UNIVERSITI PUTRA MALAYSIA

ACTIVE PAPER COATING BASED ON FISH GELATIN, PALM WAX AND LEMONGRASS ESSENTIAL OIL FOR GROUND BEEF PACKAGING APPLICATION

NURUL SYAHIDA BINTI SAHID

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By

NURUL SYAHIDA BINTI SAHID

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

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

ACTIVE PAPER COATING BASED ON FISH GELATIN, PALM WAX AND LEMONGRASS ESSENTIAL OIL FOR GROUND BEEF PACKAGING APPLICATION

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NURUL SYAHIDA BINTI SAHID

July 2020

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

In recent years, growing environmental concern regarding the disposal problem of petroleum-based packaging has led to increasing interests towards the use of biopolymers in the food packaging industry. Thus, this study focused on the development of an active paper coating based on natural and biodegradable substances which include fish gelatin (FG), palm wax (PW) and lemongrass essential oil (LEO). The experimental design follows these successive steps: (1) the effects of PW concentration (0-60%) on the physicomechanical and water barrier properties of FG films was determined, (2) different LEO concentrations (0-12%) was incorporated into film coating and its effects on the physicomechanical, water resistance and functional properties of Kraft paper was analysed, and (3) the efficiency of the gelatin/palm wax/lemongrass essential oil (GPL)-coated Kraft paper in maintaining the quality of ground beef during 7 days of storage at 4℃ was evaluated. In the first objective, FG was incorporated with different PW concentrations (0-60%). The gelatin/palm wax (GP) films were significantly ($p<0.05$) thicker, more opaque, stronger, more flexible and had better resistance towards water and ultraviolet radiation compared to the control film. These characteristics increased significantly ($p<0.05$) with higher PW concentrations except for the tensile strength which decreased significantly ($p<0.05$). The results revealed that the GP films incorporated with 15% PW exhibited the best property improvements and was chosen as the film coating on Kraft paper for the second objective. The film coating was incorporated with different concentrations of LEO (0-12%) and the properties of the GPL-coated Kraft paper were evaluated. The GPL film coating significantly ($p<0.05$) increased the physicomechanical, water barrier and antioxidant properties of the Kraft paper and this effect was more prominent ($p<0.05$) with higher LEO concentration. However, no antimicrobial property was observed in all paper samples. The GPL-coated Kraft paper incorporated with 12% LEO outperformed the other treatments as an active packaging. Thus, it was used for the following objective in which the effectiveness of the GPL-coated Kraft paper in maintaining the quality of ground beef during 7 days of storage at 4℃ was determined. The properties of the ground beef packed with GPL-coated Kraft paper (GPL/K) were compared to those packed with uncoated Kraft paper

(K) and GP-coated Kraft paper without LEO (GP/K). Ground beef that was not packed with any paper packaging was used as control (C). The pH, moisture content and colour properties (L^*, a^* and b^*) of all samples significantly ($p<0.05$) decreased whereas the total bacterial count, peroxide value and TBARS value significantly ($p<0.05$) increased with storage time. Nevertheless, compared to the other treatments, the GPL-coated Kraft paper incorporated with 12% LEO was able to significantly ($p<0.05$) delay all the property changes of the ground beef throughout the 7 days of storage. Thus, the use of GPL-coated Kraft paper could help to effectively maintain the quality of ground beef during chilled storage.

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

SALUTAN KERTAS AKTIF BERASASKAN GELATIN IKAN, LILIN SAWIT DAN MINYAK PATI SERAI UNTUK APLIKASI PEMBUNGKUSAN DAGING LEMBU CINCANG

Oleh

NURUL SYAHIDA BINTI SAHID

Julai 2020

Pengerusi : Nur Hanani Binti Zainal Abedin, PhD
Fakulti : Sains dan Teknologi Makanan

