



Faculty of Engineering

**FABRICATION AND MECHANICAL PROPERTIES OF CELLULOSE
NANOFIBER REINFORCED POLYVINYL ALCOHOL COMPOSITES**

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**Bachelor of Engineering with Honours
(Mechanical and Manufacturing Engineering)**

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Masters

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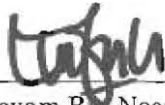
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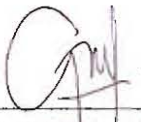
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FABRICATION AND MECHANICAL PROPERTIES OF CELLULOSE
NANOFIBER REINFORCED POLYVINYL ALCOHOL COMPOSITES

NADZRIN AHMED BIN FASIHUDDIN

A dissertation submitted in partial fulfillment
of the requirement for the degree of
Bachelor of Engineering with Honours
(Mechanical and Manufacturing Engineering)

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Universiti Malaysia Sarawak

2017

Dedicated to my beloved parents, my brother and all friends for best support, help and encourage me through good and hard time.

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ABSTRACT

Focus on green materials is increasing due to problems associated with environment especially for plastic materials that are non-biodegradable, toxic and containing harmful chemicals. Polyvinyl alcohol (PVA) is a biodegradable, polymer material with excellent mechanical properties, non-toxic and good film-forming ability. Cellulose nanofiber (CNF) is a natural polymer which has a potential as reinforcement material with PVA as CNF showing good mechanical properties, organic and abundant in nature. In this research, the mechanical properties of PVA films, CNF sheets and CNF/PVA composite sheets has been conducted to evaluate the reinforcement effect with different CNF content of 40 wt%, 50 wt%, 60 wt% and 70 wt%. Also, the effect of heat treatment on mechanical properties of the specimens was studied. At increasing CNF content, the tensile strength and Young's modulus increased up to 217.1% and 109.1% respectively compared to pure PVA films and CNF sheets. Tensile strength and Young's modulus for heat treated specimens also has shown significant increase up to 75.7% and 13.4% compared to untreated specimens. Increase in CNF content yields higher density CNF/PVA composite sheets. Based on water swelling test, it was observed that as the CNF content increased, the CNF/PVA composite film to be more hydrophobic as less water being absorb. SEM observation showed good compatibility of CNF and PVA resin which contribute to increase in tensile strength of the composite specimens compared to pure PVA film. Increase of CNF content and heat treatment has improved mechanical properties of PVA resin which enables wider range of application.

Keywords: *Polyvinyl alcohol, cellulose nanofiber, nanocomposites, green composites, heat treatment*

ABSTRAK

Permintaan dalam penghasilan bahan hijau telah meningkat disebabkan oleh masalah alam sekitar terutamanya kepada bahan plastik yang tidak mudah terurai, bertoksik dan mengandungi bahan kimia berbahaya. Polivinil alkohol ialah bahan yang mudah terurai secara biologi, mempunyai sifat mekanikal yang baik, tidak bertoksik, mudah dibentuk menjadi filem dan ketahanan yang tinggi secara semula jadi. Selain itu, selulosa serat nano berpotensi menjadi pengukuh pengisi dengan polivinil alkohol kerana mempunyai sifat mekanikal yang baik, bukan organik, dan banyak didapati secara semula jadi. Dalam penyelidikan ini, sifat mekanikal polivinil alkohol dan selulosa serat nano telah dijalankan to menilai kesan pengukuhan dengan jumlah kandungan berbeza (40 wt%, 50 wt%, 60 wt% dan 70 wt%) dalam penghasilan filem komposit. Selain itu, kesan rawatan haba terhadap sifat mekanikal turut dikaji. Penambahan selulosa serat nano menambahkan kekuatan tegangan dan Young's Modulus sehingga 217.1% dan 109.1% berbanding dengan filem polivinil alcohol. Kekuatan tegangan dan Young's Modulus selepas rawatan haba bertambah naik sehingga 75.7% dan 13.4% berbanding spesimen tidak dirawat. Selain itu, peningkatan kandungan selulosa serat nano meningkatkan ketumpatan filem yang dihasilkan. Berdasarkan ujian serapan air, pada jumlah selulosa serat nano meningkat menunjukkan lebih hidrofobik disebabkan kurang menyerap air. Mikroskop imbasan electron menunjukkan keserasian antara selulosa serat nano dan polivinil alkohol yang menyebabkan peningkatan kekuatan tegangan komposit berbanding filem polivil alkohol. Peningkatan jumlah selulosa serat nano and rawatan haba mampu meningkatkan sifat mekanikal resin polivinil alkohol yang mampu diluaskan kegunaannya.

