

UNIVERSITI TEKNOLOGI MARA

**DEVELOPMENT OF
HEMICELLULOSE FILMS FROM
PINEAPPLE PEEL BY
MICROWAVE-ASSISTED
EXTRACTION**

**NUR AZA ATIQA BINTI
MAD ZAHIR**

Thesis submitted in fulfillment
of the requirements for the degree of
Master of Science
(**Environmental Science and Technology**)

Faculty of Applied Sciences

January 2023

ABSTRACT

Pineapple is a commercial crop that generates a vast amount of waste such as pineapple peel (PP) during processing and represents another abundant raw material in Malaysia. PP hemicellulose extracted by microwave-assisted extraction could be interest in potential applications in various fields of materials science. The aim of this study is to prepare hemicellulose film from pineapple peel. The MAE conditions (temperature, time and sodium hydroxide, NaOH concentration) were optimised to obtain the highest yield of PP hemicellulose using response surface methodology (RSM). In order to optimise the extraction conditions of microwave-assisted water extraction (MAWE) and microwave-assisted alkali extraction (MAAE) for maximum hemicellulose yield, RSM with Central Composite Design was employed. The optimised samples were characterised for lignin content, and also by using fourier-transformed infrared spectroscopy (FTIR), X-ray diffraction (XRD), thermal (TGA), and scanning electron microscopy (SEM) analysis. Hemicellulose films with different hemicellulose concentration such as 0 %, 2 %, 4 %, and 6 % (w/v) were developed. The properties of films such as colour measurement, thickness measurement, water vapour permeability and water vapour transfer rate, mechanical properties, hydration properties, thermal properties, and attenuated total reflectance-fourier transform infrared (ATR-FTIR) analysis were evaluated. The results showed that the optimum conditions (125 °C, 14 minutes) for MAWE contributed hemicellulose yield of 14.9 ± 0.5 %, whereas 88.3 ± 0.5 % was obtained at optimum conditions (122 °C, 29 minutes, 7 % NaOH) for MAAE. The determination of lignin content showed optimised hemicellulose extracted via MAAE had the highest lignin content (3.12 %). The FTIR analysis revealed important peaks indicating the presence of hemicellulose in the range of 1700 cm^{-1} and 700 cm^{-1} . XRD analysis showed the highest crystallinity index of PP residue by MAWE was 37.65%. TGA analysis concluded that the decomposition temperature for optimised hemicellulose extracted via MAWE, and MAAE were 276 °C and 277 °C, respectively. The SEM characterisation indicated that the morphological structure of the dried PP and microwave treated PPs were different from each other. A film with 6% (w/v) hemicellulose concentration showed a dark yellowish-brown in colour. The thickness of films (0.08 mm – 0.15 mm), moisture content (18.4 % – 32.18 %), and water vapour permeability (1.88×10^{-8} g/h.mm.pa - 8.21×10^{-8} g/h.mm.pa) increased significantly which differ from water solubility (56.03 - 42.84 %) properties that decreased significantly with increasing hemicellulose concentration in the films. For mechanical properties, the tensile strength (33.16 Mpa - 0.97 Mpa) decreased whereas elongation at break (19.70 % – 63.66 %) increased when the hemicellulose concentration increased. Film 6% was thermally stable (onset temperature 217 °C) compared to the others. ATR-FTIR analysis demonstrated an important peak around 1707 cm^{-1} that was attributed to the crosslinking reaction between hemicellulose, chitosan, glycerol and citric acid. Therefore, the utilisation of PP hemicellulose in biocomposite film preparation was successfully developed and Film 6% showed suitability film-forming properties that contributed to the packaging product application.

ACKNOWLEDGEMENT

Alhamdulillah thanks to Allah S.W.T for all His guidance and blessing through all the hardship encountered and also for giving me good health and strength to complete my research study successfully, though this has been a long and difficult journey.