Sejak beberapa tahun yang lalu, kerisauan tentang masalah pembuangan pembungkus berasakan petroleum telah meningkatkan minat terhadap penggunaan biopolimer dalam industri pembungkusan makanan. Oleh itu, kajian ini memberi tumpuan kepada pembangunan salutan kertas aktif berasaskan bahan semula jadi dan boleh terbiodegradasi yang mana meliputi gelatin ikan (FG), lilin sawit (PW) dan minyak pati serai (LEO). Reka bentuk eksperimen mengikuti langkah-langkah berikut: (1) kesan kepekatan PW yang berbeza (0-60%) terhadap sifat fizikomekanikal dan rintangan air filem FG telah dikaji, (2) LEO dengan kepekatan berbeza (0-12%) telah ditambah ke dalam salutan filem dan kesannya terhadap sifat fizikomekanikal, rintangan air dan ciri-ciri fungsian kertas Kraft telah dianalisis, dan (3) keberkesanan kertas Kraft bersalut gelatin/lilin sawit/minyak pati serai (GPL) dalam mengekalkan kualiti daging lembu cincang selama 7 hari penyimpanan pada suhu 4 ℃ telah dinilai. Dalam objektif pertama, filem FG telah ditambah dengan kepekatan PW yang berbeza (0-60%). Filem gelatin/lilin kelapa sawit (GP) adalah ketara ($p<0.05$) lebih tebal, legap, kuat dan fleksibel serta mempunyai daya ketahanan yang lebih baik terhadap air dan sinaran ultraungu berbanding filem kawalan. Ciri-ciri ini meningkat dengan ketara ($p<0.05$) pada kepekatan PW yang lebih tinggi kecuali TS yang menurun dengan ketara ($p<0.05$). Keputusan analisis menunjukkan bahawa filem GP dengan 15% PW menunjukkan peningkatan ciri-ciri yang terbaik dan telah dipilih sebagai salutan filem untuk objektif kedua. Salutan filem telah ditambah dengan LEO dengan kepekatan yang berbeza (0-12%) dan ciri-ciri kertas Kraft bersalut GPL telah dinilai. Salutan filem GPL dengan ketara ($p<0.05$) telah meningkatkan ciri-ciri fizikomekanikal, rintangan air dan sifat antioksidan kertas Kraft dan kesan ini dapat dilihat dengan lebih ketara ($p<0.05$) pada kepekatan yang lebih tinggi. Walau bagaimanapun, semua sampel kertas tidak menunjukkan sebarang ciri-ciri antimikrob. Ciri-ciri kertas Kraft bersalut GPL yang ditambah dengan 12% LEO mengatasi yang lain sebagai pembungkusan aktif. Oleh itu, ia digunakan untuk objektif seterusnya yang mana keberkesanan kertas Kraft bersalut GPL dalam mengekalkan kualiti daging lembu cincang selama 7 hari penyimpanan pada suhu 4 ℃ telah dikaji. Sifat daging lembu cincang yang dibungkus bersama kertas

bersalut GPL (GPL/K) dibandingkan dengan sampel yang dibungkus bersama kertas yang tidak bersalut (K) dan kertas Kraft bersalut GP tanpa LEO (GP/K). Daging lembu cincang yang tidak dibungkus menggunakan sebarang kertas bungkusan digunakan sebagai kawalan (C). Kadar pH, kandungan kelembapan dan ciri-ciri warna (*L**, *a** and *b**) semua sampel menurun dengan ketara ($p<0.05$) sementara jumlah kiraan bakteria, nilai peroksida dan nilai TBARS meningkat dengan ketara ($p<0.05$) mengikut pertambahan masa penyimpanan. Walau bagaimanapun, berbanding dengan kumpulan lain, kertas Kraft bersalut GPL dengan 12% LEO mampu menangguhkan semua perubahan ciri-ciri daging lembu cincang dengan ketara ($p<0.05$) sepanjang 7 hari penyimpanan. Oleh itu, penggunaan kertas bersalut GPL dapat membantu mengekalkan kualiti daging lembu cincang semasa penyimpanan sejuk.

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

Nur Hanani binti Zainal Abedin, PhD
Associate Professor
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Chairman)

Ismail-Fitry bin Mohammad Rashedi, PhD
Senior Lecturer
Faculty of Food Science and Technology
Universiti Putra Malaysia
(Member)

Ainun Zuriyati binti Mohamed @ Asa'ari, PhD
Research Officer
Institute of Tropical Forestry and Forest Products
Universiti Putra Malaysia
(Member)

ZALILAH MOHD SHARIFF, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 08 October 2020

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Signature: _____ Date:_____

Name and Matric No.: Nurul Syahida Binti Sahid GS49450

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Signature: _____
Name of
Chairman of
Supervisory
Committee: Associate Prof. Dr. Nur Hanani binti Zainal Abedin

Signature: _____
Name of
Member of
Supervisory
Committee: Dr. Ismail-Fitry bin Mohammad Rashedi

