UNIVERSITI PUTRA MALAYSIA

CELLULOSE NANOFIBERS FROM OIL PALM MESOCARP FIBER AND THEIR UTILIZATION AS REINFORCEMENT MATERIAL IN LOW DENSITY POLYETHYLENE COMPOSITES

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Chair : **Hidayah Ariffin, PhD**
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Oil palm mesocarp fiber (OPMF) is made up of mainly cellulose, making it a potentially raw material for microfibrillated cellulose (MFC) and cellulose nanofiber (CNF) production. MFC and CNF properties may be influenced by their production method; it is therefore in this study three different methods were used for the production of MFC and CNF from OPMF: electrospinning, ultrasonication and high pressure homogenization. In electrospinning, cellulose concentration and ionic liquid formulation affected the cellulose solubility, and viscosity; which influenced the properties of the MFC produced. The best MFC was formed when 6% (w/v) OPMF-cellulose was dissolved in ([EMIM]Cl:([C_{10}MIM][Cl]):DMF; whereby MFC with average diameter of 200 to 500 nm, crystallinity of 57% and $T_{d50\%}$ at 348 °C was obtained. By using electrospinning, nano-sized fiber (< 100nm) was not obtained, hence, ultrasonication and high pressure homogenization were conducted. Ultrasonication at 125 W and 36 kHz for 9 hours produced mixture of MFC and CNF with non-homogeneous diameter size between 40 – 200 nm, having crystallinity index and $T_{d50\%}$ of 41% and 338 °C, respectively. Meanwhile, high pressure homogenizationconducted at 50 MPa for 30 passes with cellulose concentration of 0.2% (w/v) resulted in CNF with diameter of 80 – 100 nm, crystallinity index of 62% and $T_{d50\%}$ at 353 °C. The CNF obtained from high pressure homogenization method was then used as reinforcement material for low density polyethylene (LDPE) composites production. Effect of melt compounding methods on the mechanical properties of nano-sized fiber composites was determined. Composites consisted of low density polyethylene (LDPE), maleic anhydride-grafted PE (PE*g*MA) and CNF at formulations of 97/3/0.5–5 (wt/wt/wt), respectively, were prepared by twin screw extrusion and internal melt blending processes. Morphology of the composites as revealed by SEM-EDS and X-CT scan showed that the twin screw extrusion process permitted homogeneous dispersion of CNF, thus led to an increment of up to 195% in flexural strength compared to neat LDPE. In contrast, the composites prepared by internal melt-blending method showed an agglomeration and heterogeneous dispersion of CNF within LDPE matrix, caused the composites to have lower tensile strength and flexural strength compared to those prepared by twin screw extrusion. CNF-based composites preparation method can be shortened by introducing simultaneous nanofibrillation and melt compounding using one unit operation. Herewith, a one-pot process was conducted by using an extruder with specially designed twin screw. FE-SEM micrograph exhibited that the resultant LDPE/CNF composites had CNF with average diameter of 80 – 100 nm. These composites prepared by one-pot process had similar properties with those prepared in conventional two-pot process, with the advantage of having higher productivity – by almost doubled. A two-step in one unit operation (2-in-1) would be an ideal process for composites making as this method may improve productivity, reduce downtime in between the two steps, could contribute to a lower capital and processing costs, and may have lower energy consumption. The one-pot process also meets most of the Green Chemistry Principles; suggesting the method as a sustainable and greener method for polymer composites production.

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NANOFIBER SELULOSA DARIPADA SERAT SABUT KELAPA SAWIT DAN PENGGUNAANNYA SEBAGAI BAHAN PENGUKUH DALAM KOMPOSIT POLIETILINA BERKETUMPATAN RENDAH

