CHARACTERIZATION AND BIODEGRADATION OF POLY (VINYL ALCOHOL)/CASSAVA STARCH

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CHARACTERIZATION AND BIODEGRADATION OF POLY (VINYL ALCOHOL)/CASSAVA STARCH

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Special dedication to my beloved parents, Nor Hassan B. A. Gapar and Sarimahtun Bt. Saliken, siblings, friends and my love…

Thanks for their patience, support, love and memories

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ABSTRACT

A series of poly (vinyl alcohol)/cassava starch (PVA/CS) blends at 50, 60, 70 and 80 wt.% cassava starch contents were prepared in one-step compounding process. One step compounding means, all blends were compound together without solely plasticized PVA or cassava starch at beginning. All blends were characterized and testing accordingly. In this research, comparison between one-step compounding and two-step compounding (plasticized PVA/CS at the beginning) were tested by tensile strength. It shows that their tensile strengths were comparatively higher than the two-step compounding process of PVA/CS blends. In term of impact test, it indicates that CS acts as non-reinforcing filler. The compatibility of PVA/CS represents the similarity functional group and possess same polar group. Studies on thermal stability and crystallinity shows, increasing the cassava starch loading in PVA matrix, has reduced the enthalpy of melting point as well as degree of crystallinity. The onset degradation had shifted to a higher temperature when a higher percentage of cassava starch was used in PVA matrix. This is due to cyclic hemiacetal had been introduced into PVA blend by cassava starch. It resist to thermal attack. In biodegradability test, several factors have been determined to evaluate the rate of biodegradation. Increase the humidity as well as increasing the cassava starch loading in PVA matrix will improve the susceptibility contact of microorganism onto the surface of PVA/CS blend. *Aspergillus Oryzae* had been selected as a good reagent used to degrade the sample of PVA/CS blend compared to *Bacillus Amyloliquefacien*s and effective microorganism. The biodegradation process of PVA/CS blends in anaerobic condition is much more faster compared to aerobic conditions.

ABSTRAK

Penyediaan adunan poli (vinil alkohol)/tepung ubi kayu (PVA/CS) dilakukan pada satu peringkat pemprosesan di mana peratusan berat kandungan tepung ubi kayu: 50, 60, 70 dan 80 wt%. Satu peringkat pemprosesan bermakna, semua campuran di adun secara serentak tanpa melibatkan proses pemplastikan PVA atau tepung ubi kayu pada peringkat awal. Semua adunan PVA/CS telah dicirikan dan diuji sewajarnya. Dalam kajian ini, perbandingan antara satu dan dua peringkat pemprosesan telah diuji dengan kekuatan tegangan. Ia menunjukkan bahawa kekuatan tegangan bagi satu peringkat pemprosesan adalah lebih tinggi berbanding dua peringkat pemprosesan bagi adunan PVA/CS. Bagi ujian hentaman, keputusan menunjukkan bahawa CS bertindak sebagai pengisi bukan pengukuh. Keserasian PVA/CS menggambarkan PVA dan CS mempunyai kumpulan berfungsi dan kutub yang sama. Bagi kajian ke atas kestabilan terma dan penghabluran, ia menunjukkan bahawa peningkatan kandungan tepung ubi kayu di dalam matrik-PVA dapat mengurangkan entalpi takat lebur serta darjah penghabluran. Peralihan degradasi permulaaan kepada suhu yang lebih tinggi turut berlaku disebabkan oleh kitaran *hemiacetal* daripada tepung ubi kayu di dalam adunan PVA/CS. Ia menghalang serangan haba. Dalam ujian terbiodegradasi, beberapa faktor telah ditentukan untuk menilai kadar biodegradasi. Peningkatan kelembapan serta peningkatan kandungan tepung ubi kayu dalam matrik-PVA akan meningkatkan kecenderungan mikroorganisma ke permukaan adunan PVA/CS. *Aspergillus Oryzae* telah dipilih sebagai reagen yang baik yang digunakan untuk degradasi sampel adunan PVA/CS berbanding *Amyloliquefaciens Bacillus* dan mikroorganisma berkesan. Proses terbiodegradasi bagi adunan PVA/CS dalam keadaan anaerobik lebih cepat berbanding dalam keadaan aerobik.

