EXTRACTION OF COW FAT AND SOLIDIFICATION OF KINETIC STUDY ANALYSE USING THE AVRAMI MODEL

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THESIS DECLARATION

I hereby declare that the work embodied in this report entitled "Extraction of Cow Fat and Solidification of Kinetic Study Analyse Using the Avrami Model" is the result of the original research and has not been submitted for a higher degree to any universities or institutions.

Signature : ________________________________

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I certify that this thesis entitled "Extraction of Cow Fat and Solidification of Kinetic Study Analyse Using the Avrami Model" by Abdul Rahman bin Mat Jusoh has been examined and all the correction recommended by examiners have been done for degree of Bachelor of Applied Science (Animal Husbandry) with Honours, Faculty of Agro-Based Industry, Universiti Malaysia Kelantan.

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LIST OF ABBREVIATIONS AND SYMBOLS

e	exponent
Eq.	Equation
g	gram
k	crystal growth
Min	minutes
n	avrami exponent
TAG	triacylglycerides
X	fraction of solidification
°C	Temperature
%	Percentage

Extraction of Cow Fat and Solidification of Kinetic Study Analyse Using The Avrami Model

ABSTRACT

Fat crystallization studies are important from both technological and academic points of view. Understanding the effects of formulation and process factors on the kinetics of crystallization is important to control of product quality in industry. This application is to study the solidification phenomena of cow fat using Avrami method by consider the factor which is the effect of the difference cold temperature and difference mass of fat and compare the solidification mechanism. The percentage of solidification was measured started at first two minute of the solidification until the complete solidification of all parameter. Percentage of the solid was measured and recorded and Avrami equation is apply in order to determine the growth rate and crystallization form. To study the effect of the cold temperature, the weight of the fat was kept constant at 28 g and the temperature of the fat was being set at the 70°C which is the fat is in liquid form. As cold temperature decreased the percentage of solidification increased which the lower temperature which is 10°C is the higher percentage value which is 49.28% follow by 15°C and 20°C which is 35.35% and 30.71% respectively. Have a same value of n=3 that consider the crystallization form of spherulitic growth from instantaneous nucleation or disc-like that in sporadic nucleation form. Smaller volume used have the higher degree of solidification and occur in short time because of the decreasing resistance caused by air bubbles and increased air space volume for arrangement of solid crystal structure to easily occur. The study recorded, The smaller mass of the fat which is 10 g have the higher degree of the solidification percentage was recorded which is (61.8%) compare to other difference mass, 20 g and 30 g that have (51.61%) and (48.67%) respectively. Overall, the higher percentage of solidification will be in lowest temperature and mass of the fat.

Keywords: Cow fat, Avrami model, Gravimetric Method, Solidification mechanism

Process Mengekstrak Lemak Lembu Dan Kajian Kinetik Dalam Pemejalan Lemak Lembu Berdasarkan Model Avrami

ABSTRAK

Kajian kristalisasi lemak adalah penting dari sudut pandang teknologi dan akademik. Memahami kesan faktor perumusan dan proses pada kinetics of crystallization adalah penting untuk mengawal kualiti produk dalam industri. Kajian ini adalah untuk mengkaji fenomena pemejalan lemak lembu menggunakan kaedah Avrami dengan mempertimbangkan faktor yang mana kesan perbezaan suhu sejuk dan jisim perbezaan lemak dan membandingkan mekanisme pemejalan. Peratusan pemejalan diukur bermula pada awal dua minit pemejalan sehingga proses pemejalan telah mencapai 100% pejal berdasarkan parameter yang digunakan. Peratusan pepejal diukur dan direkodkan dan persamaan Avrami digunakan untuk menentukan kadar pertumbuhan dan bentuk penghabluran. Untuk mengkaji kesan suhu sejuk, berat lemak disimpan tetap pada 28 g dan suhu lemak telah ditetapkan pada 70°C yang adalah lemak dalam bentuk cair. Seiring suhu sejuk menurun, peratusan pemejalan meningkat dimana suhu yang lebih rendah iaitu 10°C adalah nilai peratusan yang lebih tinggi iaitu 49.28% mengikut 15°C dan 20°C masing-masing sebanyak 35.35% dan 30.71%. Mempunyai nilai yang sama n = 3 yang menganggap bentuk penghabluran pertumbuhan spherulitik dari nukleasi segera atau cakera-seperti yang dalam bentuk nukleasi sporadis. Jisim lemak yang lebih sedikit yang digunakan mempunyai tahap pemejalan yang lebih tinggi dan berlaku dalam masa yang singkat kerana rintangan berkurang yang disebabkan oleh gelembung udara dan peningkatan isipadu ruang udara untuk susunan struktur kristal padu untuk mudah terjadi (Ismail et 2008). Kajian ini mencatatkan, jisim yang lebih kecil daripada lemak yang 10 g mempunyai kadar peratusan yang lebih tinggi direkodkan iaitu 61.8% berbanding dengan jisim yang berbeza, 20 g dan 30 g yang mempunyai 51.61% dan 48.67 %) masing-masing. Secara keseluruhan, peratusan pemejalan yang lebih tinggi akan berada di dalam suhu dan jisim terendah.

Kata kunci : lemak lembu, pemejalan, model Avrami, metod gravimetri, mekanisma pemejalan

CHAPTER 1

INTRODUCTION

1.1 Research Background

There are million of cow is butcher everyday and around half of the cow is transformed into waste or side-effects.The side-effects are additionally prepared and utilized for assortment of uses, therefore, enhancing the cow. Customary utilizations for the protein rich solids incorporate use in sustenances, pet nourishments, domesticated animals sustains, and manures. Fats have been changed into cleansers and oleochemicals (unsaturated fat subordinates) notwithstanding being utilized as a part of sustenance, pet nourishments and sustain applications. The requirement for new outlets of items has likewise been acknowledged due to generally experienced zoonotic ailments. The new outlets incorporate utilizing protein dinners and creature fats as vitality sources in burning units for the age of steam or sustainable power. In any case, creature results contain large amounts of water and have an exceptionally appropriate organic and microbiological arrangement which, if not settled, can prompt disintegration and natural contamination.

The most traditional method for balancing out crude material is to process the crude material with warm. This serves to both vanish the water content and disinfect in the meantime: this procedure is known as "Rendering". Rendering is the way toward sanitizing fat from the cow bits, any blood, connective tissue, water or meat is isolated from the fat. Cow fats are delivered by this procedure which has been produced in the course of the most recent 200 years or so from extremely fundamental cooking and dissolving procedures to refined modern procedures that are basically following a similar procedure steps. Rendering is utilized to change over land creature butcher results into attractive items, including consumable and unpalatable creature fats and proteins for nourishment, rural, and mechanical utilize. cow fat is an adaptable, manageable, and common reason for some items.

Greasy tissues from cow meat are made out of basically three parts, viz. water, protein and fat. It is the motivation behind any rendering framework to acquire as entire a partition as achievable of theseUI87 materials. As a rule, the greasy tissues are cooked and the fat is discharged by temperature and cell burst. In elective process, the temperature is kept low and the fat is discharged chiefly through mechanical burst of the cells. Adjacent to that, to render fat, you soften it and warmth at a low temperature until the point that all proteins cement and any water vanishes. You at that point channel the solids from the fluid fat. Once cooled, you are left with clean unadulterated fat. Poisons, for example, anti-infection agents that are nourished to seriously cultivated creatures gather in their fat stores, so it's savvy to source your fat from solid, unfenced and fed creatures.

According to A.E.Bailey (1997), The estimation of solid–liquid phase equilibrium(solidification) is important for the design, development, and operation of many industrial processes because of application in many manufacturing fields such as cosmetic, pharmaceutics, and biotechnology industries.

