



**UNIVERSITI PUTRA MALAYSIA**

***DEVELOPMENT OF ANTIOXIDANT BILAYER FILMS BASED ON  
POLYETHYLENE, GELATIN AND FRUIT PEEL EXTRACTS FOR FOOD  
PACKAGING***

**NOR ADILAH BINTI ABDULLAH**

**FSTM 2018 20**



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By

**NOR ADILAH BINTI ABDULLAH**

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia  
in Fulfillment of the Requirements for the Degree of Master of Science.**

**April 2018**

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Master of Science

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**April 2018**

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**Faculty : Food Science and Technology**

In this research, utilization of agricultural by-products as a source of natural antioxidant was developed for an active packaging system. In the first objective, four types of by-products namely, jackfruit peels (JPE), mango peels (MPE), pineapple peels (PPE), and papaya peels (YPE) in the form of extracts were incorporated into fish gelatin films to determine their physical and antioxidant properties. Films with three different concentrations of extracts (1, 3 and 5%) were prepared by solution casting method. Increasing concentration of extracts up to 5% produced thicker, colored, good tensile strength and less flexible films. It also showed an improvement in water permeability with less solubility. Although all films have potential to be developed into active packaging, gelatin based film with 5% MPE was more outstanding in availability of phenolic compounds and its antioxidant performances. Therefore, in the second objective, 5% MPE-gelatin based film was developed into bilayer active packaging by casting onto the PE films at 10  $\mu\text{m}$  (PE/G10), 20  $\mu\text{m}$  (PE/G20), 40  $\mu\text{m}$  (PE/G40), and 60  $\mu\text{m}$  (PE/G60). Bilayer films also showed compatibility structure without any separation between the PE and gelatin active layer. Thicker coatings influenced the reduction ( $p \leq 0.05$ ) of the total phenolic content (TPC) values. Besides, thicker coating layer had improved the transparency and the antioxidant ability but increased ( $p \leq 0.05$ ) the water vapor permeability. Therefore, it can be suggested that bilayer film is suitable for low water activity of food products. In the third objective, PE/G60 was used to determine the effectiveness of the bilayer films on maintaining or controlling the quality of margarine during storage at 4 °C and 25 °C. Results observed that margarine packed in PE/G60 at 4 °C able to inhibit lipid oxidation during 28 days of storage. The bilayer material affects the color changes in margarine with no significant ( $p > 0.05$ ) differences were observed on pH of margarine for both temperatures. This concluded that 5% MPE at 60  $\mu\text{m}$  was

suitable to be developed into bilayer films and had the potential as an antioxidant packaging for high lipid product.



Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Sarjana Sains

**PEMBANGUNAN FILEM DWILAPISAN ANTIOKSIDA BERASASKAN POLIETILENA, GELATIN DAN EKSTRAK KULIT BUAH SEBAGAI PEMBUNGKUSAN MAKANAN**

Oleh

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Dalam kajian ini, penggunaan produk sampingan pertanian sebagai sumber antioksidan semulajadi telah dibangunkan untuk sistem pembungkusan yang aktif. Dalam objektif pertama, empat jenis produk sampingan iaitu, kulit nangka (JPE), kulit mangga (MPE), kulit nanas (PPE), dan kulit betik (YPE) dalam bentuk ekstrak telah digabungkan ke dalam filem gelatin ikan untuk menentukan sifat fizikal dan antioksidan. Filem yang mempunyai tiga kepekatan ekstrak berbeza (1, 3 dan 5%) telah disediakan oleh kaedah bancuhan ke atas acuan. Peningkatan kepekatan ekstrak sehingga 5% menghasilkan filem yang tebal, berwarna, tegang dan kurang fleksibel. Ia juga menunjukkan peningkatan kebolehtelapan air dengan kurang kelarutan. Walaupun semua filem berpotensi untuk dikembangkan menjadi pembungkusan aktif, filem berasaskan gelatin dengan 5% MPE mengandungi kadar fenolik dan tahap antioksidan yang tinggi. Oleh itu, didalam objektif kedua, filem berasaskan MPE-gelatin 5% telah dibangunkan menjadi pembungkusan aktif dwilapisan dengan kaedah bancuhan ke atas PE pada 10  $\mu\text{m}$  (PE/G10), 20  $\mu\text{m}$  (PE/G20), 40  $\mu\text{m}$  (PE/G40) dan 60  $\mu\text{m}$  (PE/G60). Filem dwilapisan juga menunjukkan keserasian struktur antara lapisan PE dan gelatin aktif. Lapisan tebal mempengaruhi pengurangan ( $p \leq 0.05$ ) nilai TPC. Selain itu, lapisan tebal juga telah menambah baik ketelusan dan kebolehan antioksidan tetapi meningkatkan ( $p \leq 0.05$ ) kebolehtelapan wap air. Oleh itu, filem dwilapisan sesuai digunakan untuk produk makanan yang kandungan airnya yang rendah. Dalam objektif ketiga, PE/G60 digunakan untuk menentukan keberkesanan filem dwilapisan dalam mengekalkan dan mengawal kualiti marjerin semasa penyimpanan pada suhu 4 ° C dan 25 ° C. Keputusan mendapati bahawa marjerin yang dibungkus dalam PE/G60 pada 4 ° C dapat menghalang pengoksidaan lipid selama 28 hari. Bahan dwilapisan mempengaruhi perubahan warna dalam marjerin, tanpa perbezaan yang signifikan ( $p > 0.05$ ) terhadap pH marjerin untuk kedua-dua suhu. Ini menyimpulkan bahawa MPE 5% pada 60  $\mu\text{m}$  sesuai untuk

