

ENHANCEMENT OF POLY (VINYL ALCOHOL) MEMBRANE PACKAGING  
MATERIAL BY USING BETA-CYCLODEXTRIN

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A dissertation submitted in fulfillment of the  
requirements for the award of the degree of  
Master of Engineering (Bioprocess)

Faculty of Chemical Engineering  
Universiti Teknologi Malaysia

APRIL 2014

*Special dedication to my beloved husband, my parent  
Thanks for the love, support and memories*

## ACKNOWLEDGEMENT

In the name of ALLAH S.W.T. and with His blessing and gracing has led my thesis successfully complete. I would like to take this opportunity to express my gratitude to people who have directly or indirectly contributes towards the success of my research project.

First of all, I would like to express my sincere appreciation to my supervisor, **Dr. Tariq Iqbal** for his encouragement and guidance throughout the entire research. I also owe an enormous debt of gratitude to **Dr. Dayang Norulfairuz Abang Zaidel** the years I spent as her student.

Specially, I dedicated to my beloved, my husband for support and patience, and to my parent and my family whom involved directly and indirectly for their love and everlasting unconditional support.

Finally, I would like to thanks all my friends specially **Dr. Davood Almasi** for their motivation, loves and cooperation throughout the hard times. Without those helps, there would be no possible for this thesis to be completed. Thank you.

## ABSTRACT

Poly Vinyl Alcohol (PVA) is becoming popular in these days, due to its low price, easy workability and chemical properties. On the other hand, PVA is categorized as biodegradable polymer. Hence, most of the researchers have been interested to utilize the PVA and PVA blend in food packaging industrial. They can be very good alternative for packaging in food industrials due to their properties. This research is considered to enhance the properties of available packaging materials using blending PVA and  $\beta$ -Cyclodextrin ( $\beta$ -CD). However, in industries the pure PVA is powder and the PVA additives has different mechanical and chemical characteristics as compared to the pure PVA. For solving this problem, the existing commercial method is to cast membrane. Nowadays, the cast technology is used worldwide due to low cost, simple operation, excellent flatness and uniform thickness distribution. Therefore, this research has been conducted to study and analyze the mechanical and chemical characteristics of the pure PVA and the blend PVA with  $\beta$ -CD for using on food packaging. The research is divided into two parts. In the first part, the interfacial adhesion at different ratio between PVA and  $\beta$ -CD was studied. In the second part, the effect of different ratio of  $\beta$ -CD of new blend was investigated. Then, the mechanical and chemical properties of the new blend PVA have been analyzed. Theses analyses have been conducted by tensile, differential scanning calorimeter, Fourier transform infrared spectroscopy and contact angle test. The results are shown that the proposed blend PVA with  $\beta$ -CD are compatible. On the other hand, it shows that using  $\beta$ -CD can improve the thermal, mechanical properties of the packaging, which will be beneficial for the shelf life of food products.

## ABSTRAK

Poly Vinyl Alcohol (PVA) telah digunakan secara meluas kebelakangan ini, kerana harganya yang murah, mudah digunakan serta mempunyai ciri-ciri kimia yang baik. Oleh yang demikian, PVA dan adunan PVA telah diaplikasikan secara meluas oleh para penyelidik dalam industri pembungkusan makanan. PVA digunakan sebagai alternatif dalam pembungkusan makanan kerana sifat-sifatnya yang baik. Dalam penyelidikan ini, ciri-ciri bahan pembungkusan yang tersedia ada diperbaiki dengan menggunakan PVA dan  $\beta$ -Cyclodexsterin ( $\beta$ -CD). Walau bagaimanapun, kebanyakan PVA asli yang digunakan di dalam industri adalah berbentuk serbuk dan biasanya mengandungi ciri-ciri mekanikal dan kimia yang berbeza dari bahan campuran PVA. Untuk mengatasi masalah ini, satu kaedah komersial telah digunakan dengan menghasilkan membran melalui proses pembentukan dalam acuan. Pada masa ini kaedah ini digunakan secara meluas kerana kosnya yang rendah, mudah dikenal, serta keseragaman ketebalan. Oleh itu, penyelidikan ini bertujuan untuk mengkaji dan menganalisa ciri-ciri mekanikal dan kimia bagi PVA asli dan PVA yang telah dicampur dengan  $\beta$ -Cyclodexsterin ( $\beta$ -CD). Penyelidikan ini terbahagi kepada dua bahagian; bahagian pertama mengkaji tentang daya lekatan antara permukaan bagi kadar campuran PVA dan  $\beta$ -CD yang berbeza. Manakala bahagian kedua mengkaji kesan penambahan  $\beta$ -CD pada kadar yang berbeza ke atas campuran baru yang dihasilkan. Seterusnya, ciri-ciri mekanikal dan kimia bagi campuran PVA yang dihasilkan telah dianalisa. Analisis telah dijalankan dengan menggunakan Mesin tegangan, kalorimeter pembezaan imbasan, Fourier sinar inframerah dan ujian contact angle. Melalui kajian ini, campuran PVA dan  $\beta$ -CD yang dicadangkan telah terbukti mampu meningkatkan ciri-ciri terma, mekanikal dan kimia bahan tersebut. Penggunaan  $\beta$ -CD dapat menambah baik ciri-ciri PVA asli, dan seterusnya memberikan manfaat dalam meningkatkan jangka hayat produk makanan.

