UNIVERSITI PUTRA MALAYSIA

DEVELOPMENT AND CHARACTERIZATION OF ROSELLE (*Hibiscus sabdariffa* L.) NANOCELLULOSE-REINFORCED POLYLACTIC ACID NANOCOMPOSITE FOR WATER FILTRATION

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IPTPH 2019 12

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By

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for Degree of Doctor of Philosophy

September 2019

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

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September 2019

Chairman : Mohammad Jawaid, PhD
Institute : Tropical Forestry and Forest Products

Roselle is abundantly cultivated in tropical areas, such as Malaysia, Borneo, Indonesia, India, Tanzania, Thailand, Sri Lanka, Sudan, Tanzania, Togo and Guyana. Roselle fiber is a potential bio-filler for composites reinforcement. In Malaysia, the roselle plant is harvested annually for its fruit and the remaining parts, such as the stem are disposed of as agricultural waste. Roselle plant is a valuable source of cellulose. The growing environmental awareness endowed the utilization of nanocellulose as alternative fiber to reinforce biopolymers. This research is to investigate the characterization of isolated microcrystalline cellulose, nanocrystalline cellulose and cellulose nanowhisker from roselle fiber and fabrication of polylactic acid nanocomposites for membrane applications. The isolation of microcrystalline cellulose from roselle fiber was conducted by employing bleaching, alkali treatment and acid hydrolysis. Also, different hydrolysis reaction time and sonication time were used to extract both nanocrystalline cellulose and cellulose nanowhisker from microcrystalline cellulose. Characterization of microcrystalline cellulose (MCC), nanocrystalline cellulose (NCC), and cellulose nanowhisker (CNW) were carried out by using various advance equipments. From the implemented experiment, microcrystalline cellulose exhibited rough surface in the form of microcrystallites with high thermal stability which was contributed by the substantial removal of lignin and other residues. Nanocrystalline cellulose and cellulose nanowhisker possessed dimension in nanometer range in the form of needle-like and elongated rod-like particles shape respectively. The degree of crystallinity has been improving for nanocrystalline cellulose (79.5%) and cellulose nanowhisker (79.9%) after the isolation from microcrystalline cellulose (78.0%). Besides this, the thermal stability is found decreasing for both types of nanocellulose particles which resulted by the presence of sulfur content.

At later stage, nanocrystalline cellulose and cellulose nanowhisker were used as reinforcing material to develop polylactic acid (PLA) nanocomposites. Comparison was carried out between nanocrystalline cellulose and cellulose nanowhiskers reinforced polylactic acid nanocomposites to select the suitable membrane for metal ions treatment. As examined from characterization, cellulose nanowhisker filled polylactic acid membranes showed better porous structure than that of nanocrystalline cellulose filled polylactic acid membrane besides its great thermal stability, mechanical strength and metal ions adsorption capability. Ultimately, an improvement is conducted to further enhance the properties of selected nanocellulose reinforced polylactic acid nanocomposite membrane by dual layer fabrication method as well as water vapor-induced phase separation technique. Dual layer membrane filled with 3 wt% cellulose nanowhisker possessed well-laminated two layers structure. The membrane porosity was improved, while the pore size decreased with the increment of nanocellulose loadings from 1 to 3 wt%. This evidenced the nanocellulose acted as pore-forming agents within the membrane. In continuous wastewater filtration, dual layer membrane exhibited high efficiency in removing both Co^{2+} and Ni^{2+} metal ions with 83% and 84%, respectively. Therefore, the experimental settings in this study are believed can be used in future research studies on the isolation and extraction of microcrystalline cellulose, nanocrystalline cellulose and cellulose nanowhisker from roselle fiber as well as the production of good performed roselle nanocellulose reinforced polylactic acid nanocomposite for membrane application.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PEMBANGUNAN DAN KARAKTERISASI ROZEL (*Hibiscus sabdariffa* L.) NANOSELULOSA-MEMPERKUKUH POLYLAKTIK ASID NANOKOMPOSIT UNTUK PENAPISAN AIR

