UNIVERSITI PUTRA MALAYSIA

CRYOMECHANICAL FREEZING OF KEROPOK LEKOR

NOR AMAIZA MOHD. AMIN

CRYOMECHANICAL FREEZING OF KEROPOK LEKOR

By

NOR AMAIZA MOHD. AMIN

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, In Fulfilment of the Partial Requirement for the Degree Master of Science

March 2003

Dedicated to my beloved persons….

My Parents..

My Hubby..

for their loves and encouragements….

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirements for the degree of Master of Science

CRYOMECHANICAL FREEZING OF KEROPOK LEKOR

By

NOR AMAIZA MOHD. AMIN

March 2003

Chairman: **Associate Professor Mohd. Nordin Ibrahim, Ph.D.**

Faculty : **Engineering**

The freezing process of keropok lekor (original fish sausage) has been studied experimentally and by numerical simulation of unsteady heat transfer The viability of using combined freezing process (called cryomechanical freezing), consisting of a two consecutive processes, an in line cryogenic freezer (using liquid N_2 (LN)) followed by a continuous mechanical freezer as a preservation method was investigated The core temperature history of keropok lekor immersed into cryogenic liquids or placed in the air-blast freezer was monitered till it reached –20°C The total time taken was 16 minutes for cryomechanical freezing and 21 minutes for mechanical freezing, which is called the freezing time The changes in sensory parameters (color, flavor, texture and overall accepatability) of keropok lekor as a result of cryomechanical freezing were also evaluated Keropok lekor made by the mechanization method with 2 1 ratio of fish meat to flour was used and were in cylindrical or spherical form The product was first submerged for 30s in LN_2 and then immediately transferred to the air blast freezer for further freezing until the core temperature reached –20°C The product core temperature was measured over time using K-type thermocouple connected to data acquisition system The physical characteristics of keropok lekor were determined by measuring the changes in water content, crude protein, fat, carbohydrate and ash during freezing The product thermal conductivity, enthalpy and freezing point were obtained from standard composition model The heat transfer coefficients were determined from the thermal history of aluminium with dimensions similar to the treated keropok lekor The Numerical model proposes was based on Fourier equation for an infinite cylinder and a sphere given with variable product enthalpy and temperature dependent physical properties Fourty evenly distributed grid points was found to be adequate for obtaining a good reproducible results Calculated freezing times were comparable to data obtained by freezing a cylinder of gelatin, used as a model and with the initial data for keropok lekor frozen using both cryomechanical and mechanical freezer The total freezing time is reduced when the immersion time is increased The influence of the immersion time and the formation of the crust thickness were also investigated The Numerical model predicts that only a few seconds of immersion in the cryogenic liquid are enough to reduce the freezing time Moreover, the freezing time by cryomechanical freezing is reduced by about 27% compared to mechanical freezing The sensory evaluations were analyzed by Analysis of Variance (ANOVA) Panelist did not recognize any difference between the keropok lekor frozen by cryomechanical and mechanical freezer from the fresh one

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi sebahagian daripada keperluan untuk ijazah Master Sains