Crystallization is the solidification of atoms or molecules into a highly structured form called a crystal. Usually, this refers to the slow precipitation of crystals from a solution of a substance. However, crystals can form from a pure melt. Crystallization can also refer to the solid-liquid separation and purification technique in which mass transfer occurs from the liquid solution to a pure solid crystalline phase. Two events must occur for crystallization to occur. First, atoms or molecules cluster together on the microscopic scale in a process called nucleation. If the clusters become stable and sufficiently large, crystal growth may occur. Crystal growth and crystallization type of the fat can be determined by using avramic equation. Atoms and compounds can generally form more than one crystal structure (polymorphism). The arrangement of particles is determined during the nucleation stage of crystallization. This may be influenced by multiple factors, including temperature, size of cow meat, environment, and the purity of the cow meat.

TAGs (triacylglycerides) generally crystallize initially in forms α and β′, although form β is more stable. This phenomenon is related to the fact that form β has a higher free energy of activation for nucleation. The polymorphic transformation is an irreversible process going from the less stable to the more stable form (monotropic phase transformation), depending on the temperature and time involved. At constant temperature, the forms α and β′ can become, as a function of time, form β through the liquid–solid or solid-solid mechanisms (Herrera and Marquez Rocha,1996). The transition rate is dependent on the degree of homogeneity of the TAGs. Fats with low variability of TAGs quickly turn into the stable form β. Fats that are the random distribution of TAGs may have the form β′ indefinitely. In addition, factors such as formulation, cooling rate, crystallization heat and level of agitation affect the number and type of crystals formed. However, as fats are complex mixtures of TAGs, at a certain temperature, different polymorphic forms and liquid oil can coexist (Sato,2001).The characteristic classification of polymorphs of cocoa butter is based on the Roman numbering system (I to VI), in which form I is the less stable one and form V. fat in VI is determined as form β′.

Fats with crystals in form β′ feature increased functionality, as they are softer; provide good aeration and creaminess properties. Thus, form β′ is the polymorph of interest for the production of foods high in fat such as margarine and confectionery and bakery products. For the production of chocolates with good physical and sensory characteristics, however, form βV is the desirable polymorph, because it is associated with properties such as brightness, uniformity, characteristic snap and improved shelf life (O'Brien 2008).

In a solution in the crystal growth phase, equilibrium is established in which solute particles dissolve back into the solution and precipitate as a solid. If the solution is supersaturated, this drives crystallization because the solvent cannot support continued dissolving. Sometimes having a supersaturated solution is insufficient to induce crystallization. It may be necessary to provide a seed crystal or a rough surface to start nucleation and growth.

1.2 Problem Statement

In Malaysia, there is numerous items that have generally large amounts of soaked fat, which is most considered as a hazard factor for coronary illness by administrative bodies in created nations.There have only been a few studies to understand the phase change behavior of fat, esspecially on combining gravimetric methods. Beside there is also lack of information on the cooling condition of the cow fat employing the use of Avrami model, as fat its recognised health benifit has a widespread use in many industrial processes manufacturing fields such as cosmetic, pharmaceutic, and biotechnology industries. Solidification study of cow fat can potentially develop fat as functional material, which, solidification phenomenon and behaviour may affect the fat quality and functional.

1.3 Objective

In oder to study the solidification behavior and extraction, the objectives of this research are determined as follows:

1. To study the effect of time, temperature, and volume of meat on crystalization
2. To study of solidification of kinetic by gravimetric method using the the avrami model kinetics.

1.4 Scope of Study

The study is focosing extraction and solifidication of crystallization of fat and by using wet rendering method and gravimetric method. The parameter doing study are the effect of difference cold temperature, and effect of fat size used. The fat yield of the fat was determine after the wet rendering process. The solid fat was determined by measuring the fat liquid throughout the solidification, which is will be minus to total of fat used, 28g. The solidification was conduct and record in two minute difference range,by determined the percentage of fat solidification, T= 2,4,6,8,10. The result was compare between the parameter used. The solidification kinetic of fat was conduct with gravimetric methods. From the data, avramic model was apply.

The avramic model is importance to study the phase change of liquid fat to solid fat and study the fat crystallization behavior. The avramic model is conduct to determined n value and growth rate of the fat crystallization on the parameter used. the crystalization behavior will effect in difference temperature and size.

1.5 Significant of Study

The crystallization of fats has been extensively investigated, due to its importance in everyday life. After all, an understanding of the fat crystallization process plays a critical role in determining overall product quality. Products in which fat crystallization is important include chocolates and confectionery coatings, dairy products such as butter and cream, vegetable spreads and peanut butter. Fat crystallization is also important in fat and oil fractionation where lipid products with varying melting and physical properties are manufactured by melt crystallization (Hartel, 1992). Next, triglyceride crystallization is used to eliminate small quantities of high-melting compounds from an oil so it remains clear at low ambient temperature (Dibildox-Alvarado & Toro-Vazquez, 1997).

The study was carry out to understand the solidification behavior of cow fat obtain from two different variable that will affect the crystallization , physical properties of the final product which, the effect of different cold temperature and the mass of fat used in solidification process. In understanding solidification behavior, Avrami method will apply by determining the growth rate, degree of solidification and solidification index. According to (Hartel 1998), the study of nucleation mechanism and growth rate is impoertance to in oder to controlling process and storage condition. It also importance in production of smooth texture and good quality of the product that made from cow fat.

1.6 Limitation of Study

In this study, there are few limitation with regards to equipment. First of all, the unavailability of equipment to study the crystal structure accurately such as pulsed nuclear magnetic resonance(NMR) is due to the fact that it is very costly. NMR gives an exact information of peaks that indicate which atom are connected, and its neighbouring atoms. The second limitation is the lack of cooling and heating device for study fat phase behavior. This make study of solidification of fat more difficult as gravimetric method contain error related to evaporation of fat during heating and to the surrounding laboratory evironment.

CHAPTER 2

LITERATURE REVIEW

2.1 Chemistry of Lipids

(According to Hui,1996) fats and oils make up about 99% of the lipids of plant and animal origin.Fats and oils are distinguished by their physical state at room temperature. The lipid mixture which is solid at room temperature known as fat, while oil refers to the fat which is liquid at room temperature. Mostly, fat and oil are constituted by triacylglycerols. Triacylglycerol molecules have a glycerol backbone with up to three fatty acids esterified to it. According to (Afaf and Jan,2005) a glycerol molecule that have three fatty acids are known as triacylglycerols (TAG), when the glycerol that have two fatty is called diacylglycerols(DAG), and if only one fatty acid is present, it called monoacylglycerol (MAG). Futhermore, the characterization of fatty acid is base on the number of carbons and degree of unsaturation in the molecule. Commently, edible oils have fatty acids of carbon length ranging from 4 to 20. Fatty acids can be divided into two difference catogories which,saturated and unsaturated fatty acids. Saturated fatty acids can be recognize with their caracteristic which is contain no double bonds as each carbon atom is surrounded by other carbon atoms and hydrogen atoms. Due to the absence of double bonds, the fatty acid molecules are joined in a zig-zag chain as there is free rotation about the carbon atoms. This saturated fat molecules more easy to create a closely packed structure rather than unsaturated fatty acid that have double bonds (Semih, 2005).

Edible oils and fats is vital role in providing essential fatty acids and energy that provide essential nutrients in human diet. Chemically, natural oils and fats that are triacylglycerols (TAGs), consist of glycerol esters and fatty acids. Additionally, polar lipids that is the minority lipids are found solubilized in the triacylglycerol matrix, diacylglycerols (DAGs),monoacylglycerols (MAGs), free fatty acids, phospholipids, glycolipids and sterols are the example of polar lipid. The composition of triacylglycerol is determines to know the physical properties of oils and fats that will affect the structure, stability, flavor, aroma, storage quality, and sensory and also visual characteristics of foods.

