ISOLATION AND CHARACTERIZATION OF NANOCELLULOSE FROM EMPTY FRUIT BUNCH FIBER FOR NANOCOMPOSITE APPLICATION

NURUL SAADIAH BINTI LANI

UNIVERSITI TEKNOLOGI MALAYSIA

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NURUL SAADIAH BINTI LANI

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To my wonderful families especially to my beloved mother and father

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ABSTRACT

Nowadays, the demands for plastics materials are increasing rapidly. Nevertheless, most of these products are non-environmentally friendly and non-biodegradable. About 60 to 100 million gallons of petroleum are needed to produce plastics every year around the world. Therefore, there has been growing interest in developing bio–based products that can offer favorable environmental advantages. The purpose of this study is to isolate nanocellulose from empty fruit bunch (EFB) fiber and to investigate reinforcing effect of nanocellulose in poly(vinyl alcohol) (PVA)/starch blend films. The optimization of acid hydrolysis conditions for nanocellulose yield with response surface methodology (RSM) was also investigated. Cellulose and nanocellulose fibers were successfully extracted by using alkali treatment and acid hydrolysis, respectively. Subsequently, a series of PVA/starch film with different content of nanocellulose were prepared by solution casting method. The isolated nanocellulose displayed a relatively high crystallinity, which were around 73% that consisted of rod like nanoparticles with the diameter of 4 to 15 nm. Analysis of the RSM result revealed that high nanocellulose yield (83.42%) was obtained when the sulfuric acid concentration, hydrolysis time and reaction temperature were set at 58 wt%, 43 minutes and 35 °C, respectively. PVA/starch films reinforced with nanocellulose fiber possessed significantly improved properties compared to the film without reinforcement. From the results, PVA/starch films with the addition of 5% (v/v) of nanocellulose suspension exhibited the best combination of properties. This nanocomposite was found to have tensile strength about 5.694 MPa and the elongation at break about 481.85%. In addition, this nanocomposite had good water resistance (19.71%) and biodegradability (47.73%). It can be concluded that the nanocellulose obtained in this study can be an excellent reinforcing material in PVA/starch blend film.

ABSTRAK

Pada masa kini, permintaan untuk bahan plastik semakin meningkat dengan pantas. Walau bagaimanapun, kebanyakan produk ini tidak mesra alam dan tidak terurai. Kira–kira 60 hingga 100 juta gelen petroleum diperlukan untuk membuat plastik setiap tahun di seluruh dunia. Justeru itu, terdapat minat yang semakin meningkat dalam membangunkan penggunaan produk berasaskan bio yang mempunyai kebaikan untuk alam sekitar. Tujuan kajian ini adalah untuk mengasingkan nano selulosa dari serat tandan buah kosong dan mengkaji kesan pengukuhan mereka dalam filem polivinil alkohol (PVA)/kanji. Pengoptimuman keadaan hidrolisis asid untuk menghasilkan nano selulosa dengan menggunakan kaedah gerak balas permukaan (RSM) juga dilakukan. Serat selulosa dan nano selulosa telah berjaya diekstrak dengan menggunakan rawatan alkali dan hidrolisis asid. Selepas itu, satu siri filem PVA/kanji dengan kandungan nano selulosa yang berbeza disediakan dengan menggunakan kaedah tuangan larutan. Nano selulosa yang telah diasingkan meunujukkan penghabluran yang secara relatifnya tinggi, iaitu kira-kira 73% dan mempunyai bentuk seperti rod dengan diameter dari 4 hingga 15 nm. Analisis keputusan RSM mendedahkan bahawa hasil nano selulosa (83.42%) adalah tinggi apabila kepekatan asid sulfurik, masa hidrolisis dan suhu tindak balas diletak masing-masing pada 58 wt%, 43 minit dan 35 °C. Filem PVA/kanji yang diperkukuhkan dengan gentian nano selulosa mempunyai ciri-ciri penambahbaikan yang ketara berbanding filem tanpa pengukuh. Daripada keputusan, filem PVA/kanji dengan tambahan 5% (v/v) ampaian nano selulosa meunujukkan kombinasi ciri-ciri yang terbaik. Nano komposit ini didapati mempunyai kekuatan tegangan pada kira-kira 5.694 MPa dan pemanjangan pada takat putus adalah 481.85%. Sebagai tambahan, nano komposit ini mempunyai rintangan air (19.71%) dan biodegradasi (47.73%) yang baik. Kesimpulannya, nano selulosa yang diperoleh dalam kajian ini boleh menjadi bahan pengukuh yang sangat baik untuk filem adunan PVA/kanji.