Kata Kunci: *Polivinil alcohol, selulosa serat nano, komposit bersaiz nano, composite hijau, rawatan haba*

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LIST OF ABBREVIATIONS

PVA	=	Polyvinyl alcohol
PVA-f	=	Fully hydrolyzed polyvinyl alcohol
PVA-p	=	Partially hydrolyzed polyvinyl alcohol
CNF	=	Cellulose nanofiber
CNC	=	Cellulose nanocrystals
MFC	=	Microfibrillated cellulose
NCC	=	Nanocrystalline cellulose
BC	=	Bacterial nanocellulose
CNT	=	Carbon nanotubes
SEM	=	Scanning electron microscopy
DP	=	Degree of polymerization
DH	=	Degree of hydrolysis
CTE	=	Thermal expansion
OLED	=	Organic light emitting diode
RTIL	=	Room temperature ionic liquid
σ	=	Tensile strength
ε	=	Tensile strain
E	=	Tensile modulus
Δl	=	Elongation of length
l_o	=	Original length
σ_y	=	Yield stress
P_y	=	Load at yielding
A_o	=	Original cross sectional area
UTS	=	Ultimate tensile strength
wt %	=	Weight percent
XRD	=	X-ray powder diffraction
TGA	=	Themogravimetric analysis
mm	=	Millimeter

MPa = Megapascal
°C = Celsius
g/cm³ = Gram per centimeter cubed

CHAPTER 1

INTRODUCTION

1.1 Background of Study

1.1.1 Overview of Polymer and Nanocomposites

Polymer has a wide application for human beings in everyday life (Young & Lovell, 2011). Polymer composites are applied in wide applications such as in aerospace components, automobiles and sporting goods. Recently, research and development of polymer nanocomposite is increasing. Polymer nanocomposites are composites which at least one of the dimensions is in nanoscale. Besides, the final product does not have to be in nanoscale but also can be acceptable in micro- or macroscopic scale (Hussain & Hojjati, 2006). Therefore, with a massive exploration, polymer nanocomposite will bring a whole new dimension—towards future technology. In early 1980s, use of scanning tunneling microscopy and scanning probe microscopy has significantly facilitated in nanotechnology development. Researchers are able to study nature of surface in atomic resolution (Nanoscience and Technology, 2004). Besides, development of computer technology is helping researcher to study and predict the properties at the nanoscale by modelling and simulation (Hussain & Hojjati, 2006).

Basically, composites are structures in which two or more materials are merged to produce a new material whose properties are expected to be better from the initial individual material. In general, composite is made of filler and matrix. The matrix helps to surround the other phases and provides bulk form to the material. The major structural member is filler which are typically in form of particles or fibres. Fibre-filled composites are frequently laminated that formed from stacks of individual plies that give continuous in-

plane reinforcement. Moreover, strength and stiffness of a composite can be controlled by altering the shape, amount, surface functionality and orientation of the filler in the matrix. The ability to modify properties makes composite to be an attractive alternative for any desired applications.

1.1.2 Green Materials for Sustainable Future

Plastic materials are widely being utilized in industrial application such as in aeronautics, building and construction, packaging, automotive and medical devices. Besides, increase of plastic materials leads to concern towards the environment (Stevens, 2001). This is due to the fact that most of plastic material used nowadays is not environmental friendly and non-biodegradable. More than 30 million and 15 million tons of plastic waste were reported in United States and Western Europe respectively (Jayasekara, et al., 2005). The waste products are disposed in the landfill that causes these materials to stay for long period of time without degrading that eventually causes the land unusable.

Besides, plastic material residue will cause severe damage to the environment as the materials contain chemicals that are harmful to the living creatures on earth. Moreover, the process to produce plastic materials is frequently involving the use of toxic and harmful chemicals. About 6-7% of petroleum produced is used to produce plastic materials and other materials. Human beings are consuming petroleum at uncontrolled rate which is 100,000 times than the nature need to produce petroleum (Netravali & Chabba, 2003). At this current usage of petroleum trend, estimated times for petroleum reserves to last is at 50 to 60 years only (Stevens, 2001). Therefore, an alternative towards reducing use of petroleum should be taken. Replacing plastic materials with biodegradable resource could be a next move towards green environment. Many researches and studies have been conducted to develop new material which has low impact to environment (Kowalczyk, et al., 2011). Waste materials are being recycled to form useful materials (Takagi, et al., 2013).

