



**UNIVERSITI PUTRA MALAYSIA**

***DEVELOPMENT AND CHARACTERIZATION OF SUGAR PALM  
[Arenga pinnata (Wurmb.) Merr.] STARCH/NANOCELLULOSE  
BIOCOMPOSITES FILMS INCORPORATED WITH ESSENTIAL OILS***

**MOHAMAD OMAR SYAFIQ BIN RAZALI**

**IPTPH 2022 6**



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UNIVERSITI PUTRA MALAYSIA  
BERILMU BERBAKTI

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By

**MOHAMAD OMAR SYAFIQ BIN RAZALI**

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in  
Fulfillment of the Requirements for the Degree of Doctor of Philosophy**

**March 2022**

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## DEDICATION

To Al-Quran, the greatest source of knowledge

*Bring me sheets of iron" - until, when he had leveled [them] between the two mountain walls, he said, "Blow [with bellows]," until when he had made it [like] fire, he said, "Bring me, that I may pour over it molten copper." (Al-Kahf:Verse 96)*

&

To my beloved father and mother for their invaluable sacrifices, encouragements and support throughout my life

&

To my beloved family for their love, patience and understanding

&

To my beloved Teachers

&

To my awesome team members

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

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**March 2022**

**Chairman : Mohd Sapuan bin Salit, PhD**  
**Institute : Tropical Forestry and Forest Product**

The demand of antibacterial active packaging is escalating due to the needs of improving quality and shelf life of food. Furthermore, plastic waste is one of the world's concern to human health. Biocomposite have attracted attentions among the researchers, due to their environmental friendliness and sustainable nature. Starch is an alternative to substitute the petroleum based plastic film. There are several limiting factors as using starch, which are fragile and brittle. Plasticizers were added to the starch film-forming solutions to help overcome the unplasticized starch films' fragile and brittle nature. Besides, cinnamon essential oil was incorporated as antibacterial agent in the biocomposite films to form antibacterial active packaging. In order to transform into high performance of the antibacterial active packaging, cinnamon essential oil incorporated sugar palm nanocellulose reinforced sugar palm starch biopolymer composites were casted by using solution-casting method to investigate the properties of biocomposite films. The characterizations of biocomposite films was performed using Field Emission Scanning Electron Microscopic (FESEM), Thermal Gravimetric Analysis (TGA), Fourier Transform Infrared Spectroscopic (FTIR), density, antibacterial activity, seal and tear strength. The effect of various types of plasticizer with different concentrations (glycerol, sorbitol and (glycerol + sorbitol) at 0 - 4.5 %wt) on the mechanical, physical, water barrier and biodegradable properties were evaluated. It shows water absorption were increase respective to the plasticizer concentration. Besides, water vapour permeability (WVP) and solubility of the different concentration plasticizer used in the biopolymer shows increasing trend due to high water content. Water content for sample glycerol (G-plasticized), sorbitol (S-plasticized) and mixture of glycerol and sorbitol (GS-plasticized) were shown increasing trend, which were from 14.86-30.30%, 12.36-15.00% and 13.58-35.04% respective to their concentration. Increasing plasticizer contents resulted in increments in film thickness and moisture contents. On the contrary, the increase in plasticizer concentrations resulted in the decrease of the densities of the plasticized films. Three types of essential oil, i.e. cinnamon, eucalyptus and rosemary oils were tested for antibacterial activity, cinnamon essential oil (EO) showed the highest inhibition zone. Cinnamon EO had been chosen as antibacterial agent in the

biocomposite films. The effect of various concentration of cinnamon EO (0 - 2.0 wt %) on the mechanical, morphological, flammability, thermal and antibacterial activity were evaluated. The effectiveness of antibacterial packing is determined by the diameter of the zone of inhibition; the wider the diameter, the more sensitive the bacterium to the packaging. The antibacterial test was carried out by agar disc method (ADM) to evaluate the inhibition effect of the films on the gram positive bacteria, gram negative bacteria and yeast. The result suggested that the optimum amount of EO used (2.0 wt %) for positive result of inhibition activity. Tested film incorporated with 2.0 wt % EO showed the inhibition zone for *B. subtilis*, *S. aureus* and *E. coli*, which were 7.85, 6.63, and 7.43 mm respectively. From scanning electron microscopy analysis, the cross-sections of the cinnamon EO-containing films showed appearance of micro-porous spots as micro-porous holes because of the occurrence of partial evaporation on the cryo-fractured surface as a result of the vacuum condition. Increment in cinnamon EO concentration resulted in increasing trend of the number and size of the micro-porous holes. Significant increase was observed in the thermal stability with the cinnamon EO loading. The thermal stability of the CEO/SPNCC/SPS biopolymer composites was remarkably improved with increasing CEO loadings (sample 5,  $T_{max}=296.25^{\circ}\text{C}$ ) compared to the thermal stability of the unfilled compound (sample 1,  $T_{max}=289.18^{\circ}\text{C}$ ). FESEM micrograph show the films was slightly yellowish and transparent films. Overall, cinnamon essential oil/sugar palm nanocellulose/starch biocomposite films have good potential for active food packaging applications.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