Signature: _____
Name of
Member of
Supervisory
Committee: Dr. Ainun Zuriyati binti Mohamed @ Asa'ari

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LIST OF ABBREVIATIONS

ΔE	Colour difference
ABTS	2,2'-azino-bis-(3-ethylbenzothiazoline-6-sulfonic acid
AFM	Atomic force microscopy
ANOVA	Analysis of variance
AOAC	Association of Official Analysis Chemists
ASTM	American Society for Testing and Materials
ATCC	American Type Culture Collection
ATR	Attenuated total reflectance
BHT	Butylated hydroxytoluene
BSE	Bovine Spongiform Encephalopathy
C	Unwrapped ground beef sample
CFU	Colony forming unit
CIE	Commision Internationale de I'Elcairage
DPPH	2,2-diphenyl-1- picrylhydrazyl
EAB	Elongation at break
EDTA	Ethylene diamine tetraacetic acid
e.g.	For example
Eq.	Equation
eV	Electronvolt
EVOH	Ethylene vinyl alcohol
FESEM	Field electron scanning microscopy
FFS	Film-forming solution
FG	Fish gelatin
FTIR	Fourier Transform Infrared

GAE	Gallic acid equivalent
GP	Gelatin/palm wax
GP/K	Ground beef sample wrapped in gelatin/palm wax-coated Kraft paper
GPL	Gelatin/palm wax/lemongrass essential oil
GPL/K	Ground beef sample wrapped in gelatin/palm wax /lemongrass essential oil-coated Kraft paper
GRAS	Generally recognised as safe
IDF	International Dairy Federation
ISO	International Organisation for Standardisation
K	Ground beef sample wrapped in uncoated Kraft paper
LDPE	Low-density polyethylene
LEO	Lemongrass essential oil
MAP	Modified atmosphere packaging
MDA	Malondialdehyde
meq	Milliequivalent
NIST	National Institute of Standards and Technology
PBAT	Poly(butylene adipate-co-terephthalate)
PE	Polyethylene
PET	Polyethylene terephthalate
PLA	Poly (lactic acid)
PP	Polypropylene
PVC	Polyvinyl chloride
PVdC	Polyvinylidene chloride
PW	Palm wax
R_a	Average roughness
RH	Relative humidity

rpm	Revolutions per minute
R_q	Root-mean-square roughness
SEM	Scanning electron microscopy
TAPPI	Technical Association of the Pulp and Paper Industry
TBARS	Total thiobarbituric acid reactive substances
TMBC	Total mesophilic bacterial count
TMP	1,1,3,3-tetramethoxypropane
TPBC	Total psycrotrophic bacterial count
TPC	Total phenolic content
TS	Tensile strength
TSM	Total soluble matter
TVC	Total viable count
UC	Uncoated
USA	United States of America
USD	United State Dollars
UV	Ultraviolet
v/v	Volume per volume
w/v	Weight per volume
w/w	Weight per weight
WVP	Water vapour permeability
YM	Young's modulus

CHAPTER 1

INTRODUCTION

Petroleum-based plastics are extensively used in the packaging industry due to their excellent mechanical and barrier properties (Hahladakis & Iacovidou, 2018). However, the non-biodegradable nature of plastic may result in overaccumulation of this synthetic waste and the only effective disposal method is through combustion or pyrolysis (Geyer, Jambeck, & Law, 2017). Although landfilling and incineration can be conducted in controlled facilities, illegal dumping and open burning remain as a major problem globally (Song, Zhang, Duan, & Xu, 2018; Wierckx et al., 2015). A report by Lebreton and Andrady (2019) revealed that approximately 60-99 million metric tonnes of municipal plastic wastes were dumped into the environment in 2015. The plastic debris accumulated on land may be transported by the wind or tides and eventually enters the ocean (Jambeck et al., 2015). Thus, reliance on plastics will create a huge strain on the natural resources and environment if no sustainable alternative is used.

Over the past few years, paper has been gaining popularity as a food packaging material due to its biodegradability and the renewability of its raw material (Lavoine, Desloges, Khelifi, & Bras, 2014). It is a low-cost material that offers excellent mechanical properties for various packaging applications (Teck Kim, Min, & Won Kim, 2014). Kraft paper, in particular, is widely used as bags, sacks, wrappers, fibreboards and various other food packagings due to its exceptional resistance to tearing and tensile forces (Dagnon, Thellen, Ratto, & D'Souza, 2010). However, the application of Kraft paper in the food packaging industry is limited because of its high hydrophilicity and oxygen permeability (Vaezi, Asadpour, & Sharifi, 2019). Therefore, Kraft papers are often coated with a hydrophobic coating to improve their water and gas barrier properties.

