



UNIVERSITI PUTRA MALAYSIA

***FORMULATION IMPROVEMENT, PROCESS DEVELOPMENT, AND
QUALITY ASSESSMENT OF CHILI SHRIMP PASTE***

BABAK SOBHI

FSTM 2012 28

**FORMULATION IMPROVEMENT, PROCESS DEVELOPMENT, AND
QUALITY ASSESSMENT OF CHILI SHRIMP PASTE**



**Thesis Submitted to the School of Graduate Studies, Universiti Putra
Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of
Philosophy**

May 2012



Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

**FORMULATION IMPROVEMENT, PROCESS DEVELOPMENT, AND
QUALITY ASSESSMENT OF CHILI SHRIMP PASTE**

By

BABAK SOBHI

May 2012

Chairman: Noranizan Mohd Adzahan, PhD

Faculty: Food Science and Technology

Chili shrimp paste (CSP), also known as *sambal belacan* in Malaysia, is well-liked in many Southeast Asian countries and is recognized as a national heritage food by the Malaysian Ministry of Cultural Arts and Heritage. This savory condiment is prepared by pounding fresh chilies with a small amount of fermented shrimp paste in stone mortar. No standard formulation or safety regulations have yet been developed for this product which could be used as a reference in food industries. It is usually prepared fresh, just before serving due to its perishable nature. In addition, pounding the chili in mortar to get the desired texture is a tiring job. This study was conducted to improve formulation, assess quality and develop processing methods for CSP. Results indicated that a typical product preferred by panelists contains approximately 70% moisture content, 27°Brix, 4.4% total salt content, 10% sucrose content, thick and chunky paste with lightness value (L) of 23, redness value (a) of more than 20 and yellowness value

(b) of 12. The instrumental methods developed included texture analysis using the back extrusion (sphere and cylinder) and the rheometer using an oscillation frequency sweep (vane in large cup) were found suitable to evaluate textural and rheological quality of CSP. Pastes prepared using an electrical stone mill with parallel plates (120 μm gap) had textural and rheological characteristics similar to the traditionally prepared products. Samples with acceptable textural properties were tested for its rheological behavior in the temperature range of 25-85 °C. Shear stress-shear rate data were adequately fitted to rheological models, i.e. Power law, Bingham plastic, Herschel-Bulkley, Casson and Mizrahi and Berk models, with the Casson being the most fitted ($R^2 = 0.981$) model. The tested paste showed non-Newtonian shear thinning behavior as the flow behavior index was less than one. Experimental yield stress values were different from those calculated using models, thus it is more accurate to determine yield stress from experiments. The defined flow behavior of CSP will allow engineers to design industrial process equipment. Thermal process (80 °C, 21.6 min) was effective and reliable in controlling microorganisms and deactivate the peroxidase as a deteriorative enzyme. In contrast, physicochemical and sensorial properties of CSP were negatively affected by heating. Electron beam irradiation (10 kGy) as an alternative non-thermal processing method was able to effectively control the microorganisms in CSP. Despite insignificant reduction in peroxidase activity and destructive effect on texture, irradiation was a better tool compared to thermal treatment in preserving phenolics, capsaicinoids, color and flavor of CSP. In conclusion, this study provided improved formulation and quality control method for textural analysis of CSP. In addition, preliminary data for scaling up of CSP in commercial setup was obtained. Irradiation was a better processing method than

thermal processing for preserving the quality of the product. Findings of this research will help future industrial implementation of commercialized CSP to meet consumer demands.



Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

**PENAMBAHBAIKAN FORMULASI, PEMBANGUNAN PROSES, DAN
PENILAIAN KUALITI SAMBAL BELACAN**

Oleh

BABAK SOBHI

Mei 2012

Pengerusi: Noranizan Mohd Adzahan, PhD

Fakulti: Sains dan Teknologi Makanan

Sambal belacan amat digemari oleh rakyat negara-negara di Asia Tenggara dan ianya telah diiktiraf sebagai makanan warisan kebangsaan oleh Kementerian Perpaduan, Kebudayaan, Kesenian dan Warisan Malaysia. Hidangan yang sedap ini disediakan dengan menumbuk cili merah segar dan sedikit belacan dengan menggunakan batu lesung. Masih belum ada lagi sebarang formulasi piawai atau peraturan keselamatan khas yang diwujudkan untuk sambal belacan, yang boleh dijadikan rujukan bagi industri makanan. Sambal belacan biasanya disediakan sejurus sebelum dihidangkan kerana ia cepat rosak. Tambahan pula, kerja menumbuk cili pada batu lesung amat memenatkan. Kajian ini dijalankan untuk menambahbaik formulasi, menilai kualiti dan membangunkan cara pemprosesan sambal belacan. Hasil kajian menunjukkan sambal belacan tipikal yang disukai oleh ahli-ahli panel mengandungi kira-kira 70% kandungan air, 27 °Brix, 4.4% kandungan garam, 10% kandungan sukrosa, sos sambal yang pekat dan kasar