dikembangkan menjadi filem dwilapisan dan mempunyai potensi sebagai pembungkusan antioksidan untuk produk berlipid tinggi.



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I certify that a Thesis Examination Committee has met on 18<sup>th</sup> April 2018 to conduct the final examination of Nor Adilah binti Abdullah on her thesis entitled "Development of antioxidant bilayer films based on polyethylene, gelatin and fruit peel extracts for food packaging" 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 Master of Science.

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## TABLE OF CONTENTS

	<b>Page</b>
<b>ABSTRACT</b>	i
<b>ABSTRAK</b>	iii
<b>ACKNOWLEDGEMENT</b>	v
<b>APPROVAL</b>	vi
<b>DECLARATION</b>	viii
<b>LIST OF TABLES</b>	xiv
<b>LIST OF FIGURES</b>	xv
<b>LIST OF ABBREVIATIONS</b>	xvi
<b>CHAPTER</b>	
<b>1 INTRODUCTION</b>	<b>1</b>
<b>2 LITERATURE REVIEW</b>	<b>4</b>
2.1 Agricultural and industrial waste	4
2.1.1 Waste issues	4
2.1.2 Utilization of waste as renewable resources	5
2.2 Fruit peels waste as natural source of antioxidant	5
2.3 Tropical fruits	6
2.3.1 Mango ( <i>Mangifera indica</i> L.)	7
2.3.2 Pineapple ( <i>Ananas comosus</i> )	8
2.3.3 Papaya ( <i>Carica papaya</i> L.)	9
2.3.4 Jackfruit ( <i>Artocarpus heterophyllus</i> Lam.)	9
2.4 Fish gelatin	10
2.4.1 Properties of fish gelatin	10
2.4.2 Potential of fish gelatin as packaging material	11
2.4.3 Improvement of fish gelatin films	11
2.5 Packaging	12
2.5.1 Food packaging	12
2.5.2 Food packaging materials	13
2.5.3 Properties of polyethylene (PE) packaging	14
2.6 Innovations in food packaging	16
2.7 Active packaging	16
2.7.1 Types of active packaging	17
2.7.2 Combination of biopolymers and synthetic polymers as active packaging material	18
2.7.3 Antioxidant packaging	19
2.8 Migration in food packaging systems	21

<b>3</b>	<b>THE ASSESMENT OF DIFFERENT PEEL EXTRACTS ON THE PHYSICAL, MECHANICAL AND ANTIOXIDANT PROPERTIES OF FISH GELATIN-BASED FILMS</b>	
3.1	Introduction	24
3.2	Materials and Method	26
3.2.1	Materials	26
3.2.2	Preparation of fruit peels	26
3.2.3	Extraction of fruit peels	26
3.2.4	Preparation of film forming solution (FFS)	26
3.2.5	Determination of physical film properties	27
3.2.5.1	Thickness	27
3.2.5.2	Color measurement	27
3.2.5.3	Film opacity	27
3.2.5.4	Water vapor permeability (WVP)	28
3.2.5.5	Film solubility	28
3.2.6	Determination of mechanical properties	28
3.2.6.1	Tensile strength (TS) and elongation at break (EAB)	28
3.2.7	Determination of total phenolic content (TPC)	29
3.2.8	DPPH radical scavenging activity	29
3.2.9	Statistical analysis	29
3.3	Results and discussion	30
3.3.1	Determination of physical properties	30
3.3.1.1	Film thickness	30
3.3.1.2	Color measurement	31
3.3.1.3	Film opacity	34
3.3.1.4	Water vapor permeability (WVP)	34
3.3.1.5	Film solubility	35
3.3.2	Determination of mechanical properties	36
3.3.2.1	Tensile strength (TS) and elongation at break (EAB)	36
3.3.3	Determination of total phenolic content (TPC) and radical scavenging activity (RSA)	39
3.4	Conclusion	41