PROSES PENYEJUKBEKUAN GABUNGAN (KRIO-MEKANIKAL) KE ATAS KEROPOK LEKOR

Oleh

NOR AMAIZA MOHD. AMIN

March 2003

Pengerusi: **Prof. Madya Mohd. Nordin Ibrahim, Ph.D.**

Fakulti: **Kejuruteraan**

Proses penyejukbekuan keropok lekor dikaji secara eksperimen dan simulasi kaedah berangka untuk pemindahan haba yang tidak mantap Kesesuaian penggunaan kaedah penyejukbekuan gabungan (*cryomechanical freezing*) yang melibatkan dua langkah penyejukbekuan berturutan, penyejukbekuan secara langsung (menggunakan cecair nitrogen (LN)), diikuti dengan penyejukbekuan mekanikal sebagai kaedah pengawetan makanan telah dikaji Perubahan suhu di tengah keropok lekor semasa direndam di dalam cecair kriogenik dan proses penyejukbekuan didalam 'air-blast freezer' dicatit sehingga ia mencapai –20°C Jumlah masa yang diambil adalah 16 minit untuk proses penyejukbekuan gabungan dan 21 minit untuk proses penyejukbekuan mekanikal, dimana disebut sebagai masa penyejukbekuan Perubahan pada nilai deria yang berlaku (warna, rasa, tekstur dan penerimaan keseluruhan) juga dinilai Keropok lekor dengan nisbah 2 1 daging ikan kepada tepung yang disediakan melalui kaedah mekanikal digunakan dan adalah dalam bentuk silinder dan sfera Sampel tersebut direndam dahulu selama 30 saat didalam cecair nitrogen dan kemudiannya dimasukkan serta merta ke

dalam ‘air blast freezer’ untuk proses penyejukbekuan seterusnya sehingga suhu ditengah sampel mencapai $-20^{o}C$ Perubahan suhu ditengah keropok lekor dengan masa dicatit menggunakan penggandıng suhu jenis-K yang dipasang kepada sistem pengesan data Perubahan sifat fizikal keropok lekor ditentukan dengan mengambilkira perubahan kandungan air, protin kasar, lemak, karbohidrat dan abu semasa proses penyejukkan dijalankan Sifat terma seperti konduktiviti terma, entalpi dan takat beku ditentukan menggunakan kaedah standard berdasarkan kandungan bahan-bahan tersebut Pekali pemindahan haba ditentukan melalui kaedah perubahan sifat terma aluminium yang mempunyai dimensi yang sama seperti keropok lekor yang dikaji Model kaedah berangka yang digunakan melibatkan persamaan Fourier untuk silinder berbentuk fana dan sfera dengan nilai entalpi yang berubah serta perubahan sifat fizikalnya terhadap perubahan suhu Bilangan 40 titik kekisi yang sama digunakan dan adalah memadai untuk memberikan nilai hasil yang tepat Hasil penentuan masa penyejukbekuan berasaskan model menunjukkan perbandingan yang baik dengan hasil eksperimen untuk silinder gelatin, sebagai model makanan dan untuk data keropok lekor bagi kedua-dua kaedah penyejukbekuan secara gabungan (kriomekanikal) dan mekanikal Masa penyejukbekuan berkurangan apabila masa rendaman dalam cecair kriogenik ditambah Pengaruh masa rendaman dan pembentukkan kerak beku diambil kira Model yang dibina telah mengesahkan hanya beberapa saat rendaman dalam cecair kriogenik sudah mencukupi untuk memperolehi kerak beku dan mengurangkan masa penyejubekuan Tambahan pula, masa yang diperlukan dalam proses penyejukbekuan gabungan adalah lebih cepat iaitu 27% berbanding penyejukbekuan secara mekanikal sahaja Penilaian perubahan warna, rasa, tekstur dan penerimaan keseluruhan keatas keropok lekor dianalisis menggunakan ‘Analysis of Variances’ (ANOVA) Ahli panel tidak dapat mengesan sebarang perbezaan antara keropok lekor yang telah dibekukan melalui kaedah krio-mekanikal dan mekanikal berbanding keropok lekor yang asli

ACKNOWLEDGEMENT

I would like to express my gratitude to the members of my supervisory committee, Associate Professor Dr. Mohd. Nordin Ibrahim (Chairman), Dr. Sergei Yurevich Spotar and Mr. Dzulkifly Mat Hashim for their invaluable guidance, constructive comments and assistance during my study, without whose support and criticism I would not have been able to complete this thesis.

I am also deeply indebted to Mr Wissam N. Omar from Chemical and Environmental Engineering Department for his assistance during the conduct of the experiment. Not forgetting other lecturers in the Process and Food Engineering Department, clerical staffs and technicians and to many of my friends and colleagues for their help and encouragement.

