UNIVERSITI PUTRA MALAYSIA

POTENTIAL OF LOCAL K-CARRAGEENAN EXTRACTED FROM RED SEAWEED (*Eucheuma cottonii*) AS FOOD PACKAGING MATERIAL

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By

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Chairman : Nur Hanani Zainal Abedin, PhD
Faculty : Food Science and Technology

Interest in renewable packaging materials over the synthetic petroleum-based packaging has been rose recently. κ-carrageenan from local red seaweeds (*Eucheuma cottonii*) has great potential as the renewable sources and was not explored extensively. Therefore, this research was focused on the development and properties of the κ-carrageenan films extracted from Malaysia's *E. cottonii*.

In the first objective, physical properties of κ-carrageenan films with different concentration (10-60%) and type (glycerol, sorbitol, and polyethylene glycol (PEG)) of plasticizers were studied. The overall observations revealed that films contained sorbitol showed the best properties than films contained glycerol and PEG, especially in water barrier properties. Sorbitol-plasticized films had lowest ($p<0.05$) water vapor permeability (WVP) (4.2×10^{-10} g mm s^{-1} m^{-2} Pa^{-1}) and water uptake ratio (WUR) (2278.1%) at the 30% concentration of plasticizer. Concentration of plasticizers did influence ($p<0.05$) the κ-carrageenan films except for the films that contained sorbitol ($p>0.05$). The tensile strength (TS) and elongation at break (EAB) of films was decreased and increased ($p<0.05$), respectively with the increasing of plasticizers. The addition of plasticizers reduced the opacity of films ($p<0.05$) while the color of films was not affected ($p>0.05$).

In the second objective, the effects of palm oil at different levels (0.5-2%) on the properties of local (C1: Lucky Frontier, Malaysia) and commercial (C2: Tacara, Malaysia; C3: MSC Co., Korea; C4: Rico Co., Philippines) κ-carrageenan films were studied. The water barrier properties of κ-carrageenan films were improved along with the increasing of palm oil concentrations ($p<0.05$), irrespective of the κ-carrageenan sources. The addition of palm oil produced thicker and more opaque films ($p<0.05$), decreased the TS and thermal properties ($p<0.05$), but increased the EAB ($p<0.05$) and the contact angle ($p<0.05$). The C4 films showed the best in overall properties

including lowest WVP (3.2×10^{-10} g mm s^{-1} m^{-2} Pa^{-1}), WUR (22.48.1%), and film solubility (FS) (61.1%) ($p<0.05$).

In the third objective, the potential of κ-carrageenan coatings to protect and maintain the quality of fresh-cut pineapples was investigated for 9 days. The C1 and C4 κ-carrageenan with and without 2% of palm oil were used as the formulation of coatings. κ-carrageenan coatings had protected the fresh-cut pineapples from weight loss and microbes and maintained their firmness ($p<0.05$). The C1 coatings showed better performance with the lower weight loss (1.1%) and total plate counts (TPC, 3.9 log CFU/g) and higher firmness (1.6 N) than C4 coatings. Therefore, κ-carrageenan had high potential as food packaging material.

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

POTENSI K-KARAGINAN TEMPATAN DIEKSTRAK DARIPADA RUMPAI LAUT MERAH (*Eucheuma cottonii*) SEBAGAI BAHAN PEMBUNGKUSAN MAKANAN

Oleh

ILI BALQIS BINTI ABDUL MUTTALIB

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Permintaan bahan pembungkusan yang boleh diperbaharui semakin meningkat melebihi bahan berasaskan petroleum. κ-karaginan daripada rumpai laut merah (*Eucheuma cottonii*) tempatan berpotensi besar sebagai sumber yang boleh diperbaharui untuk bahan pembungkusan dan tidak dikaji dengan lebih dalam lagi. Oleh itu, kajian ini bertujuan untuk membangunkan dan mengkaji sifat-sifat filem κ-karaginan tempatan yang diperolehi daripada *E. cottonii* Malaysia.