<b>4</b>	<b>THE EFFECTIVENESS OF BILAYER FILMS BASED ON POLYETHYLENE/GELATIN (PE/G) FILMS ON THE PHYSICAL AND FUNCTIONAL PROPERTIES</b>	
4.1	Introduction	42
4.2	Materials and method	43
4.2.1	Materials	43
4.2.2	Preparation and extraction of mango peels	44
4.2.3	Preparation of bilayer film	44
4.2.4	Determination of film properties	44
4.2.4.1	Thickness	44
4.2.4.2	Color	44
4.2.4.3	Transparency	45
4.2.4.4	Water vapor permeability (WVP)	45
4.2.5	Determination of total phenolic content (TPC)	45
4.2.6	DPPH radical scavenging activity (RSA)	45
4.2.7	Scanning electron microscopy (SEM)	45
4.2.8	Statistical analysis	45
4.3	Results and discussion	46
4.3.1	Physical properties of bilayer films	46
4.3.1.1	Films thickness	46
4.3.1.2	Color and transparency of bilayer films	46
4.3.1.3	Water vapour permeability (WVP)	48
4.3.2	Total phenolic content (TPC) and radical scavenging activity (RSA) of bilayer films	50
4.3.3	Bilayer Films microstructure	52
4.4	Conclusion	54
<b>5</b>	<b>EFFECT OF ACTIVE PACKAGING USING BILAYER FILMS ON THE QUALITY OF MARGARINE DURING STORAGE</b>	
5.1	Introduction	55
5.2	Materials and method	56
5.2.1	Materials	56
5.2.2	Preparation of bilayer films	56
5.2.3	Preparation of margarine	56

5.2.4	Oxidative stability of product	57
5.2.4.1	Peroxide value (PV)	57
5.2.4.2	Thiobarbituric acid reactive substances (TBARS)	57
5.2.5	Color measurements for margarine	57
5.2.6	pH of margarine	58
5.2.7	Statistical analysis	58
5.3	Result and discussion	58
5.3.1	Peroxide value (PV)	58
5.3.2	Thiobarbituric acid reactive substances (TBARS)	60
5.3.3	Color of margarine	61
5.3.4	pH changes in margarine	63
5.4	Conclusion	64
<b>6</b>	<b>GENERAL CONCLUSION AND RECOMMENDATIONS FOR FUTURE RESEARCH</b>	
6.1	General conclusion	65
6.2	Recommendations and future research	65
	<b>REFERENCES</b>	66
	<b>APPENDICES</b>	91
	<b>BIODATA OF STUDENT</b>	129
	<b>PUBLICATION</b>	130



## LIST OF TABLES

Table		Page
1	Structure of LDPE, LLDPE, and HDPE.	15
2	Comparison of passive and active packaging.	16
3	Applications of selected examples of active packaging system.	18
4	Classification of migration types.	21
5	The thickness of films ( $\mu\text{m}$ ) with different peel extracts at concentrations of 1, 3, and 5 %.	30
6	<i>L</i> , <i>a</i> , and <i>b</i> values for films with different peel extracts at concentrations of 1, 3, and 5 %.	32
7	Opacity measurements for films with different peel extracts at concentration of 1, 3, and 5 %.	34
8	Total thickness of bilayer films.	46
9	Color and transmittance (%) of bilayer films.	47
10	Color measurement of margarine during storage.	62

