Also they used in sparkling water and sports drinks. With the addition of nanobubbles, the water can potentially keep gases for a longer time period (Bauer Nanobubbles, 2017).

Nanobubbles also have application in paints. Due to the presence of nanobubbles, paint dries faster and also resists mold. In addition, there is an increase in brightness due to the nanobubbles (Bauer, 2014). They are also used as artificial flotation in water. This is accomplished by altering the ionic equilibria of dissolved ions in solutions and by changing the net charge on particle surfaces (Moleaer, 2017a).

Nanobubbles are also used in food industry. Nanobubbles are used to regulate pH levels in liquids utilizing carbon dioxide. This is achieved by adding nano $CO_2$. Bubbles, which are suspended in the water for a long time regulating solution pH (Moleaer, 2017c). Nanobubbles are also used in fish farming. Studies have shown that a decrease of oxygen leads to decreased respiration and feeding activity that slows growth rate of fish. However, with nanobubbles, oxygen levels in water are maintained leading to high fish survival rates (Moleaer, 2017b). Although there are many applications of nanobubbles as suggested above, there is limited understanding of the reason for their long-term stability.

# CHAPTER 3 : METHODOLOGY

In this chapter, the method of the experiment was explained. The step consists of the preparation of the sample from stage one until stage three. For both sample which is normal water and nanobubbles liquid they have the same method for the preparation. The only changes in the sample preparation is normal water will replaced with the nanobubbles liquid as the ingredients. The analysis which is overrun, viscosity, melting rate and firmness of ice cream was conducted. Data obtained from analysis was recorded and discuss in next chapter.

## 3.1 Stage 1: 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.

### 3.1.1 Ice cream mix formulation

The aim of this project is to discover the effects of using nanobubble liquid on the physical properties of hard ice cream. Hence, the sample was 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 (%) | Mass for each batch of 500g (g) |
|---|---|---|
| Water | 60.9 | 304.5 |
| Skimmed Milk Powder | 14.5 | 72.5 |
| Sugar | 16.3 | 81.5 |
| Whey Powder | 3.6 | 18 |
| Creamer | 3.6 | 18 |
| Emulsifier | 0.4 | 2 |
| Stabilizer | 0.3 | 1.5 |
| Flavoring | 0.4 | 2 |

### 3.2 Stage 2: Production of ice cream

Stage 2 was occurred where the production of the ice cream took place. The process of ice cream is including weighed the ingredients, mixed, pasteurized, homogenized, ageing, froze and hardened.

#### 3.2.1 Weighed Ingredients

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

Figure 3-1: The ingredients was placed together in mixing bowl

### 3.2.2 Mixed

The sample formulation of ice cream mix was mixed together using the mixer (Model 5K5SS, KitchenAid, St Michigan, USA) as shown in figure 3-2. The ice cream mix was mixed for 5 minutes so that all the ingredients which is dry ingredients and wet ingredients is mixed well with each other.

Figure 3-2: Mixing process

3.2.3 Pasteurized

After mixed, the sample mixture was pasteurized. The mixture was pasteurized on the stove 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.

Figure 3-3: Pasteurization process on the stove

3.2.4 Homogenized

After pasteurized the ice cream mix, the mix is immediately transfer into a jug to undergo homogenization process. The mixture was homogenized by using the homogenizer (Success Technic Indutries, 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 7 shows how the homogenization process of ice cream mix was occurred.

Figure 3-4: Homogenization process

Figure 3-5: Lab scale Homogenizer

3.2.5 Ageing process

After homogenized, 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 body and texture of the ice cream. Ageing process was shown in Figure 3-6.

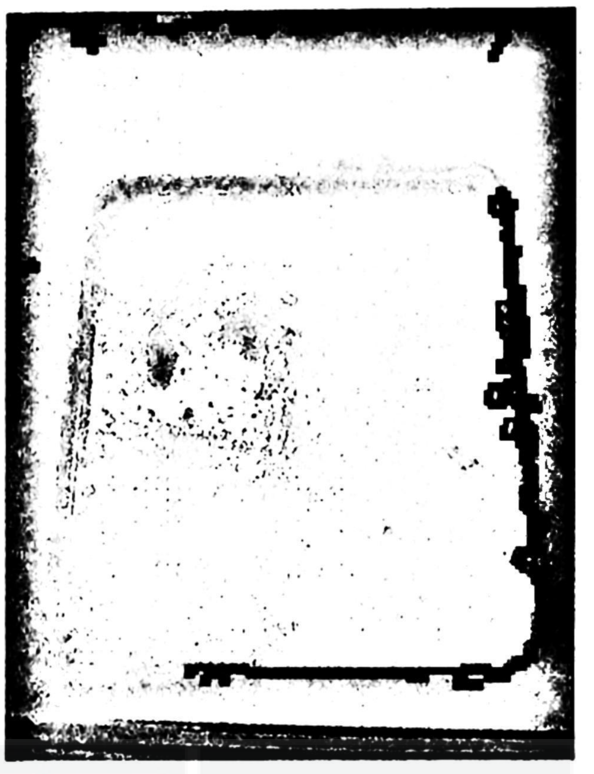


Figure 3-6: Ice cream mix after homogenization process

3.2.6 Freezing process

After aged the ice cream mix for overnight, the aged ice cream mix was whipped and froze in the batch ice cream freezer (Breville, Model BC1600, Australia). The freezer was undergo under cooled and froze temperature to about -20°C.Tthe freezing process for ice cream mix was shown in Figure 3-7 and Figure 3-8.