The use of biodegradable biopolymers as paper coating could help resolve the waste disposal problems related to petroleum-based materials to a considerable extent. One of the most promising biopolymers is fish gelatin (FG), which is produced via partial hydrolysis of collagen obtained from the skins and bones of fish (Adilah, Jamilah, Noranizan, & Hanani, 2018). It has an excellent film-forming ability as well as good mechanical and gas barrier properties that are beneficial for food packaging application (Nilsuwan, Guerrero, de la Caba, Benjakul, & Prodpran, 2019). However, the main drawback with FG lies in its poor water barrier property, which limits its application to foods with low moisture content.

Various approaches have been taken to improve the water resistance property of FG film. One of the most popular methods is by blending it with hydrophobic wax to produce a composite film that combines the benefits of both components. Natural waxes in the market so far are biodegradable and renewable but are often expensive and limited in terms of supply. Palm wax (PW), which is derived from the fruit of oil palm (*Elaeis guineensis* Jacq.) (Basri, Abd Rahman, & Salleh, 2013), is a low-cost option that is readily available due to the high productivity of the oil palm (Yuan, Arondel, & Domergue, 2019). Palm wax has outstanding hydrophobic properties which can be

attributed to the high content of wax esters, long-chain fatty acids and fatty alcohols. Its low melting temperature (58-60℃) enables it to be used with other thermosensitive active compounds that require a low processing temperature.

The functional properties of a food packaging material can be improved by incorporating natural active agents into the film formulation. These active agents can eliminate any undesirable factors such as moisture, oxygen and microorganisms that may cause food degradation (Eskandarabadi et al., 2019). This will provide an optimum condition for maintaining the quality and safety of the food products. Lemongrass essential oil (LEO), in particular, has a great potential to be used as part of active packaging materials due to its capability in reducing microbial growth and lipid oxidation, which are among the main causes of food deterioration. The antimicrobial and antioxidant activities of LEO are mainly contributed by its high citral content, which is a monoterpene aldehyde that is capable of damaging microbial cell membrane as well as scavenging free radicals (Kumar et al., 2018; Mpho, Sivakumar, Sellamuthu, & Bautista-Baños, 2013). Thus, the use of this active packaging on perishable food products such as meat and poultry could be an interesting application as it is not only capable of inhibiting food-borne pathogenic microorganisms, but also lipid oxidation and any undesirable colour change (Navikaite-Snipaitiene et al., 2018).

In this study, an active packaging based on natural and biodegradable materials was developed. The experimental design is shown in Appendix A and the three main objectives are:

1. To evaluate the effect of different concentrations of palm wax on the physicomechanical and water barrier properties of fish gelatin film.
2. To determine the effect of active coating based on fish gelatin, palm wax and lemongrass essential oil on the physicomechanical and functional properties of Kraft paper.
3. To evaluate the effectiveness of active coated paper in maintaining the quality of ground beef during storage at a chilled temperature (4℃).

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BIODATA OF STUDENT

Nurul Syahida was born on the 22nd November 1994 in Selangor, Malaysia. She received her primary and secondary education in Sekolah Kebangsaan Jalan Raja Musa and Sekolah Menengah Kebangsaan Agama Kuala Selangor, respectively. She then entered the Centre for Foundation Studies in Science in 2012 for her Foundation in Life Science studies before enrolling in University of Malaya to get a degree in Bachelor of Science (Biotechnology). After graduating in 2017, she pursued her study in Universiti Putra Malaysia under the programme of Master of Science (Food Technology). She believes that the food industry holds a promising future and she sincerely hopes that her bits of knowledge could contribute to the development and improvement of the industry

LIST OF PUBLICATIONS

Nurul Syahida, S., Ismail-Fitry, M. R., Ainun, Z. M. A., & Nur Hanani, Z. A. (2021). Effects of gelatin/palm wax/lemongrass essential oil (GPL)-coated Kraft paper on the quality and shelf life of ground beef stored at 4°C. *Food Packaging and Shelf Life*, *28*, 1-7.

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