The determination of oil and fat physical properties are importance to determine its use. This can be more is particular in a large quantity and variety of oils and fats that used in various forms and function. The components of fat characterize it as a material composed of an intimate mixture in the liquid and solid phases, and its physical state can vary from a viscous fluid to a solid or brittle plastic.

2.2 Functions of Lipids

From Harry (1995), say that fat provide tenderness, structural integrity, aid in the incorporation of air, and to extend shelf life of food. Fats are very importance and usefull in production of confectionaries and also as emulsifiers in food products. According to Shahidi,(2004) say that the short and medium chain-length of dietary fatty acid are not usually esterified but it rapidly oxidized that will become the source of can be call fuel in tissues that to support all the events necessary and keep functioning organisms to work. Futhermore, fatty acids that have long-chain are usually esterified to glycerols in tissues (Shahidi, 2004). This is importance and funtioning for growth, building cell walls and aid in the construction of phospholipids. Essential fatty acids shoud be obtained therefore, fatty acids cannot be synthesised by the body and it good for the diet. They aid in lowering excessive cholesterol levels in the body, thereby decreasing the occurrence of cardiovascular diseases.

According to Maxwell and Marmer, (1983) reported, Fatty acids are also can be used asbiosynthetic precursors of many insect pheromones and secondary metabolites in plants Unesterified fatty acids are produced rapidly as a consequence of the binding of specific agonists to plasma membrane receptors which is it can anable to act as second messengers required for the translation of external signals. fatty acids can act to amplify or otherwise modify signals within cell that influence the process and activities of enzymes such as protein kinases, phospholipases, and many more. There are other uses and functions of animal fat. They are involved in regulating gene expression, mainly targeting genes that encode proteins with roles in fatty acid transport or metabolism via effects on transcription factors, i.e., peroxisome proliferator-activated receptors (PPARs) in the nuclei of cells.

Such effects can be highly specific to particular fatty acids (Hui, 2006). Thus, unesterified arachidonic acid may have some biological importance as part of the mechanism by which apoptosis (programmed cell death) is regulated. Stores of fat within the human body are important as they provide quick access to energy while fasting, act as insulation from hypothermia, while fat surrounding organs provides an internal layer of protective padding (Shahidi, 2004). The ingestion of fats however, must be done with moderation. It has been found that saturated fats are major causes of cardiovascular disease such as arthelesclerosis (Gunstone, 2004). They increase low density lipoprotein (LDL) cholesterol, the so called "bad" cholesterol, which leads to coronary heart disease. Trans fats, introduced during partial hydrogenation causes and increase in LDL cholesterol while concurrently decreasing HDL cholesterol (Gunstone, 2004).

In addition, most natural oils and fats have limited application, imposed by their particular composition in fatty acids and TAGs. This way, oils and fats for various industrial applications are chemically modified, through hydrogenation or interesterification, or physically modified, through fractionation or mixture (Erickson 1995). Although used for a long period, the partial hydrogenation results in the expressive formation of trans fatty acids, associated with negative health effects (Hunter 2005).

Worldwide, the controversial issues about the role of *trans* fatty acids in food caused progressive modifications in legislation, aiming at the inclusion of more information for consumers, requiring the declaration of the levels of these components on the nutrition labeling of foodstuffs. In response, the industries have opted for the progressive replacement of Trans fat in various products, through the development of fat bases with economic viability and functionality equivalent to partially hydrogenated fats, without increasing the content of saturated fatty acids in foods (Ribeiro et al. 2007).

In this sense, interesterification proved to be the main alternative for obtaining plastic fats with low levels of Trans isomers or even absence of these compounds. Particularly, the chemical interesterification of liquid oils with fully hydrogenated oils (hardfats) is currently the alternative of greater versatility to obtain zero trans fats, producing fat bases with favorable characteristics to prepare general-purpose shortenings (Ribeiro et al. 2009). The use of blends, i.e. mixtures of fats with different physical properties, and fractionation are also additional alternatives for obtaining fat bases with proper plasticity and physical characteristics to be used in several products, although with potential limited by the chemical composition of the raw materials (Foubert et al. 2007).

Although the processes of interesterification, fractionation and blending are very functional under the technological point of view, the replacement of partially hydrogenated fats in food products, mainly in shortenings and confectionery, is currently a challenge, as suitable crystallization and texture properties are difficult to obtain in the absence of trans fatty acids (Reyes-Hernandez et al. 2007).

Especially, the appropriateness of the crystallization kinetics of these fat bases is of is of paramount importance for their use can be adjusted to the limitations of industrial processes and to improve the control of the processing steps involving the recrystallization of the fat fraction, ensuring the quality of the end product (Foubert et al. 2006). Otherwise, processing times and equipment already standardized need to be changed according to the characteristics of the fat used. This fact has become particularly important as new fat bases began to replace partially hydrogenated fats in most industrial applications, mainly in the production of biscuits and bakery products, in which it was noted that fats with the same apparent solid profile presented very different crystallization properties (Bell et al. 2007). In the specific case of interesterified fats, the formation of partial acylglycerols, such as MAGs and DAGs, as a result of chemical interesterification, can influence the crystallization kinetics through changes in the process of nucleation of crystals (Herrera et al. 1999). According to Minal (2003), 0.1 % of sodium methoxide catalyst, employed for randomization, can produce between 1.2 and 2.4 % of MAGs + DAGs. Since the typical content of the catalyst used industrially varies between 0.1 and 0.4 %, the resulting levels of these minor lipids can be greater than 9 %. Although minor lipids have influence on the properties of crystallization of these fats, their complete removal is still difficult and costly, especially on a large scale (Metin and Hartel 2005).

Considering that this replacement process is relatively recent, the crystallization behavior problems because of the non-suitability of new fat bases are numerous and exacerbated, mainly because of regional climatic differences and conditions of transportation and storage. In this context, we can highlight problems like undesirable polymorphic transitions, exudation of oil, development of fat bloom, formation of crystalline clusters, as well as fat bases with maximum content of solid fat or induction periods incompatible with certain industrial applications. Studies on the modification, stabilization and control of the crystallization of these materials thus have crucial importance for the development of the industry of edible oils.

2.3 Sources of Animal Fat

Fats from animal origin come from various of animals, for example cow which is contain many fat in body. The amount of fats that found on the cow is depends highly on their diet. The amount of fat found in the animal depends also on location on the animal and also the species of cow may influence the number of fat. The major amount of fat can be found that located under the skin and overlying superficial muscles and also the intermuscular fat that located between muscles.

2.4 Extraction Pre-Treatment Techniques

Particle size reduction is also an important step in sample pretreatment. Size reduction promotes good solvent contact as it provides a greater surface to volume ratio, enhances fat extraction resulting.

2.5 Crystallization Behavior

Crystallization studies are important from both technological and academic points of view. Understanding the effects of formulation and process factors on the kinetics of crystallization is important to control of product quality**.** Crystallization in general can be divided into two steps: nucleation and growth. Before nuclei formation, the mother phase must be supercooled to provide a thermodynamic driving force for crystallization. Once the nuclei have formed, they grow and develop into crystals. In fact, both events usually happen simultaneously and the system is in continuous evolution. Additional changes in crystals can occur as stable crystals modify their habit and metastable crystals undergo polymorphic transitions. The nucleation and growth sequence may become erratic, leading to imperfect and variable-size crystals. In the industrial fractionation of fats, it is an important aim of the process to limit this exotherm by varying the rate of heat removal and agitation with temperature.