I would like to express my sincere gratitude to my supervisor, Assoc. Prof. Dr. Sabiha Hanim Saleh for her valuable advice and encouraging guidance and supervision throughout this study. I am also grateful to my co-supervisor Assoc. Prof. Dr. Noraini Hamzah for her guidance, knowledge and valuable comments which had been of great value to me. I would like to take this opportunity to thank Mr. Mohd Shahrulrizan Ibrahim, Mr. Ahmad Kambali Khalil, Mr. Mohamad Faizal Omar, Mdm. Norahiza Mohd Soheh, Mdm. Siti Marhani Mardi, Mdm. Julia Kasim, Ms. Nor Suhadah Mohammad Samri, and all other laboratory staff in the Faculty of Applied Sciences, UiTM Shah Alam for their entire assistance in contributing to my understanding and thoughts.

I would like to express my deepest gratitude to my parent, Mr. Mad Zahir Ali, [REDACTED] [REDACTED] [REDACTED] and not to forget my other family members for their unconditional love, understandings, patience and supports throughout my life and research study. I have always felt the privilege of having such a family. Special thanks are due to Ahmad Naim Firdaus Mohd Yusoff and to all my friends Nurainshafika Sahak, Nurul Asyikin Abdul Halim, and Nur Izyan Yusof for their endless support and encouragement making the completion of this thesis possible.

Once again, thank you to all of them for their contribution toward completing my degree of master. All of their help will be remembered, and may Allah bless all of us.

TABLE OF CONTENTS

	Page
CONFIRMATION BY PANEL OF EXAMINERS	ii
AUTHOR'S DECLARATION	iii
ABSTRACT	iv
ACKNOWLEDGEMENT	v
TABLE OF CONTENTS	vi
LIST OF TABLES	x
LIST OF FIGURES	xii
LIST OF SYMBOLS	xiv
LIST OF ABBREVIATIONS	xv
CHAPTER ONE: INTRODUCTION	1
1.1 Research Background	1
1.2 Problem Statement	3
1.3 Significance of Study	4
1.4 Objectives of Study	5
1.5 Scope of Study	5
CHAPTER TWO: LITERATURE REVIEW	6
2.1 Pineapple (<i>Ananas Comosus</i>)	6
2.2 Lignocellulosic Biomass	9
2.2.1 Lignin	9
2.2.2 Cellulose	10
2.2.3 Hemicellulose	12
2.3 Hemicellulose Extraction Method	13
2.3.1 Conventional Method	14
2.3.2 Non-Conventional Method	16

2.4	Fundamentals of Microwave-Assisted Extraction	19
2.4.1	Microwave Instrumentation	19
2.4.2	Principle of Microwave Heating	21
2.4.3	Mechanism of Microwave-Assisted Extraction of Hemicellulose	22
2.5	Response Surface Methodology (RSM)	23
2.6	Application: Hemicellulose Based Films	25
	CHAPTER THREE: METHODOLOGY	28
3.1	Materials	28
3.1.1	Raw Materials	28
3.1.2	Chemicals	28
3.2	Chemical Composition of Pineapple Peel	28
3.2.1	Holocellulose Determination	28
3.2.2	Cellulose Determination	29
3.2.3	Hemicellulose Determination	30
3.2.4	Lignin content determination	30
3.3	Microwave-Assisted Extraction of Hemicellulose from Pineapple Peel	30
3.3.1	Microwave-Assisted Extraction with Different Type of Solvents	31
3.3.2	Effect of Microwave-Assisted Extraction Conditions on the Hemicellulose Yield	32
3.4	Experimental Design of Response Surface Methodology	33
3.5	Hemicellulose Characterisation	36
3.5.1	Lignin Content	36
3.5.2	Fourier Transformed Infrared Spectroscopy Analysis	36
3.5.3	X-ray Diffraction Analysis	36
3.5.4	Thermogravimetric Analysis	37
3.5.5	Scanning Electron Microscopy Analysis	37
3.6	Hemicellulose Film Preparation	38
3.7	The Effect of Different Hemicellulose Concentration on the Film	39