ON THE USE OF NANO FIBRILLATED CELLULOSE (NFC) FIBER AS REINFORCEMENT IN POLYLACTIC ACID (PLA) MATRIX COMPOSITES

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Submitted in accordance with requirement of the University Teknikal Malaysia Melaka (UTeM) for Bachelor Degree of Manufacturing Engineering (Hons.)

by

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Signature : ……………………………………

Author's Name : CARRON TING SHUANG SIA

Date :

APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirement for Degree of Manufacturing Engineering (Engineering Materials) (Hons). The members of the supervisory committee are as follow:

……………………..................................

(Profesor Qumrul Ahsan)

……………………..................

(Dr. Mohd Sukor Bin Salleh)

ABSTRAK

Tujuan penyelidikan ini adalah untuk menentukan hasil selulosa nano berserat (NFC) selepas proses hidrolisis asid, proses putaran mekanikal dan fibrilasi, untuk menganalisis morfologi pengedaran dan penyebaran NFC dalam NFC- PLA komposit dan untuk mengkaji kesan pengukuhan pada sifat mekanikal komposit NFC-PLA. Pada mulanya, gentian kenaf adalah dalam skala nano selepas perawatan kimia dan proses fibrillasi. Selepas itu, serat nano selulosa dengan pelet PLA mengalami tekanan panas untuk membentuk komposit NFC-PLA. Peratusan hasil NFC ditentukan dengan menggunakan kaedah sentrifugal. Penampilan morfologi pengedaran dan penyebaran NFC dalam komposit NFC-PLA diperhatikan dengan menggunakan mikroskop optik (OM) dan mikroskop elektron (SEM). Kesan pengukuhan pada sifat mekanik komposit NFC-PLA diselidiki dengan menggunakan ujian mekanikal iaitu tegangan. Untuk kajian ini, data analisis panjang dan diameter rata-rata gentian fibrilasi menunjukkan tren menurun kerana kelajuan pencampuran dan peningkatan masa. Peningkatan maksimum kekuatan tegangan sebanyak 59.32% diperhatikan untuk NFC-PLA yang komposit dengan gentian fibrilasi yang diperolehi dari kelajuan putaran dan masa 15000 rpm dan 15 minit berbanding dengan PLA tulen. Sifat tegangan menunjukkan bahawa kekuatan dan modulus telah bertambah baik dengan peningkatan kandungan nanofiber. PLA menunjukkan kekukuhan dan kekuatan yang baik dan digunakan dalam aplikasi seperti botol minuman, pembungkusan makanan dan beg plastik yang boleh rosak. Selulosa nano fibril yang diperkukuhkan di PLA dapat meningkatkan sifat mekaniknya. Kompleks NFC PLA terkenal digunakan sebagai bahan dalam industri percetakan 3D. Dengan mengurangkan penggunaan plastik konvensional, pembaziran tapak pelupusan boleh dikurangkan juga. Hal ini turut menyumbang kepada kehidupan atau amalan hijau yang membantu memulihara sumber semula jadi dengan mengurangkan penggunaan sumber yang tidak boleh dikembalikan dan tidak boleh diperbaharui.

ABSTRACT

This research is to determine the yield of nano fibrillated cellulose (NFC) after chemico mechanical treatments of natural fibers through acid hydrolysis, mechanical rotational and fibrillation process, to analyze the distribution and dispersion morphology of NFC in nano fibrillated cellulose poly lactic acid (NFC-PLA) composite and to investigate the reinforcing effect on mechanical properties of NFC- PLA composite. In the beginning, kenaf fibers were fibrillated nanoscale through chemical treatment and fibrillation process. After that, the nano cellulose fibers with PLA pellet undergo hot pressing to form NFC-PLA composite. The yield percentage of the NFC was determined using centrifugal method. For the NFC-PLA composite, the morphological appearance of distribution and dispersion of NFC in NFC-PLA composite is observed by using optical microscope (OM) and scanning electron microscope (SEM). The reinforcing effect on mechanical properties of NFC-PLA composite was investigated by using mechanical testing which is tensile test. For this research, analytical data of average length and diameter of fibrillated fibers show a trend of decreasing as the blending speed and time increase. The maximum increase in tensile strength of 59.32% was observed for NFC-PLA composite with fibrillated fiber derived from rotational speed and time of 15000 rpm and 15 minutes compared to pure PLA. The tensile properties indicated that the strength and modulus were improved with increased nanofiber contents. PLA shows good stiffness and strength and is being used in applications such as drinking bottles, food packaging and degradable plastic bags. The nano fibrillated cellulose reinforced in the PLA can further improve its mechanical properties. NFC PLA composite is well known used as material in the 3D printing industry. By reducing the usage of conventional plastics, the landfill wastage can be reduced as well. This contributes to green living or practices that help conserve natural resources by reducing the use of non-degradable and non-renewable resources.