2.5.1 Nucleation

According to (Boistelle,1988) nucleation involves the formation of molecule aggregates that exceed a critical size, and are therefore stable. Liquid fats are cooled will turn to solid phase where its composition and quality rely greatly on the solidification temperature and cooling rate. Nucleation begins when the liquid release enough heat energy which is knows as exothermic to form small crystals in the liquid solution. According to (Garside,1987), to initiate the process, a thermodynamic driving force that known as super cooling has to be used. The process is reversible where nuclei can dissolve with change of temperature. A stable crystal will form only when the energy due to the heat of crystallisation exceeds that required to overcome the surface energy (Timm,2005). Hence, solid oil molecule will come together and that decreased their free energy, which it leading to the production of crystal lattice. When concentration of these molecule exceed the solubility limit, liquid fat separation from solid fat to allow solid-solid interaction of fat to form nuclei aggregates. it is because that the solubility must depend on the size of the crystal.

Once crystals have formed due to primary nucleation, secondary nucleation can also occur. Secondary nuclei form whenever small pieces of crystal are removed from the growing crystal surface. If the pieces are smaller than the critical size, they redissolve; if larger, they act as nuclei and grow to become crystals. Secondary nucleation is undesirable in fractionation. Agitation or stirring is the primary cause, and therefore agitation is usually kept to the minimum required to facilitate heat transfer. (Ulrich and Strege 2002) reported the nucleation can greatly influence the fat properties such as the content of solid fat and crystal properties. Measuring the rate of nuclei formation is tedious as it involves counting the quantity of crystal formation is tedious as it involve counting the quantity of crystal formation before they can develop to a detectable size (Hartel, 2011).

2.5.1 Crystal Growth

Once a crystal nucleus has formed, it will start growing by the incorporation of other molecules from the adjacent liquid layer that is continuously filled by the supersaturated liquid surrounding the crystal. It begins when separated nuclei formed attach with each other on the surface of crystal. This is cause by more of liquid fraction separating away from the solid fraction. These molecules are taken from the adjacent liquid layer, which is replenished continuously from the surrounding, supersaturated liquid by diffusion. The growth rate is proportional to the amount of supercooling and inversely proportional to the viscosity, which affects the rate of diffusion. (Timm,2005). The development of crystal will continues if oil crystal molecule have enough energy to arrange themselves together in a crystal lattice due to the present of high saturated TAGs. However according to (Timm, 2005) when there is absence of stirring, temperature will have increased, then the volume and concentration of liquid fat adjacent to the growing crystal will be desaturated and the nuclei can be dissolved

2.6 Solidification (Crystallization) Behaviour of Fat

The crystallization behaviour of lipids has important implications in the industrial processing of food products whose physical characteristics depend largely on fat crystals. Such products include chocolates, margarines, spreads, fats for confectionery and bakery, dairy products, and commonly used shortenings. There are many factor that will affected the degree of solidification of crystallization behaviour. Crystallization is generally divided into four distinct phases. Initially, in order to obtain the formation of crystals from the liquid state, the system must reach the supersaturation zone, in which there is a driving force for crystallization. Once the appropriate driving force to overcome the energy barrier for crystallization is reached, nucleation occurs and molecules in the liquid state join together to create a stable nucleus. After the formation of stable nuclei, a rapid transition to the next stage of crystallization occurs, crystal growth, i.e., during which additional molecules (or growth units) are incorporated into the crystal lattice, decreasing the driving force of supersaturation. Unless restricted by a kinetic constraint, growth continues until the system reaches equilibrium, at which the driving force for crystallization approaches zero and the maximum volume of the crystal phase is obtained

2.6.1 Effect of Difference Cold Temperature

Temperature can be a significant variable in the crystallization of biological macromolecules (proteins). Temperature often influences nucleation and crystal growth by manipulating the solubility and supersaturation of the fat. Temperature has also been shown to be an important variable with phase separation in detergent solutions during membrane protein crystallization. Temperature was shown to affect quantity, size, and quality of the crystals as well as sample solubility and preliminary crystallization data. Temperature is amenable to control and can be used to carefully manipulate crystal nucleation and growth. According to Hampton, crystallization screens and experiments are performed at room temperature and sometimes 4 °C. A reasonable range of temperature to screen and optimize for protein crystallization is 4 to 45 °C. Cooling of confectionery product, hence animal fat will result in formation and growth of oil crystals. Difference storage temperature lead to difference degree of solidification of fat.

2.6.2 Effect of Difference Mass of Fat

The study of the parameter conducted by using difference mass of fat to determined effect of mass in solidification of crystallization. According to Ismail et.al (2008), smaller volume used have the higher degree of solidification and occur in short time because of the decreasing resistance caused by air bubbles and increased air space volume for arrangement of solid crystal structure to easily occur. As expected result, the lower the mass of fat will have the higher degree of solidification. To identify the growth rate of the fat, within the all fat, Avrami method will apply and compare.

2.7 Solidification Kinetics- Avrami Model

Avrami model is considering to determined isothermal solidification behaviour by analysis the crystallization of fat at phase transition. Avrami equation used by fill the data of the solidification process to study the growth rate and the avramic exponent that show the crystallization form. The process of crystallization is expected to start randomly at various locations from the nucleation sites and spread outwards. This is demonstrated by the conception of raindrops dropping in a pool creating wave circles that cross over each other while expanding to the whole surface. The raindrops are possible to fall all at once or sporadically. Or similarly, they may hit the surface of the pool randomly at different points. The waves' expanding circles are viewed as the growth front of the spherulites, while the impact points are considered the crystallite nuclei. Using probability derivations, the degree of crystallinity, X which is considered the volume fraction of the crystalline material,could be shown as the following:

$$1 - X = e^{-E} \quad (1)$$

the average number of all fronts in the system is represented by E. A suitable approximation of X ≈ E can be considered for the low degree of crystallinity. E in the exponent of Equation (1) for the bulk crystallisation possibly will be considered as the volume fraction or the volume of the crystalline materials, V_t and therefore,

$$1 - X = e^{-V_t} \quad (2)$$

In bulk crystallisation, Equation (2) has been extensively recognized and applied to describe crystallisation. This equation could be taken into account for sporadic or instantaneous nucleation and it could be shown as the following:

$$1 - X = e^{-Kt^n} \quad (3)$$

X represent the degree of solidification while K denotes the growth rate (min^{-1}) of the solidification, and n represent the Avrami exponent representing the nucleation’s nature and characterizes the crystal structure, and t is time (s) The Avrami exponent (n) has been frequently utilized in order to draw a distinction between different crystallisation mechanisms; moreover, its theoretical value (1, 2, 3, or 4) is defined by the crystal growth and nucleation’s nature. Rod-like crystals represent by n =1 corresponds to growth of instantaneous nuclei while, n=3or 4 indicates spherulitic growth from either instantaneous or sporadic nucleation as shown in the table 2.1.

Table 2.1: The Avrami Solidification Mechanism

Crystallization mechanism	N	Growth form
Spheres sporadic instantaneous	 4 3	 Three dimensional
Discs Sporadic Instantaneous	 3 2	 Two dimensional
Rods Sporadic Instantaneous	 2 1	 One dimensional

The present study uses the relative fat deposition to measure the degree of crystallinity (δ_r) being the deposition's mass fractions on the test tube wall divided by the initial mass of the wax-oil liquid:

$$\delta_r = \frac{\delta_t - \delta_0}{\delta_\infty - \delta_0} \quad (4)$$

Where δ_t represents the total deposition at time t (min), wt%, δ_0 the initial mass of fat content in the liquid, (g) and $\delta_\infty$the asymptotic or maximum deposition attained from the deposition curves when the quasi-steady state or the asymptotic condition has been accomplished (g). Replacing X by δ_r from Equation(4) can be linearized low twice where it can been seen in Equation(5).

$$\log\left(-\ln(1-x)\right) = \log K + n\log(t) \quad (5)$$

As a result, by plotting the log(t) versus the left side in Equation (5), the intersection K and the slope of the straight line can be found which is n is the slop that act as avrami exponent and K as y-intercept which is the growth rate of the crystallization. The Avrami coefficient is helpful in understanding the mechanism of phase transformation and its effect on both nucleation and growth.