## LIST OF FIGURES

Figure		Page
1	Example of polymerized chain of polyethylene.	14
2	Type of active packaging systems.	17
3	The migration of active compounds from various packaging systems.	22
4	Gelatin-based films with various peel extracts at different concentrations.	33
5	Water permeability of films with various peel extracts at different concentrate ion.	35
6	The solubility of films with various peel extracts at different concentration.	36
7	The tensile strength of films with various peel extracts at different concentration.	38
8	The elongation at break of films with various peel extracts at different concentration.	38
9	The total phenolic content of films with various peel extracts at different concentration.	40
10	The radical scavenging activity of films with various peel extracts at different concentration.	41
11	PE and bilayer films.	48
12	Water vapor permeability of bilayer films at different thickness.	49
13	Total phenolic content of bilayer films at different thickness.	50
14	DPPH radical scavenging activity of bilayer films at different thickness.	51
15	The surface and cross-section of bilayer films by scanning electron microscopy (SEM).	52
16	Peroxide values of margarine samples.	59
17	TBARS of margarine samples.	61
18	Packed margarine in PE and PE/G at day 0 and day 28 stored at 4 °C and 25°C.	63
19	The pH changes in margarine.	64

## LIST OF ABBREVIATIONS

%	Percentage
°C	Degree celsius
μl	Microliter
μm	Micrometer
BHA	Butylated hydroxyanisole
BHT	Butylated hydroxytoluene
DPPH	2,2-diphenyl-1-picrylhydrazine
EAB	Elongation at break
FFS	Film forming solution
FG	Fish gelatin
GAE	Gallic acid equivalent
g	Gram
HDPE	High-density polyethylene
h	Hour
JPE	Jackfruit peel extracts
kPa	Kilopascal
LDPE	Low-density polyethylene
LLDPE	Linear low-density polyethylene
mg	miligram
ml	Milliliter
mM	miliMolar
mm	Millimeter
MPa	Milipascal
MPE	Mango peel extracts
PE	Polyethylene
PPE	Pineapple peel extracts
PV	Peroxide value
RH	Relative humidity
RSA	Radical scavenging activity
SEM	Scanning electron microscopy
TBARS	Thiobarbituric acid reactive substances
TPC	Total phenolic content
TS	Tensile strength
UV	Ultraviolet
w/v	Weight per volume
w/w	Weight per weight
WVP	Water vapor permeability
YPE	Papaya peel extracts

## CHAPTER 1

### INTRODUCTION

Food processing and agricultural practices produce billions of tons of agricultural by-products (Baiano, 2014). Agricultural by-products can be classified into crop residues and agro-industrial residues. Crop residues usually comprise the non-edible components of harvested plants, while agro-industrial residues are constituents like peels, hulls, pomace, corn cobs, and others which are obtained after processing of the main components (Mande, 2005). Most of these by-products are considered to be non-beneficial; hence they are disposed at landfills or utilized for feedstock and composting. However, the increase in generated waste by the year could have an impact on the limited amount of land for waste disposal and eventually cause environmental pollution. Thus, the exploitation of agricultural by-products seems to be a promising method to reduce the burden on landfills. It is said that such wastes contain valuable components such as fiber, flavor compounds, phytochemicals, polysaccharides, and proteins, all of which can be used as functional ingredients in nutritional and pharmacological products (Baiano, 2014). In the past few years, various studies have focused on agricultural wastes as renewable bioactive natural products (Lai et al., 2017; Baiano, 2014; Kammerer, Kammerer, Valet & Carle, 2014; Abdullah, Zulkifli, Abdullah, Aziman & Kamarudin, 2012; Ayala-Zavala, Vega-vega, Rosas-domínguez, Palafox-carlos & Villarodríguez, 2011).

The demand for safer natural antioxidants to replace artificial ones is increasing. Nowadays, consumers prefer a minimal amount of synthetic additives in their food due to concerns over the adverse effects towards their health. Over the past few years, synthetic antioxidants such as butylated hydroxyanisole (BHA) and butylated hydroxytoluene (BHT) have been widely used in the food industry to prevent the deterioration of food products by lipid oxidation or microbial action. Therefore, natural additives in the form of extracts and essential oils are a few alternatives to be exploited. While the addition of natural antioxidants to food is non-hazardous to health, this process may alter the quality, flavor, taste, and smell of the food product. Subsequently, the consumers' preferences for the product may change. To prevent this drawback, the food packaging industry has developed new packaging systems such as active and intelligent packaging to retain the quality of the food products.