My heartfelt gratitude and utmost love to my husband, Mat Salleh Bin Yamin for his support, encouragement and patience. For my son Muhamad Adam Haziq, who enliven my life. Last but not least, my million thanks to my parents, Mohd. Amin Bin Ismail and Masrelaah Bte Kromin, my brothers, sisters and family in laws for their continuing support. Thank you very much. And above all, to ALLAH, the most gracious and most merciful who made all things possible.

TABLE OF CONTENTS

		Page
DEDICATION		ii
ABSTRACT		iii
ABSTRAK		v
ACKNOWLEDGEMENTS		viii
APPROVAL SHEETS		ix
DECLARATION FORM		xi
LIST OF TABLES		xiv
LIST OF FIGURES		xv
NOTATIONS		xvi
CHAPTER		
1	**INTRODUCTION**	1
1.1	Overview	1
1.2	Keropok lekor	4
1.3	The Objectives of this Project	5
2	**LITERATURE REVIEW**	7
2.1	Freezing as a Preservation Method	7
2.2	Nature of the Freezing Process	8
2.3	Types of Food Freezing Systems	10
2.3.1	Air Blast Freezer	10
2.3.2	Plate Freezer	11
2.3.3	Cryogenic Freezer	11
2.4.4	Cryomechanical Freezing	12
2.4	Prediction of Freezing Time	15
2.4.1	Freezing Time Definition	15
2.4.2	Analytical Models	16
2.4.3	Numerical Models	19
2.4.3	By Experimentation	24
2.5	Thermo-physical Properties of Foods	25
2.5.1	Published Data	25
2.5.2	Measured Data	26
2.5.3	Predicted Data, Based on Composition Information	26
A	Density	27
B	Specific Heat Capacity	27
C	Enthalpy	28
D	Thermal Conductivity	28
2.6	Determination of Heat Transfer Coefficients	30
2.7	Quality Evaluation	33
2.8	Summary of Literature Review	34

3	**METHODOLOGY**	37
	3.1 Materials	37
	3.2 Methods	38
	3.2.1 Proximate Analysis	38
	3.2.2 Freezing Methods and Equipment	38
	3.3 Determination of Thermo-physical Properties of Keropok Lekor	39
	3.4 Determination of Heat Transfer Coefficients	40
	3.5 Modelling and Simulation of the Freezing Process	40
	3.5.1 Model Assumptions and Basic Equations	41
	3.6 Crust Thickness Formation	49
	3.7 Sensory Evaluation Studies	49
4	**RESULTS AND DISCUSSIONS**	51
	4.1 Proximate Analysis	51
	4.2 Thermo-physical Properties	52
	4.3 Heat Transfer Coefficients	55
	4.4 Freezing Time.	56
	4.4.1 Validation of the Model with Experimental Data	57
	4.4.1.1 Model Food-Cylinder of Gelatin	57
	4.4.1.2 Keropok Lekor	58
	4.4.2 Effect of Two of the Main Variables	62
	4.4.2.1 Influence of The Immersion Time *(t1)*	62
	4.4.2.2 Influence of The Transport Time *(t2)*	63
	4.5 Crust Thickness Formation	65
	4.6 Sensory Quality Evaluation	67
5	**CONCLUSIONS AND RECOMMENDATIONS**	68
	5.1 Conclusions	68
	5.2 Recommendations for Further Studies	69
	BIBLIOGRAPHY	71
	APPENDICES	
A	Proximate Analysis Procedure	77
B	Thermo-physical Properties of the Product (MathCAD Programme)	82
C	Heat Transfer Coefficients Estimation	89
D	Experimental Data (Original Data)	93
E	Enthalpy Curve for Model Food (*Macchi, 1995*)	95
F	Freezing Time Prediction Model	96
G	ANOVA Table for Sensory Evaluation of Keropok Lekor	106
	VITA	109