DEDICATION

Only

My beloved father, William Ting Kee Seng

My appreciated mother, Wong Ngik Toh

My adored sister and brother, Ting Jin Sia and Ting Siew Yang

For giving me moral support, financial help, cooperation, encouragement and also understandings

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NFC	-	Nano Fibrillated Cellulose
PLA	-	Poly Lactic Acid
FTIR	-	Fourier Transform Infrared Spectroscope
OM	-	Optical Microscope
SEM	-	Scanning Electron Microscope
FESEM	-	Field Emission Scanning Electron Microscope
ASTM	-	American Society for Testing and Materials

CHAPTER 1

INTRODUCTION

Chapter 1 will be discussing the importance of nano fibrillated cellulose (NFC) based on NFC-PLA matrix biodegradable composite. The background of research describes about NFC, PLA and NFC-PLA polymer bio composite. Besides, the problem statement will show the research gap that will be reduced throughout the research which is mainly about the yielding of NFC and dispersion of NFC-PLA biodegradable composite. This section will point out the objectives of this research and its scope.

1.1 Background of Research

Synthetic polymers are widely used in varies applications. As synthetic polymers are non-degradable, polymer pollution has become a serious matter. Hence, researchers are developing raw materials for polymers from renewable sources. Nowadays, most people are focusing to develop ecofriendly, natural degradable polymers. Biodegradable polymers can be categorized to two which are gas derived and microorganism derived polymers. For gas such as petroleum derived polymers, it is non-environmentally friendly because they emit greenhouse gases during the production. Whereas, microorganism derived biodegradable polymers utilize its bioactivity to produce a product for polymerization from plant source.

Poly lactic acid (PLA), the sample of this research is made by using the activity of microorganisms. This kind of polymer uses renewable feedstock and the production process releases carbon. PLA can be one of the most promising biodegradable polymers which have biocompatibility and good mechanical properties. It has tensile strength of 48 to 53MPa and modulus of 3500MPa (Jamshidian *et al.*, 2010).

However, the insufficient impact strength, inherent brittleness and low thermal stability of PLA are limiting the scope of its application. Strategies include copolymerization, polymer blends and addition of plasticizers, nanoscale reinforcing fillers and natural fibers. PLA polymers have a characteristic of aliphatic polyesters that absorb moisture which helps in its pathway of degradation. Biodegradable polymer degrades under aerobic condition which must have water and air exists so that the microbial biodegradation will take place.

In particular, cellulose nano fibrils (NFC) are ideal reinforcing materials for nanocomposites as they have high mechanical strength performance, high aspect ratio and large specific surface area. High quality of NFCs can be obtained at low cost through mechanical disintegration of wood pulp fiber after enzymatic or chemical pretreatments. When NFC is aligned, it can be a good reinforcement to its composites. Even though the nano fibers are randomly oriented, it might be possible to enhance the composites' properties (Jonoobi et al., 2010). The NFC in the bio nanocomposite materials also gives enhancement in thermal stability because of a strong NFC bond that is formed within the matrix. (Karolina Larsson et al., 2013) When compared to conventional fibers, natural fibers have the advantages of biodegradability, light weight and low environmental impact. Besides, when blends with polymer, it provides good mechanical properties to the polymer matrix composites.

Bio-reinforced green composites from renewable polymers and natural fibers have demonstrated substantially improved barrier properties, mechanical properties and thermal stability. Bio reinforced composite such as PLA with natural and man-made cellulose fibers such as wood pulp fibers, plant fibers from kenaf, flax, hemp, bamboo, ramie and even agriculture residues, regenerated cellulose fibers and recycled newspaper fibers have been prepared and extensively studied. Biodegradable polymers are other alternatives for petroleum derived polymers since they are overall biodegradable and renewable without releasing any toxic compounds to the environment (Alkbir *et al.*, 2016).