dengan nilai Hunter L (kecerahan) sebanyak 23, nilai Hunter a (kemerahan) lebih daripada 20 dan nilai Hunter b (kekuningan) sebanyak 12. Penentuan tekstur dan reologi sambal belacan dilakukan menggunakan kaedah *back extrusion* (sfera dan silinder) dan reometer yang menggunakan frekuensi ayunan saku (ram dalam cawan besar). Sambal belacan yang disediakan dengan menggunakan batu kisar elektrik (plat selari, 120 m saiz jurang) didapati mempunyai tekstur dan ciri-ciri reologi yang sama dengan sambal belacan tradisional. Sampel sambal belacan yang mempunyai ciri-ciri tekstur yang boleh diterima, telah dikaji ciri-ciri reologinya pada suhu 25-85 °C. Data tekanan ricihan dan kadar ricihan telah dimasukkan ke dalam model-model reologi, iaitu *Power law*, *Bingham* plastik, *Herschel-Bulkley*, *Casson* dan model *Mizrahi* dan *Berk*. Model *Casson* telah mencapai nilai koefisien determinasi yang tertinggi ($R^2 = 0.981$). Sampel yang diuji menunjukkan kelakuan penipisan tegasan yang tidak Newtonian memandangkan indeks kelakuan alirannya kurang dari satu. Nilai tekanan yang diperoleh daripada eksperimen adalah berbeza daripada nilai tekanan yang diperoleh menggunakan model-model. Oleh itu, nilai tekanan yang diperoleh daripada eksperiment adalah lebih tepat. Penentuan kelakuan aliran sambal belacan membolehkan jurutera merekacipta peralatan pemprosesan industri. Proses terma (80 °C, 21,6 min) sangat berkesan dan boleh dipercayai dalam mengawal mikroorganisma dan menyahaktifkan enzim perosak peroksidase. Sebaliknya, sifat-sifat fizikokimia dan sensori sambal belacan akan terjejas oleh proses pemanasan. Penyinaran alur elektron (10 kGy) sebagai alternatif kepada kaedah pemprosesan bukan terma mampu mengawal mikroorganisma di dalam sambal belacan secara berkesan. Walaupun tidak memberi kesan penurunan aktiviti enzim peroksidase yang ketara dan mendatangkan kerosakan kepada

tekstur sambal belacan, penyinaran merupakan kaedah yang lebih baik berbanding dengan rawatan terma dalam mengekalkan kandungan fenolik, kapsaisinoids, warna dan rasa sambal belacan. Kesimpulannya, kajian ini menghasilkan formula yang telah ditambahbaik dan kaedah kawalan mutu bagi analisis tekstur sambal belacan. Di samping itu, data awal bagi penskalaan sambal belacan komersial telah diperolehi. Radiasi merupakan kaedah pemprosesan yang lebih baik dari pemprosesan terma dari segi memelihara kualiti produk. Hasil penyelidikan ini akan membantu perlaksanaan industri sambal belacan secara komersial bagi memenuhi permintaan pelanggan.

ACKNOWLEDGEMENTS

There has been a lot of support for the present study, of which the majority has come from professional, knowledgeable and experienced individuals from the Faculty of Food Science and Technology. Many people I came to know through professional contact have become true friends, which I cannot express my word enough for their valuable friendship.

Firstly, I would like to gratefully acknowledge my supervisor, Dr. Noranizan Mohd Adzahan, whose encouragement, guidance and support from the initial to the final level enabled me to develop the subject.

My sincere appreciation also goes to the other members of the Supervisory Committee: Associate Professor Dr. Muhammad Shahrim Abdul Karim, Professor Dr. Jamilah Bakar, Professor Dr. Russly Abdul Rahman and Dr. Zulkafli Ghazali.

Appreciation is extended to Universiti Putra Malaysia for supporting this research through the Research University Grant Scheme (RUGS 91953 and 91027).

Finally, I would like to express a special note of appreciation to my wife, my dear parents for their help, advice and support throughout the duration of the project.