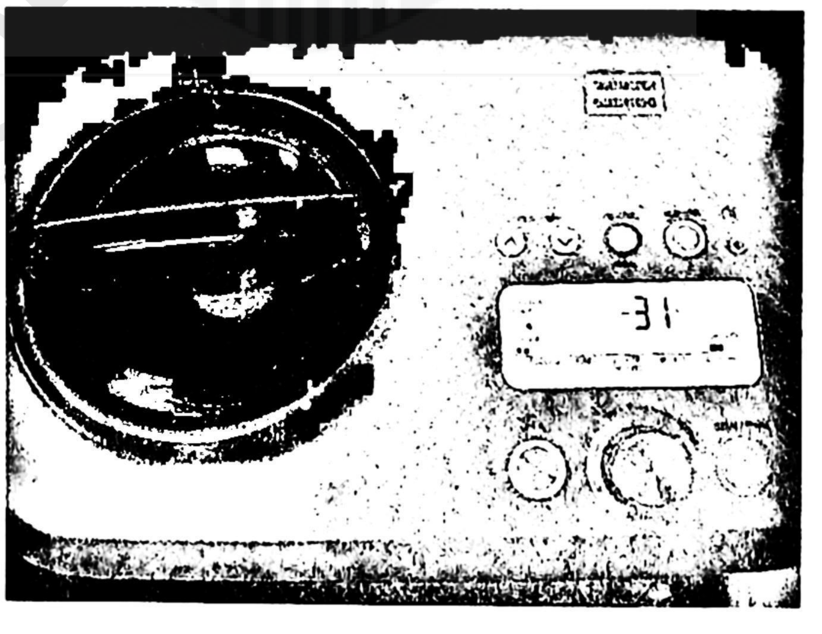


Figure 3-7: Batch ice cream freezer

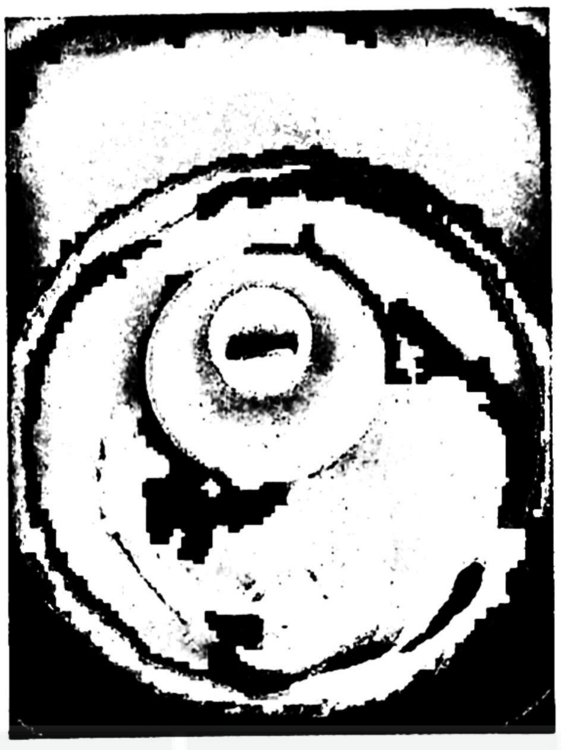


Figure 3-8: Ice cream mix after undergo freezing process

3.2.7 Hardened

After the freezing 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 3-9 show the ice cream sample for hardening process after froze in the batch-freezer.

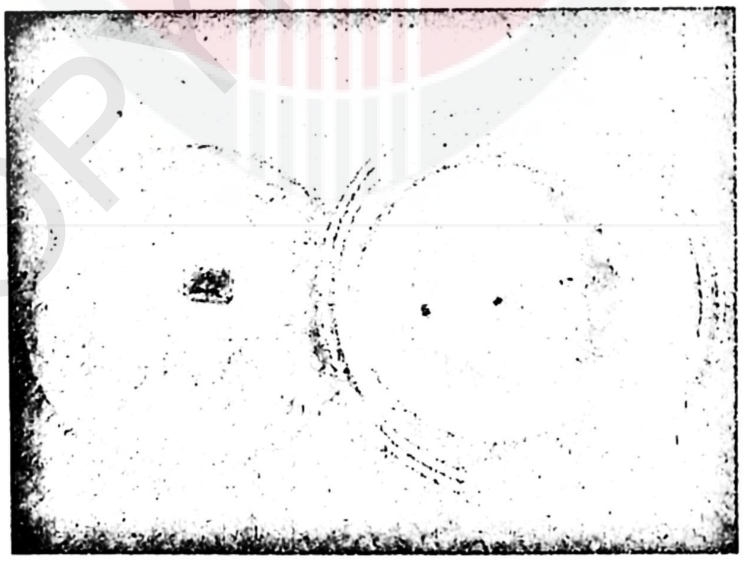


Figure 3-9: Ice cream after hardening process

**3.3 Stage 3: Analysis of ice cream**

The analysis of ice cream was performed on the sample of ice cream depends of the type of sample needed. The analysis that was conducted is particle size analysis, overrun, viscosity, melting rate and firmness of ice cream.

3.3.1 Particle Size Analysis

This analysis was conducted to determine whether this two sample have different size or not. Firstly, for the normal water sample, the particle size was tested using the particle size analyser. There are a beaker that was filled with distilled water. The ice cream was put into syringe and dropped into the distilled water. Then, Malvern Particle Size Analyser (Malvern Mastersizer 2000, UK) was detected the size of ice cream mix. For nanobubbles water, the particle size was measured by using zeta-potential analyser as it has nano size particle (Zetasizer Nano ZS, Malvern Instrument Ltd, Malvern, UK). Firstly, the ice cream mix was diluted with distilled water to achieved suitable intensity. After that, the zeta-potential analyser was detected the size and shown the result at the software in the computer.

Figure 3-10: Malvern Particle Size Analyser (Malvern Mastersizer 2000, UK)

3.3.2 Analysis on Overrun of the ice cream

Overrun by definition is the percent of expansion of ice cream achieved from the amount of air incorporated into the product during the freezing process (WIO, 2018). Overrun was determined according to the method described by Marshall et al. (2003). A known volume of ice cream was mix and frozen hard ice cream were weighed and the overrun was calculated according to the equation:

$$\text{Overrun (\%)} = \frac{\text{weight of the ice cream mix} - \text{weight of the ice cream}}{\text{weight of the ice cream mix}} \qquad \text{Eq (3-1)}$$

3.3.3 Analysis of viscosity

The rheological measurement for the ice cream mix were performed on a dynamic rheometer (AR-G2, TA instrument, New Castle, USA) using cone plate configuration of with 1° and 60mm diameter. The sample was loaded on rheometer base plate and was allowed to rest for 10min to prevent the influence of structural modification during sample handling and loading. The shear rate for this sample was set from 0 too 300$s^{-1}$.