2.8 Recrystallization

Recrystallization was defined by Fennema as any change in the number, size, shape, orientation or perfection of the crystals after completion of initial solidification. The basic mechanism of the recrystallization process is size-dependent equilibrium (melting temperature or solubility) documented by the Gibbs-Thomson effect. Small crystals, due to the small radius of curvature of the surface, are slightly more soluble or have a slightly lower melting point than larger crystals. Over time, these differences promote the disappearance of small crystals and growth of larger crystals. These changes generally occur without a change in volume of the crystalline phase, and are driven by the difference in thermodynamic equilibrium based on the size of the crystals. These crystals occur slowly at a constant temperature, but their presence increases with temperature swings as the phenomenon referred to as Melting-Recrystallization becomes dominant. When the temperature rises during a temperature cycle, the crystals melt or dissolve to maintain phase equilibrium. The small crystals, which are less stable, disappear first. When the temperature starts to decrease during the temperature cycle, the volume of the crystal phase increases, but only by growing and without the formation of new nuclei. The mass of small crystals that melted is redispersed among the larger crystals. As the average size of the crystals increases, the number of crystals decreases as a result of these thermodynamic effects. Thus, a dispersion of many small crystals tends to minimize the surface energy (and surface area) by recrystallization.

The final stage of crystallization in foods occurs during storage, and a population of crystals undergoes a recrystallization step, reaching a more broad equilibrium state. This phenomenon is of primary concern during storage of foods, and is responsible for changes to the texture of ice cream, fat bloom in chocolates and toppings and exudation of oil in products rich in fat. In lipid systems, the recrystallization process involves changes to the internal arrangement of the crystalline structure via polymorphic transformation

CHAPTER 3

METHODHOLOGY

From this chapter will describe the procedure of fat extraction method and solidification design base on difference parameter. The extraction method is conduct with et rendering method which compare the fat yield percentage of two sample use in difference time of extraction. Solidification of cow fat is determine by using two parametere which is effect of cold temperature and size of fat used. The solidifididification behaviour of cow fat anylysis is determined by using Avramic model of solidification kinetic.

3.1 Material and Equipment

material and equipment used in extraction and solidification process

Table 3.1: material and equipment used

Material	Equipment
Cow fat	Stove
Clean water	electronic balance
Falvan tube	knife
	pan
	cool water bath
	hot water bath

3.2 Sample Collection and Preparation

The cow meat is from the same type that were bought from the local market in town of jeli, kelantan. The cow meat is from frozen meat that have already package in weight of 900g each. the frozen cow meat is choosen because it have the same volume, type and more easy to handle. Cow meat was chop to reduse it size into small pieces. This process is occur to promotes good solvent contact as it provides a greater surface to volume ratio, enhances fat extraction resulting.

3.3: Fat Extraction Method

Weight 900 g of cow meat. Trim off cow meat into small pieces. Next, add water into the pot and start the heating process until it reach the boiling point which is (90°C-100°C). Put the trimmed meat into the pot and alow to extraction for 1 hour. The hot water containing fat were tapped off and a repeat of the above process carried out with fresh water for 1 hours for sample one. The process was repeated for the second sample of 900 g of cow meat for 30 min, then make the repetition process for 30 min with fresh water.

This process was repeated twice to increased the extraction result.The advantages of this method it recovery fat is better. This system uses heating, separation and cooling on a continuous basis and is regarded as an ideal purpose. The hot water that have been tapped off allow to cool in room temperature. At room temperature, after the fat had separated from water, then the fat can be collected. The fat is weight and the percentage of fat yield of the meat is determined using the formula in equation (6). Compare the percentage fat yield within sample 1 and sample 2.

$$\text{percentage of fat yield}(\%) = \frac{\text{weight of fat}}{\text{weight of sample}} \times 100 \qquad (6)$$

3.4. Solidification of Fat Method

Solidification of cow fat was determine by using two parameteres which were effect of cold temperature and size of fat used. The solidifididification behaviour of cow fat anylysis was determined by using Avramic model of solidification kinetic.

3.4.1: Effect of Cold Temperature

The effect of this parameter was conducted by using conventional method of solidification, which is chiling waterbath was used to obtain the crystallization temperature. And hot waterbath was constant at 70°C to melt the fat sample into liquid form. The percentage of solid or degree of solidification was measured were determined by recording the solidification change in temperature every 2 minutes. The fat sample were at a constant weight which is 28 g, the fat was cooled in chilling water bath at the difference temperature, which T= 10°C, 15°C, 20°C.The same type of Valcan tube is used in this experiment and keep constant to reduce error. Firstly, the 28 g of fat was melt at 70°C in the falcan tube to obtain liquid form. the sample was cooled at 10°C for 2 minutes and weight the fat that have not been solidified . record the percentage of fat solidification using the formula in equation(7).

$$\text{percentage of fat solidification} = \frac{\text{weight of liquid fat} - \text{weight of sample}}{\text{weight of sample}} \times 100 \quad (7)$$

From this equation, weight of solid fat can also benn determined. The above process was repeated for 4 min, 6min,8min and 10 min and determine the percentage of fat solidification to determined the curve of solidification and crystallization.

The process is repeated to next temperature which is 15°C and 20°C, the n value and growth rate is compare and determined for the three parameter by using Avramic exponent method.

3.4.1 Effect of Difference Weight of Fat

The effect of this parameter was conducted by using conventional method of solidification, which is chiling waterbath was used to obtain the crystallization temperature. And hot waterbath was constant at 70°C to melt the fat sample into liquid form. Difference weight of fat is become the variable which is, 10g, 20g, and 30g in a falcon tube. The same type of valcan tube was used to reduce error. The percentage of solid or degree of solidification was measured were determined by recording the solidification change in temperature every 2 minutes. Firstly, the 10g of fat was melt at 70°C in the valcan tube to obtain liquid form. the sample was cooled at 10°C for 2 minutes and weight the fat that have not been solidified . record the percentage of fat solidification using the formula.

$$\text{percentage of fat solidification} = \frac{\text{weight of liquid fat} - \text{weight of sample}}{\text{weight of sample}} \times 100 \quad (7)$$

From this equation, weight of solid fat can also benn determined. The above process was repeated for 4 min, 6min,8min and 10 min and determine the percentage of fat solidification to determined the curve of solidification and crystallization.

The process is repeated to next weight which is 20g and 30g, the n value and cryztallization growth rate is compare and determined for the three parameter by using Avramic exponent method.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Sample Preparation and Pretreatment

The meat was shredded to reduce it size with the measurement of size. The repetition is make to ensure high percentage of fat extraction. The yield of fat was recovered for sample 1 is 28 gram and for sample 2 is 23 gram after weighing. The fat was store in dry room (30°C-40°C), to prevent from damage caused by insect, bacterial and fungal attacks to maintaining it good quality for a long time.

4.2 Fat Extraction Yield

Figure 1 illustrated that the percentage of fat yields(g) of sample 1 and sample 2 that conduct difference time which is sample 1 (3.11%) and sample 2(2.55%) respectively. According to Cheng et al (2009) found that yield of abdominal fat tissue increased as the rendering temperature and time increased. The difference in the fat recovery rates in the different studies was probably because of the different raw materials such as skin or abdominal fat tissues. The same weight of samples was used to determine the percentage of fat yield (900g each).

