UNIVERSITI PUTRA MALAYSIA

STUDIES ON STARCH-SOLUTES INTERACTION AND ANNEALING ON THE GELATINIZATION OF SAGO (Metroxylon sagu) STARCH BY DIFFERENTIAL SCANNING CALORIMETRY

MA' ARUF BIN ABD. GHANI

FSMB 1998 11

STUDIES ON STARCH-SOLUTES INTERACTION AND ANNEALING ON THE GELATINIZATION OF SAGO (*Metroxylon sagu*) STARCH BY DIFFERENTIAL SCANNING CALORIMETRY

MA'ARUF BIN ABD. GHANI

MASTER OF SCIENCE
UNIVERSITI PUTRA MALAYSIA

1998

STUDIES ON STARCH-SOLUTES INTERACTION AND ANNEALING ON THE GELATINIZATION OF SAGO (*Metroxylon sagu*) STARCH BY DIFFERENTIAL SCANNING CALORIMETRY

BY

MA'ARUF BIN ABD. GHANI

Thesis Submitted in Fulfilment of the Requirements for the Degree of Master of Science in the Faculty of Food Science and Biotechnology Universiti Putra Malaysia

October 1998

ACKNOWLEDGEMENTS

With great thanks to Almighty Allah S.W.T and His mercy for giving me life and the opportunity to write this thesis as required.

I would like to take this opportunity to express my most sincere appreciation and deepest gratitude to Professor Dr. Yaakob Bin Che Man, the Chairman of my Supervisory Committee. Without his invaluable guidance, advice, suggestions and constructive criticisms, I would not have been able to present my work and in the preparation of this thesis.

I offer my sincere thanks to other members of the Supervisory Committee: En. Dzulkifly Mat Hashim and Assoc. Prof. Dr. Russly Abd. Rahman and also to my former supervisor Assoc. Prof. Dr. Asbi Bin Ali for their supervision, support and comments.

My heartfelt gratitude goes to all my friends, Ms. Tri Haryati, En. Zainal Samicho, Mr. Shamsul Haque and Ms. Umme Ara for their advice, kindness, generous support and invaluable friendships. I would like to acknowledge Universiti Kebangsaan Malaysia for granting me to pursue my M.Sc studies and provide financial assistance. Special thanks and appreciation to the lecturers and staff of the Faculty of Food Science and Biotechnology for helping me in various ways.

Deepest love and gratitude is forwarded to my parents, brothers, sisters and son, Harith Daniel for their love, concern, encouragement, unfailing faith and unwavering support rendered throughout this study. I also owe my special thanks to my beloved wife Hasimah Ismail for her inspiration, moral encouragement, support, patience and sacrifices which had helped me in undertakings and completing this research study.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF PLATES

LIST OF SYMBOLS AND ABBREVIATIONS

Symbols

ΔH	Enthalpy
T_O	Onset temperature
T_P	Peak temperature
T_{P1}	Peak temperature (first endotherm)
T_{P2}	Peak temperature (second endotherm)
T_m	Conclusion temperature
ν_1	Volume fraction of water
G	First endotherm
M_1	Second endotherm
T_m-T_O	Melting temperature range
T_m^o	Melting point of undiluted polymer

Abbreviations

DSC	Differential scanning calorimetry
HLB	Hydrophilic-lipophilic balance
PE	Perkin Elmer
PS	Polysorbate-60
NS	No significant
S	Significant
SE	Sucrose ester
TA	Thermal analysis

Abstract of the Thesis Presented to the Senate of Universiti Putra Malaysia in Fulfilment of the Requirements for the Degree of Master of Science.

STUDIES ON STARCH-SOLUTES INTERACTION AND ANNEALING ON THE GELATINIZATION OF SAGO (*Metroxylon sagu*) STARCH BY DIFFERENTIAL SCANNING CALORIMETRY

BY

MAARUF BIN ABD. GHANI

October, 1998

Chairman : **Professor Yaakob Bin Che Man, Ph.D.**

Faculty : **Food Science and Biotechnology**

This study was conducted to study the gelatinization of sago starch by differential scanning calorimetry. Sago (*Metroxylon sagu*) starch was used because it is locally produced and the cheapest source of starch in this country. The effects of six parameters on the gelatinization of sago starch were conducted. These parameters were heating rates, water content, the presence of sugars and salts, annealing treatments and the effect of combinations of sugars-emulsifiers. For comparison, potato starch was also studied.

Results of this study showed that sago starch was much easier to gelatinise, as their gelatinization temperature was relatively low. The differential scanning calorimetry (DSC) thermogram curves of sago starch were similar to that of potato starch, hence its, gelatinization property resembled that of potato starch. These

results were based on various heating rates (1.0-30.0 °C/min) and water:starch ratios (0.1:1 to 4:1). This study also showed that a heating rate 10 °C/min was suitable for the measurement of the gelatinization for both starches. The gelatinization temperature and enthalpy (ΔH) for sago and potato starches in excess water were 60.2-77.1 °C and 15.54-15.78 J/g and 61.1-77.2 °C and 16.98-17.31 J/g, respectively. In excess and intermediate water contents single endothermic transitions (G) occurs and transformed into double endothermic (G and M_1), respectively. However, in limited water contents only M_1 occur.