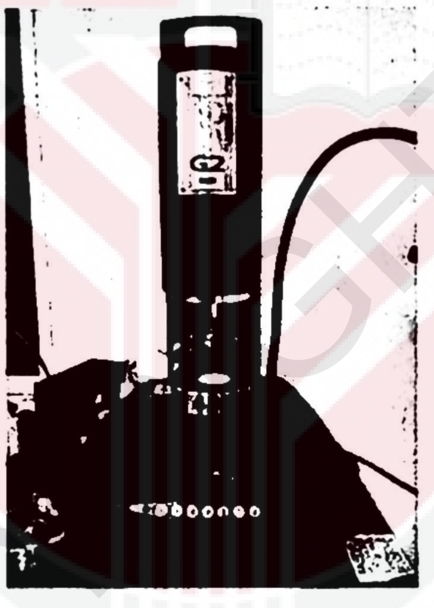



Figure 3-11: Rheometer (AR-G2, TA instrument, New Castle, USA)

3.3.4 Analysis of melting rate.

For melting rate analysis, the melting rate was measured by taking sample for 37g±1g that was harden in the freezer. For this analysis, wire gauze, stopwatch, beaker and electronic balance were used. Firstly, the hard ice cream was placed on the wire gauze. A beaker was placed on the electronic balance and the weighed of melt ice cream was recorded. The mass of melt ice cream was recorded for every 5 minutes. The duration of the experiment was expected to finish in 45 minutes for total loss of the ice cream structure. Weight of melted ice cream was recorded and was tabulated with time taken. Figure 3-12 shown the setup for melting rate analysis.

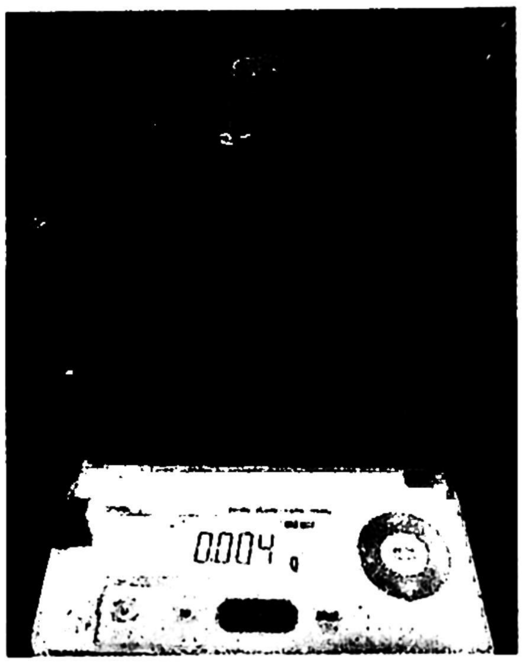


Figure 3-12: Set-up for melting rate

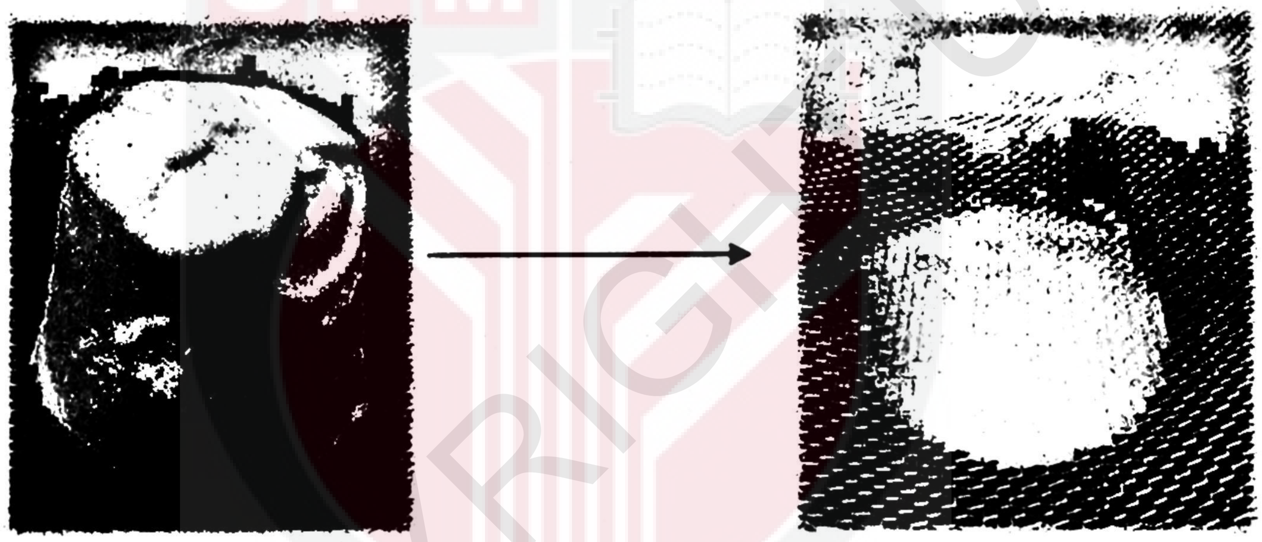


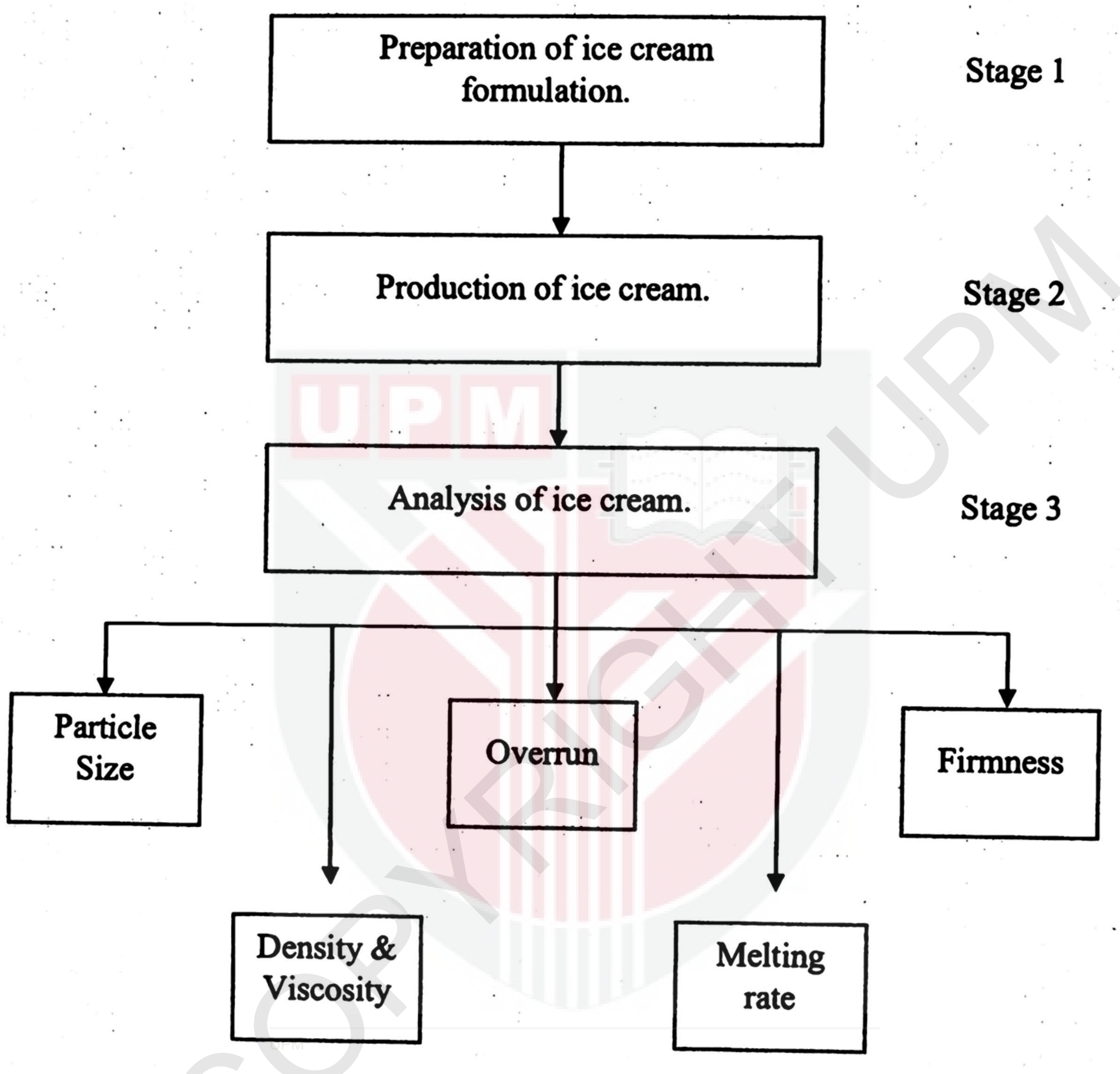
Figure 3-13: Changed of structural of ice cream in melting rate analysis