LIST OF TABLES

Tables		Page
2.1	Characteristics of the product and their freezing condition *(Agnelli & Mascheroni, 2001)*	22
2.2	Properties of pure components	29
3.1	Characteristic of the product and their freezing condition	40
4.1	The proximate analysis values	51
4.2	Two way ANOVA table for sensory evaluation of keropok lekor	67

LIST OF FIGURES

Figures		Page
2.1	Schematic cooling curve for water and an aqueous solution	9
3.1	Purpose-fitted pilot Cryomechanical Freezer	39
3.2	Design of the grid for calculation	43
3.3	Modelling algorithm	48
4.1	Typical change in thermo-properties with temperature for keropok lekor	54
4.3	Experimental *(Macchi, 1995)* and calculated temperature-time profiles for gelatin cylinder frozen in the cryomechanical freezer	59
4.4	Experimental *(Macchi, 1995)* and calculated temperature-time profiles for gelatin cylinder frozen in the mechanical freezer	59
4.5	Experimental and calculated temperature-time profiles evaluated at the center for keropok lekor *(D = 20mm, L = 100mm)* frozen in cryomechanical and mechanical freezer	60
4.6	Experimental and calculated temperature-time profiles evaluated at the center for keropok lekor of different diameter *(D = 20mm and 30mm, L = 100mm)* frozen in mechanical freezer	60
4.7	Experimental and calculated temperature-time profile evaluated at the center for keropok lekor sphere *(D = 20mm)* frozen in the mechanical freezer	62
4.8	Calculated temperature-time profile at the center for keropok lekor of cylinder *(D = 20mm, L = 100mm)* at different values of ***t1***	64
4.9	Temperature versus crust thickness formation for keropok lekor of cylinder *(D = 20mm, L = 100mm)*	66
4.10	Development of the frozen crust during product immersion in liquid nitrogen	66

NOTATION

A	Area
a	constant for radial geometry
Bi	Biot number
Cp	Specific heat capacity
$Cp(T)$	Specific heat capacity as a function of temperature
Cp_u	Specific heat capacity above freezing point (unfrozen)
Cp_f	Specific heat capacity below freezing point (frozen)
$\alpha(T)$	Thermal diffusivity as a function of temperature
d	Diameter of infinite cylinder or sphere, or thickness of an infinite slab
H	Enthalpy
h	Heat transfer coefficient
k	Thermal conductivity
k'	Thermal conductivity below freezing point (in Plank's equation)
$k(T)$	Thermal conductivity as a function of temperature
L	Latent heat of freezing
L'	Latent heat of freezing for water
N	Denotes number of nodes in r direction in finite difference schemes (grid points)
n	Denotes position of a nodes in r direction in finite difference schemes (space index)
P	Geometric factors in Plank-type equations
Pk	Plank's number
ρ	Density
ρ'	Density below freezing point (in Plank's equation)
$\rho(T)$	Density as a function of temperature
R	Geometric factors in Plank-type equation
r	Radius of cylinder or sphere
Ste	Stefan's number
T	Temperature
T_a	Ambient temperature
T_f	Freezing point
T_∞	External temperature (temperature of LN or cold air)
t_F	Freezing time
t	Time index
T_s	Surface temperature
V	Volume
X	Mass fraction
B	Bound water
C	Carbohydrate
LW	Water in liquid state
P	Protein
S	Solids-not-fat, solids other than fat
W	H_2O

CHAPTER 1

INTRODUCTION

1.1 Overview

Preservation of food is to prevent any microbial or enzymatic activity in food, so that its quality and freshness can be retained or even enhanced for market availability at all times. Development of food processing industry introduced many ways of keeping more perishable foods like meat, fish, vegetables and fruit in their best conditions for long periods. Freezing has a role to play as one of the simplest preservation technology.

According to *Tressler et. al (1968)*, low temperature storage reduces the rates of all chemical reactions causing deterioration, and retains the original quality of pre-cooked frozen foods. It represents a preservation process for food where the product temperature is decreased to a point below the temperature of the product as much as is economically feasible in an effort to reduce deterioration reaction rates within the product. The most documented evidence of freezing as a preservation process has appeared within the last 150 years *(Heldman, 1992)* and the process has become an integral part of food handling and distribution in most developed countries.