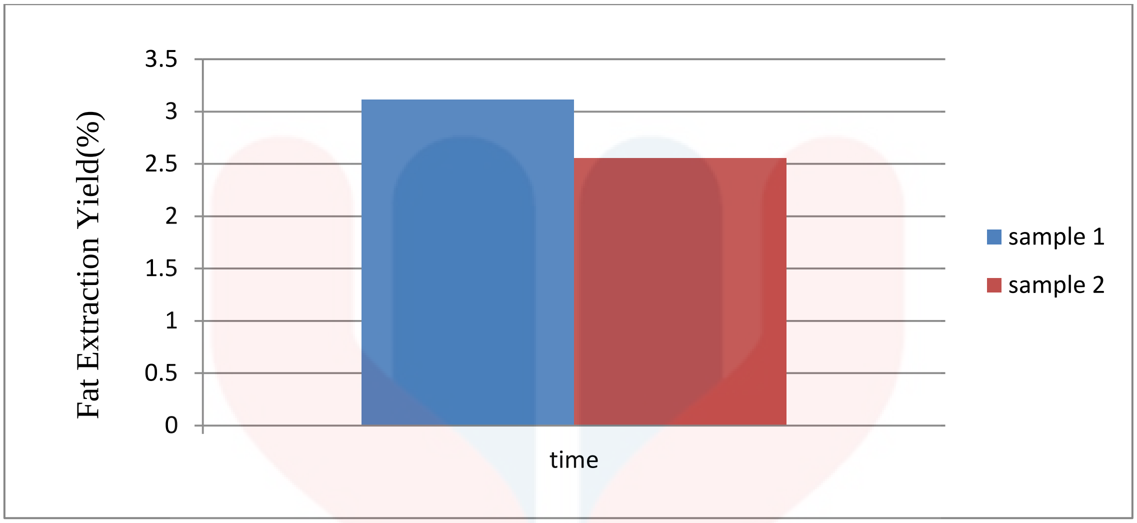

Figure4.1: Fat extraction yield(%) between sample 1 and 2.

4.3 Effect of Cold Temperature on Solidification Phenomenon of Fat

A crystal nucleus is the smallest crystal that can exist in a triglyceride mixture of a certain concentration and temperature. Aggregates of molecules smaller than a nucleus are called embryos and will redissolve if formed. A stable crystal will form only when the energy gain due to the heat of crystallization exceeds that required to overcome the surface energy required to increase the surface. The changing gradient of the slopes indicated the difference stage of crystal formation, initial production will start with nuclei, rapid growth and the decreasing of the crystallization rate because of the matured solid molecule is colliding with each other. Once a nucleus has formed, it starts to grow. The growth rate is proportional to the degree of super cooling which is lower temperature, and inversely proportional to the viscosity. Like the nucleation rate, the growth rate depends on the polymorph crystallized. The more stable the polymorph the less soluble it is and therefore the higher the growth rate. But, rapid cooling of a fat can leads to the initial formation of unstable crystals because nucleation is exponentially related to temperature. According to Ralph Timms (2005) Solid fat occupies about 90% of the volume of liquid fat The amount of contraction depends on the SFC of the fat (the amount of fat crystallized) and the polymorph it will be seen the more stable polymorphs are denser. According to Ismail et.al (2008) crystal formation can be divided into 2 difference phase which is the growth phase and the quasi steady state which at the quasi steady state, the crystal growth to equilibrium.

To study the effect of the cold temperature, the weight of the fat was kept constant at 28 g and the temperature of the fat was being set at the 70°C which is the fat is in liquid form. The same type of Falcon Tube is used to reduce error which is Vulcan tube. From the graph and table below, it seen that the solidification is occur at first 2 minute for all temperature but have difference degree of solidification curve. The percentage of the solidification for first 2 minute at 10c is the higer which is 49.28% follow by 15°C and 20°C which is 35.35% and 30.71% respectively. The solidification at 10°C is complete at 8minute, however for 15°C and 20°C is only 95.21% and 86.78% respectively and complete the solidification late than 10c which is at 10 minute of the solidification process.

Table 4.2: Percentage of Solidification on the Effect of Difference Temperature

Time/min	Temperature(°C)		
	10°C	15°C	20°C
2	49.2857%	35.3571%	31.7142%
4	68.5714%	63.9285%	59.2857%
6	85.7142	82.8571%	78.2142%
8	100	93.2142%	86.7857%
10		100	100

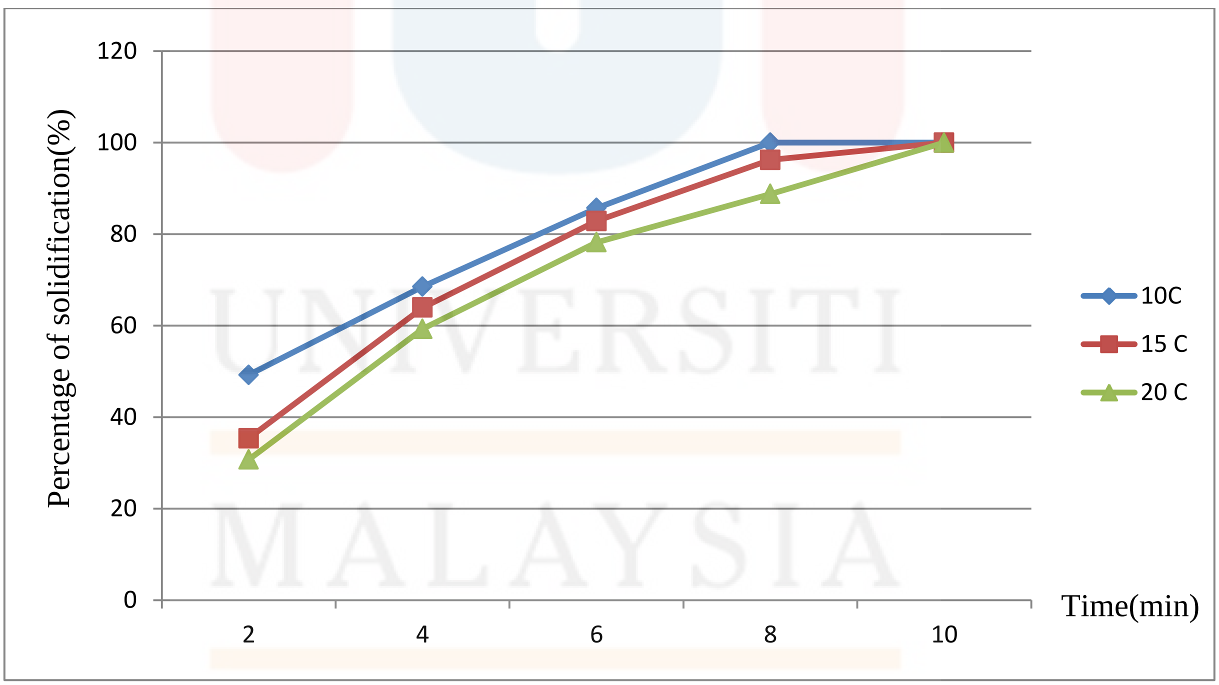

Figure4.2: Percentage of Solidification of Difference Temperature Again Time.

4.3.1 Avrami Mechanism of Cold Temperature

Figure 4.3 and show the crystallization kinetic when Avrami equation is apply into the information on degree of solidification over time using linear regression. The result show, the difference cold temperature had the same n value that had determined as n=3. This may conclude that the crystallization structure will be in form of spherulitic growth from instantaneous nucleation or disc-like that in sporadic nucleation form. As can see in table, the crystal rate growth, k increased hence the temperature decreased, which indicated that solidification occur rapidly at 10°C compare at 15°C and 20°C. This result fits according to Xiong and Adelaun (2016) that reported that the crystal rate growth k, increased with decreased the storage temperature which means the higher crystallization will occur at lower temperature.

