

UNIVERSITI TEKNOLOGI MARA

**OPTIMISATION AND
CHARACTERISATION OF OCTENYL
SUCCINIC ANHYDRIDE (OSA)
MODIFIED SAGO STARCH AND ITS
CAPABILITY TO STABILISE
EMULSION**

NUR FARHANA BINTI ZAINAL ABIDDIN

Thesis submitted in fulfillment
of the requirements for the degree of
Doctor of Philosophy
(Science)

Faculty of Applied Sciences

February 2021

AUTHOR'S DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the results of my own work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any degree or qualification.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

Name of Student : Nur Farhana Binti Zainal Abiddin

Student I.D. No. : 2011419562

Programme : Doctor of Philosophy in (Science) – AS990

Faculty : Applied Sciences

Thesis Title : Optimisation and Characterisation of
Octenyl Succinic Anhydride (OSA)
Modified Sago Starch and Its Capability to
Stabilise Emulsion

Signature of Student :
.....

Date : February 2021

ABSTRACT

The demand for natural, biodegradable and renewable emulsifier has led to new market for modified starch. Sago starch has met the requirements and becomes good candidates for emulsion stabilization. However, a major barrier of native sago starch is the lack of hydrophobic properties, thus the starch require to be modified with octenyl succinic anhydride (OSA) to provide amphiphilic properties. The aim of this study was to optimize the reaction condition in preparation of OSA sago starch, analyzing the physicochemical properties and determine the emulsifying ability of modified starch. The characterisation includes determination of amylose content, thermal properties and morphology of starch. Three types of starches were used as comparison which was sago starch, gelose 80 and commercial starch. The optimisation via response surface methodology (RSM) has obtained the optimum condition for the preparation of OSA sago starch was at 5% OSA concentration, pH 7.20 and reaction time of 9.65h which produced degree of substitution (DS) of 0.0120 (OSA sago starch) and 0.0145 (OSA gelose 80). The DS was found to be important, since it affect the physicochemical and emulsification properties of OSA starch. The FT-IR analysis evidenced the successful synthesis of OSA starch with the presence of two new groups at 1717 cm^{-1} (stretch of ester carbonyl group) and 1569 cm^{-1} (assymetric stretch of vibration of a carboxylate RCOO^-). The particle size was significantly ($p < 0.05$) increased (sago starch: from 29.05 to 29.89 μm ; gelose 80: from 9.79 to 20.37 μm) due to swelling phenomenon with less damage was seen to occur on the surface of granule. The significant reduction ($p < 0.05$) in amylose content of sago starch from 30.18% to 25.27% and gelose 80 starch from 78.47% to 70.70% as esterification was preferentially occur at amorphous region. For thermal properties, OSA starches displayed significantly ($p < 0.05$) lower gelatinization temperature and enthalphy with the ability to prevent retrogradation. The physicochemical properties was generally affected by DS and this was proved by reduction in surface tension which was more pronounced in OSA gelose 80 (54.87 mN/m) as compared with OSA sago starch (58.40 mN/m). The emulsifying properties of OSA sago starch were dependent on the concentration used. At low concentration (4 and 5%), the droplet size was inconsistent as the size keep increasing during storage. Emulsion with 6% OSA sago starch shows the largest droplet, however it give significantly lower CI, high viscosity values and the droplet was closely packed which make it able to stabilize emulsion to a certain extent. The emulsion stabilized by OSA sago starch and OSA gelose 80 shows a bimodal distribution, indicating that the emulsion contain heterogenous droplet. The OSA commercial starch demonstrated better emulsifying properties when compared with the other OSA starches. It produced stable emulsion with smallest droplet, supported by clear unimodal distribution, low surface tension and less aggregation as shown by confocal laser scanning calorimetry (CLSM). The work in this thesis shows the potential of sago starch and its ability to become an emulsifier.

ACKNOWLEDGEMENT

Most importantly, thanks to The Almighty God for giving me chance to embark on my PhD and for completing this journey successfully.

First and foremost, I want to thank to my main supervisor, Assoc. Prof Dr. Anida Yusoff, who keeps supporting, encouraged and always believe in me. You are the one who always keep reminding me not to quit, your kindness make me feel that I have to finish this study not matter how long it takes. A special note of appreciation to my co-supervisor, Assoc. Prof Dr. Noorlaila Ahmad for patiently reviewing my drafts of thesis and her valuable opinions.

To my dearest husband, Mohd Adnin, thank you for always be there for me each and everytime. You are a part of my PhD journey. You are there since I embarked in this journey and till the end. You are the one who never bored lending me your shoulder and ear, and always cheer me up when I am in difficulty. Thank you for always pushed me to finish this study towards the end. May Allah bless our family.

To my cheeky son, Muhammad Daniyal, this is dedicated for you. You are with me throughout this journey. From inside my womb during pregnancy to holding hand together for my meeting with supervisor. When one day you can read this, you will understand why I had to steal your time with me. Babysitting you, doing house chores and at the same time studying really make my daily life pack, But yet, you still give me your beautiful smile and kiss on my cheek every night. I owed you too much. You are my strength to keep going; you are the reason why I am still standing strong.

For my lovely family, Ayah, Mak, Along, Abang, Nadia, Wani & Aiman, thank you for always pray for my success. For always giving me support even sometimes I do feel this journey was not mean for me. This journey is really hard for me, but I cannot compare your hardship to raise me. Without all of you, this journey would been but a dream.

I cannot help but thanks to all the laboratory assistant and science officer at Faculty of Applied Science, UiTM Shah Alam (Miss Suhadah, Mrs Norahiza, Mrs Siti Marhani, Miss Hariyah, Mr Muhammad Faisal, Mr Hayub) and Faculty of Pharmacy, UiTM Puncak Alam (Mrs Nor Zaleha and Mrs Nor Hidayah) and Imaging Centre, UiTM Puncak Alam (Dr. Zolkapli Eshak) for their help and support over these years.

TABLE OF CONTENTS

	Page
CONFIRMATION BY PANEL OF EXAMINERS	ii
AUTHOR'S DECLARATION	iii
ABSTRACT	v
ACKNOWLEDGMENT	vi
TABLE OF CONTENTS	vii
LIST OF TABLES	xi
LIST OF FIGURES	xiii
LIST OF SYMBOLS	xvi
LIST OF ABBREVIATIONS	xviii
LIST OF NOMENCLATURES	xix
CHAPTER ONE: INTRODUCTION	2
1.1 Background of Study	4
1.2 Problem Statement	6
1.3 Significance of Study	8
1.4 General Objective	8
1.5 Objectives	8
1.6 Hypotheses	9
1.7 Scope and Limitation of the Study	9
1.8 Novelty	
CHAPTER TWO: LITERATURE REVIEW	
2.1 Introduction	10
2.2 Hydrocolloids	10