I certify that a Thesis Examination Committee has met on 02 May 2012 to conduct the final examination of Babak Sobhi on his thesis entitled "Formulation Improvement, Process Development, and Quality Assessment of Chili Shrimp Paste" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

Members of the Thesis Examination Committee were as follows:

Sharifah Kharidah binti Syed Muhamad, PhD

Associate Professor

Faculty of Food Science and Technology

Universiti Putra Malaysia

(Chairman)

Badlishah Sham bin Baharin

Associate Professor

Faculty of Food Science and Technology

Universiti Putra Malaysia

(Internal Examiner)

Lasekan Olusegun Olaniyi, PhD

Associate Professor

Faculty of Food Science and Technology

Universiti Putra Malaysia

(Internal Examiner)

Olga Martin-Belloso, PhD

Senior Lecturer

University of Lleida

Spain

(External Examiner)

SEOW HENG FONG, PhD
Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Doctor of Philosophy. The members of the supervisory committee were as follows:

Noranizan Mohd Adzahan, PhD
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Chairperson)

Jamilah Bakar, PhD
Professor
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Member)

Russly Abdul Rahman, PhD
Professor
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Member)

Muhammad Shahrim Abdul Karim, PhD
Associate Professor
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Member)

Zulkafli Ghazali, PhD
Radiation Processing Division
Malaysian Nuclear Agency
(Member)

BUJANG BIN KIM HUAT, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

DECLARATION

I declare that the thesis is my original work except for quotations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.



BABAK SOBHI

Date: 02 May 2012

TABLE OF CONTENT

| | Page |
|---|-------------|
| DEDICATION | ii |
| ABSTRACT | iii |
| ABSTRAK | vi |
| ACKNOWLEDGEMENTS | ix |
| APPROVAL | x |
| DECLARATION | xii |
| LIST OF TABLES | xvii |
| LIST OF FIGURES | xix |
| LIST OF ABBREVIATIONS | xxi |
| CHAPTER | |
| 1 INTRODUCTION | 1 |
| 2 LITERATURE REVIEW | 5 |
| 2.1 Chili Shrimp Paste | 5 |
| 2.1.1 Chili Paste (<i>Sambal</i>) | 5 |
| 2.1.2 Shrimp Paste (<i>Belacan</i>) | 6 |
| 2.2 Product Development and Sensory Evaluation | 8 |
| 2.3 Rheological Models | 10 |
| 2.4 Thermal Processing | 15 |
| 2.4.1 Thermal Processing and pH | 15 |
| 2.4.2 Hot Fill Process | 16 |
| 2.4.3 Thermal Process Optimization | 17 |
| 2.5 Thermal Processing of Chili Paste | 18 |
| 2.6 Non- Thermal Processing | 19 |
| 2.6.1 Irradiation | 20 |
| 2.6.1.1 Gamma-Ray | 21 |
| 2.6.1.2 X-Ray | 22 |
| 2.6.1.3 Electron Beam | 23 |
| 2.6.1.4 Advantages of Electron Beam | 27 |
| 2.6.1.5 Dose of Irradiation | 29 |
| 2.6.1.6 Effect of Irradiation on Microorganisms | 30 |
| 2.6.1.7 Effect of Irradiation on Food Components | 31 |
| 3 PHYSICOCHEMICAL AND SENSORY PROPERTIES OF CHILI SHRIMP PASTE | 32 |
| 3.1 Introduction | 32 |
| 3.2 Materials and Methods | 33 |
| 3.2.1 Sample Collection | 33 |
| 3.2.2 Color Measurement | 34 |
| 3.2.3 Determination of Total Soluble Solids and pH | 35 |
| 3.2.4 Determination of Moisture, Salt and Sucrose Contents | 35 |
| 3.2.5 Sensory Analysis | 36 |
| 3.2.6 Statistical Analysis | 36 |