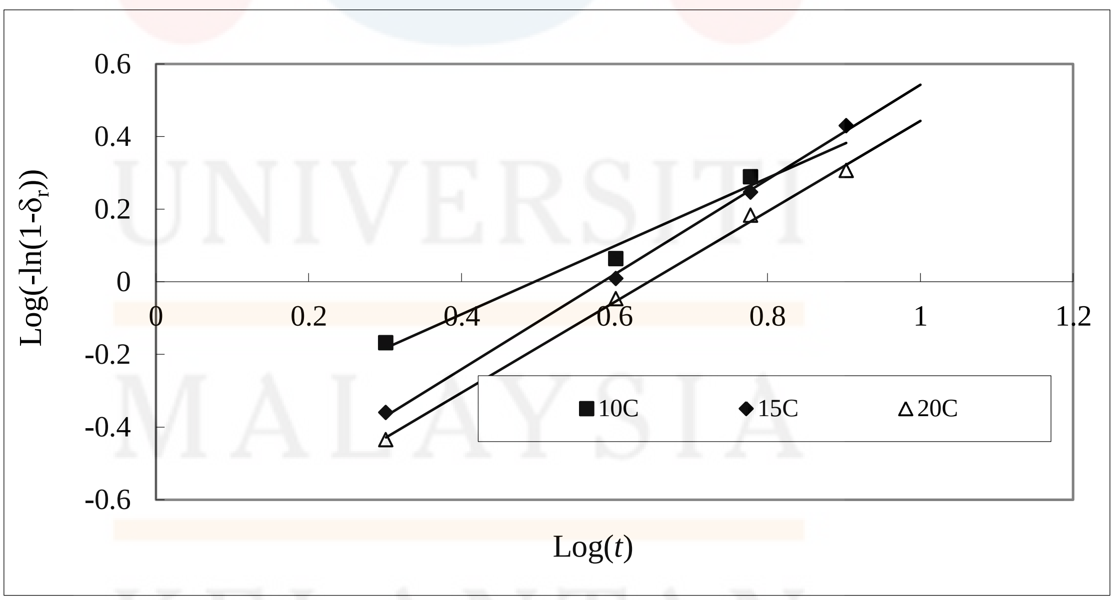

Figure4.2: avrami graph on the effect of difference cold temperature

Table 4.1: n Value and Growth Rate between Difference Cold Temperatures

Temperature(t)	Avrami exponent(n)	Growth rate, k(s^{-1})
10C	3.6785	0.020
15C	3.2145	0.018
20C	3.2096	0.017

4.4 Effect of Difference Mass of Fat on Solidification Phenomenon.

The percentage of solidification degree was measured in term of solid percentage which is formed within liquid fat over time. Cooling process was conducted at the constant temperature of 10°C which is the optimum temperature for crystal growth. Figure 4.3 show the progress over time which is the percentage of solid form increased until all liquid fat was achieved 100% solid. To determine the percentage of solidification, the initial temperature which is 70°C and final temperature which is 10°C was constant.

From Figure 4.4 it can be show that solidification was began at 2 minute for all difference mass of the fat. However, the smaller mass of the fat which is 10 g have the higher degree of the solidification percentage was recorded which is (61.8%) compare to other difference mass, 20 g and 30 g that have (51.61%) and (48.67%) respectively. According to Ismail et.al (2008) which is the smaller volume used have the higher degree of solidification and occur in short time because of the decreasing resistance caused by air bubbles and increased air space volume for arrangement of solid crystal structure to easily occur. Complete solidification for 10g of fat is faster which is at 4th minute of the solidification process than 15g and 20g of fat that only have 100% solidification at 6th min and 8th min respectively.

The volume of fat increased, the percentage of solidification decreased. From the graph 4.3 it seen that the rate of solidification that occur using 10g of fat is start at first 2 minute and achieved 100% solid at 4th minute. Compare to 20 g of fat, only (86.9%) of fat was been converted into solid form at 4 minute. However, the next reading at 6 minute, the percentage of solidification fat is about 99% to 100% which is completely in solid form. This show the solidification rate growth increased higher at low temperature and need short time to complete the solidification. This also apply to next 30 g of fat which is at the 4 minute only (68.43%) have been solidified, however, the growth rate increased rapidly at 6 minute which is (95.62%) and completed it solidification at 8 minute. This show the increment of solidification degree and rate is lower for 30g of fat compare to 10g and 20g.

Table 4.4: Percentage of Solidification on the Effect of Difference Mass of Fat

Time/Min	Mass (g)		
	10g	20g	30g
2	61.8679%	51.6122%	48.6735%
4	100%	86.9128%	68.4312%
6		100%	91.6278%
8			100%

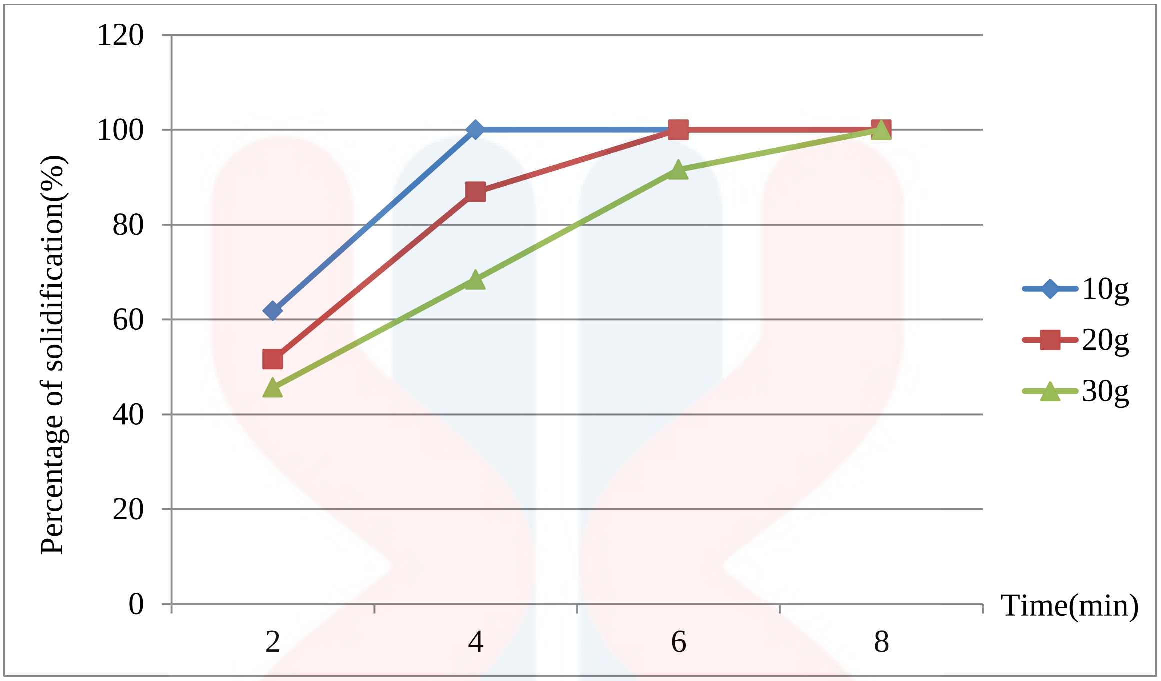

Figure4.3: The percentage of solidification of difference mass of fat used again time

4.4.1: Avrami Mechanism of Difference Mass of Fat.

From the table 4.4 and graph 4.4 can be observe the crystallization kinetic when Avrami equation is apply into the information on degree of solidification over time using linear regression. The result shows the n value for the lowest mass which is 10 g is 2.682 that the crystallization form may be in one or two dimensional which in Discs Sporadic or Rods instantaneous form. However, the 20g and 30g has same range of n value that consider the crystallization form of spherulitic growth from instantaneous nucleation or disc-like that in sporadic nucleation form.

The graph 4.4 indicates growth rate of the three difference mass. As the result, the lowest mass of the fat has the higher rate of crystallization growth. from this result, strongly agree to Ismail et. that reported that smaller volume used have the higher degree of solidification and occur in short time because of the decreasing resistance caused by air bubbles and increased air space volume for arrangement of solid crystal structure to easily occur.