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LIST OF ABBREVIATION

AFM	-	Atomic Force Microscope
AOX	-	Adsorbable Organic Halide
AMG	-	Amyloglycoside
ANOVA	-	Analysis of Variance
ASTM	-	American Society for Testing and Materials
BCC	-	Bamboo Cellulose Crystal
Ca	-	Calcium
CBN	-	Cassava Bagasse Cellulose Nanofiber
CCD	-	Central Composite Design
CMC	-	Carboxymethyl cellulose
COD	-	Chemical Oxygen Demand
CW	-	Cellulose Whisker
DMA	-	Dynamic Mechanical Analyzer
EMC	-	Equilibrium Moisture Content
EFB	-	Empty Fruit Bunch
FESEM	-	Field Emission Scanning Electron Microscope
FMP	-	Fish Myofibrillar Protein

FTIR	-	Fourier Transform Infrared
JNF	-	Jute Nanofibril
KBr	-	Potassium Bromide
KOH	-	Potassium Hydroxide
LDPE	-	Low Density Polyethylene
MFC	-	Microfibrillated Cellulose
Mg	-	Magnesium
MMC	-	Metal Matrix Composite
MS	-	Mean Square
$NaCIO_2$	-	Sodium Chlorite
NaOH	-	Sodium Hydroxide
NFC	-	Nanofibril Cellulose
NR	-	Natural Rubber
OPT	-	Oil Palm Trunk
OPF	-	Oil Pam Frond
PALF	-	Pineapple Leaf Fiber
PFF	-	Presses Fruit Fiber
PHB	-	Poly-3-hydroxybutyrate
POME	-	Palm Oil Mill Effluent
PP	-	Polypropylene
PVA	-	Polyvinyl Alcohol
PU	-	Polyurethane

RSM	-	Response Surface Methodology
SEM	-	Scanning Electron Microscope
SiO_2	-	Silicon Dioxide
SPI	-	Soy Protein Isolate
SPU	-	Segmented Polyurethane
TEM	-	Transmission Electron Microscope
TPS	-	Thermoplastics Cassava Starch
WAC	-	Water Absorption Capacity
WVP	-	Water Vapor Permeability
WVTR	-	Water Vapor Transmission Rate
XRD	-	X–Ray Diffraction

LIST OF SYMBOLS

E_a	-	Activation energy
kg/m^3	-	kilogram per cubic meter
mL	-	milliliter
mg/L	-	milligram per liter
MPa	-	megapascal
µm	-	macrometer
w/v	-	weight per volume
°C	-	degree celcius
%	-	percentage

CHAPTER 1

INTRODUCTION

1.1 Background of Study

In recent years, many researchers have shown considerable interest for the incorporation of nanocellulose fibers as reinforcement. The applications of nanoscale particles in composite processing are also expected to have achieved a significant improvement on the environmental issue as this nanocellulose reinforcement could develop a more biodegradable plastic. This is due to the fact that the usage of non–biodegradable plastics nowadays contributes to about one quarter of all domestic trash in landfill sites. Moreover, the process to manufacture plastics are often involves the use of toxic and environmentally harmful chemicals. Therefore, it is quite important to develop composites that can be easily and completely degraded and also produced from renewable resources.

Polyvinyl alcohol (PVA) has been considered as a suitable source of materials for the composite production because of its benefits of being non–toxic and highly durable. Besides that, PVA is a synthetic polymer that can be fully biodegradable in the environment. However, the applications of PVA materials are limited due to their high cost and slow degradation process especially under anaerobic condition (Takasu *et al.,* 2002). To overcome these limitations, PVA is often blended with other cheap and biodegradable polymers.

By blending with other renewable polymers, it will improve PVA's biodegradation rate and lowers the overall cost. Among the different types of biopolymer available, starch is well known as the most abundant raw materials and relatively cheap. Previous studies have reported that blending of PVA and starch can enhance their tensile strength, elongation and toughness (Guohua *et al.,* 2006; Mao *et al.*, 2002; Rahman *et al.*, 2010; Russo *et al.*, 2009; Sin *et al.*, 2010). In addition, starch has been successfully blended with PVA due to the presence of hydroxyl groups in both PVA and starch molecules, which tend to form strong hydrogen bonding and relatively good compatibility of PVA and starch (Tang and Alavi, 2011). However, the major disadvantages of these PVA/starch blends are particularly poor water barrier properties, generally attributed to the very large number of hydroxyl groups along with their intrinsic hydrophilicity. Therefore, several studies have proposed the incorporation of fillers in a nanoscale size into PVA/starch blends in order to improve their water barrier properties.