3.3.5 Analysis of hardness of ice cream

Before conduct the firmness analysis, hard ice cream sample with weight 200g was hardened in the freezer. The test was conducted by using the Texture Analyser (TA.XT Plus, Stable Microsystem, England). For each sample, three measurement were carried out using 45° Perspex cone probe. The ice cream was penetrated by a probe to a distance of 50m.

### 3.4 Summary of methodology

Chapter 3 were discussed on the methodology used in this study to produce ice cream with normal water and nanobubbles water ice cream. After that, the test that required for analysis such as particle size analysis, viscosity, melting rate and firmness was explained.



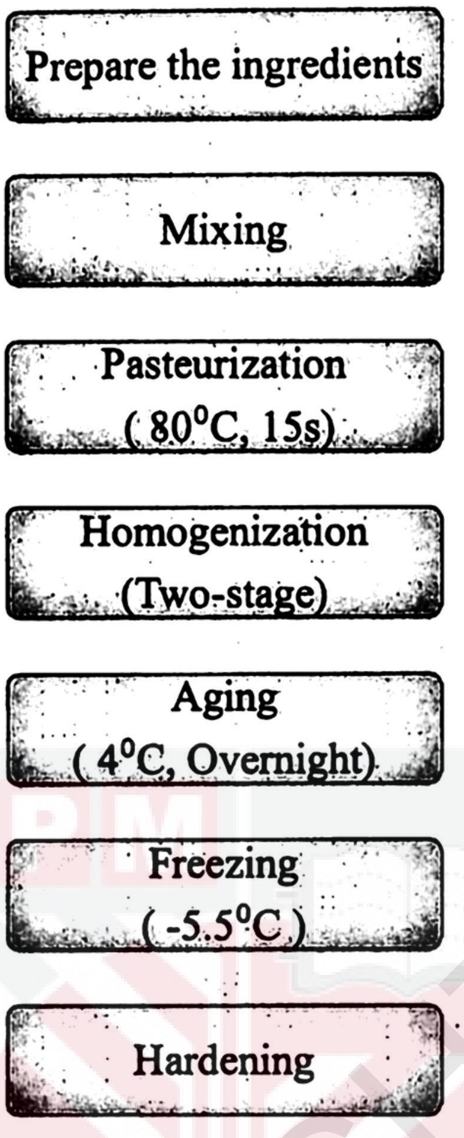



Figure 3-14: Flow diagram for sample preparation

# CHAPTER 4 : RESULT AND DISCUSSION

This chapter consists of the results of physical properties of hard ice cream by comparing hard ice cream that use normal water and nanobubbles liquid that was obtained from the experiment which is density and viscosity of the mix, overrun, melting test and firmness test.

## 4.1 Effect of using nanobubbles liquid on particle size distribution and viscosity of ice cream mixture

The particle size distribution in the ice cream mix had been identify using the Malvern particle size analyser for normal and Zeta potential Analyser for nanobubbles liquid. The size of particles was identified after the ice cream mix go through some shear effects such as mixing, pasteurizing and homogenizing process. Based on table 4-1, it was proved that there were different particle size distribution among both sample.

Table 4-1: Particle Size Distribution

| Sample | Particle Size Distribution |
|---|---|
| Normal water (Control) | 97.9 μm ± 5.04 |
| Nanobubbles liquid | 335.8 nm ± 5.76 |

Viscosity of mix of ice cream had been carried out based on research methodology. The bar graph viscosity of mix of hard ice cream after aging had been plotted as shown in Figure 4-1.