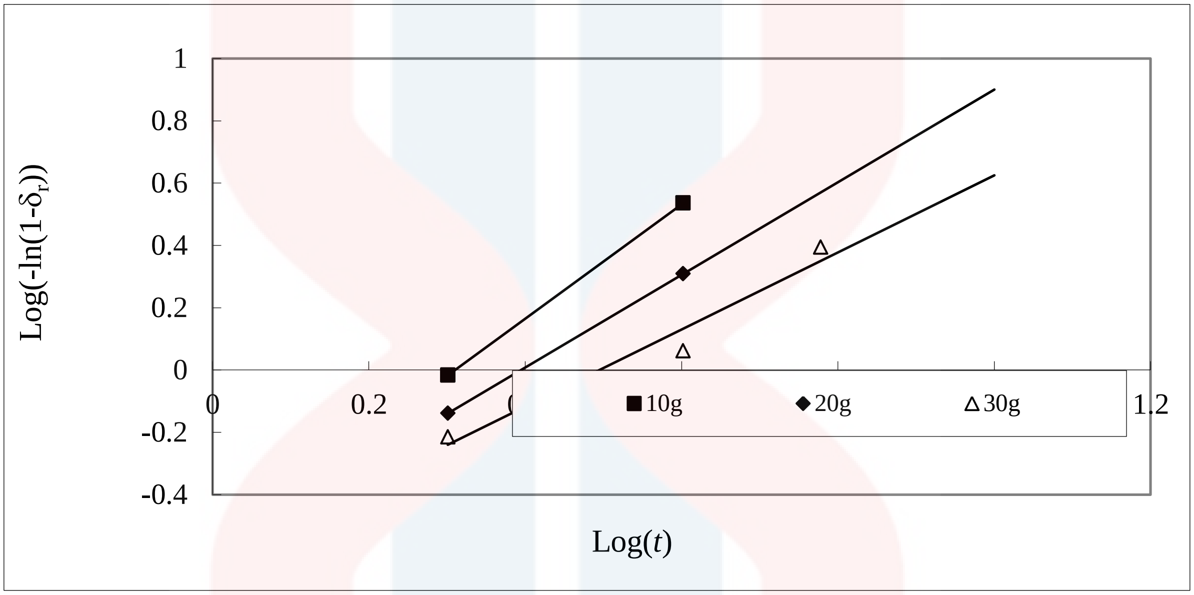

Figure 4.4: Avrami graph on the effect of difference mass

Table 4.4: Avrami exponent (n) and growth rate on difference mass of fat

Mass g	Avrami exponent(n)	Growth rate(s^{-1})
10g	2.682	0.038
20g	3.246	0.026
30g	3.819	0.021

CHAPTER 5

CONCLUSION AND RECOMMENDATION

This study involves the analysis of phase change of the fat and solidification kinetic study by evaluating the effect of fat mass used and the effect of difference cold temperature.by using gravimetric mechanism. Solidification kinetic study is analysis by using Avrami equation to determine the n value which is can be represented the crystallization form and it optimum growth rate of the fat according to the parameter used. The process of the solidification had 2 stage which is nucleation and crystallization development that occur simultaneously in solidification process. This study is to know the mechanism of the process which it can be very benefit in industry to commit with a quality product. The relation between growth characteristics, and the kinetics of phase transformation can be used for the control and optimization of the growth process. Moreover, the model proposed allows us to consider the process of three-dimensional growth as re-iterative building, layer by layer, in order to determine the important geometrical characteristics of a three-dimensional film, such as mean thickness, surface roughness, and roughness self-corielation function.

The crystallization analysis of fat based on the effect of difference cold temperature and difference of fat mass is achieved. In this cases, the lower the temperature, will influence the percentage of solidification which we can see the percentage of solid against time is higher on lowest temperature. However, when the fat was cooled in the difference cold temperature, have the same solidification mechanism which n=3, this show, the characteristic of the crystal may be in spherulitic growth from instantaneous nucleation or disc-like in form of sporadic nucleation. K represent the growth rate of the solidification that will be low in higher temperature. The difference mass of fat used also influence the growth rate, and degree of solidification which the low the mass used will be the higher in solidification and crystal growth. In conclusion, the fat crystallization were affected by temperature and mass which is the lower the temperature and mass the higher the crystallization occur, however, the crystallization form of the fat is may same which is spherulitic growth from instantaneous nucleation or disc-like that in sporadic nucleation form.

Next, in order to improve the case study of crystal solidification, the use of DCS will result more accurately on investigation the effect of the parameter used on avrami theory. Furthermore, the condition need to control which mean, the place of the solidification process must be in closed environment that not be affected the result of the solidification. As we know, in this cases the solidification is occur in laboratory that in open environment that will consider as hazard factor.

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APPENDICES

APPENDIX A

Table A.1: Flow Chart of Methodology of Wet Rendering and Solidification Analysis

Meat cow preparation and pretreatment

1. Reduce the size of cow meat of two sample and weight for 900gram each sample.

↓

Start the wet rendering process with the cow meat according to parameter

↓

Collection of fat and determined the percentage of fat yield of two sample

↓

Solidification study using gravimetric method

1. Effect of cold temperature
2. Effect of size

Table A.2: Fat Extraction Yield

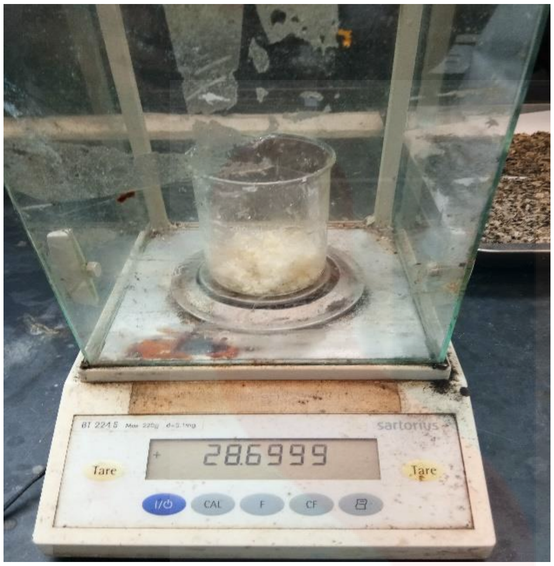
Sample	Heating duration	Fat yield(%) percentage	yield(g)
Sample 1 3.1111	2hours	28	
Sample2 2.5555	1hours	23	

APPENDIX B

Figure B.1: Two sample of cow meat which is 900 g each that used in the project

Figure B.2: Cow meat in cutting into smaller shape process

Figure B.3: Cow meat is weight



Sample A SAMPLE B

Figure B.4 and Figure B.5: The extraction fat of two sample is collected and weight.

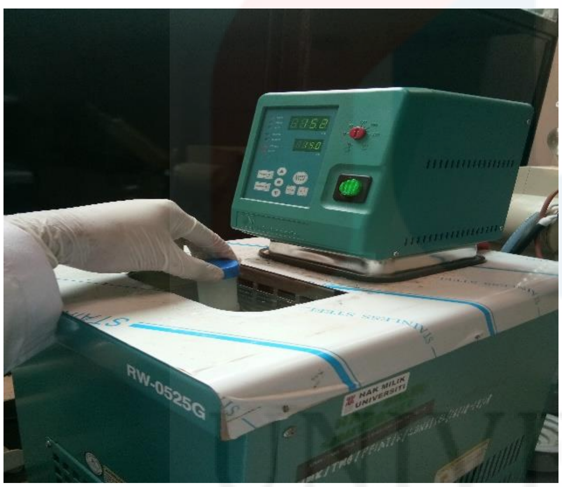


Figure B.5 : Using cold Water Bath to study the effect of Difference cold temperature

Figure B.6: using hot water bath to melt the fat in liquid form at 70°C.