**PEMBANGUNAN DAN PENCIRIAN FILEM BIOKOMPOSIT  
KANJI/NANOSELULOSA ENAU [*Arenga Pinnata* (Wurmb.) Merr.] YANG  
DIGABUNGAN DENGAN PELBAGAI JENIS PATI MINYAK**

Oleh

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Permintaan pembungkusan aktif antibakteria semakin meningkat kerana keperluannya untuk meningkatkan kualiti dan jangka hayat makanan. Tambahan pula, sampah berpunca daripada plastik adalah salah satu faktor kerisauan dunia terhadap kesihatan manusia. Biokomposit telah menarik perhatian para penyelidik, kerana sifatnya yang mesra alam, lestari alam sekitar, dan mempunyai kekuatan yang sesuai. Kanji adalah salah satu alternatif untuk menggantikan filem plastik berasaskan petroleum. Terdapat beberapa faktor yang membatasi dalam menggunakan kanji, iaitu rapuh dan mudah pecah. Pemplastik ditambahkan ke dalam bancuhan pembuatan filem berasaskan kanji untuk membantu mengatasi sifat rapuh dan mudah pecah tersebut. Selain itu, pati minyak kayu manis ditambah dan digabungkan sebagai agen antibakteria dalam filem biokomposit. Untuk menghasilkan pembungkusan aktif antibakteria yang berprestasi tinggi, pati minyak kayu manis yang digabungkan ke dalam komposit biopolimer kanji/nanoselulosa enau telah dibuat menggunakan kaedah penuangan cairan akan diuji terhadap sifat filem bionanokomposit. Pencirian filem bionanokomposit dilakukan adalah terdiri daripada mikroskopi elektron pengimbasan emisi lapangan (FESEM), analisis gravimetrik termal (TGA), spektroskopi inframerah transformasi Fourier (FTIR), ketumpatan, aktiviti antibakteria, kekuatan lekatan termal dan kekuatan untuk mengoyak. Kesan daripada pelbagai jenis pemplastik dengan isi padu yang berbeza (gliserol, sorbitol dan (gliserol + sorbitol) pada 0 – 4.5 wt %) terhadap sifat mekanikal, fizikal, penghalang air dan biodegradasi telah dinilai. Keputusan menunjukkan penyerapan air meningkat bergantung kepada kepekatan pemplastik. Selain itu, kebolehtelapan wap air (WVP) dan kelarutan menunjukkan arah aliran yang meningkat bergantung kepada perbezaan kadar kepekatan pemplastik yang digunakan dalam biopolimer kerana kandungan air yang tinggi. Kandungan air untuk sampel G-, S- dan GS-plastik menunjukkan peningkatan dari (14.86-30.30%), (12.36-15.00%) dan (13.58-35.04%) bergantung kepada kepekatannya. Peningkatan kandungan pemplastik mengakibatkan peningkatan ketebalan filem dan kandungan lembapan. Sebaliknya, peningkatan kepekatan pemplastik mengakibatkan penurunan kepadatan filem plastik. Tiga jenis pati minyak daripada kayu manis, kayu putih dan rosemary diuji untuk