Oleh

TENGKU ARISYAH BINTI TENGKU YASIM ANUAR

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Pengerusi : Hidayah Ariffin, PhD
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Serat sabut kelapa sawit (OPMF) kebanyakannya terdiri daripada selulosa, menjadikannya bahan mentah yang berpotensi digunakan untuk pengeluaran mikrofiber selulosa (MFC) dan nanofiber selulosa (CNF). Ciri-ciri MFC dan CNF mungkin dipengaruhi oleh kaedah pengeluaran; oleh itu, dalam kajian ini tiga kaedah pengeluaran yang berbeza telah digunakan untuk pengeluaran MFC dan CNF daripada OPMF: elektroputaran, ultrasonikasi dan homogenisasi bertekanan tinggi. Untuk elektroputaran, kepekatan selulosa dan formulasi cecair ionik mempengaruhi kelarutan dan kelikatan selulosa; di mana turut mempengaruhi ciri-ciri MFC yang dihasilkan. Pembentukan MFC terbaik terhasil apabila 6% (w/v) selulosa-OPMF dilarutkan dalam [EMIM]Cl:([C_{10}MIM][Cl]):DMF, dimana selulosa fibrilasi mempunyai diameter antara 200 – 500 nm, penghabrulan 57% dan $T_{d50\%}$ pada suhu 348 °C. Dengan menggunakan elektroputaran, fiber bersaiz nano (< 100 nm) tidak diperolehi, oleh itu, ultrasonikasi dan homogenisasi bertekanan tinggi dijalankan. Ultrasonikasi yang dijalankan pada 125 W dan 36 kHz selama 9 jam menghasilkan MFC yang tidak homogen dengan diameter diantara 40 – 200 nm, nilai penghabrulan dan $T_{d50\%}$ pada 41% dan 338 °C. Sementara itu, homogenisasi bertekanan tinggi yang dijalankan pada tekanan 50 MPa untuk 30 pusingan dengan kepekatan sellulosa sebanyak 0.2% (w/v) berjaya menghasilkan CNF dengan diameter 80 – 100 nm, 62% penghabrulan dan $T_{d50\%}$ pada suhu 353 °C. CNF daripada kaedah homogenisasi bertekanan tinggi kemudiannya digunakan sebagai bahan pengukuh dalam pembuatan komposit-polietilina berketumpatan rendah (LDPE). Kesan kaedah adunan cair terhadap ciri-ciri mekanikal bagi biokomposit berasaskan nanofiber telah dikenalpasti. Komposit yang terdiri daripada polietilina berketumpatan rendah (LDPE), maleic

anhidrida-polietilena (PE*g*MA) dan CNF pada formulasi (97/3/0.5-5 wt/wt/wt) setiap satunya, disediakan melalui kaedah penyemperitan skru berkembar dan pengadunan leburan. Analisis morfologi daripada SEM-EDS dan imbasan X-CT menunjukkan proses penyemperitan skru berkembar menghasilkan taburan CNF yang homogen, seterusnya meningkatkan kelenturan sebanyak 195% berbanding PE sahaja. Sebaliknya, komposit yang disediakan melalui kaedah pengadunan leburan menunjukkan gumpalan dan taburan CNF secara heterogen dalam matrik LDPE, menyebabkan komposit tersebut mempunyai nilai kekuatan tegangan dan lenturan yang lebih rendah berbanding dengan komposit daripada kaedah penyemperitan skru berkembar. Kaedah penyediaan komposit berasaskan CNF boleh dipendekkan dengan menjalankan proses nanofibrilasi dan pencairan pengkompaunan secara serentak menggunakan satu unit operasi. Kaedah pemprosesan satu pot dijalankan menggunakan ekstruder yang dilengkapi dengan skru berkembar direka khas. Mikrograf FE-SEM mempamerkan komposit LDPE/CNF yang terhasil mempunyai diameter 80 – 100 nm. Komposit yang disediakan melalui proses satu-pot mempunyai ciri-ciri yang sama dengan komposit yang disediakan melalui kaedah konvensional dua-pot, dengan daya pengeluaran yang lebih tinggi - hampir dua kali ganda. Dua langkah dalam satu unit operasi (2-dalam-1) akan menjadi proses yang ideal untuk pembuatan komposit kerada kaedah ini dapat meningkatkan pengeluaran, mengurangkan masa henti antara kedua-dua proses, mampu menyumbang kepada modal dan kos pengeluaran yang rendah, dan mungkin mempunyai penggunaan tenaga yang lebih rendah. Proses satu-pot juga memenuhi sebahagian besar Prinsip Kimia Hijau; mencadangkan kaedah ini sebagai kaedah lestari dan hijau bagi pengeluaran komposit polimer.
.