Dalam objektif pertama, ciri-ciri fizikal filem κ-karaginan mengadungi kepekatan (10-60%) dan jenis (gliserol, sorbitol, dan polietilen glikol (PEG)) 'plasticizer' yang berbeza telah dikaji. Pemerhatian secara menyeluruh menunjukkan filem κ-karaginan yang mengandungi sorbitol mempunyai ciri-ciri yang terbaik berbanding filem yang mengandungi gliserol dan PEG, terutamanya dalam sifat-sifat penghalang air. Filem mengandungi sorbitol (30%) mempunyai kebolehtelapan wap air (WVP) (4.2 x 10^{-10} g mm s^{-1} m^{-2} Pa^{-1}) dan nisbah pengambilan air (WUR) yang terendah ($p<0.05$). Kuantiti 'plasticizer' mempengaruhi filem κ-karaginan ($p<0.05$) kecuali filem yang mengandungi sorbitol ($p>0.05$). Peningkatan kepekatan 'plasticizer' telah mengurangkan daya tahan (TS) dan meningakatkan daya tarikan (EAB) filem ($p<0.05$). 'Plasticizer' mengurangkan kelegapan ($p<0.05$) dan tidak mengubah warna filem ($p>0.05$).

Dalam objektif kedua, kesan minyak sawit pada kepekatan yang berbeza (0.5-2%) terhadap sifat-sifat filem κ-karaginan tempatan (C1: Lucky Frontier, Malaysia) dan komersial (C2: Tacara, Malaysia; C3: MSC Co., Korea; C4: Rico Co., Filipina) telah dikaji. Sifat-sifat penghalang air filem κ-karaginan telah meningkat dengan peningkatan kuantiti minyak sawit ($p<0.05$), tanpa mengira sumber κ-karaginan. Penambahan kepekatan minyak sawit menghasilkan filem yang lebih tebal dan legap

($p<0.05$), mengurangkan TS dan ciri-ciri haba filem ($p<0.05$), namun meningkatkan EAB dan ‘contact angle’ ($p<0.05$) filem. Filem C4 telah menunjukkan sifat-sifat yang terbaik termasuklah WVP (3.2×10^{-10} g mm s^{-1} m^{-2} Pa^{-1}), WUR (22.48.1%), dan kadar larut dalam air (FS) (61.1%) yang terendah ($p<0.05$).

Dalam objektif ketiga, potensi saduran κ-karaginan untuk melindungi dan mengekalkan kualiti nanas potong telah dikaji selama 9 hari. Saduran daripada κ-carrageenan C1 dan C4 mengandungi dan tanpa minyak sawit (2%) telah digunakan sebagai rumusan saduran. Saduran κ-karaginan telah melindungi nanas potong daripada penurunan berat dan mikrob dan juga mengekalkan tekstur ($p<0.05$). Saduran C1 menunjukkan sifat-sifat yang terbaik dengan penurunan berat (1.1%) dan ‘total plate counts’ (TPC, 3.9 log CFU/g) lebih rendah dan mempunyai tekstur (1.6 N) yang lebih tinggi berbanding saduran C4. Oleh itu, κ-karaginan mempunyai potensi tinggi sebagai bahan pembungkusan makanan.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

%	Percentage
°C	Degree Celsius
°	Degree
μL	Microliter
μm	Micrometer
ι	Iota
κ	Kappa
λ	Lambda
θ	Theta
ANOVA	Analysis of Variance
AOAC	Association of Official Analytical Chemists
ATR-FTIR	Attenuated Total Reflectance-Fourier Transform Infrared
BI	Browning Index
CA	Contact Angle
CFU	Colony Forming Units
CS	Coating Solution
EAB	Elongation at Break
ECS	Emulsion Coating Solution
FAMA	Federal Agricultural Marketing Authority
FCC	Food Chemicals Codex
FFE	Film Forming Emulsion
FFS	Film Forming Solution
FS	Film Solubility
g	Gram
h	Hour
Hm	Enthalpy of Melting

kV	Kilovoltage
M	Molarity
m	Mass
mA	Miliamplitude
MC	Moisture Content
mg	Miligram
mL	Mililiter
mm	Milimeter
mPa	Milipascal
Mw	Molecular Weight
N	Newton
nm	Nanometer
Pa	Pascal
PCA	Plate Count Agar
ppm	Part per Million
PPO	Polyphenol Oxidase
RH	Relative Humidity
rpm	Revolutions per Minute
s	Seconds
SEM	Scanning Electron Microscopy
TA	Titratable Acidity
Tm	Melting Temperature
TPC	Total Plate Count
TSS	Total Soluble Solid
WUR	Water Uptake Ratio

CHAPTER 1

INTRODUCTION

Food packaging is a system that provides protection to the food products from any external influences such as moisture, gases, and microorganism, as a means to maintain or increase the food's quality and safety, as well as to prolong the shelf- life of the food products (Franco & Falque, 2016). Among packaging materials, plastics are widely used as food packaging owing to their characteristics which are light, strong, flexible, and cheap to manufacture (Roy, Saha, Kitano, & Saha, 2012; Mahalik & Nambiar, 2010) . It is commonly made from petroleum-based synthetic polymers such as polyethylene (PE), high density polyethylene (HDPE), polyethylene terephthalate (PET), polyvinyl chloride (PVC), and polystyrene (PS). However, these kind of polymers have poor sustainability and degradability that lead to adverse environmental and ecological problems, plus becoming a source of chemical food contamination (Prashanth & Tharanathan, 2007).