mengetahui aktiviti antibakteria dan pati minyak kayu manis telah menunjukkan zon penghadangan tertinggi. Pati minyak kayu manis telah dipilih untuk digunakan sebagai agen antibakteria dalam filem bionanokomposit. Pelbagai kepekatan pati minyak kayu manis (0 - 2.0 wt %) dinilai terhadap aktiviti mekanikal, morfologi, mudah terbakar, termal dan antibakteria. Keberkesanan pembungkusan antibakteria ditentukan oleh diameter zon penghadangan; lebih lebar diameter, lebih sensitif bakteria kepada pembungkusan. Ujian antibakteria dilakukan dengan kaedah ADM untuk menilai kesan penghadangan bagi filem terhadap bakteria gram positif, bakteria gram negatif dan yis. Keputusan menunjukkan bahawa kandungan pati minyak yang paling tinggi (2.0 wt %) di dalam kandungan filem menghasilkan zon penghadangan tertinggi iaitu, 7.85, 6.63, dan 7.43 mm untuk *B. subtilis*, *S. aureus* dan *E. coli*. Dalam keadaan vakum, analisis imbasan mikroskopi electron bagi filem yang mengandungi pati minyak kayu manis menunjukkan kemunculan bintik-bintik mikro-poros sebagai lubang mikro-berpori kerana berlakunya penyejatan separa pada permukaan fraktur krio. Peningkatan kepekatan pati minyak kayu manis menghasilkan tren peningkatan bagi bilangan dan saiz lubang berliang mikro. Peningkatan ketara dilihat pada kestabilan terma dengan penambahan pati minyak kayu manis. Kestabilan terma bagi komposit biopolimer CEO/SPNCC/SPS meningkat dengan ketara bergantung kepada peningkatan kandungan CEO (sampel 5,  $T_{max}=296.25^{\circ}\text{C}$ ) berbanding dengan (sampel 1,  $T_{max}=289.18^{\circ}\text{C}$ ) Mikrograf FESEM menunjukkan filem yang agak baik dengan ciri-ciri yang sedikit kekuningan dan lut sinar. Secara keseluruhan, filem bionanokomposit yang diperbuat daripada gabungan kanji/nanoselulosa enau serta ditambah dengan pati minyak kayu manis berpotensi untuk meningkatkan kesesuaiannya dalam aplikasi 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 Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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## Declaration by Members of Supervisory Committee

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- the research conducted and the writing of this thesis was under our supervision;
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## LIST OF SYMBOLS

A	Area of film exposed (m <sup>2</sup> )
cm <sup>3</sup>	Centimeter cube
°	Degree
°C	Degree celcius
ρ	Density
t	Duration for permeation (s)
W <sub>i</sub>	Initial weight
W <sub>f</sub>	Final weight
d	Film thickness (mm)
g	Gram
h	Hour
mm	millimeter
MPa	Mega pascal
T <sub>Onset</sub>	Onset temperature
P	Water vapor partial pressure across the films (Pa)
%	Percentage
M <sub>f</sub>	Mass final
M <sub>i</sub>	Mass initial
W <sub>L</sub>	Weight loss
W <sub>o</sub>	Weight before being buries
W <sub>t</sub>	Weight after being buried
wt %	Weight percentage
T <sub>max</sub>	Temperature max

## LIST OF ABBREVIATIONS

ADM	Agar disc method
ANOVA	Analysis of Variance
ASTM	American Society for Testing and Materials
DDM	Disk diffusion method
DSC	Differential Scanning Calorimetry
DTG	Derivative thermogravimetry
EN ISO	European Union-International Organisation for Standardisation
EO	Essential oil
EOCs	Essential oil constituents
FESEM	Field scanning electron microscope
FTIR	Fourier Transform Infrared
IEC	International Electrotechnical Commission
MIC	Minimum inhibitory concentration
PVA	polyvinyl alcohol
SEM	Scanning Electron Microscopy
SPF	Sugar palm fibre
SPNCC	Sugar palm nanocrystalline celluloses
SPS	Sugar palm starch
TGA	Thermal-gravimetric analysis
TPS	Thermoplastic Starch
TS	Tensile strength
WA	Water absorption
WC	Water content
WS	Water solubility
WVP	Water vapour permeability

## CHAPTER 1

### INTRODUCTION

#### 1.1 Background

Food quality and freshness products may change by numerous unwanted physical and chemical properties change before, during or after processing stages. The biodegradable film keeps food quality and an environmentally friendly packaging ( Ilyas et al, (2018a); Ilyas et al., (2018b); Resianingrum et al. (2016); Sanyang et al., (2018)). Packaging plays an important role in ensuring food safety and quality, and the development of active packaging, especially antibacterial packaging, enables actively inhibiting/killing the microorganisms causing food spoilage and thus extending the product's shelf life. Antibacterial active packaging is one of the favourable concepts that can prolong the shelf life of food by inhibiting the growth of microbial. Starch-based polymers are potential as feedstock for the huge scale manufacture of bio-plastic film.

