UNIVERSITI PUTRA MALAYSIA

PHYSICO - CHEMICAL, THERMAL AND RHEOLOGICAL PROPERTIES OF VARIOUS FISH PROTEIN - SAGO STARCH FORMULAS IN KEROPOK LEKOR

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By

MOHAMED ABD ELGADIR MOHAMED SAEED

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

September 2010

DEDICATED TO MY PARENTS,
MY BROTHERS,
WIFE, KIDS AND FRIENDS

Abstract of thesis submitted to the Senate of Universiti Putra Malaysia in Fulfillment of the requirement for the degree of Doctor of Philosophy

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Chairman: Professor Jamilah Bakar, PhD

Faculty: Food Science and Technology

This study was conducted to investigate the interaction between fish protein and sago starch using *keropok lekor* as a model with the following objectives: to study the thermal and physical properties of the model, to optimize the contents used in the model based on their thermal and rheological properties, to study the effects of sodium chloride and sucrose on transition temperatures of gelatinization and to investigate the effect of sodium chloride and sucrose on cooking time of the model. The ingredients used in preparing the formulations were red tilapia (*Oreochromis spp*.) minced fish, sago starch (*Metroxylon sagu*), and water in the ranges of 20 - 50, 10 - 40 and 10 - 35, respectively. A two-level factorial design was carried out to obtain the experimental formulations of the model. Differential scanning

calorimetry (DSC), texture profile analysis (TPA), sensory evaluation and the changes in thermophysical properties such as thermal conductivity (k), specific heat capacity (C_p), density (ρ), thermal diffusivity (α) and the effects of sodium chloride and sucrose concentrations on the cooking time of the optimized fish protein-sago starch model were conducted. The onset temperatures (T_o) of the different formulations of the fish protein - sago starch model varied from 60.9 ± 0.5 - 80.8 ± 0.4°C with an average value of 70.9 ± 0.5°C. The formulations had peak temperatures of gelatinization that varied from 65.5 ± 0.1 - 86.0 ± 0.4°C with average value of 75.8 ± 0.3°C. Increasing the ratios of the minced fish in the formulations was found to be the most significant factor affecting the values of T_o and T_p. The highest hardness value 50.9 N was obtained in the sample formulated with 5: 4: 1 minced fish: sago starch: water, respectively followed by 45.0 N which was obtained in the sample formulated with the ratios of 3.5 minced fish 4 sago starch: 2.25 water. The sensory evaluation study showed that the panelists preferred the model formulated with 2: 1: 1 minced fish: sago starch: water followed by 3.5: 2.5: 2.25 of the components, respectively. The optimum formulation of the model was highly dependant on the ratios of fish protein: sago starch. The addition of NaCl to the optimized formulation resulted in the shifting of the T_o from 82.2 ± 0.9°C to 98.2 ± 0.1°C, T_p from 94.8 ± 0.2°C to 106.2 ± 0.3°C and T_c from 108.0 ± 0.9°C to 116.3 ± 0.2°C. However, the addition of sucrose shifted T_o from 82.2 ± 0.9°C to 111.4 ± 0.2°C, T_p from 94.8 ± 0.2°C to 118.2 ± 0.2°C and T_c from 108.0 ± 0.9°C to 119.3 ± 0.3°C. During frying an increase in the thermal conductivity (k), specific heat capacity (c_p) and thermal diffusivity (α) values was observed. The k value of the model increased dramatically from 0.418 to 0.584 $W/m^2.^oC$ in the control. The calculated k values confirmed that the NaCl and sucrose caused an increase in k values of the model from 0.419