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I certify that a Thesis Examination Committee has met on 26 June 2018 to conduct the final examination of Tengku Arisyah binti Tengku Yasim Anuar on her thesis entitled "Cellulose Nanofibers from Oil Palm Mesocarp Fiber and their Utilization as Reinforcement Material in Low Density Polyethylene Composites" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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LIST OF ABBREVIATIONS

BNC	Bacterial nanocellulose
CNC	Cellulose nanocrystal
CNF	Cellulose nanofiber
DP	Degree of polymerization
DSC	Differential scanning calorimetry
FESEM	Field emission scanning electron microscopy
ILs	Ionic liquids
PE*g*MA	Maleic anhydride grafted polyethylene
Mw	Molecular weight
DMF	N, N-Dimethylformamide
OPEFB	Oil palm empty fruit bunch
OPF	Oil palm frond
OPMF	Oil palm mesocarp fiber
PKS	Palm kernel shell
OPT	Oil palm trunk
PE	Polyethylene
SEM-EDS	Scanning electron microscopy-energy dispersive spectroscopy
TG	Thermogravimetric
X-CT	X-ray computed tomography
XRD	X-ray diffractometer

CHAPTER 1

INTRODUCTION

1.1 Overview

The Malaysian oil palm industry generates large quantity of lignocellulosic biomass, with more than 90% of the country's total biomass deriving from both plantations and mills, which disposal is a challenging task. It is estimated that the total oil palm biomass generated in Malaysia is approximately 44.85 Mt (dry weight basis) (Loh 2016). Therefore, with the growing concern on environmental sustainability, abundant and continuous supply of oil palm biomass as well as due to finite supply of non-renewable resources, various industries have started to use oil palm biomass as an alternative raw material for various products manufacturing such as biocomposite (Nordin et al. 2013; Then et al. 2014; Essabir et al. 2016; Syamsu et al. 2016; Warid et al. 2016), biofuel (Abdul-Manan et al. 2014; Chin et al. 2013; Johari et al. 2015; Wu et al. 2017), biochar (Ali et al. 2015; Idris et al. 2015; Kong et al. 2014; Salema et al. 2017), biocompost (Chin et al. 2017; Siddiquee et al. 2017; Widyasti et al. 2017) and many more. However, most research focused on utilizing oil palm empty fruit bunches (OPEFB), while leaving behind the oil palm mesocarp fibers (OPMF), despite of having almost similar chemical properties. Unlike the OPEFB which is still competitively utilized for various applications, the OPMF received less attention due to the belief that OPMF is exclusively used as boiler fuel at the mill (Wu et al. 2017). It has been reported that due to the abundance of OPMF generation daily, it was inefficiently burnt as a mean of disposal, which consequently leads to the environmental pollution (Loh 2016). Hence, instead of treating this OPMF as a trash, particular attention and research can be given to produce valuable materials from OPMF as it is made up of mainly cellulose, which is an important component in various bioproducts production, including cellulose nanofibers (CNF).

CNF is generally extracted from wood pulp though they can also be prepared from any lignocellulosic materials. CNF which is a term referring to nano-structured cellulose is a tiny fiber with light weight, yet possess great strength, stiffness, biodegradability and renewability (Abitbol et al. 2016; Chen et al. 2014; Cheng et al. 2017; Wang et al. 2015c; Zhao et al. 2017). Owing to their good mechanical properties, CNF can be used for several applications ranging from consumer products to high-tech industrial applications. However, there have been few major issues in the production of CNF especially in removing non-cellulose impurities from the wood pulp as well as delaminating the fibers cell wall and separating the nano-sized fibrils without affecting their properties (Abdul Khalil et al. 2014). Due to this matter, selection of nanofibrillation methods need to be carried out thoroughly to ensure the chosen materials are well fibrillated.

Electrospinning is one of the most common method used to produce nano-scale, single and continuous cellulose and non-cellulose nanofiber (Almasian et al. 2015; Chen et al. 2015a). This method requires cellulose to be dissolved in solvents and ionic liquid was among the environmentally friendly and promising solvent that had been commonly used. Among many types of ionic liquids, imidazolium-based ionic liquid was among the most effective ones, as it was not solely able to dissolve cellulose, but it was also able to facilitate electrospinnability (Gao et al. 2013). This type of ionic liquid may influenced the properties of cellulose solution such as viscosity and surface tension which were among the determining parameters for the elctrospinnability of cellulose (Freire et al. 2011a). Besides electrospinning, ultrasonication and high pressure homogenization are another methods that can produce relatively long CNF (Davoudpour et al. 2015). In contrast to electrospinning, cellulose does not necessarily be dissolved in solvents prior to nanofibrillation by these two methods. Ultrasonication and high pressure homogenization were able to mechanically disintegrate the individual CNF from the cellulose microfibrils by breaking down the interfibrillar hydrogen bonds during the process (Abdul Khalil et al. 2014).