The essential oil has a lengthy history of use as a current antibacterial Cox et al. (2000). In current times, it has enlarged its reputation as a harmless, natural and active antibacterial substance. This has directed to a reappearance in popularity and presently it is incorporated as the principal antibacterial or as a natural preservative in many pharmaceutical and cosmetic products proposed for external use Ishak et al., (2011). Test of *Escherichia coli* cells or other microbes using electron microscopy after contact with essential oil discovered a damage of cellular electron-dense material and coagulation of cytoplasmic parts, although it was apparent that these effects were secondary actions that happened after cell death Cox et al. (2000). In addition, various antibacterial additive type of essential oils (EO) and concentration were implemented into the nanocomposite films to defeat microbial activities in the films. An edible film with antibacterial characteristics has potential to prevent contamination in food product Resianingrum et al., (2016). The researcher targeted small amounts of EO used to minimise costs while having higher mechanical properties that allow the film to maintain its structure for a long time. Antibacterial activity was observed to be more active against the bacteria tested than the control film to extend product shelf life (Restrepo et al., 2018). Thus, biocomposite polymer based materials for food packaging is explored to address environmental pollution from non-biodegradable food packaging materials. The main driving factor for the commercialization of biodegradable plastics in many countries is government laws related to environmentally products.

Starch is not meltable in its pure form and thus cannot be processed as thermoplastic. Hence, products made from native starches easily crumble into bits when dried in ambient conditions. The process of curing the brittleness of native starches is known as plasticization of starch. Therefore, plasticization is repeatedly employed for the modification of starches to further upgrade their processability and other properties. Plasticizers are non-volatile compounds with low molecular weight. The ultimate role of

plasticizers is to enhance the flexibility and processibility of starch by reducing strong intermolecular interactions between starch molecules Sanyang et al. (2015).

## 1.2 Problem Statements

Recently many scientists and polymer engineers focus on developing sustainable eco-friendly packaging based on renewable natural biopolymers. When looking at the issue from a bigger picture, i.e. the whole food chain, packaging plays a key role in reducing food losses. Biodegradable films for packaging still have been reported not to have antibacterial agent. Such drawback strongly limits their wide application, especially for food packaging purposes. This is because foods served on our tables are subjected to various risks, including physical damage, physicochemical deterioration, microbial spoilage, and particularly, contamination/cross-contamination of pathogens could cause severe threats to human health. It is believed that the amount of packaging wastes generated in industrialized countries in a single day is sufficient to fill up a space equivalent to the Sears Tower, Chicago, USA, which was once the tallest building in the world Rosa et al. (2010). To minimize food losses, a number of practical measures have been proposed to consumers, for example, planning before shopping, avoiding the temptation of special offers coming with large quantity packs, decreasing the cooking/warming proportions in one's diet Günkaya & Banar, (2016).

Sugar palm is a multipurpose tree found in most South East Asian countries and it is regarded as a potential source of natural fibre and biopolymer. However, such a bio-source is still underutilized and, thus, very limited studies have been reported related to their development as a green packaging material. Hence, sugar palm starch (SPS) incorporated with cinnamon essential oil was modified and employed in the current study to develop fully biodegradable films and nanocomposite films as environmentally friendly packaging material for the food industry. This study focused on the SPS based films incorporated with cinnamon essential oil for food packaging industries to prolong the shelf life of products and reduce the risk of pathogenic bacteria growth on food surfaces. Biodegradable composite films with antibacterial properties can be made up of three groups of substances: biopolymers (polysaccharides, proteins, fats), additives (plasticizers, emulsifiers), and synthetic or natural antibacterial. The introduction of essential oils into food packaging films form strong prevention of bacterial and fungi properties, positively affecting the storage of food products. The reinforcement of natural biopolymers with nanocellulose has been shown to be beneficial for reducing water transmission properties of biodegradable film.

The major challenges for the development of starches as packaging films are the shortcomings related to brittleness, processability, high moisture sensitivity, quick retrogradation, poor mechanical and barrier properties. Pure sugar palm starch (SPS) cannot be directly used as thermoplastic materials for film preparation without addition of plasticizer. It is like most other biopolymers, which is hydrophilic in nature due to either their hydroxyl or polar groups. The addition of different types and concentrations of plasticizer(s) into the starch matrix and the reinforcement of nanocellulose fibre would help to transform native SPS into high performance thermoplastic starch for food packaging application.

### **1.3 Research objectives**

The principal aim of this study is to develop and characterize totally biodegradable and sustainable materials based on natural resources to be utilized in antibacterial active packaging applications. The research objectives can be specified into:

1. To determine the antibacterial activity, physical, mechanical and barrier properties of sugar palm based nanocellulose/starch biocomposite films incorporated with different concentrations of cinnamon essential oil based on fixed amount of plasticizer and nanocellulose.
2. To evaluate the thermal, morphological and flammability properties of sugar palm based nanocellulose/starch biocomposite films incorporated with different concentrations of cinnamon essential oil based on fixed amount of plasticizer and nanocellulose.
3. To characterize the effect of plasticizer type and concentration on selected properties of sugar palm nanocellulose reinforced sugar palm starch composites incorporated with cinnamon essential oil.
4. To develop and characterize the effect of sugar palm nanocellulose/ essential oil (SPNCC/EO) loading on the physical, water barrier properties, biodegradability and morphological properties of sugar palm nanocrystalline cellulose reinforced sugar palm starch incorporated with cinnamon EO composites for packaging purposes.