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Rozel banyak ditanam di kawasan tropika, seperti Malaysia, Borneo, Indonesia, India, Tanzania, Thailand, Sri Lanka, Sudan, Tanzania, Togo dan Guyana. Serat rozel adalah potensi bio-pengisi untuk pengukuhan komposit. Di Malaysia, tumbuhan rozel dituai setiap tahun untuk buahnya dan bahagian yang lain seperti batang dilupuskan sebagai sisa pertanian. Tumbuhan rozel adalah sumber yang berharga untuk selulosa. Kesedaran alam sekitar yang semakin meningkat menyumbang penggunaan nanocellulose sebagai serat alternatif untuk menguatkan biopolimer. Kajian ini adalah untuk mengkaji pencirian microkristal selulosa, nanokristal selulosa dan selulosa nanokumis terpencil daripada rozel serat, dan juga fabrikasi nanokomposit polylactic asid untuk aplikasi membrane. Pengasingan microkristal selulosa daripada rozel serat telah dijalankan dengan menggunakan peluntunan, rawatan alkali dan hidrolisis asid. Juga, masa reaksi hidrolisis yang berbeza dan masa sonication digunakan untuk mengekstrak kedua-duanya nanokristal selulosa dan selulosa nanokumis daripada microkristal selulosa. Pencirian microkristal selulosa (MCC), nanokristal selulosa (NCC), dan selulosa nanokumis (CNW) telah dikaji dengan menggunakan pelbagai peralatan canggih. Daripada eksperimen yang dilaksanakan, microkristal selulosa mempamerkan permukaan kasar dalam bentuk mikrokristal dengan kestabilan haba yang tinggi yang disumbangkan oleh penyingkiran lignin dan sisa-sisa lain. Nanokristal selulosa dan selulosa nanokumis memiliki pelbagai nanometer dimensi dalam bentuk batang jarum dan memanjang masing-masing. Darjah kristalografi telah bertambah baik terhadap nanokristal selulosa (79.5%) dan selulosa nanokumis (79.9%) selepas pengasingan daripada microkristal selulosa (78.0%). Selain ini, kestabilan haba didapati menurun pada kedua-dua jenis nanoselulosa disebabkan kewujudan kandungan sulfur.

Pada peringkat seterusnya, nanokristal selulosa dan selulosa nanokumis digunakan sebagai bahan pengukuhan untuk mengembang polylactic asid (PLA) nanokomposit. Perbandingan dilakukan antara nanokristal selulosa dan selulosa nanokumis memperkukuh polylactic asid nanokomposit untuk memilih membran yang sesuai untuk rawatan ion logam. Seperti yang diperiksa dari pencirian, selulosa nanokumis memperkukuh polylactic asid membran menunjukkan struktur berliang yang lebih baik daripada nanokristal selulosa memperkukuh polylactic asid membran selain kestabilan terma, kekuatan mekanikal dan keupayaan penyerapan ion logam. Pada akhirnya, penambahbaikan dilaksanakan untuk mempertingkatkan ciri-ciri selulosa nanokumis memperkukuh polylactic asid nanokomposit dengan kaedah fabrikasi lapisan dua dan juga teknik pemisahan fasa yang didorong oleh wap air. Membran lapisan dua diisi 3 wt% selulosa nanokumis menunjukkan struktur dua lapis yang dilaminasi serentak. Keliangan membran telah bertambah baik, manakala saiz liang menurun dengan penambahan nanoselulosa daripada 1 wt% hingga 3 wt%. Ini membuktikan bahawa nanoselulosa bertindak sebagai agen pembentuk liang dalam membran. Dalam penapisan air sisa berterusan, membran lapisan dua diisi 3 wt% selulosa nanokumis mempamerkan kecekapan tertinggi untuk menyerap Co^{2+} dan Ni^{2+} ion logam dengan 83% dan 84%, masing-masing. Oleh itu, seting percubaan dalam kajian ini dipercayai dapat digunakan dalam kajian penyelidikan masa depan mengenai pengasingan dan pengekstrakan microkristal selulosa, nanokristal selulosa dan selulosa nanokumis daripada rozel serat serta penghasilan rozel nanoselulosa memperkukuh polylactic asid nanokomposit yang berprestasi tinggi untuk aplikasi membran.