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LIST OF SYMBOLS AND ABBREVIATIONS

ASTM	-	American standard of testing and method
a_w	-	Water activity
CaS	-	Calcium stearate
$C_3H_8O_3$	-	Glycerine
CH_3COONa	-	Sodium acetate
CS	-	Cassava starch
pCS	-	Plasticized cassava starch
FTIR	-	Fourier transform infrared
DSC	-	Differential scanning calorimeter
DTG	-	Differential-thermogravimetric
FTIR	-	Fourier transform infrared
J/g	-	Joule/gram
H_3PO_4	-	Phosphoric acid
ISO	-	International standard organization
mm	-	Millimeter
MFI	-	Melt flow index
µm	-	micronmeter
phr	-	Part per hundred
PVA	-	Poly(vinyl alcohol)
pPVA	-	Plasticized poly (vinyl alcohol)
pCS	-	Plasticized cassava starch
PVA/CS	-	Plasticized poly(vinyl alcohol)/cassava starch
T_g	-	Glass transition temperature
TG	-	Thermogravimetric
T_m	-	Melting temperature
w/w	-	Weight over weight

wt %	-	Weight percentage
XRD	-	X-ray diffraction
ΔH_m	-	Enthalpy of melting
ΔH_{mi}	-	Theoretical enthalpy of melting
ΔH_f	-	Enthalpy of 100% crystalline PVA

CHAPTER 1

INTRODUCTION

1.1 Background of Research

Over the past 50 years, more than 90% of the plastic household and industrial products are made of the feedstock of petroleum and gas (Mecking, 2004). The versatility of plastics that can be designed as rigid or elastic, breakable or resilient, transparent or brightly coloured, cheaper price and recyclable is the reason their usage become major in worldwide daily activities compared to other materials (Momani, 2009). By 2010, the annual plastic productions have been increased up to 300 million tonnes (Thompson *et al.*, 2009).

Plastics from petroleum-based are not biodegradable and they will exist until 100 years or more (Barnes *et al.*, 2009; Domenek *et al*., 2004). It gives negative impact towards our ecosystem such as diminish of landfills, toxic chemicals leakage due to contamination of groundwater, toxic gases emission cause severe health problem and threat to the existence of wildlife (Mooney, 2009; Gregory, 2009). However, the amount of the petroleum reduces year by year with uncertainty price correspond to the demand increase year by year has been causing to the cost production of the synthetic polymer. (Gervet, 2007; Hopewell *et al*., 2009).

With the depletion of oil, backlash for environmental pollution and greenhouse effect have driven researchers to look at the plastics that utilize naturally occurring cheap feedstock and environmental friendly. Current trend of research has seen the development of biodegradable plastics or bioplastics for various packaging applications that have been made in broad academic and industrial interests (Lee *et al.*, 2010; Rahman *et al.*, 2010; Cyras *et al.*, 2006; Novamont, 2007; Chung *et al.*, 2010; Yu *et al.*, 2010).

Starch is a natural biodegradable polymer that derived from plants such as from potatoes, rice, tapioca, and corn. It is renewable resources with wide availability and cheaper price compared to the conventional sources. It can be degrade by microorganism (Shah *et al.*, 1995; Mani and Bhattacharya, 1998). It has an ability to melt into thermoplastic starch. Majdzadeh-Ardakani *et al.* (2010) reported that thermoplastic starch has deteriorated in physical and mechanical properties. Thermoplastic starch has polar substance that present hydroxyl (-OH) group in the molecules. Some incorporation polar synthetic polymers, which can compatibilist with starch, will solve this problem (Chilleni *et al.*, 2003). Among synthetic degradable polymer such as poly (ε-caprolactone)(PCL), poly (lactic acid)(PLA) and poly(vinyl alcohol)(PVA), poly (vinyl alcohol)(PVA), which has been widely accepted to be fully biodegradable in various environments and confirmed, in current standard test reported by McCarthy (2003) and Swift (2003). Both starch and PVA also possess hydroxyl group (─OH), thus the formation of hydrogen bonds after the blend tended to promote localized stability subsequently improved miscibility of starch and PVA. Addition of PVA to thermoplastic starch or starch to PVA will reduce the cost of PVA itself, eco-friendlier and enhance the mechanical properties (Mao *et al.*, 2000; Tang *et al.*, 2011).

Different compounding process will give the different result due to geometrical structure and conformational of polymer macromolecules. Toh *et al.* (2011) shows the resulted based on poly (vinyl alcohol) (PVA) and sago pith waste

(SPW) in three different methods: dry blend method, pre-extrusion of PVA and pre-extrusion of both PVA and SPW. In all methods, dry blend method has been chosen because of finding obtained by tensile properties and water absorption. In tensile properties, dry blend gives value at 4.85 MPa, meanwhile pre-extrusion of PVA and pre-extrusion both PVA and SPW gives value at 4.47 MPa and 5.33 MPa. Meanwhile, in water absorption test of about 420 minutes, it shows that dry blend method gives higher value then pre-extrusion of PVA and pre-extrusion both PVA and starch which is, 58.33%, 57.75% and 56.03%. It claims that due to similar result, the dry blends have been chosen because of minimal step taken to give the minimal time and manufacturing cost.