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**LIST OF ABBREVIATIONS**

ATR	-	Attenuated Total reflectance
ASTM	-	American society for testing and materials
$\beta$ -CD	-	$\beta$ -cyclodextrin
CD	-	Cyclodextrin
DSC	-	Differential scanning calorimetry
FTIR	-	Fourier transform infrared
GA	-	Glutaraldehyde
H <sub>2</sub> SO <sub>4</sub>	-	Hydrogen chloride
ICs	-	Inclusion Complexes
PVA	-	Poly vinyl alcohol
PVAc	-	Poly (vinyl acetate)
PVC	-	Poly (vinyl chloride)
PVN	-	Poly vinyl nitrate
T <sub>g</sub>	-	Glass transition temperature
T <sub>m</sub>	-	Melting temperature
TMA	-	Thermo-mechanical analysis
Wt %	-	Weight percentage

## CHAPTER 1

### INTRODUCTION

#### 1.1 Introduction

Since the last 40 years ago, polymer blends have become a very important part of the commercialization of polymers. Blending of existing polymers and taking advantages of synergy effect of the blends to develop new materials has been a goal for decades (Baker *et al.*, 2001). In 1998, polymer blends constitute over 30% of polymer consumption. Benefits of blending can be discussed from the perspective of material properties and economics and it can bring to the manufacturer. There are many reasons for blending such examples are for modification of specific resin for a specific type of behavior, to gain direct economy by diluting expensive engineering resins with commodity ones and developing materials with a full set of desired properties (Utracki, 2002).

PVA is recognized as one of the very few vinyl polymers that soluble in water and also susceptible of ultimate biodegradation in the presence of suitable acclimated microorganisms (Chiellini *et al.*, 2003). Combination PVA and starch will provide the ideal compound that makes them available for thermoplastic processing and improve their rheological, mechanical and degradation property.

Cyclodextrins are cycles of oligosaccharide produced from enzyme-fermented starch which has successfully been used in drug development and supply (Del Valle, 2004). The remarkable feature of cyclodextrins is their ability to train Include the Complex (ICs) in the solution with a very broad range of sound, liquid, and for gaseous by the phenomenon of the molecular complexes. Among the various types of cyclodextrins,  $\beta$ -cyclodextrins derivative is the most acceptable, the lowest-priced and usually helpful product (Del Valle, 2004).

Combination of these two existing materials and taking advantages of synergy effects of the blends to develop new material is the aim for this research. In developing polymeric mixtures plastic compatible is important since it regulates the morphology of mixtures and thus, affects the physical and mechanical properties of these polymers. Blending of starch and Poly (vinyl alcohol) (PVA) reduce the costs and increase the degradation of the mix film membrane. However, the mechanical resistances of the mixtures reduce due to poor surface adhesion of the materials. The resultant mixture is fragile and rigid, as both have major disadvantages in many food packaging. Same results are expected for mixture of Cyclodextrines (CDs) and PVA. In this research work, the surface adhesion PVA and  $\beta$ -cyclodextrins ( $\beta$ -CDs) in the various weight rates will be examined and the approach for using a master batch (PVA with large  $\beta$ -CD substance) to improve the accession is studied. It will be examined whether exposure to moisture and high temperature during processing can cause early loss of the antibiotic substances wrapped in the  $\beta$ -CD molecules in later version, as this will reduce the antimicrobial activity.