ACKNOWLEDGEMENTS

I would like to express my deepest gratitude to my supervisor, Dr. Mohammad Jawaid for his guidance and encouragement throughout my PhD study. I am also greatly thankful to the members of the supervisory committee; Associate Prof. Dr. Hidayah Ariffin, Associate Prof. Dr. Mohamed Thariq Hameed Sultan and Dr. Zoheb Karim for their valuable motivation and assistance during this research. Meanwhile, I am feeling thankful to the Graduate Research Fellowship (GRF) from Universiti Putra Malaysia for supporting my PhD work.

Besides that, I wish to extend my special appreciation to all staff in Institute of Tropical Forestry and Forest Product (INTROP) for their co-operation and help. Also, I am not forgetting all my friends for their tips and endless cooperation whenever I needed. Herein, once again I would like to put forward my gratitude to the technical staffs and my friends for their aiding throughout my research work.

Last but not least, I wish to express my appreciation to my beloved family especially my parents Lau Chin Yong and Ang Moi Sim, my sisters Lau Kia Ning and Lau Kia Li for motivation, encouragement, understanding, and any kind of support towards me during this study lifetime.

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

NCC	Nanocrystalline cellulose
CNW	Cellulose nanowhisker
CNF	Cellulose nanofibril
CNC	Cellulose nanocrystals
BC	Bacterial cellulose
CA	Cellulose acetate
MCC	Microcrystalline cellulose
PLA	Polylactic acid
PBS	Polybutylene succinate
PCL	Polycaprolactone
PEG	Polyethylene glycol
PGA	Polyglycolic acid
PLGA	Polylactic-co-glycolic acid
PVDF	Polyvinylidene difluoride
PES	Polyether sulfone
$NaClO_2$	Sodium chlorite
CH_3COOH	Acetic acid
NaClO	Sodium hypochlorite
H_2O_2	Hydrogen peroxide
NaOH	Sodium hydroxide
KOH	Potassium hydroxide
H_2SO_4	Sulfuric acid
$CaCl_2$	Calcium chloride
HCl	Hydrochloric acid
H_3PO_4	Phosphoric acid
DMAc	N,N-dimethylacetamide
LiCl	Lithium chloride
DMF	N,N-Dimethylformamide
DMSO	Dimethyl sulfoxide
TEMPO	2,2,6,6-tetramethylpiperidine-1-oxyl
NaBr	Sodium bromide

$CHCl_3$	Chloroform
ECF	Elemental chlorine-free
Ag^{+}	Silver(I) ion
Co^{2+}	Copper(II) ion
Ni^{2+}	Nickel(II) ion
Zn^{2+}	Zinc(II) ion
Ba^{2+}	Barium(II) ion
Cu^{2+}	Copper(II) ion
Cd^{2+}	Cadmium(II) ion
Fe^{2+}	Ferrous(II) ion
Fe^{3+}	Ferrous(III) ion
Cr^{3+}	Chromium(III) ion
$CoCl_2.6H_2O$	Cobalt(II) chloride hexahydrate
$NiCl_2.6H_2O$	Nickel(II) chloride hexahydrate
FTIR	Fourier transform infrared ray
XRD	X ray diffraction
TEM	Transmission electron microscopy
SEM	Scanning electron microscopy
FESEM	Field emission scanning electron microscopy
AFM	Atomic force microscopy
TGA	Thermogravimetric analysis
DTG	Derivative thermogravimetric analysis
DSC	Differential scanning calorimetry
BET	Brunauer-Emmett-Teller
ICP-OES	Inductive coupled plasma optical emission spectrometry
R_{ir}	Irreversible fouling
R_r	Reversible fouling ratio
R_t	Total fouling ratio
FR_w	Flux recovery of pure water
FR_p	Flux recovery of metal ion solution

