



**THE STUDY OF CHEMICAL MODIFICATIONS  
ON THE PROPERTIES OF SOY PROTEIN  
ISOLATE (SPI)/KAPOK HUSK (KH)  
BIOCOMPOSITE FILMS FOR AGRICULTURE  
APPLICATIONS**

by

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Doctor of Philosophy

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## LIST OF ABBREVIATIONS

AC	Adipic acid
ASTM	American Society for Testing and Materials
CSI	Commercial soy isolate
DP	Degree of polymerization
EA	2-Ethylhexyl Acrylate
EB	Elongation at break
EG	Ethylene glycol
FA	Formaldehyde
FFS	Film forming solution
FTIR	Fourier Transform Infrared Spectroscopy
KH	Kapok husk
LCA	Life cycle assessments
MC	Moisture content
MMA	Methyl Methacrylate
MW	Molecular weight
PA	Phthalic anhydride
PEG	Polyethylene glycol
PG	Propylene glycol
PS	Protein solubility
SDS	Sodium Dodecyl Sulphate
SEM	Scanning electron microscopy
SPI	Soy Protein Isolate
TGA	Thermogravimetry analysis
TS	Tensile strength
TSM	Total soluble matter
WPI	Whey protein isolate
WVP	Water vapour permeability

## LIST OF SYMBOLS

%	Percentage
$M_o$	Weight before drying for total soluble matter
$M_i$	Weight after drying for total soluble matter
$N_o$	Weight of specimens before swelling for gel fraction
$N_1$	Weight of dried gel for gel fraction
$T_g$	Glass transition temperature
$W_o$	Weight before drying for moisture content
$W_i$	Weight after drying for moisture content
$Z_o$	Initial weight loss of a specimen before enzymatic degradation
$Z_1$	Weight loss of a specimen after enzymatic degradation

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(PST)/Sekam Kekabu (SK) Untuk Aplikasi Pertanian**

**ABSTRAK**

Dalam kajian ini, penggunaan sekam kekabu (SK) sebagai pengisi dalam protin soya terasing (PST) telah dikaji untuk menggantikan plastik dan filem polimer bersumber petroleum untuk pembungkusan. Kesan daripada kandungan SK, agen-agen sambung silang seperti ptalik anhidrida (PA), asid adipik (AA) dan formaldehid (FA), dan modifikasi kimia terhadap SK dengan 2-etilhexil akrilat (EA), metil metakrilat (MMA) dan sodium dodecyl sulfat (SDS) pada sifat-sifat tegangan, kajian morfologi, analisis thermogravimetri (TGA), kandungan kelembapan, jumlah bahan larut, pecahan gel dan sifat-sifat biodegradasi enzim filem-filem biokomposit PST/SK telah dikaji. Gliserol telah digunakan sebagai pemplastik untuk memberikan fleksibiliti kepada filem-filem biokomposit itu. Keputusan eksperimen menunjukkan bahawa peningkatan kandungan SK telah meningkatkan kekuatan tegangan, elastisiti modulus, dan pecahan gel daripada filem-filem biokomposit PST/SK kawalan, manakala pemanjangan pada takat putus, sisa arang, kandungan kelembapan, jumlah bahan larut dan berat biorosoton pengenziman menurun. Morfologi permukaan keretakan selepas pengajian kekuatan tegangan daripada filem-filem biokomposit PST/SK kawalan daripada kandungan SK yang lebih tinggi menunjukkan interaksi yang lebih baik antara SK pengisi dan SPI matrik. Penambahan agen-agen sambung silang yang berbeza seperti PA, AA dan FA telah meningkatkan kekuatan tegangan dan elastisiti modulus bagi filem-filem biokomposit tersambung silang. Pecahan gel daripada semua filem-filem biokomposit tersambung silang lebih tinggi daripada filem-filem biokomposit yang tidak tersambung silang. Walau bagaimanapun, biokomposit tersambung silang menunjukkan susutan dalam pemanjangan pada takat putus kecuali AA, sisa arang, kandungan kelembapan, jumlah bahan larut, dan pengurangan berat pada biorosotan di dalam pengenziman daripada filem-filem biokomposit yang tidak tersambung silang. Kajian pengimbasan mikroskop electron (PME) bagi filem-filem biokomposit PST/SK tersambung silang dengan PA, AA dan FA menunjukkan permukaan kasar, disebabkan tahap sambung silang dipertingkatkan. Spektra FTIR bagi kesemua filem-filem biokomposit sambung silang mengilustrasikan perubahan-perubahan di dalam kumpulan berfungsi. SK terawat dengan EA, MMA, dan SDS filem-filem biokomposit mempunyai kekuatan tegangan yang lebih tinggi dan elastisiti modulus berbanding dengan yang tidak terawat PST/SK filem-filem biokomposit. Walau bagaimanapun pemanjangan pada takat putus, sisa arang dari TGA, kandungan kelembapan, jumlah bahan larut dan pengurangan berat pada biorosotan di dalam pengenziman telah menurun. Semua filem-filem biokomposit PST/SK yang terawat telah menunjukkan sedikit perubahan sahaja dalam pecahan gel apabila kandungan SK bertambah. SK terawat dengan EA, MMA dan SDS telah meningkatkan ikatan permukaan antara SK dan PST matrik dalam filem-filem biokomposit, yang telah dibuktikan dengan analisis PME. Ini boleh diringkaskan bahawa pencampuran SK dalam filem PST mempunyai keupayaan untuk meningkatkan sifat-sifat filem-filem biokomposit PST/SK. Walau bagaimanapun dalam perbandingan, filem-filem biokomposit PST/SK modifikasi dengan EA telah memberikan penambahbaikan tertinggi dalam semua pencirian.