Gelatin films have been widely used in film-processing as they are an excellent barrier to volatile compounds, UV light, and gases. Gelatin is derived from a protein called collagen, and can be obtained from animal by-products through acidic or alkaline hydrolysis. The most abundant sources of gelatin are pig skin (46%), bovine hide (29.4%), as well as pig and cattle bones (23.1%) (Gómez-Guillén, Giménez, López-Caballero & Montero, 2011). Marine fish species are increasingly receiving attention from researchers as they can potentially replace the gelatin of mammalian species in

light of halal, vegetarian, and kosher issues. Fish gelatin is an inexpensive major by-product of the fish-processing industry. It can be extracted from the bones and skins of the fishes. The abundance of these wastes cause pollution, so their valorization can allow them to be used as a valuable source of gelatin (Uranga, Leceta, Extabide, Huerrero & de la Caba, 2016; Hosseini, Rezaei, Zandi & Farahmandghavi, 2013; Badii & Howell, 2006). Moreover, fish gelatin has good film-forming abilities, and is also one of the first materials to be proposed as carriers of bioactive components (Gómez-Guillén et al., 2011). Furthermore, the formation of gelatin films is relatively simple and does not involve particular condition to produce (by casting method or extrusion) and for drying (Nur Hanani, Roos & Kerry, 2014). However, the drawback of gelatin film is that it has poor water vapor permeability. Therefore, several approaches like the production of bi- or multi-layers, crosslinking of chemicals, or blending of biopolymers are used to improve the properties of the films (Uranga et al., 2016).

The development of food packaging via the incorporation of active compounds into packaging materials, packed products, or their surrounding conditions - which is referred to as 'active packaging' - has been and is still ongoing (Realini & Marcos, 2014; Camo, Beltrán, & Roncalés, 2008). This innovation has generated interest among researchers due to the need to prolong the shelf life, better maintain the quality and safety, as well as enhance the organoleptic properties of food. Moreover, active packaging methods which are based on environmentally-friendliness and natural preservatives could be a better option to overcome health concerns and environmental issues (Park et al., 2012; Suppakul, Miltz, Sonneveld & Bigger, 2003; Appendini & Hotchkiss, 2002). The inclusion of natural compounds or extracts (from essential oils, plants, and agricultural wastes) as additives in biopolymer matrices has been investigated to improve the properties and provide novelty to the films (Valdés, Mellinas, Ramos, Garrigós & Jiménez, 2014). More importantly, the inclusion of these additives in polymer matrices may prolong the shelf life of the food products. The migration and slow release of antioxidants into the foodstuffs reduces their oxidation and spoilage (Manzanarez-López et al., 2011).

Plastics, which are valued over paper, cardboard, metal, glass, and other materials, are widely used as packaging materials. The most common type of polymer for plastic packaging is polyethylene (PE). PE plastics like low-density PE (LDPE), high-density PE (HDPE), and linear low-density PE (LLDPE) are generally used in food packaging. PE is the most favorable packaging material in light of its low price, ease of processing, and broad range of properties (Lokensgard, 2008). PE plastics are excellent barriers to water vapor barrier but poor barriers to gas. Therefore, these types of plastics are not suitable for oxidation-sensitive food products. The development of the barrier properties of PE films along with other materials such as gelatin-based films to form bi- or multi-layer films seems to be a promising method to enhance the properties of PE films.

An increase in 'green' consumerism - that which prefers natural ingredients over synthetic substances - is a desirable occurrence whereby valuable components from food

and agricultural wastes are recovered. The wastes contain health-promoting components such as phenolic compounds, dietary fiber, and proteins (Lai et al., 2017). Active compounds are mostly used in food fortification. However, the usage of natural antioxidants releases their own odors and flavors that could alter the sensory properties of food if directly incorporated into the food. Thus, other approaches such as the development of active packaging using by-products as natural active substances in the film matrices - have been taken to help reduce this problem and at the same time maintain the quality of the food products. Besides, a combination of two different types of polymers is expected to improve the barrier properties of the films and protection of the food. In this study, agricultural waste (specifically, fruit peels) was extracted and used as a source of active compounds. The active components were added to the gelatin film-forming solution (FFS) which acted as an active compound carrier. Then, the gelatin-based film was coated on an existing PE film to produce a bilayer active packaging material. The bilayer film was expected to have antioxidant ability, lower permeability, improved light barrier and transparency. Finally, the bilayer film was applied to food products to determine the effectiveness of the film as packaging material. The objectives of this study were:

- 1) to study the effect of different peel extracts on the physical, mechanical and antioxidant properties of fish gelatin-based films.
- 2) to evaluate the effectiveness of bilayer films based on polyethylene/gelatin (PE/G) films on the physical and functional properties.
- 3) to determine the effect of active packaging composed of bilayer films on the quality of margarine during storage.

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