As a recent developed form of cellulose, CNF has great number of applications and one of them is as a reinforcement material for polymer composites. In general, polymer composites can be defined as a combination of polymer matrix and reinforce material having size less than 100 nm for at least one dimension, either length, width or diameter (Salas et al. 2014). Since the use of CNF as a reinforcing material in polymer composites is relatively new, the most convenient compounding method to produce polymer nanocomposite reinforced with CNF has yet been determined. Conventionally, micro-scale fillers were found to be well dispersed and aligned in a polymer matrix prepared by twin screw extrusion and were heterogeneously dispersed and randomly oriented when prepared by internal melt-blending method (Pickering et al. 2016). Similar effect however still not been reported for polymer composites reinforced with CNF. Hence, this study attempted to study the effect of compounding methods on the mechanical properties of polymer composites reinforced with CNF.

Other than that, despite of the well-developed method, nanocomposite production process is still time consuming. The two separate steps; nanofibrillation and nanocomposite production require more processing time and plural equipments, as both processes need to be conducted in separate two unit operations. In fact, due to the aqueous nature of CNF, it has poor compatibility with hydrophobic polymer which consequently leads to poor dispersion of CNF in polymer matrix (Abitbol et al. 2016; Herrera et al. 2015a; Pickering et al. 2016). To overcome this problem, further study has been carried out to produce composites in one-step process using an extruder. In the other words, both nanofibrillation and nanocomposite production process have been carried out simultaneously in *in-situ* condition. The chemical, physical and mechanical properties of the composites produced by this one-pot extrusion were compared with other composites produced by two-steps processing.

1.2 Problem Statements

Generally in biocomposite production, the dispersion and orientation of natural fiber in polymer matrix will play an important role on the properties of biocomposite developed. In the case of micro-scale fillers, the dispersion and orientation of the fillers are varied, depending on the fabrication process. Conventionally, micro-scale fillers reinforcing polymer matrix prepared by twin-screw extrusion will be well-dispersed and well-aligned (Faruk et al. 2012), while those prepared by internal melt blending method will be randomly dispersed and poorly aligned (Then et al. 2013). Similar effect however is still unknown for CNF, as to date, there have been no reports on this. Nano-scale fiber may give different reinforcement effect. Due to its tiny size, it is able to fill in the polymer matrix gaps, regardless of the compounding methods. This may create reinforcement effect and affect the biocomposite properties. Another issue with CNF as reinforcement material is its high tendency to agglomerate each other. Compounding method like internal blending may promote agglomeration of CNF. It is hence this study was conducted in order to determine the effect of compounding methods on the dispersion of CNF which will eventually affect the mechanical properties of LDPE/CNF composites. The morphology, crystallinity, mechanical, thermal and wettability properties of composites produced by internal-melt blending and twin screw extrusion were compared.

Conventional CNF-based biocomposite production requires two steps of processing. Firstly the CNF preparation can be achieved by nanofibrillation process such as high pressure homogenization, ultrasonication, steam-explosion and many more. Second step involves composite compounding, usually by melt-blending. This conventional two separate processing steps are however laborious and take longer time. In this study, a one-step simultaneous nanofibrillation and nanocomposite production was introduced by using a twin screw extruder. The morphology, crystallinity, mechanical, thermal and wettability properties of composites produced by this method were compared with the nanocomposite prepared by the conventional two steps processing in order to suggest the effectiveness of the one-step simultaneous process for nanocomposite production.

1.3 Objectives

General objective of this study was to utilize oil palm mesocarp fiber for cellulose nanofiber and nanocomposite production. While the specific objectives of this study were:

i. to investigate the influence of ionic liquid formulation on electrospinnability of oil palm mesocarp fiber cellulose solution,
ii. to characterize cellulose nanofiber from oil palm mesocarp fiber using ultrasonication and high pressure homogenization,
iii. to determine effects of compounding methods on the mechanical properties of low density polyethylene/cellulose nanofiber composites, and
iv. to compare the properties of low density polyethylene/cellulose nanofiber composites produced by one-pot and two-pot processing methods.

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