to 0.585 $W/m^2.^oC$ for NaCl and from 0.423 to 0.588 $W/m^2.^oC$ for sucrose treated ones. The calculated values of thermal diffusivities of the model varied from 1.19 x 10^{-7} to 1.51 x 10^{-7} m^2 s^{-1} during deep - fat frying. In summary, it can be concluded that the thermal properties of the model were highly dependant on the ratios of fish protein, sago starch and water used. The optimum formulation was obtained in the model formulated with 2: 1.01: 1 minced fish: sago starch: water, respectively. The shifts in the transition temperatures values of T_o, T_p and T_c caused by NaCl were 16, 11.4 and 8.3°C, respectively. However, sucrose shifted the transition temperatures values of T_o, T_p and T_c to 29.2, 23.4 and 11.3°C, respectively. Sucrose was found to be more effective in increasing the onset and conclusion temperature of gelation of the model. The actual cooking time of the control sample of the model was 180 s and the predicted cooking time of the model according to Ansari's equation was 195 s. The model with either added sucrose or NaCl needed more time to be cooked.

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

SIFAT- SIFAT FISIKA - KIMIA, TERMA DAN RHEOLOGI BAGI BERBAGAI FORMULASI PROTEIN IKAN - KANJI SAGU DI DALAM *KEROPOK LEKOR*

Oleh

MOHAMED ABD ELGADIR MOHAMED SAEED

September 2010

Pengerusi: Professor Jamilah Bakar, PhD

Fakulti: Sains dan Teknologi Makanan

Kajian ini dijalankan untuk menyiasat interaksi antara protein ikan dan kanji sagu dengan menggunakan keropok lekor sebagai model dan mempunyai objektif - objektif berikut: sifat-sifat terma dan fizikal sistem, pengoptimuman kandungan yang digunakan dalam model berdasarkan sifat reologi, kesan penambahan bahan (natrium klorida dan sukrosa) terhadap suhu peralihan pengelatinan, dan jangkaan masa memasak akibat dipengaruhi oleh penambahan bahan (NaCl dan sukrosa) dikaji. Nisbah bahan - bahan yang digunakan untuk menyediakan sampel adalah ikan tilapia merah (*Oreochromis* spp.) yang dicincang (MF), kanji sagu (*Metroxylon sagu*) (SS) dan air (W), masing-masing di dalam lingkungan 20-50, 10 - 40 dan 10.35. Rekabentuk faktorial dua - peringkat telah digunakan untuk mendapatkan formulasi ujikaji. Teknik kalorimetri pergimbasan pembeza (DSC), analisis tekstur profil