## 1.2 Problem Statement

Research on biodegradable materials based on starch began in the 1970's continuing all over the world. The reason is due to the pollution problem caused by non-biodegradable plastic. Food packaging plays an important role in order to maintain and improve the quality of the food conservation (Comstock *et al.*, 2004).

PVA is known, as a biodegradable thermoplastic polymer that is used in wide application such as medicine and food packaging. PVA is a non-toxic, with high physical and chemical characteristics, however is hard and water soluble that decrease the strength in wet membrane. Little strength and rigidity is an important issue in food packaging, which needed to be considered. Also, package is directly conducted with product and environment, hence protect from water uptake is necessary.

Composite PVA and starch was investigated to produce a new material with excellent mechanical property (Follain *et al.*, 2005). Cyclodextrins produced from enzyme-fermented starch.  $\beta$ -cyclodextrins have a potential to be inclusion complex and protection of incompatibility. Also,  $\beta$ -cyclodextrins act as a hydrophobic agent. Because of crystalline structure and strong biological activity of  $\beta$ -cyclodextrin, combination of PVA and  $\beta$ -cyclodextrin may produce material with excellent membrane properties that beneficial for food packaging.



### 1.3 Objectives

The aim of this research is to enhance the properties of available packaging materials by using PVA and  $\beta$ -cyclodextrins. Different ratios of  $\beta$ -cyclodextrin with the pure PVA are developed in order to achieve the aim of this research. In this regards, the following objectives have been defined:

- To enhance the bio-degradable PVA compound by improving the compatibility between PVA and  $\beta$ -CDs by casting membrane technique.
- To investigate the effect of addition of the  $\beta$ -CDs on the mechanical, thermal, and chemical properties of the PVA membrane.
- To investigate the effect of crosslinking on the mechanical, thermal, and chemical properties of the PVA membrane.

### 1.4 Scope of the Study

The scopes of this research are embarked the formulation of a bio-based polymer made from PVA and  $\beta$ -cyclodextrin. The membrane by different formulation with various ratios of PVA (60-100 wt%) and  $\beta$ -cyclodextrin (0-40 wt%) were prepared by solution casting method. The membranes were crosslinked by Glutaraldehyde (1wt%). The following analysis has been conducted to characterization the membrane:

1. Mechanical Property
  - Tensile test was conducted to determine the strength of membranes.
2. Thermal Property
  - DSC test was conducted to determine the thermal stability of membrane.

3. Spectroscopy Analysis
  - FTIR test was performed to investigate the chemical molecules bonding and compatibilized between PVA and  $\beta$ -cyclodextrin.
4. Contact Angle Analysis
  - Contact angle test was performed to investigate the wettability of the surface membrane.

## 1.5 Thesis Outline

This research contains five chapters, which are organized as follows:

### Chapter 1: Introduction

This chapter is discussed an overview of this study. There are three project objectives that need to successfully achieve as the goals of this research. The scope and importance of this project have also been pointed out.

### Chapter 2: Literature Review

In this chapter, the current study and research on the polymer such as PVA, additive, cast membrane technology and food packaging have been reviewed. The chemical structure of the selected material are presented and investigated. Applications of the material of this research are also studied.

### Chapter 3: Preparation of the Blend Poly Vinyl Alcohol

The proposed method for preparing the membrane poly vinyl alcohol is presented in this chapter. Then, different chemical materials with their specification are investigated that used in this research. The chemical, mechanical and thermal equipment, which are used to analysis the proposed samples have been introduced.

#### Chapter 4: Results and Discussions

In this chapter, the chemical, mechanical and thermal properties of the proposed samples have been conducted. In order to study these tests, the equipment that are explained and introduced in Chapter 3 is utilized to analyze the different test. These tests are tensile, elongation at break, different scanning calorimeter, contact angle and Fourier transformer Infrared Spectroscopy are analyzed.

#### Chapter 5: Conclusions and Furture Works

This chapter comprises the research conclusions as well as the contributions made by this research project. The chapter closes with recommendations for future work that can be undertaken with reference to the work presented in this research.

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