### **1.4 Significance of study**

1. The current study's findings are likely to contribute to the development of high performance antibacterial biodegradable polymers generated from sugar palm starch reinforced nanocellulose incorporated cinnamon EO for food packaging applications.
2. The development of biodegradable antibacterial polymers with better qualities in this work is intended to aid in addressing environmental issues related to replacement materials for petroleum-based polymers.
3. In term of waste management issue, this research provides platform for utilizing wastes from agricultural products into nanofibres and biopolymers, which these biocomposites are low-cost, biodegradable, environmentally friendly, and renewable.
4. The successful development of such green materials from sugar palm tree would provide opportunities to improve the standard of living of the sugar palm tree farmers in Malaysia by generating non-food source of economic development for rural areas.



5. This research may also add to the effort to uncover the potential of using sugar palm starch in developing green products with antibacterial properties; else, such abundant bioresources may be underutilized.

## **1.5 Scope of Study**

In this study, sugar palm fibre (SPF) and sugar palm starch (SPS) were manually extracted from the sugar palm tree. Then, the sugar palm cellulose (SPC) were extracted from sugar palm fibres and the yield was optimized using delignification and mercerization process. The best properties of the obtained cellulose were subsequently hydrolyzed (sulphuric acid solution) and mechanically homogenised (high pressurise homogenization) to isolate sugar palm nanocrystalline cellulose (SPNCC).

Thermoplastic sugar palm starch was developed by using solution casting method with the addition of glycerol, sorbitol and essential oil. The characterized biocomposite film of SPNCC and cinnamon EO were used as reinforcement for SPS films to improve the matrix and antibacterial properties. The optimal loading ratio of the obtained composite films was then selected based on the physical and tensile properties supported by morphological, structural, antibacterial properties and thermal properties. Thus, the effect of various concentration of cinnamon EO (0.8, 1.2, 1.6, 2.0 wt %) also types (sorbitol, glycerol and sorbitol + glycerol) and concentrations (1.5, 3.0 and 4.5 wt %) of plasticizer on the physical, mechanical, water barrier, thermal and biodegradable properties of SPS based composite films was carried out. The criteria for selection were mainly based on the best physical and tensile properties as well as good antibacterial activity.

## **1.6 Structure of thesis**

The structure of this thesis is in accordance with the alternative thesis format of Universiti Putra Malaysia which is based on the publications of this study. Each research chapter represents a separate study that has its own: 'Introduction', 'Materials and method', 'Results and discussion', and 'Conclusions'. The details structure of the thesis are presented below.

### **Chapter 1**

The concerns that prompted this research, as well as the research objectives, were clearly stated in this chapter. This chapter further elaborated on the significance of this work and the scope of study.

### **Chapter 2**

This chapter offers a comprehensive literature review on previous research works related to the current study. Furthermore, the research gaps identified throughout the review were clarified inside this chapter.

### **Chapter 3**

This chapter describes the methods utilised in this study for material preparation, testing procedures, and data collection.

### **Chapter 4**

This chapter presents the first article entitled “Antibacterial activity, physical, mechanical and barrier properties of sugar palm based nanocellulose/starch biocomposite films incorporated with cinnamon essential oil”. In this article, the antibacterial activity of biocomposite were investigated.

### **Chapter 5**

This chapter presents the second article entitled “Thermal, flammability, and morphological properties of nanocellulose fibre reinforced starch biopolymer composites incorporated with cinnamon essential oil”. In this article, the thermal, flammability, and morphological properties were investigated.

### **Chapter 6**

This chapter present the third article entitled “Effect of plasticiser on properties of sugar palm nanocellulose/cinnamon essential oil reinforced starch biocomposite films”. In this article, the effect of various plasticizer types and concentrations were investigated.

### **Chapter 7**

This chapter presents the forth article entitled “Water barrier and biodegradable properties of cinnamon essential oil/sugar palm nanocellulose/starch biopolymer composites”. In this article, the biodegradable characteristics of the biocomposite film were investigated.

### **Chapter 8**

This chapter covers the overall findings and conclusion of the study, as well as future recommendations for improving the study.

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