(TPA), penilaian sensori dan perubahan sifat termofizikal seperti terma konduktivi (*k*), kapasiti haba spesifik (C_p), ketumpatan (ρ), difusiviti terma (α) dan kesan kepekatan natrium klorida dan sukrosa ke atas jangkaan masa memasak model protein ikan - kanji sagu yang dioptimakan telah dijalankan. Suhu awal gelatinasi (T_o) bervariasi dalam lingkungan 60.9 ± 0.5 - 80.8 ± 0.4°C dalam formulasi protein ikan - kanji sagu yang berlainan dengan nilai purata 70.9 ± 0.5°C. Formulasi - formulasi tersebut mempunyai suhu puncak gelatinasi yang bervariasi dalam lingkungan 65.5 ± 0.1 - 86.0 ± 0.4°C dengan nilai purata 75.8 ± 0.3°C. Peningkatan nisbah ikan yang dicincang dalam formulasi ditemui sebagai faktor signifikasi yang menjejaskan nilai T_o dan T_p. Nilai kekerasan tertinggi iaitu 50.9 N dicapai dalam sampel berformulasi 5:4:1, masing-masing ialah ikan dicincang: kanji sago: air, diikuti 45.0 N di mana ia dicapai oleh sampel berformulasi dengan nisbah 3.5 ikan yang dicincang: 4 kanji sago: 2.25 air. Kajian penilaian sensori menunjukkan bahawa ahli panel lebih suka model yang diformulakan dengan 2: 1: 1 ikan yang dicincang: kanji sago: air diikuti 3.5: 2.5: 2.25. Formulasi optima model sangat bergantung kepada nisbah campuran protein ikan - kanji sagu dan diperolehi dalam model berformula 2: 1.01 : 1. Penambahan NaCl ke atas formula optima menyebabkan pengalihan T_o daripada 82.2 ± 0.9 kepada 98.2 ± 0.1, T_p daripada 94.8 ± 0.2 kepada 106.2 ± 0.3 dan T_c daripada 108.0 ± 0.9 kepada 116.3 ± 0.2. Namun, penambahan sukrosa mengalihkan T_o daripada 82.2 ± 0.9 kepada 111.4 ± 0.2, T_p daripada 94.8 ± 0.2 kepada 118.2 ± 0.2 dan T_c daripada 108.0 ± 0.9 kepada 119.3 ± 0.3. Sukrosa ditemui lebih berkesan dalam meningkatkan suhu awal dan akhir gelatinasi model. Semasa penggorengan, peningkatan terma konduktivi (*k*), kapasiti haba spesifik (C_p), ketumpatan (ρ), dan difusiviti terma (α) diperhatikan. Nilai k model meningkat dengan dramatik daripada 0.418 kepada 0.584 W/m^2 °C dalam kawalan. Nilai k yang dikira menyakinkan bahawa NaCl dan sukrosa menyebabkan peningkatan dalam nilai k daripada 0.419 kepada 0.585 W/m^{2} ^{o}C untuk model yang diperlakukan oleh NaCl dan daripada 0.423 kepada 0.588 W/m^{2}.^{o}C untuk model yang diperlakukan oleh sukrosa. Nilai difusiviti terma model yang dikira bervariasi daripada 1.19×10^{-7} kepada 1.51×10^{-7} $m^2\ s^{-1}$ semasa penggorengan lemak dalam. Sebagai rumusan, dapat disimpulkan sifat-sifat terma model sangat bergantung kepada nisbah protein ikan, kanji sagu dan air yang digunakan. Formulasi optima telah didapati daripada model berformulasi 2: 1.01: 1 ikan yang dicincang: kanji sagu: air. Pertukaran nilai suhu peralihan T_o, T_p dan T_c disebabkan oleh NaCl adalah 16, 11.4 dan 8.3^{o}C. Bagaimanapun, sukrosa telah menukar nilai-nilai suhu peralihan masing - masing, T_o, T_p dan T_c kepada 29.2, 23.4 dan 11.3^{o}C. Sukrosa didapati lebih efektif dalam meningkatkan suhu awal dan kesimpulan gelatinasi model tersebut. Jangka masa memasak sebenar untuk sampel kawalan model adalah 180 saat dan jangka masa jangkaan model mengikuti persamaan Ansari adalah 195 saat. Model yang ditambah dengan sama ada sukrosa atau NaCl memerlukan lebih masa untuk dimasak.

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I certify that an Examination Committee has met on 30/09/2010 to conduct the final examination of Mohamed Abd Elgadir Mohamed Saeed on his Ph.D. thesis entitled "PHYSICO - CHEMICAL, THERMAL AND RHEOLOGICAL PROPERTIES OF VARIOUS FISH PROTEIN - SAGO STARCH FORMULAS IN *KEROPOK LEKOR*" in accordance with Univesrsiti Pertanian Malaysia Doctor of philosophy Act 1980 and Universiti Pertanian Malaysia Doctor of philosophy Regulations 1981. The committee recommends that the student be awarded the Doctor of philosophy.

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DECLARATION

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

MOHAMED ABD EGADIR MOHAMED SAEED

Date: 30 September

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

MF	Minced fish
SS	Sago starch
W	Water
T_o	Onset temperature of gelatinization
T_p	Peak temperature of gelatinization
T_c	Conclusion temperature of gelatinization
G'	Storage modulus
G''	Loss modulus
RSM	Response surface methodology
DSC	Differential scanning calorimetry
ΔH	Enthalpy
J/g	Joule per gram
Pa	Pascal
kPa	Kilo Pascal
NaCl	Sodium chloride
3D	Three dimensional plots
R^2	Regression coefficient
α	Thermal diffusivity
c_p	Specific heat capacity
ρ	density
k	thermal conductivity

t	time
T	temperature
s	second

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