Therefore, development of biopolymers as food packaging has been getting great attention recently, as they offer valuable characteristics such as biocompatibility, biodegradability, environmental-friendliness, and even edibility (Salmieri & Lacroix, 2006). Simultaneously, biopolymers derived from polysaccharides (starch, carrageenan, alginate, agar, pectin, chitosan, cellulose, and pullulan), proteins (casein, gluten, gelatin, and whey protein), lipids (beeswax, essential oil, vegetable oil, and fatty acids), and/or their combinations have been developed as biodegradable films or coatings in food industry (Alves, Costa, & Coelhoso, 2010; Bao, Xu, & Wang, 2009).

Among these natural biopolymers, polysaccharides have gained a high level of interest based on their unique colloidal nature, abundance, low cost, and good film-forming property as well as their moderate oxygen and moisture permeability (Robertson, 2013; Alves et al., 2010; Bao et al., 2009). Carrageenan is one of the polysaccharides and is the promising biopolymers since it can be obtained abundantly from renewable resources like red seaweeds, with biocompatibility and environmentally friendly characteristics, also strong gel forming ability (Shankar, Reddy, Rhim, & Kim, 2014). In general, it is known as valuable ingredients for foods, cosmetics, and pharmaceuticals.

Carrageenan is derived from red seaweeds (Rhodophyceae) and is a natural hydrophilic polymers with a linear chain of partially sulfated galactans, which possesses good film-forming properties (Osorio, Molina, Matiacevich, Enrione, & Skurtys, 2011). Besides, these biodegradable resources posed certain benefit whereby their hydrophilic nature makes them excellent barriers against non-polar substances such as aromas and oxygen (Khwaldia et al., 2004). Carrageenan films have shown good gas-barrier properties and have been reported that suitable to be used for edible film or coating. Carrageenan coatings have been proven efficient to extend the shelf life of fresh-cut and fresh whole fruits and presented lower oxygen permeability than starch films (Paula, Benevides, Cunha, de Oliveira, Pinto, Morais, & Azeredo, 2015). One of the carrageenan types that has been discovered to be useful for packaging purposes is known as kappa (κ) carrageenan. It is able to form gel and has high mechanical strength compared to the other types (iota- and lambda-carrageenan) (Nieto, 2009; Choi, Choi, Cha, Chinnan, Park, & Lee, 2005).

κ-carrageenan is found abundantly in the red seaweeds species of *Eucheuma cottonii* (Munoz, Freile-Pelegrin, & Robledo, 2004) where, almost pure κ-carrageenan, with small amount of ι-carrageenan (<10%) was detected in this species (Lee, Lo, & Chye, 2008). *E. cottonii* has been cultivated as raw material for industrial production of κ-carrageenan (Estevez, Ciancia, & Cerezo, 2004), and has been largely cultivated on the east coast of Sabah, Malaysia for this last three decades (Lee et al., 2008) due to its wide range of applications. At the early stage (1970s), *E. cottonii* was manufactured as a component of canned pet food and later, the quality of alkali-treated flour has been improved with respect to the color, odor, and taste and was exported as food grade κ-carrageenan (Lee et al., 2008). Unfortunately, the application as packaging material is still unfamiliar and extensive study on the properties of the films should be carried out.

Some studies on local carrageenan that highlighted *E. cottonii* were limited to the physicochemical properties of the carrageenan powder (Chan, Mirhosseini, Taip, Ling, & Tan, 2013) and the production of a semi-refined carrageenan (Mustapha et al., 2011; Normah & Nazarifah, 2003). Recently, a study on the effects of various plant oils on κ-carrageenan films extracted from Sabah's *E. cottonii* was carried out (Nazurah & Hanani, 2017). Therefore, an additional study should be carried out to understand more on the characteristic of this carrageenan. This will provide some information to other researchers and packaging industries to utilize this material under optimum condition to suit their needs.

In this study, the properties of local κ-carrageenan films were studied with 3 objectives;

1. To develop and investigate the κ-carrageenan films with different concentrations and types of plasticizer.
2. To study the effects of palm oil at different levels on the properties of local and commercial κ-carrageenan films.
3. To study the effect of κ-carrageenan coatings on the quality of fresh-cut pineapple.

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LIST OF PUBLICATONS

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