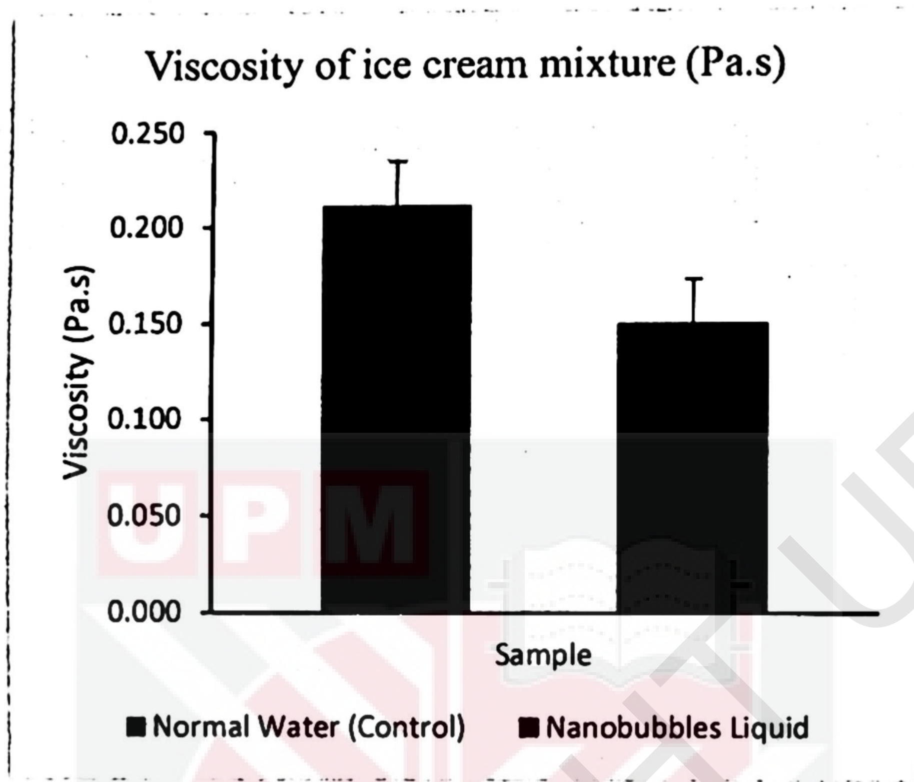



Figure 4-1: Bar graph for viscosity of ice cream mixture for normal water and nanobubbles liquid

Figure 4-1 shown the value of the viscosity of ice cream mix. Based on Figure 4-1, the viscosity of mix ice cream after ageing process by using normal water and nanobubbles liquid was 0.211 Pa.s and 0.149 Pa.s respectively. From the result, it was shown that the viscosity of normal water is higher than the viscosity of nanobubbles liquid. Goff and Hartel (2003) stated that for the viscosity of mix ice cream after aging process, there were no ideal mix value but it was usually in the range of 0.1 Pa.s to 0.8 Pa.s. Bahram and Tehrani (2011) stated that there are one of most important rheological properties of ice cream mix and unfrozen portion of ice cream which is viscosity.

The viscosity of ice cream can be affected by the mix composition (mainly stabilizer and protein), type and quality of the ingredients, processing and handling of the mix, concentration (total solid content) and temperature. Generally, for both sample normal water and nanobubbles liquid have the same parameters apart from the type of water used.

The viscosity in this study was related with the fat globule aggregation rate. There is few research found that viscosity increase was attributed to the solidification of fat due to aggregation and the clumping of fat globule were larger when and the size of the clumps remained the same when the viscosity decrease (Syed et al., 2018). By using nanobubbles liquid, the fat globule aggregation rate was decreases. This is because the charges on the surface of the ultrafine bubbles are negatively charged and repel each other thus makes the viscosity of the nanobubbles liquid ice cream decrease. Thus, it was shown that normal water ice cream have higher fat agglomeration than nanobubbles liquid ice cream.

Not only that, nanobubbles liquid ice cream has lower the coalescence as electrostatic properties of the nanobubbles that prevented the coalescence in the water. When the coalescence in water is decreasing, the rate air cell is coarsening also decrease. Hence, made the viscosity of the nanobubbles liquid lower than normal water. Goff (1999) stated that fat globules will move to air interface during foaming of mix. The amount of fat globule in both sample is same because of the same percentage of ingredient of process. Food (2014) found that larger molecules had tend to have higher viscosity while larger molecules had higher intermolecular forces attracting each other and with the greater strength that generates molecular flow. Thus, results for this experiment for viscosity was acceptable.

## 4.2 Effects of using nanobubble liquid on the density of hard ice cream

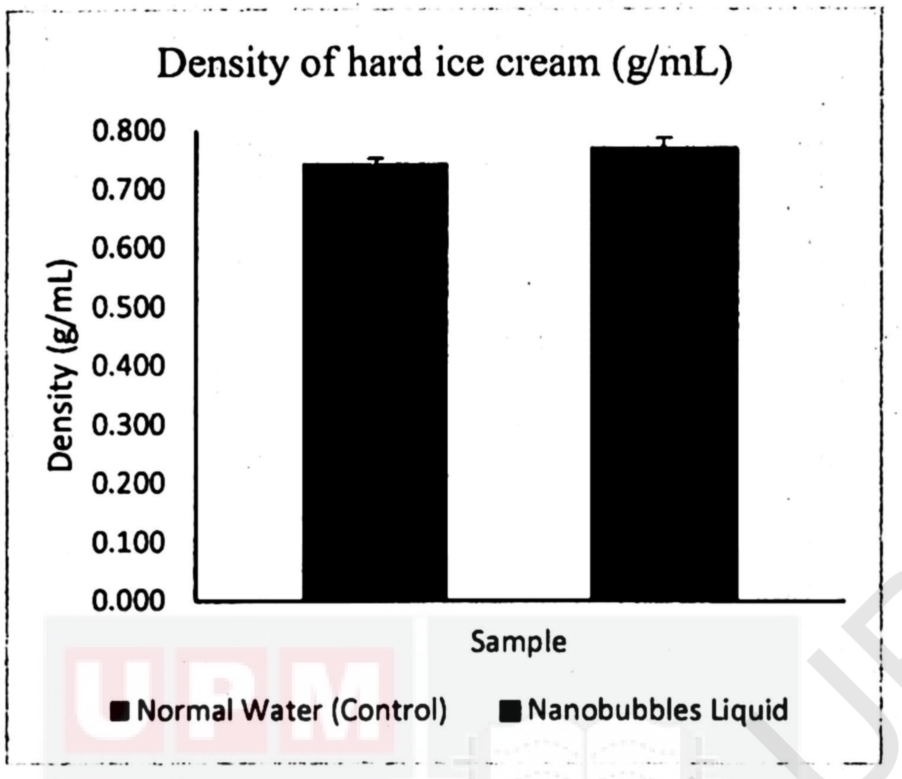



Figure 4-2: Bar graph for density of normal water ice cream and nanobubbles liquid ice cream

The density of the samples was shown in Figure 4-2. The density for normal water was 0.742 g/mL and density for nanobubbles liquid was 0.769 g/mL. From this result, it was shown that hard ice cream by using nanobubbles liquid have higher density than normal water. However, the density did not have big significant difference as the value is nearly the same. It was assumed that the density was affected by the nanobubbles liquid in the ice cream. Bajad et al (2016) stated that by increasing the level of MSNF, sugars and stabilizers increase density while increasing fat decrease the mix density. Not only that, the water content and amount of air also was affected the density value. For this research, because of all the ingredient weight by mass was the same, it was concluded that the density was affected by the amount of air entering the sample.