Most of the attention so far has focused on the preparation of nanoparticles from natural fibers. Natural fibers has attracted growing interest because of their unique characteristics, including low cost, lower density, high specific strength, good thermal properties and biodegradable.Oil palm empty fruit bunch (EFB) fiber is a natural fiber which has great relevance to Malaysia, as a large quantity of the biomass is generated by oil palm industries. In 2012, it is estimated that around 18.79 million tons of crude palm oil and 70 million tons of biomass residue are produced in the oil palm industry in Malaysia (Aljuboori, 2013). In addition, the total crop of fresh fruit bunch is approximately 92.78 million tons per year, which generate more than 20 million tons of EFB(Wan-Razali *et al.*,2012). Approximately only 10% of the EFB is used and the rest are abundant.This residue may cause many environmental problems. Therefore, there is huge potential for EFB to be exploited in the production of high value-added products, which not only complies with zero-waste strategy but also generated additional profits for the palm oil industry.

Therefore in this study, cellulose was extracted from palm oil empty fruit bunch (EFB) fiber via alkali treatment method while the isolation of nanocellulose was done by acid hydrolysis method. A large number of treatments to extract highly–purified cellulose fibers have already been reported. The most common treatment is mercerization method, which is also known as alkali treatment. The important point to note regarding alkali treatment is their capability to remove certain amount of non–cellulosic impurities on the fiber surface by disrupting the hydrogen bonding in the network structure (Li *et al.,* 2007).

Meanwhile, a comprehensive research and review article dealing with isolation of cellulose fiber in nano–scale size by sulfuric acid hydrolysis was published by many researchers (Araki *et al.,* 1999; Cho and Park, 2011; Fahma *et al.,* 2011; Luduena *et al.,* 2011; Mandal and Chakrabarty, 2011; Morais *et al.,* 2012; Moràn *et al.,* 2008; Revol *et al.,* 1992; Rosa *et al.,* 2012). Controlled acid hydrolysis of native cellulose fibers disrupts the fibers which can then be dispersed into their constituent rod–shaped elementary crystalline microfibrils.During acid hydrolysis, the amorphous regions in native cellulose are more accessible to acid and more susceptible to hydrolytic action than the crystalline domains. Therefore, acid hydrolysis of cellulose is a well–known process used to remove amorphous regions.

Subsequently, nanocellulose that are isolated from EFB fiber was used as reinforcement in composite films. In this study, two series of PVA and starch blend films were prepared and characterized. The first series was based on only PVA and starch in different ratios, as follows: 80% PVA/20% starch, 70% PVA/30% starch, 60% PVA/40% starch, 50% PVA/50% starch, and 40% PVA/60% starch. The second series contain5, 10, 15 and 20% (v/v) of nanocellulose suspension with respect to the volume of PVA and starch solution. The ratio between PVA and starch was chosen based on mechanical properties, water absorption and biodegradation properties that yield optimum results in the first series.

1.2 Problem Statement

Nowadays, the demands of plastics materials are increasing rapidly. The application of plastics materials includes aeronautics, building and construction, electronic device, packaging, automotives and medical devices. However, most of these products are non-environmentally friendly and non–biodegradable. Moreover, all these plastics residues are mainly discarded into the landfill and frequently the causes of pollution as well. The high usages of plastics are leading to serious environmental pollution, a problem that has to be faced by all societies.

Nevertheless, there is an alternative to reduce the environmental problems caused by plastics. For example, the production and application of biodegradable composites based on biodegradable resources such as natural fiber can be pursued to provide benefits to the environment with respect to the degradability. However,the most serious concerned problem with natural fibers is its hydrophilic nature, which tends to prevent better dispersion of the fibers into the matrix. Therefore, to overcome this challenge, fiber treatment process is one of the common alternatives that can be used to modify the fiber surface topology.

The need for PVA composites has never been as prevalent as it currently is. PVA offers high tensile strength and flexibility as well as excellent film forming. However, this synthetic polymer has important drawback that need to be addressed, which is their degradation rate. Therefore, blending with starch would help to improve the biodegradable properties. In the meantime, nano–reinforced starch/PVA blends are not widely studied compared to starch nanocomposites and PVA nanocomposites.Furthermore, most of the previous studies have investigated the use of nanofillers such as nano silicon dioxide (Xiong *et al.*, 2008; Abbasi, 2012), montmorillonite clay (Ardakani and Nazari, 2010; Spiridon *et al.,* 2008), sodium montmorillonite clay (Taghizadeh *et al.,* 2012) and nanoparticles of poly(methyl methacrylate–co–acrylamide) (Yoon *et al.*, 2012). However, these nanoparticles had no significant influence on biodegradability of films. For that reason, this research was conducted for the purpose of improvement in the properties of PVA and starch blend by using nanocellulose from EFB fiber as reinforcement.