Polyvinyl alcohol (PVA) is a polymer used in a wide range of industrial application due to its biodegradable, excellent mechanical properties, non-toxic and high durability. Besides that, PVA is a synthetic polymer that can be fully degradable in the environment. However, the price of PVA is rising nowadays and caused it to be an expensive polymer (Takasu, et al., 2002). In order to reduce the cost, PVA is combined with other compatible filler such as cellulose to develop a composite as both having hydroxyl group that can form hydrogen bonding between the polymers. Furthermore, the mechanical properties will eventually increase. The low mechanical strength and integrity of PVA also cause the increasing demand for reinforcing agents (Jeong, et al., 2007). Besides, nanoscale filler is helpful to increase the properties of the material as nanomaterials has larger surface areas where more amount of material can come into contact.

Developing a material which could improve the environment is needed for better living. Therefore, green nanocomposite has promising advantages for the future. More understanding on this field will help researchers to discover new method of producing green nanocomposites or significant properties which is helpful in the future. Green nanocomposite generally offers improvement in tensile strength, modulus of elasticity and heat distortion temperature. Therefore, green nanocomposite has a huge application in many fields.

1.2 Problem Statement

Natural resources such as cellulose nanofiber (CNF) have caught attention as they are abundant and also known as renewable resource. Thus, CNF has a good potential in developing green composite, with expectation to increase mechanical properties of the composites. However, reliable data and study based on CNF and PVA polymer are still inadequately available. Therefore, this study was conducted to evaluate the effect of CNF as reinforcement towards the mechanical properties of PVA based nanocomposites. The manufacturing method and preparation of nanocellulose based polymer composite still in need of intense study, therefore it is important to conduct significant amount of study to

investigate more regarding the mechanical properties, ~~chemical effect, bonding condition,~~ fabrication method and treatment on materials. The results obtained will help to give significant information on the properties of CNF/PVA composites. There is still in need for more understanding in the following aspect of information:

1. The mechanical properties of CNF/PVA composite with higher CNF content above 40 wt%
2. The effect of filler loading to the mechanical properties CNF/PVA composite
3. The mechanical properties of CNF/PVA composite with PVA of different degree of hydrolysis
4. The effect of heat treatment towards mechanical properties of CNF/PVA composite

1.3 Objectives

The main objective of this study is to study the mechanical properties of fully hydrolyzed PVA (PVA-f) and partially hydrolyzed PVA (PVA-p) and CNF and their composite with different CNF filler loading (40 wt%, 50wt%, 60 wt% and 70 wt%). Post heat treatment was conducted to study the mechanical properties and compared with untreated samples. Morphological study is also conducted to study on fibre distribution in the CNF/PVA composite at different weight fraction of CNF.

1.4 Project Outlines

There are five overall chapters in this report which includes Introduction, Literature Review, Methodology, Results and Discussion and Conclusion and Recommendation.

Chapter 1, “Introduction”, defines the whole project in general view which includes basic information of project understanding. The description for this section consists of overview of nanocomposites materials and green materials, problem statement, objectives, scope of study and project outlines.

Chapter 2, “Literature Review”, consist of the previous experimental works and topics that are related with this research will be studied. Besides, topics related to problem statement are reviewed in this chapter. An overview of research related to CNF, PVA and CNF/PVA composites is reviewed to compare the existing study on this field, effect of PVA degree of hydrolysis and effect of heat treatment is also reviewed in general in this section.

Chapter 3, “Methodology”, describe on the materials used and experimental methods and procedures being performed to conduct this study.

Chapter 4, “Results and Discussion”, contains the results obtained and the analysis being made from the conducted experiment.

1.5 Research Hypothesis

CNF acts as reinforcement materials for with PVA which eventually will increase the mechanical properties of the CNF/PVA composites as the increase of CNF content. Heat treatment on the CNF/PVA composite sheets is expected to further increase the mechanical properties of the composites compared to the untreated specimens.

1.6 Significance of Study

The finding of the research is important to discover the mechanical performance of CNF as reinforcing materials in PVA nanocomposites due to their large surface area and the nano-scale dimensions. Therefore, enhanced mechanical properties of PVA polymer by reinforcement with CNF could be useful to replace overwhelmed conventional materials available in the market. Furthermore, the CNF/PVA composite manufactured in this study could replace plastics materials which are mostly non-environmental friendly. As environmental concern is rising, a green composite which is based on natural resource and

biodegradable matrix is a solution towards better environment and also provided with better mechanical properties is an advantage for future materials.