### 4.3 Effects of using nanobubble liquid on the overrun of hard ice cream

Overrun on the ice cream had been carried out followed the research methodology. The graph for both sample were plotted on Figure 4-3 so that it is easily to compare between two samples.

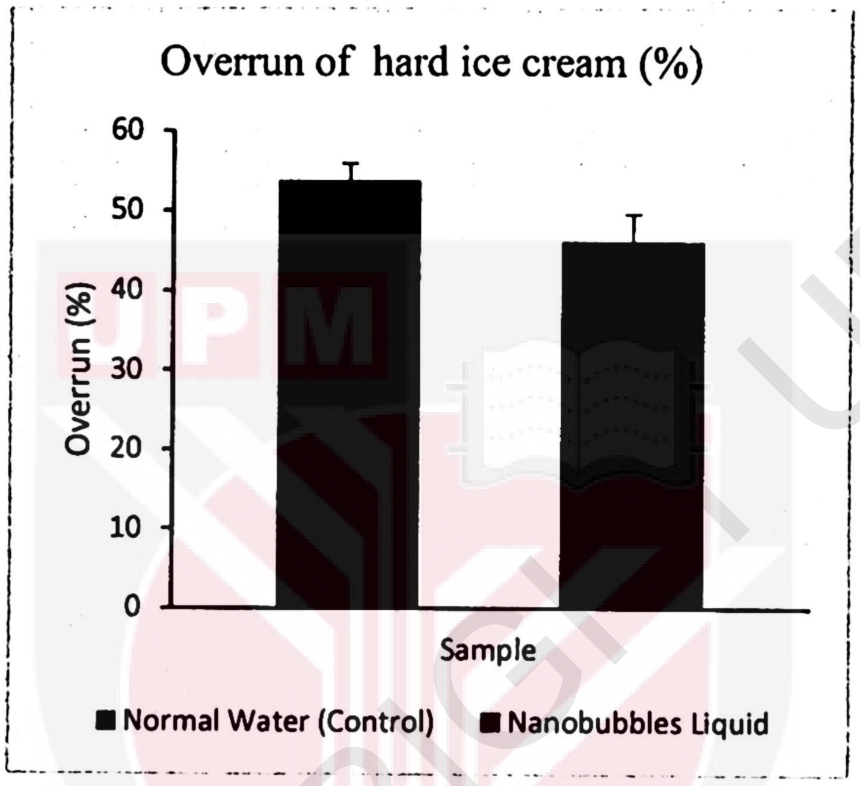



Figure 4-3: Bar graph for overrun of normal water ice cream and nanobubbles liquid ice cream

Overrun was known as the amount of the air added in the ice cream. Air play a big role in ice cream since it will affected both physical properties and storage stability. The texture of ice cream was not only affected by the amount of air enter the water but also the distribution of sizes of the air cell. Hence, the graph was shown that the overrun for normal water and nanobubbles liquid. Based on Figure 4-3, the overrun for normal water ice cream and nanobubbles liquid ice cream is 54% and 46% respectively. This might be due to the size of air bubbles in nanobubbles liquid sample which affected the expansion of ice cream.

Not only that, the value of overrun also might be due to the inconsistency during the whipping process (Chang & Hartel, 2002) which were caused by limitation of equipment. The value for both sample is lower than the general literature value 80-100% (Ozdemir et al, 2008). There were also another general literature stated that less expensive brands usually contain more air than the premium brand (Rohrig, 2014). Generally, Douglas (1996) found that ice cream with overrun less than 50%, is recognized as super-premium brands which is most expensive ice cream among the other.

Ice cream with nanobubbles water had lower overrun because less amount of air enter the ice cream. Nanobubbles liquid ice cream have ultrafine bubbles which is high in stability before added the air. Ice cream with good uniformity will enhance more stable air bubbles. Changes in air cell normally will occur in three primary mechanism which is disproportionation, coalescence and drainage. Not only that, shrinkage and expansion also important factors of defect in ice cream. In literature, Meegoda et al. (2018) stated that bubbles will swell and shrink depends on the bubble size. Larger bubbles will swell while smaller bubble will shrink that make the nanobubbles stay longer in the solution.

Ice cream that used nanobubbles liquid has higher shrinkage effect on the air cell because of the smaller diameter of the air bubbles in the nanobubbles liquid thus the overrun is low. Eventhough there were defect because of the shrinkage effect in the nanobubbles liquid ice cream, the nanobubbles liquid ice cream still have a good texture where will be prove on firmness analysis because of the others factors such as the shear in the sample preparation process such as pasteurization, homogenization and aging and also the ingredient such as stabilizers and emulsifier that make the air cell increase, thus the texture is still acceptable.

## 4.4 Effects of using nanobubble liquid on the melting rate of hard ice cream

The melting rate of ice cream was conducted as stated on the research methodology. Figure 4-4 shows that the result for melting rate for both sample which were ice cream with normal water and nanobubbles liquid.