1.3 Objective of Study

The objectives of this study are:

i. To extract and characterize cellulose and nanocellulose from oil palm empty fruit bunch fiber
ii. Toperform an optimization study on nanocellulose yield
iii. To study the effect of varying the PVA, starch and nanocellulose content on the composites properties
iv. To compare the properties of composites between pure PVA film, PVA/starch blend film, PVA/starch reinforced with cellulose composite film and PVA/starch reinforced with nanocellulose composite film.

1.4 Scope of Study

Once the objective is decided, it is necessary to determine the scopes that will limit the range of the study. This study was firstly focused on the extraction of cellulose from empty fruit bunch fiber. The cellulose from empty fruit bunch fiber was extracted by using alkaline method, whereby the experimental conditions were fixed according to the method used by Moran*et al*. (2008). Meanwhile, the nanocellulose was isolated from obtained cellulose by using acid hydrolysis method, whereby the hydrolysis conditions were fixed at 60wt% sulfuric acid solutions and reaction temperature at about 45°C with hydrolysis time of 30 minutes under strong agitation (Moran*et al.,* 2008). After that, the extracted cellulose and nanocellulose were characterized by using Fourier transform infrared spectroscopy (FTIR), field emission scanning electron microscopy (FESEM), x-ray diffraction (XRD) and thermogravimetric analysis (TGA). Furthermore, the nano–dimensions of nanocellulose were determined using a transmission electron microscope (TEM).

For the optimization study, the central composite design (CCD) method was used to determine the relationshipbetween hydrolysis conditions on maximum yield of nanocellulose.Prior to optimization study, a preliminary screening test was conducted to determine trends in the yields of nanocellulose. The yield of nanocellulose was measured as a function of acid concentration, hydrolysis time and reaction temperature, whereby the sulfuric acid concentration in the range of 45–85 wt%, hydrolysis time from 30 to 90 minutesand reaction temperature between 25 and 65°Cwere used as hydrolysis conditions.

The third part of this study covered the production of PVA/starch blend films. The starch used in the blend was a corn starch, whereby corn starch has higher amylose content compared to other types of starch, around 28%.High amylose content in starch is known to produce films with better mechanical properties (Yun and Yoon, 2010). This experiments were done by mixing PVA with starch by varying the blend ratio of PVA:starch, which is 80:20, 70:30, 60:40, 50:50 and

40:60. Glycerol was used as a plasticizer, in which it was added to the mixed solution at a 30 wt% ratio based on total weight of starch and PVA (Yao *et al.,* 2011). The mixing temperature and time were fixed at 97°C and 2 hours, respectively (Rahman *et al.,* 2010). The effect of varying the PVA and starch content on the composites properties was analyzed according to their mechanical properties, water absorption and biodegradation properties.

The effectiveness of the nanocellulose fibers as reinforcementwas tested in the PVA/starch blend solutions. In this study, content of nanocellulose was varied from 0 to 20% (v/v) of nanocellulose suspension with respect to the volume of PVA and starch solution. Nanocomposite films reinforced with nanocellulose fibers were characterized according to their mechanical properties and water absorption while the biodegradation of films were carried out by using the soil burial test.

In the comparative study, 5% (v/v) cellulose reinforced PVA/starch composite was produced. Therefore, a comparison was made between pure PVA film, PVA/starch blend film, 5% (v/v) cellulose reinforced PVA/starch composite and 5% (v/v) nanocellulose reinforced PVA/starch nanocomposite based on their mechanical properties, water absorption and degradation properties.

1.5 Research Hypothesis

Nanocellulose can be successfully isolated from empty fruit bunch fiber by acid hydrolysis and their incorporation as reinforcement can result in an improvement in polyvinyl alcohol/starch blend film.

1.6 Significance of Study

The finding of the research is important to discover the performance of nanocellulose fiber as reinforcing materials in polymer composites due to their large surface area and the nano–scale dimensions. Therefore, it can provide the opportunity for nano–engineered materials in composite processing that could have not achieved from conventional materials.

Apart from that, this research will also contribute on improving the properties and biodegradation of nanocomposite. Their good mechanical performance showed the potential replacement to glass fiber composite in the emerging advanced composite market. It may give the plastics industry a more economic solution in managing the environmental problems caused by conventional synthetic plastics.

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