Using water:starch ratios of 1:1, 2:3, 3:2 and 3:1, sucrose of 0.2-1.7 g/ g starch and sodium chloride (NaCl) of 1.0-5.0 M, the results showed that as the concentrations of sucrose increased, the gelatinization temperatures and ΔH values of these starches also increased. The addition of 0.4-0.5 g sucrose/ g starch makes gelatinization temperatures of sago and potato starches decreased. However, with the addition 0.6 g sucrose/g starch and above, the gelatinization temperatures slightly increased compared to starches without adding sucrose. In the case of sago and potato starches, it has been argued that sucrose binds water thus making water less available for starch hydration. This explanation cannot be correct because the DSC curves should have shown the second peak when the sugar is added. However, that did not occur, instead the first peak shifts to a higher temperature. The gelatinization of sago and potato starches increased at certain NaCl concentrations and decreased as the NaCl concentrations increased. NaCl delayed gelatinization temperatures of sago starch greater than potato starch at 1.0-2.0 M NaCl concentration. NaCl was shown to decrease ΔH of transition in starch samples and created similar effects on the endotherms in excess water content and on the first endotherm with limited water content.

The effect of annealing under different temperatures (45-70 °C/min), time (0-24 h) and water:starch ratio (0.6:1-4:1) and the effects of sugars-emulsifiers and starch-water mixtures (1:1.5:1.5) were also studied using DSC. The annealing caused the entire range of the gelatinization temperature to be shifted upwards. The range of the endotherm was narrowed, as the shift was higher for the onset (T_O) and peak (T_P) temperatures than the conclusion temperature (T_m). T_O, T_P and T_m increased and ΔH decreased with the increase of annealing temperatures. However, T_m-T_O decreased with the increase of annealing time for both starches. The effect of sugars-emulsifiers showed that sucrose delay gelatinization temperature in the range of 5.9-7.3 °C and 6.5-7.7 °C higher than glucose for sago and potato starches, respectively. Low emulsifier concentration in the presence of sugars did not appear to change DSC starch gelatinization temperatures.

Abstrak Tesis yang diKemukakan kepada Senat Universiti Putra Malaysia
sebagai Memenuhi Keperluan untuk Ijazah Master Sains

KAJIAN INTERAKSI KANJI-BAHAN 'SOLUTE' DAN PENYEPUHLINDAPAN KE ATAS PENGELATINAN KANJI SAGO (*Metroxylon sagu*) MENGGUNAKAN KALORIMETRI PEMBIAS KEBEDAAN (DSC)

Oleh

MAARUF BIN ABD. GHANI

Oktober, 1998

Pengerusi : **Profesor Yaakob Bin Che Man, Ph.D.**

Fakulti : **Sains Makanan dan Bioteknologi.**

Kajian ini dijalankan untuk mengkaji pengelatinan kanji sago menggunakan kalorimetri pembias kebedaan. Kanji sagu (*Metroxylon sagu*) digunakan kerana ianya dikeluarkan di negara ini dan merupakan sumber kanji yang paling murah. Kesan dari enam parameter ke atas pengelatinan kanji sagu telah dijalankan. Parameter tersebut adalah kesan oleh pelbagai kadar pemanasan, pelbagai kandungan air, kehadiran gula dan garam, perlakuan penyepuhlindapan, dan kesan kombinasi diantara gula dan pengemulsi. Untuk tujuan perbandingan, kanji kentang juga dikaji.

Hasil kajian ini menunjukkan kanji sagu mudah mengelatin di mana secara perbandingan suhu pengelatinannya adalah rendah. Keluk termogram kalorimetri

pembias kebedaan (DSC) pada kanji sagu adalah sama berbanding kanji kentang, maka ia menunjukkan ciri pengelatinan hampir sama berbanding kanji kentang. Hasil berdasarkan pelbagai kadar pemanasan (0.0-30.0 °C/minit) dan nisbah air:kanji (0.1:1-4:1). Kajian ini juga menunjukkan bahawa kadar pemanasan 10 °C/minit adalah sesuai untuk pengukuran pengelatinan kedua-dua kanji tersebut. Suhu pengelatinan dan nilai entalpi (ΔH) pada kanji sagu dan kentang pada kandungan air berlebihan masing-masing 60.2-77.1 °C dan 15.54-15.78 J/g dan 61.1-77.2 °C dan 16.98-17.31 J/g. Pada kandungan air berlebihan satu transisi endoterma (G) wujud dan berubah kepada keluk berganda (G dan M_1) pada kandungan air pertengahan. Walaubagaimanapun pada kandungan air yang terhad hanya endoterma M_1 wujud.