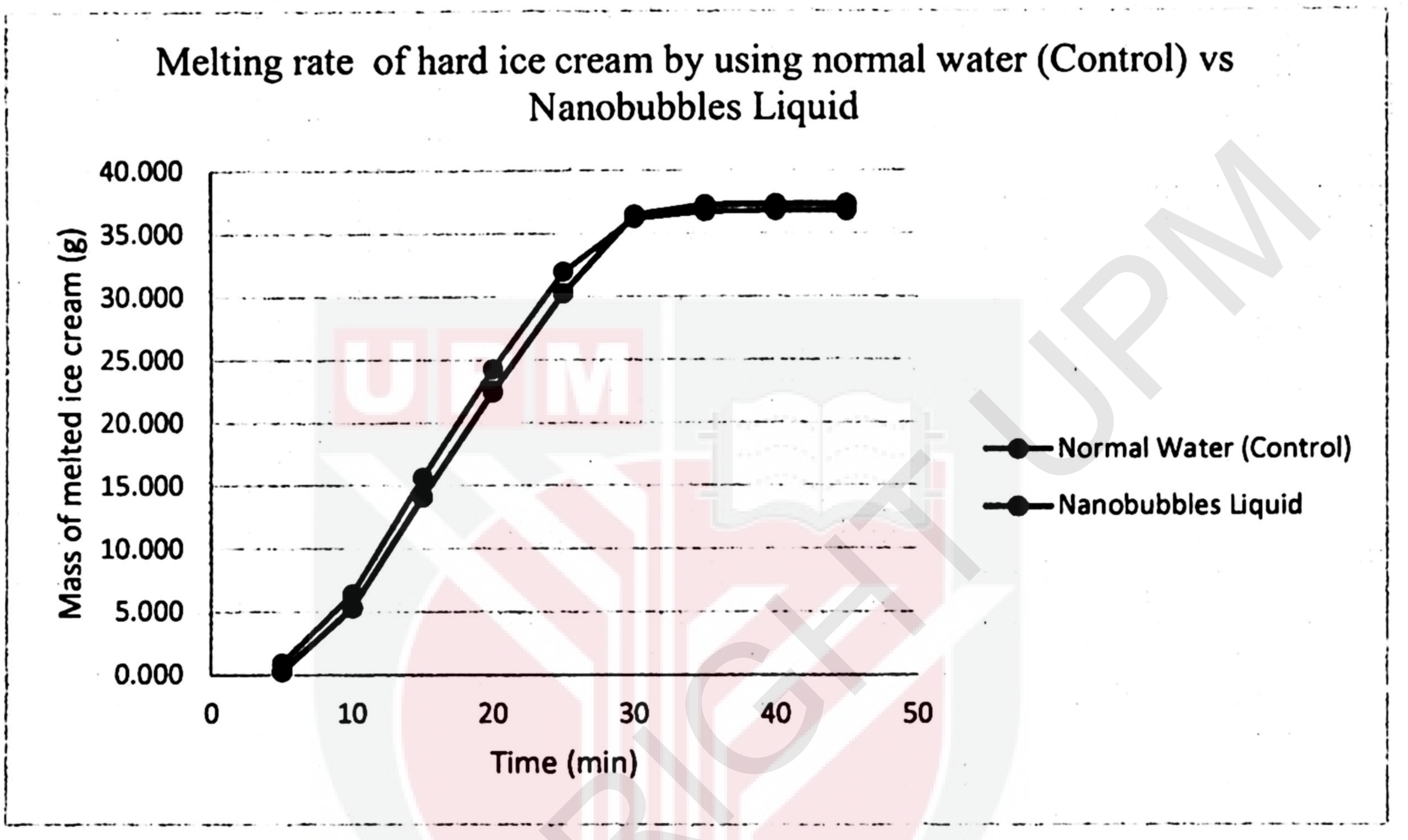



Figure 4-4: Graph of mass of melted ice cream over time

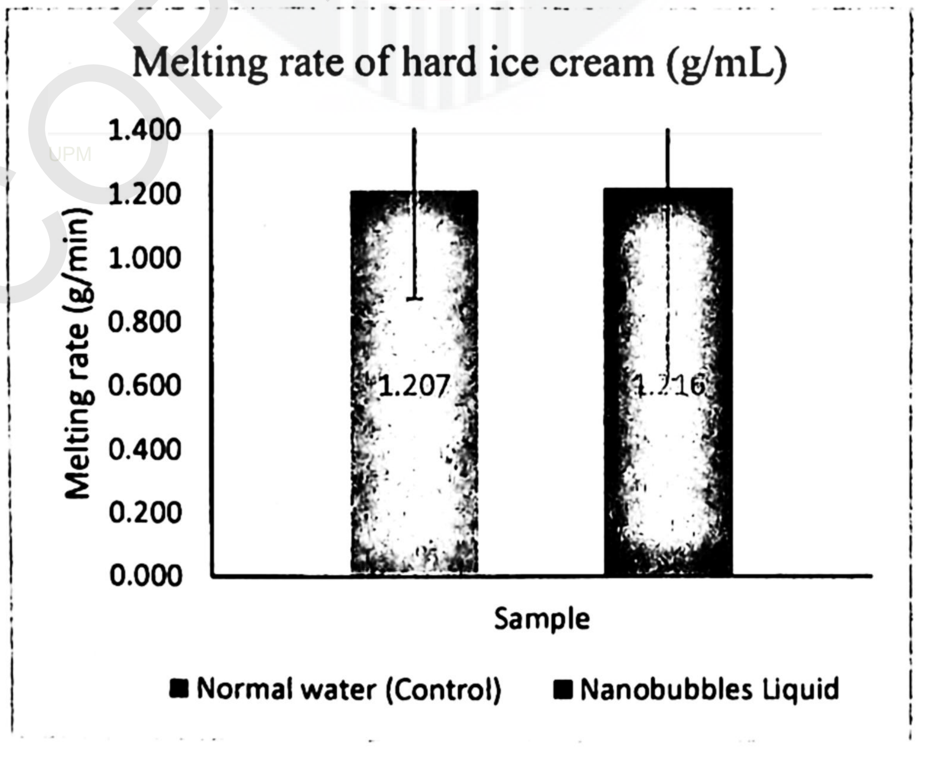



Figure 4-5: Melting rate of normal water ice cream and nanobubbles liquid ice cream

From the Figure 4-4, it was shown that the normal water had increased in weight as the time increased. For 30 minutes early, it can be seen that the normal water was melted faster than nanobubbles water but after 30 minutes, the ice cream for both ice cream started to melt constantly by weight because of the weight left in the sample. Referred to Figure 4-5, the melting rate for normal water ice cream is 1.207 g/min while for nanobubbles liquid ice cream is 1.216 g/min. This result was shown normal water ice cream has slower melting rate than nanobubbles liquid ice cream. For overall there is only less significant difference between this two types of sample.

Based on my research, the melting rate of nanobubbles water for the first 30 minutes slower may be due better structural strength and higher water binding capacity on the nanobubbles liquid ice cream as nanobubbles liquid is more stable. The melting rate of ice cream was affected by many factors such as the amount of air incorporated, the nature of ice crystals, and the network of fat globules formed during freezing (Muse & Hartel, 2004). Overrun did not appear to have a significant effect on the melting rate of ice cream in this study because the overrun of nanobubbles liquid ice cream is lower than normal water ice cream but their melting rate nearly the same which is different from Sakurai et al. (1996) and Sofjan (2002), who shown that ice creams with lower overruns had faster melting rates.

According to Pelan, Watts, Campbell, and Lips (1997), it is the stability of air cells that slows down the meltdown rate of ice cream. Thus, for my research, the meltdown rate of ice cream may be referred to the air cells stability as the nanobubbles liquid ice cream have very stable properties

## 4.5 Effects of using nanobubble liquid on the firmness of hard 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 in the experiment.