| | | |
|----------|--|-----------|
| 3.3 | Results and Discussion | 37 |
| 3.3.1 | Color | 37 |
| 3.3.2 | Physicochemical Properties | 38 |
| 3.3.3 | Sensory Analysis | 40 |
| 3.4 | Conclusions | 43 |
| 4 | TEXTURAL AND SENSORIAL PROPERTIES OF CHILI SHRIMP PASTE | 44 |
| 4.1 | Introduction | 44 |
| 4.2 | Materials and Methods | 45 |
| 4.2.1 | Sample Source and Formulation | 45 |
| 4.2.2 | Sensory Analysis | 47 |
| 4.2.3 | Oscillation Frequency Sweep | 47 |
| 4.2.4 | Textural Profile | 48 |
| 4.2.5 | Statistical Analysis | 50 |
| 4.3 | Results and Discussion | 51 |
| 4.3.1 | Consumer Acceptability of Chili Shrimp Paste Based on Textural and Viscosity Characteristics | 51 |
| 4.3.2 | Rheological Characteristics of Chili Shrimp Paste | 53 |
| 4.3.3 | Textural Characteristics of Chili Shrimp Paste Measured Mechanically | 57 |
| 4.4 | Conclusions | 61 |
| 5 | RHEOLOGICAL BEHAVIOUR OF CHILI SHRIMP PASTE PRODUCED MECHANICALLY | 63 |
| 5.1 | Introduction | 63 |
| 5.2 | Materials and Methods | 65 |
| 5.2.1 | Sample Preparation | 65 |
| 5.2.2 | Textural Analysis | 66 |
| 5.2.3 | Rheological Experiment | 67 |
| 5.2.4 | Rheological Models | 68 |
| 5.3 | Results and Discussion | 70 |
| 5.3.1 | Coarseness of Chili Shrimp Paste | 70 |
| 5.3.2 | Shear Stress-Shear Rate Model | 73 |
| 5.3.3 | Yield Stress | 77 |
| 5.3.4 | Apparent Viscosity and Flow Consistency Index | 78 |
| 5.4 | Conclusions | 83 |
| 6 | OPTIMIZATION OF THERMAL PROCESS CONDITIONS FOR CHILI SHRIMP PASTE | 84 |
| 6.1 | Introduction | 84 |
| 6.2 | Materials and Methods | 86 |
| 6.2.1 | Sample Preparation | 86 |
| 6.2.2 | Thermal Treatment | 87 |
| 6.2.3 | Color Measurement | 88 |
| 6.2.4 | Peroxidase Assay | 88 |
| 6.2.5 | Total Phenolic Content | 89 |

| | | |
|----------|--|------------|
| 6.2.6 | Capsaicinoids Content | 89 |
| 6.2.7 | Microbial Analysis | 90 |
| 6.2.8 | Statistical Design and Analysis | 91 |
| 6.2.9 | Optimization and Validation Procedures | 94 |
| 6.2.10 | Sensory Analysis | 94 |
| 6.3 | Results and Discussion | 95 |
| 6.3.1 | Statistical Analysis | 95 |
| 6.3.2 | Effect of Thermal Treatment on Color | 99 |
| 6.3.3 | Effect of Thermal Treatment on Peroxidase Activity | 101 |
| 6.3.4 | Effect of Thermal Treatment on Total Phenolic Content | 102 |
| 6.3.5 | Effect of Thermal Treatment on Capsaicinoids Content | 104 |
| 6.3.6 | Effect of Thermal Treatment on Microbial Count | 106 |
| 6.3.7 | Optimization Procedure | 108 |
| 6.3.8 | Validation of the Final Reduced Model | 109 |
| 6.3.9 | Sensory Acceptability of Thermally Processed Chili Shrimp Paste at the Optimized Condition | 111 |
| 6.4 | Conclusions | 112 |
| 7 | A COMPARISON BETWEEN THE EFFECTS OF IRRADIATION AND THERMAL PROCESSING ON QUALITY OF CHILI SHRIMP PASTE | 113 |
| 7.1 | Introduction | 113 |
| 7.2 | Materials and Methods | 115 |
| 7.2.1 | Sample Preparation | 115 |
| 7.2.2 | Non-Thermal Treatment | 116 |
| 7.2.3 | Thermal Treatment | 117 |
| 7.2.4 | Microbial Analysis | 117 |
| 7.2.5 | Color Measurement | 117 |
| 7.2.6 | Peroxidase Assay | 117 |
| 7.2.7 | Total Phenolic Content | 117 |
| 7.2.8 | Capsaicinoids Content | 118 |
| 7.2.9 | Rheological Analysis | 118 |
| 7.2.10 | Textural Analysis | 118 |
| 7.2.11 | Volatiles Analysis | 119 |
| 7.2.12 | Statistical Analysis | 121 |
| 7.3 | Results and Discussion | 121 |
| 7.3.1 | Irradiation Dose | 121 |
| 7.3.2 | Effect of Irradiation and Thermal Process on Color | 125 |
| 7.3.3 | Effect of Irradiation and Thermal Process on Peroxidase Activity | 127 |
| 7.3.4 | Effect of Irradiation and Thermal Process on Total Phenolic Content | 128 |
| 7.3.5 | Effect of Irradiation and Thermal Process on Capsaicinoids Content | 129 |
| 7.3.6 | Effect of Irradiation and Thermal Process on Rheological and Textural Properties | 131 |
| 7.3.7 | Effect of Irradiation and Thermal Process on Volatile Compounds | 133 |
| 7.4 | Conclusions | 145 |

SUMMARY, CONCLUSIONS AND RECOMMENDATIONS FOR FURTHER RESEARCH

| | |
|---|-----|
| Summary | 146 |
| Conclusions | 149 |
| Recommendations for Further Research | 150 |

REFERENCES

151

APPENDICES

169

BIODATA OF STUDENT

178

LIST OF PUBLICATIONS

179

PROCEEDING

180