Although freezing has been a recognized preservation method for centuries, and the frozen food industry has been established since the beginning of 1970's *(Desrosier and Tressler, 1977)*, serious consideration of the mathematics of freezing has taken place only in the past decade or two Food engineers dealing with freezing are often faced with two major tasks estimating the refrigeration requirements for a freezer system and designing the necessary equipment and processes to accomplish rapid freezing It is recognized that the quality of frozen products is largely dependent on the rate of freezing In general, slow freezing (by using a conventional freezer) of food tissues results in formation of larger ice crystals in the extra-cellular spaces while rapid freezing (by direct contact to a cryogenic liquid or gas) produces small ice crystals distributed throughout the tissue *(Fennema, 1966)* Formation of these ice crystals, particularly the larger ones, damages the cellular structure and upon thawing, the food material will have a poorer texture *(Fennema and Powrie, 1964)* Therefore it is necessary to predict the freezing time or rate during freezing process and to know the temperature history of the material during frozen storage, in order to optimize the design of freezing equipment and to exercise control over the quality of the end product

Nowadays, the development of cryogenic followed by the mechanical freezing or known as combine freezing has been introduced The use of this system for food processing industry is actually increasingly growing and becoming more important Its use is not limited in the food processing only but also for medical purpose The process consists of a two-steps process, an in-line cryogenic freezer (using liquid N_2 (LN) or CO_2) combined with a continuous mechanical freezer (with cold air as heat transfer fluid produced by a conventional refrigeration equipment) The main problem that it is not yet widely used in the food processing industry is because of the cost of cryogenic liquid When used with the mechanical freezers, immersion, as well as cryogenic tunnel freezers can reduce 20 to 25 percent of the total freezing time in seconds *(http //www airproducts com/food/app_mechanical asp)* Thus, it can increase the freezing efficiency of the mechanical system Products are also prevented from sticking to the conveyer, thus improving the quality and appearance of the end product and increasing the life of the mechanical freezer's belt It is also observed that crust-freezing of products prior to full freezing, locks in moisture and prevents dehydration This leads to a reduction in weight loss and quality deterioration, thereby increasing yields while also enhancing the taste and appearance of the products *(Agnelli and Mascheroni, 2001)* The higher cost due to cryogenic liquid consumption is compensated by the attainment of a product with a lower weight loss and higher final quality or an overall better appearance It actually also offers an economical solution to increase freezing capacity With these advantages, the establishment of general and dependable models for the prediction of freezing time is essential for the design of freezer

In this research, the combination of cryogenic followed by mechanical freezing will be employed as a preservation method for keropok lekor To see if its use is justified, the heat transfer during the process was studied and the freezing time was predicted Besides, the quality aspects were also determined in order to examine the shelf life for enhancing and expanding its market Thus, the raw material of the sample used should be identified in order to predict the thermo-physical properties of the product, then the freezing time can be predicted in advance

1.2 Keropok Lekor (Fish Sausage)

In Malaysia, the popularity of fish sausage known as 'keropok lekor' as a snack food is confined to the east coast of peninsular Malaysia The production of fish sausage is seasonal *(Siaw, 1979)* Slicing the keropok lekor into a thin slice and dried under direct sunlight will produce another kind of fish snack called fish crackers (keropok hiris) Deep-frying of the dried fish crackers before serving will cause them to expand to form very crispy crackers with mild fishy aroma

Traditionally, keropok lekor or fish sausage consists of a mixture of fish meat, starches, water, salts, monosodium glutamate and sugar The ratio of fish meat and flour varied from place to place and from one individual maker to another The whole mixture is homogenized and the homogenized dough is then rolled into sausage form The sausages are then boiled for about 1 5 hour until cooked