CHAPTER 1

INTRODUCTION

1.1 Background

The development of nanosized particles from renewable source as reinforcing materials in nanocomposites has become interest of researchers. Nanocellulose is stiff nanometric particles and biodegradable materials from cellulose biopolymer. Nanocellulose can be categorized into nanocrystalline cellulose (NCC), nanowhisker cellulose (CNW), cellulose nanofiber (CNF) and bacterial cellulose (BC) (Salminen et al., 2017; Lin and Dufresne, 2014). The categorization depends on their physical characteristics of dimension, size and stiffness. Cellulose can be processed into nanocellulose by chemical or mechanical means (Jasmani and Adnan, 2017; Santos et al., 2017). The low cost of cellulose due to its availability and abundance in nature that make it as a bio-fillers in polymer matrixes. Cellulose possesses high mechanical strength and good thermal resistance properties. It sources from natural fibers like roselle, cotton, wood, sisal, flax, hemp, jute, ramie, oil palm, kenaf and coir (Fallahi et al., 2017; Razali et al., 2015).

Nanocellulose is widely utilized to develop nanocomposites or polymer composites because of its low density, high thermal stability, high tensile strength, biocompatible and biodegradable. Nanocellulose reinforced nanocomposites are environmentally friendly materials that can be known as green composites (Prathapan et al., 2016). It can be applied in various industries such as food packaging, automotive, furniture, construction and biomedical (Lin and Dufresne, 2014). Amongst the nanocellulose, NCC and CNW are discerned as the promising reinforcing agents of bionanocomposite owing to the interaction between the nano-sized components that form a percolated network connected by hydrogen bonding.

Roselle is a valuable source of natural fiber which consists of cellulose, hemicellulose and lignin as the majority lignocellulosic components in its bast fiber structure. The stem fiber of roselle is found abundantly as agricultural crops residues after cultivation for its calyx to manufacture beverage, food and other nutraceutical products (Bandera et al., 2015; Moemin, 2016). Nanocellulose processed from roselle has potentially to be used in blending with polylactic acid to reduce its nature deficiencies such as brittle, low thermal stability and abrasive that limits its extensive application (Basu et al., 2016; Sharma et al., 2016).

Polylactic acid nanocomposites reinforced with roselle nanocellulose is an underutilized and promising membrane material to provide high-performance composites in various fields of industries, particularly in metal ions treatment process. They also have broad commercial application range in the future owing to its comparable properties with petroleum-based composites (Fabra et al., 2016; Saini et al., 2016).

There are very little or no studies have previously been reported on the isolation and characterization of MCC, NCC and CNW from roselle fiber. Moreover, there is less comparative study made between isolated NCC and CNW from roselle fiber used as bio-nanofiller in reinforcement of polylatic acid based nanocomposites. In this study, roselle nanocellulose is utilized as the alternative biomaterials to reinforce polylactic acid based nanocomposites for membrane application in the aim to remove metal ions from water.

1.2 Problem Statement

Roselle has become an important plant today which cultivated across the states of Johor (Rengit and Mersing regions), Kedah (Sik district), Perak (around Sungkai) and other states, covering a total area of 800-1200 hectare according to the Department of Agriculture Malaysia. The cultivation is significant with about 7400-11100 plants/hectare on arable land. In Malaysia, the roselle plant is harvested for its fruit annually and the remaining parts such as stem are disposed as agricultural waste (Moemin, 2016; Razali et al., 2015). However, there is environmental problem associated with the discarding of plant fiber without any further utilization.

In order to curb the problems of environmental pollution, nanocomposite can be developed by the incorporation of nanocellulose from roselle fiber with bio-based polymeric material like polylactic acid for metal ion removal application. The challenge in the development of bionanocomposite is to increase the compatibility between nanocellulose and biopolymer blend in order to ameliorate the dispersion of nanocellulose particles. Nanocellulose reinforced composites demonstrate poor dispersion of filler in the matrix owing to the hydrophilic and hydrophobic nature respectively. Solution casting and evaporation technique is one of the easiest methods to prepare bionanocomposite film with good dispersion and reinforcement results (Lin, & Dufresne, 2014). For example, Qu et al. (2010) produced nanocomposites of polylactic acid reinforced with cellulose nanofibrils using solution casting method and adding polyethylene glycol to the matrix as compatibilizer. Khan et al. (2012) prepared nanocrystalline cellulose reinforced chitosan based nanocomposite films and Lani et al. (2014) prepared polyvinyl alcohol/starch blend films reinforced with nanocellulose by solution casting method.