Biodegradable composites can be designed for automobile industry, conducting composite, barrier applications, drug delivery systems, building and construction industry, and adhesives (Saba *et al.*, 2015). There are two different applications for degradable polymers. The first is where biodegradability is part of the function of the product. Biodegradable plastics are used in bone fixation parts, drug release control system and compost bags (Rogina, 2014). As this composite has biodegradability property, it can be applied in preparing bioplastic that can be used for packaging, disposable tableware, compost bags and food packaging (Awadhiya *et al.*, 2017).

1.2 Problem Statement

NFC is abundantly available as in its raw materials are from plants. As for this research, NFC is fibrillated from chemically treated kenaf fiber. Instead of its abundant availability, NFC is not commercially available in large amount as it consumes time and the yield of it is low. The separation of nano cellulosic materials from plant sources gives a big challenge as it requires high energy. Other than that, the cellulose tends to absorb moisture which increases the challenge of fibrillation process. Based on the study by Li *et al.* (2017), the thermal degradation temperature of NFC is occurring around 200 to 300 degree Celsius which could be limitation to NFC applications.

The materials properties of virgin polymer will show significant improvement when nano fibers are well incorporated in polymer matrices. But most of the nano fibers are costly to produce, hardly to be recycled and does not degrade after its usage period. For this research, the nano fibers used are nano fibrillated cellulose. In composite world, it is vital for the reinforcement fibers to disperse evenly in the matrix for the material properties. Dispersion plays an vital role in improving the strength of biodegradable nanocomposites, as nanoscale particles that are separated homogenously will give better interfacial contact outcome between the matrix and the particles. It is hard to quantify the dispersion of NFCs as the nano fibers greatly blurred the crystalline texture. Hence, he mixing process is important to ensure full utilization of the reinforcing properties of the NFC. PLA, a biopolymer, is a non-polar and hydrophobic, which complicates the use of NFC as reinforcement since it has hydrophilic properties. One of the solving methods can be by dispersing the polymer particles in aqueous form and the matrix in latex form. This method enables direct wet mixing of the polymer particles and matrix in their dispersed state, thus preventing aggregation on drying.

In this research, different parameters of rotating speed will be used during the mechanical rotational process to obtain a higher yield of nanofiber cellulosic from the kenaf fibers. For a better dispersion of the NFC in PLA, it is important to have surface chemical reactions that are thermodynamically and kinetically favored in order to accomplish this.

1.3 Objectives

The objectives of this research are:

a) To determine the yield of NFC after chemico mechanical treatment of natural fibers through acid hydrolysis, mechanical rotational and fibrillation process.

b) To analyze the distribution and dispersion morphology of NFC in NFC- PLA composite.

c) To investigate the reinforcing effect on mechanical properties of NFC- PLA composite.

1.4 Scopes of the Research

For this research, we will be focusing on the respective processes for the production of nano fibrillated cellulose which are through acid and alkaline hydrolysis and high speed rotating blender. NFC- PLA composite are to be prepared by internal mixer and hot compression molding.

The scopes of research are as follows:

a) Research on the percentage yield of NFC through acid and alkaline hydrolysis and high speed rotational blending treatment. In this research will be focused more on utilizing distinctive parameter which resulting in high yield of NFC. It will utilize the use of mechanical rotational treatment fibrillation which is the high speed rotation blender. Before that, it will be treated with acid and alkaline hydrolysis.

b) Analyze the morphological appearance in dispersion of NFC in NFC- PLA composite by utilising scanning electron microscope (SEM).

c) Research on investigating the reinforcing effect on mechanical properties of NFC- PLA composite by using mechanical testing (Tensile Testing).

CHAPTER 2

LITERATURE REVIEW

In Chapter 2, it will mainly describe about nano fibrillated cellulose in biodegradable polymer to form biodegradable matrix composite. The details from previous studies are used as references and discussion was done based on their research regarding fibrillation of cellulose fibrils to nanoscale and dispersion of NFC in the NFC-PLA matrix composite. There are many ways that can extract the nanofiber cellulose such as various chemical treatment and mechanical treatment. For this research, acid treatment and mechanical treatment with different rotational blending speed will be used. As for the polymer matrix composite, different characterization techniques are used to identify its physical, chemical and mechanical properties.