1.2 Problem Statement

At present, many research conducted on biodegradable polymer by using starches as fillers or matrices. However, an in depth study is still needed especially in terms of process ability and biodegradability of poly (vinyl alcohol) (PVA)/ cassava starch (CS) blend to fulfill the industrial needs. Two-step compounding process was previously done by Mohd Shahrul Nizam Bin Salleh (2010) in developing PVA/CS blend. However, the previous process is an uneconomical process used for industrial. A new idea to develop with one-step compounding will reduce the production cost of the blend since the energy will be reduced and the time of processing will be shorter compared to two-step compounding process.

This research has been designed to study the effect of polyvinyl alcohol (PVA) filled cassava starch (CS) that prepared by one-step compounding process and its biodegradability toward several environment and conditions. PVA has low biodegradability compared to starch (Ishikagi *et al.*, 1999). Incorporation of starch with synthetic polymer such as PVA will improve biodegradability. This research has utilized cassava starch (CS) due to its abundant availability in Malaysia.

An effective microorganism will be used as one of the reagent to degrade the sample since effective microorganism is much lower in cost compared to specific fungi and bacteria used toward biodegradability studies. Report by Chiellini *et al***.** (2003) shows the fungi and bacteria such as *pseudomonas* species, *alcaligenes* and *bacillus* had been used to degrade the poly (vinyl) alcohol. Recently, there is no research use of an effective microorganism to degrade the sample. Therefore, a few questions need to be answered as follows:

i. Could PVA/CS blends be prepared via one-step compounding and processable into conventional injection moulding and what are the effects of mechanical properties between the methods used?

ii. What are the effects of PVA incorporation with CS content toward functional group, thermal, flowability, crystallinity and biodegradability properties?

iii. How far can the soil and compost affected the biodegradability of PVA/CS blends and what is the effect by using fungi, bacteria and effective microorganism?

iv. Which conditions is preferable for PVA/CS blends to be biodegraded, aerobic or anaerobic conditions?

For biodegradability method, it would be performed in several months to ensure all samples reach their equilibrium state with the environment to encourage the growth of microorganism.

1.3 Objective of Research

The aim of this research is to develop a biodegradable PVA/CS blends by using one-step compounding using the conventional injection molding and their biodegradability in various environments. Therefore, this research project embarked on the following objectives:

i. To investigate the different effect of methods used; one-step and two-step compounding process toward tensile strength and determine impact property of PVA/CS blends.

ii. To determine the functional group, thermal, flowability, and crystallinity of PVA incorporation with CS content.

iii. To determine the influence of moisture sorption, soil, compost, and enzyme toward PVA/CS blends

iv. To analyze the influence CS loading on biodegradability in aerobic and anaerobic conditions enriched with fungi, bacteria and effective microorganism.

1.4 Scope of Research

The scope of research involved the preparation of PVA/CS blends via one-step compounding process. The starch loadings were varied i.e. 50wt%, 60wt %, 70wt%, 80wt% and 100wt% respectively. All blends were tested and characterized accordingly. Outline of this research were subdivided into four sections:

i. Compounding of PVA/CS blends were prepared in powder-powder system where PVA and cassava starch powder was added simultaneously with glycerol and additives in a high-speed mixer followed by blending with a twin-screw extruder. A series blends of PVA and cassava starch were varying prepared with amount of cassava starch (0 wt%, 50 wt.%, 60wt.%, 70wt.%, 80wt.%, 100 wt%). The amount of glycerol and additives were fixed.

ii. For the compound characterization, the interaction in the blends of PVA/CS were explained on Fourier Transform Infrared (FTIR). For thermal analysis, Differential Scanning Calorimetry (DSC) and Thermogravimetry-Derivative Thermogravimetry (TG-DTG) was used to determine the enthalpy changes with temperature, degree of crystallinity in increasing starch loading and thermal stability of the blends. The flow ability of blends were analysed by Melt Flow Index (MFI) for its viscosity before injected. In determining the crystallinity of the blends, X-ray Diffraction (XRD) analysis was used.

iii. For testing the injected product, mechanical properties such as tensile strength and impact properties was studied. The comparison was made between two method used in term of tensile strength.

iv. In term of biodegradation, studies on moisture sorption, enzymatic hydrolysis, soil burial, and solid phase medium via aerobic and anaerobic condition enriched with fungi, bacteria and effective microorganism were conducted.

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