## **The Study Of Chemical Modifications On The Properties Of Soy Protein Isolate (SPI)/Kapok Husk (KH) Biocomposite Films For Agriculture Applications**

### **ABSTRACT**

In this research, the utilization of kapok husk (KH) as a filler in soy protein isolate (SPI) was studied to replace the plastic and petroleum based polymer films for packaging. The effect of KH loading, crosslinking agents such as phthalic anhydride (PA), adipic acid (AA) and formaldehyde (FA), and chemical modification of KH with 2-ethylhexyl acrylate (EA), methyl methacrylate (MMA) and sodium dodecyl sulphate (SDS) on tensile properties, morphology, thermogravimetry analysis (TGA), moisture content, total soluble matter, gel fraction and enzymatic biodegradation properties of SPI/KH biocomposite films was studied. Glycerol was used as a plasticizer to give flexibility to the biocomposite films. The experimental results showed that the increases of KH loading have increased the tensile strength, modulus of elasticity, and gel fraction of control SPI/KH biocomposite films, whereas the elongation at break, char residue, moisture content, total soluble matter and weight loss of enzymatic biodegradation decreased. The morphology of tensile fracture surface of control SPI/KH biocomposite films at higher KH loading showed better interaction between KH filler and SPI matrix. The addition of different crosslinking agents such as PA, AA and FA had increased the tensile strength and modulus of elasticity of crosslinked biocomposite films. The gel fraction of all crosslinked biocomposites films higher than uncrosslinked biocomposite films. However, crosslinked biocomposites exhibited decrement in elongation at break except AA, char residue, moisture content, total soluble matter, and weight loss of enzymatic biodegradation than uncrosslinked biocomposite films. The SEM studies of crosslinked SPI/KH biocomposite films with PA, AA and FA indicated the rough surface, due to the enhanced crosslinking. The FTIR spectra of all crosslinked biocomposite films illustrated the changes in functional group. The treated KH with EA, MMA, and SDS biocomposite films have higher tensile strength and modulus of elasticity in comparison with untreated SPI/KH biocomposite films. Nevertheless the elongation at break, char residue from TGA, moisture content, total soluble matter and weight loss of enzymatic biodegradation reduced. All treated SPI/KH biocomposite films performed slight changes in gel fraction as KH loading increased. The treated KH with EA, MMA and SDS had enhanced the interfacial bonding between KH and SPI matrix in biocomposite films, which were proved by SEM analysis. It can be summarized that incorporation of KH in SPI film has the ability to improve the properties of SPI/KH biocomposite films. However in comparison, SPI/KH biocomposite films treated with EA could give the highest improvement in all characterization.

## CHAPTER 1 : INTRODUCTION

### 1.1 Background

In recent years, the increased of global awareness regarding sustainability issue leads researchers to replace various synthetic commercial applications with environmental friendly and biodegradable polymers and composites (Shivam, 2016; Averous & Pollet, 2012; Thompson et al., 2009). To solve the issues such as polluted marine water, overflowing landfills and plastics litter motivates researches the needs to widen polymers based material with manageable lifetimes by taking consideration into environmentally adequate to application, manufacturing, disposal and recycling methods (Sirvio et al., 2014).

Nowadays biofilms based on agricultural materials gained much consideration as packaging materials. To overcome such environmental problems rather than synthetic polymer packaging, biofilms are accepted to be a promising solution (Koshy et al., 2015). Additionally, biofilms is suitable to commercialized into various products such as garbage bags, grocery bags, composting yard waste bags, agriculture mulches and agro bags (Ali et al., 2013). Soy protein is naturally occurring edible materials that have been broadly investigated due to their properties such as inexpensive, fully biodegradable and availability. From the comparisons between soy protein products, soy protein isolate (SPI) has higher protein content which gives superior ability to form films (Wihodo & Moraru, 2013). From the investigation, researchers have been reported that SPI films have high barrier properties on both vapour and oxygen at low relative humidity and good