Penggunaan nisbah air/kanji pada 1:1, 2:3, 3:2 dan 3:1 dan sukrosa pada 0.2-1.7 g/g kanji dan natrium klorida (NaCl) pada 1.0-5.0 M menunjukkan nilai suhu pengelatinan dan ΔH meningkat dengan peningkatan kepekatan sukrosa. Penambahan 0.4-0.5 g sukrosa/g kanji menyebabkan suhu pengelatinan kanji sago dan kentang berkurang. Manakala dengan penambahan 0.6 g sukrosa/g kanji dan lebih, suhu pengelatinannya meningkat berbanding dengan kanji yang tidak ditambah sukrosa. Di dalam kes kanji sago dan kentang, adalah dinafikan bahawa sukrosa mengikat air sehingga mengurangkan kebolehdapatan air untuk hidrasi kanji. Pernyataan ini tidak benar kerana keluk DSC mesti menunjukkan puncak kedua bila ditambah gula. Walaubagaimanapun ini tidak wujud sebaliknya kewujudan puncak pertama dianjak kepada suhu yang tinggi. Pengelatinan kanji sagu dan kanji kentang meningkat pada suatu tahap kepekatan NaCl dan kemudian menurun dengan peningkatan kepekatan NaCl. NaCl melambatkan kadar suhu pengelatinan kanji sagu lebih tinggi berbanding kanji kentang pada kepekatannya 1.0-2.0 M. NaCl juga menurunkan entalpi (ΔH) peralihan pada sampel kanji dan menghasilkan kesan yang sama ke atas endoterma pada kandungan air berlebihan dan endoterma pertama pada kandungan air terhad.

Kesan penyepuhlindapan pada pelbagai suhu (45-70 °C/minit), masa (0-24 jam) dan nisbah air:kanji (0.6:1-4:1) dan kesan gula-pengemulsi dan campuran kanji-air (1:1.5:1.5) juga dikaji dengan DSC. Penyepuhlindapan menyebabkan semua had suhu pengelatinan beralih lebih tinggi. Had endoterma disempitkan, selari dengan suhu awal (T_O) dan puncak (T_P) berbanding suhu akhir (T_m). T_O, T_P dan T_m meningkat dan ΔH menurun dengan peningkatan suhu penyepuhlindapan. Walaubagaimanapun T_m-T_O menurun dengan penambahan masa penyepuhlindapan pada kedua-dua kanji. Kesan kombinasi gula-pengemulsi menunjukkan sukrosa menangguhkan suhu pengelatinan dalam had 5.9-7.3 °C dan 6.5-7.7 °C lebih tinggi berbanding glukosa bagi kanji sagu dan kentang masing-masing. Kepekatan pengemulsi yang rendah dengan kehadiran gula tidak menunjukkan sebarang perubahan kepada suhu pengelatinan kanji.

CHAPTER I

GENERAL INTRODUCTION

What makes starch a valuable ingredient to the food industry? It is its versatility, consistent supply, and relatively inexpensive cost compared to other food ingredients. Swinkels (1985) reported that, starch is mainly used as a thickener, and also functions as an adhesive, binder, encapsulating agent, film former, gelling agent, water binder, texturizer, and fat sparing agents, with numerous other applications both in the food and non food areas.

The word starch may be derived from the Anglo-saxon *stearc* and has the connotation of strength or stiffness, as applied to fabric or paper (Swinkels, 1985). Starch is a mixture of two polymers (amylose and amylopectin), and its primary purpose is to store the energy collected by photosynthesis. Starch gelatinization is widely used in food processing to provide unique textural and structural characteristics of product (Lund and Wirakatakusumah, 1984) and is the most important phase transition in foods (Lund, 1986).

Sago is the generic name for starch derived of the stem of the sago palm *Metroxylon sagu* and *Metroxylon rumphii* (FAO, 1992). The name *Metroxylon* is derived from the Greek language that consisted of the words *Metra* and *Xylon*. *Metra* means pith and *Xylon* is the meaning for xylem or wood (Flach, 1977). Sago starch is formed in plants by the chemical linking of hundred or even thousands of glucose units into two basic types of polymers; amylose and amylopectin (Sing, 1988). The nature and characteristic of sago, which contain high carbohydrate, a lot of nutritional values and becomes an important source of industrial starch (Sudwikatmono, 1991).

Gelatinised starch plays an important role in determining the structural and textural properties of many foods. The proportion of raw and gelatinised starch in ready-to-serve starchy products may be critical in determining acceptability. Texture of many foods such as breakfast cereals, beverages, rice, noodles, pasta and dried soups depends on the fraction of gelatinised starch in the product (Guraya and Toledo, 1993). Gelatinised rice is easy to digest. Gelatinization temperature is important in the case of rice and rice products in terms of consumer acceptance. More than one transition in starch occurs during bread making when dough is heated under baking conditions. Controlling the gelatinization process can control the stickiness of rice. Low molecular weight amylose in mashed potatoes give a gluey, sticky, or gummy texture. Gelatinization appeared to be an obvious choice as index of the cooking process of rice (Birch and Priestly, 1973). Different sources of starch have different gelatinization temperature and enthalpy.

Gelatinization is the term applied to the sequence of changes which occur when starch is heated in an aqueous medium (Pomeranz, 1971). When the starch