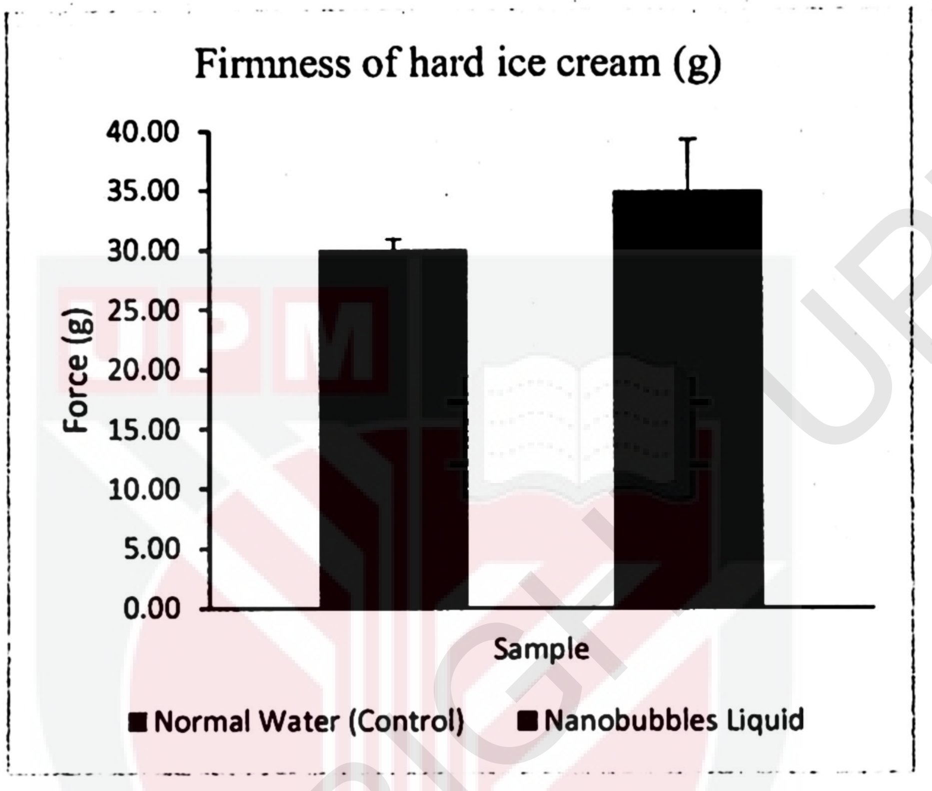



Figure 4-6: Bar graph for firmness of normal water ice cream and nanobubbles liquid ice cream

Based on the graph above, the firmness value for normal water and nanobubbles water was 29.93g and 34.80g respectively. The firmness is compared at the same distance of penetration which is 50mm. Nanobubbles liquid ice cream has higher firmness value which indicates its ability to retain its shape better and has a better texture.

The firmness can be affected by overrun, ice crystal size, ice phase volume and extent of fat destabilization (Muse & Hartel, 2004). For this research, the firmness may be affected due the ice crystal size and overrun due the nanobubbles size in the sample. Wilbey et al. (1997) stated that when the overrun is higher, the firmness of the ice cream will become lower.

Based on one general literature review, the smallest air cell will generated the most ice crystal thus increase the firmness of the ice cream (Muse & Hartel, 2004). Nanobubbles liquid ice cream had smaller particle size that will increase the amount of ice formation. Ice crystal will packed closer to each other that larger force require to be applied on the surface of ice cream that make the firmness of ice cream increase (Muse, 2003). In conclusion, nanobubbles liquid ice cream have softer texture because of the smaller ice crystallization and its better texture makes it applicable to the ice cream industry.

## CHAPTER 5 : CONCLUSION AND RECOMMENDATION

This chapter summarize the research work done. The main objective about this study is to determine the effects of nanobubbles liquid on the physical properties of ice cream. The physical properties that was determine is the size particle and viscosity of ice cream mixture. Also, the density, overrun, melting rate and firmness of ice cream which will affects the texture of the ice cream. Not only that, recommendation for future work also given in this chapter.

### 5.1 Conclusion

From this study, different industrial application of nanobubbles in food area was conducted in laboratory scale experiments to observe the effects of its properties in ice cream. It is important to choose the suitable ingredients as they have own function that will affect the physical properties of the ice cream. For this research, nanobubbles water was replaced with the normal water. This nanobubbles liquid lead to the different result between two samples.

The objective to determine the effects of nanobubbles liquid on the physical properties of ice cream have been achieved as there is difference between normal water sample and nanobubbles water sample. The density of nanobubbles liquid ice cream is higher than normal water ice cream where it shows that it more dense than normal water. Not only that, viscosity for ice cream mixture of nanobubbles liquid is lower than normal water while the overrun is also lower. Next, even though the overrun is lower, nanobubbles liquid ice cream have slow melt-down. Lastly, nanobubbles liquid ice cream have higher firmness that can makes it retain shape better.

In a conclusion, the objective of the project is achievable because we can see effects of nanobubbles liquid in the physical properties of both sample. In this study, it is shows that when density is lower, the viscosity also will be lower thus the overrun also will be low. Even though nanobubbles liquid ice cream mixture have lower viscosity, the firmness of the ice cream is higher and the melting rate of the ice cream is almost the same with normal water. The behaviour of the nanobubbles liquid ice cream is mostly affected by the size distribution in the nanobubbles itself.

## 5.2 Recommendations

The analysis was done by using the nanobubbles liquid. The usage of nanobubbles liquid is a new study as it is rarely used in food industry as it is hard to produce the nanobubbles and expensive especially in Malaysia.

The study in ice cream area is wide. Many factors that can be affected the structure of ice cream such as milk fat, Milk Solid Non Fat (MSNF), stabilizer and emulsifier. For future research, the formulation of ice cream can be alter to see the effects of ingredients on ice cream and add more analysis to further investigate the effects of nanobubble liquid on the internal structure of ice cream. The analysis can be add more such as viscoelasticity, measurement of zeta-potential. The image analysis by using light microscope or cryo-SEM also can be taken into consideration to observe the ice crystal distribution especially the structure of ice cream in liquid state and solid state.

Not only that, the aging time for the sample also need to be control as aging process will influence the viscosity of ice cream mix. The number sample also can be increased by using the ratio of normal water: nanobubbles liquid to see either the presence of nanobubbles liquid in normal water will affect the ultrafine bubbles or not.

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