Several types of fish are utilized and the common varieties are *Clupea leigaster (Ikan tamban bluru), Dussumieria hasselti Blkr. (Ikan tamban bular), Sardinella fimbriata Val. (Ikan tamban sisik), Chirocentrus dorab Forak (Ikan parang), Rastrelliger kanagurta Cuv. (Ikan kembong) and Scianena sp. (Ikan Gelama)*. Anyhow, *Yu (1992)* reported that keropok was acceptable up to a level of 60% substitution of *Rastrelliger kanagurta Cuv.* with *Oreochromis mossambicus* However above the 60% level, acceptability of keropok declined

Sago flour *(Metroxylon sago)* and/or tapioca flour *(Manihot utilissima)* constitute the starches frequently used

Cooked fish sausage, which is commonly sold in market, can be kept for only 1 day at room temperature The deterioration is due to the microbial activity, as the fish sausage made from fish and sago starch, is a good medium for microbial growth Research has been carried out in order to prolong the shelf life of keropok lekor According to *Embong (1988)*, keropok lekor can be packed and frozen by conventional air blast freezer and its shelf life could be extended to more than 7 weeks Because of lack of published material on the preservation of this product, it is assumed that there is no serious action to commercialize it Cryomechanical freezing perhaps could be a new preservation method for this product Although a conventional freezer could extend the shelf life of keropok lekor, study on the heat transfer process during the cryomechanical freezing is interesting and could be valuable since there has been no attempt along this line prior to this study In fact, the application could be extended to other product

1.3 The Objectives of this Project

The objective of this project can be divided into four as follows

- To study on cryomechanical freezing process as a preservation method of a common local food product, specifically fish sausage (Keropok Lekor)
- To present the application of the mathematical modeling to enable numerical simulation of the freezing process be carried out

- To validate the result of numerical simulation with the experimental data for a model food (cylinder of gelatin) and for keropok lekor obtained in a pilot prototype
- To examine the sensory quality parameters of keropok lekor as a result of the application of cryomechanical freezing
    1) Color change
    2) Flavor
    3) Texture firmness (chewingness)
    4) Overall acceptance

CHAPTER 2

LITERATURE REVIEW

This chapter presents an extensive review of freezing process generally. Background information on freezing time definition, analytical and numerical methods to predict the freezing time, nature of the freezing process, type of food freezing systems and thermo-physical properties of importance are also included.

2.1 Freezing as a Preservation Method

The frozen food industry is well established. It is widely accepted that freezing is the only large-scale method to bridge seasons as well as variations in supply and demand of raw materials such as meat, fish, butter, fruit and vegetables. *Syed Ziauddin et al (1993)* had studied on effects of freezing, thawing and frozen storage on microbial profiles of buffalo meat, found that preserving meat by freezing and frozen storage maintains the characteristics almost similar to fresh meat by preventing the microbial growth. *Santos (1995)* reported that freezing also is considered to be an excellent process for preserving the quality of fish for longer period of time (commercially up to 18 months or more). Freezing also makes it possible to move large quantities of food over geographical distances.

2.2 Nature of the Freezing Process

According to *Holdsworth (1968)*, the freezing process can be divided into three distinct phases. It can be defined as shown in the Figure 2.1, namely (1) the pre-cooling stage (2) the phase change stage and (3) the tempering stage

- *The pre-cooling stage* is the time period that elapses between the moment at which a product with a high temperature is subjected to a freezing process and the moment at which the water starts to crystallize.
- *The phase stage* is the period during which the temperature at the considered location is almost constant because the heat being extracted is causing the main part of the water to change phase into ice.
- *The tempering stage* is defined as the period during which the temperature is reduced from the temperature at which most of the freezable water has been converted to ice to the intended final temperature. The final temperature can be when the storage temperature is reached in any part of the product, including the thermal center, or the equalization temperature. The equalization temperature is the temperature, which is achieved under adiabatic conditions, without heat exchange with the environment.