Besides that, another concern is about the currently increasing demands of bio-based membrane by electronic, aircraft, automobile and mining industries for water treatment purpose in order to remove metal ions from discharged water. It is challenging to design membrane with specific pore sizes while maintaining its adsorption properties for removing metal ions. Several criteria are required for consideration when in designing membrane to separate metal ions by adsorption. Membrane must have interconnected porous structure for water penetration; be able to provide surfacial anionic charge to adsorb positively charged metal ions; possess well-designed pore configuration for metal attachment; and have minimum loss of water flux during wastewater filtration (Karim et al., 2016; Liu et al., 2016). Since that, there are difficulties in controlling overall porous structure by using one step technique of fabrication. Multiple processing steps and additional post-processing are usually required for membrane fabrication. It may lead to high cost and less membranes production in meeting the market demand (Liu et al., 2017; Zeid et al., 2018).

Biopolymers like polylactic acid (PLA), poly(butylene succinate) (PBS), polycaprolactone (PCL), polyethylene glycol (PEG), polyglycolic acid (PGA) and poly(lactic-co-glycolic) acid (PLGA) have advantages in membrane engineering because they exhibit great mechanical properties in supporting porous structure, good processability and are commercially available (Armentano et al., 2010; Liu et al., 2012; Murphy et al., 2017). However, there is a major concern in adsorbing metal elements on these biopolymers' surface owing to the less interaction of positively charged metal ions on hydrophobic materials. Therefore, in this study, the strategy of incorporating sulfated NCC and CNW into biopolymers is to be utilized as surface-modifying materials in PLA nanocomposite for metal ions removal. Another advantage is that the nanocellulose can be used to improve the hydrophilicity of the PLA nanocomposite for increasing high water flux permeability. In addition, the utilization of NCC and CNW in PLA nanocomposite is believed to increase its mechanical strength as well besides acting as surface-modifying material. Nevertheless, dual layer fabrication integrated with water vapor-induced phase inversion process is to be conducted for PLA nanocomposite with PBS polymer to control its porous characteristics which subsequently could contribute to the final interfacial interaction between polymers and nanocellulose materials (Danso et al., 2018; Karim et al., 2016).

1.3 Objective

1. To isolate and characterize the microcrystalline cellulose, nanocrystalline cellulose and cellulose nanowisker from roselle.
2. To compare the properties of nanocrystalline cellulose and cellulose nanowhisker in reinforcing polylactic acid nanocomposites.
3. To evaluate the dual layer fabrication of nanocellulose reinforced polylactic acid/polybutylene succinate nanocomposites membrane for heavy metal ions removal.

1.4 Significance, research gap and future scope

Present research work explored the processing conditions required for the production of nanocellulose from bast fibers and its further reinforcement in PLA biopolymer. Besides this it also highlighted the various processing techniques suitable to yield specific types of nanocellulose from bast fibers source and from other source of plant fibers as well. Moreover, this research deals with certain issues related to the preparation nanocellulose from bast fibers required before going through PLA nanocomposites fabrications.

So far no comprehensive study on methodology has been yet reported to deeply discuss the varieties of technologies that are deemed to contribute great impact in the isolation of nanocellulose from bast fibers in the future. Present work also intended to analyze the experimental settings to substantially address the research gap in nanocellulose treatment technologies. Success of this study on bast fiber nanocellulose could more or less address the main challenges in optimizing PLA nanocomposite fabrications observed in nanotechnology based industries.

1.5 Thesis Layout

This thesis is structured into eight chapters. The first chapter contains the background of this research, problem statement and objectives. Chapter two will provide the research and literature on structural characteristic of bast fibers; nanocellulose and polylactic acid; nanocellulose reinforced PLA polymer blend nanocomposites; processing techniques on bast fibers into nanocellulose and PLA nanocomposites; and potentiality of roselle nanocellulose in reinforcing polylactic acid nanocomposites for removing metal ions in membrane application. Third, fourth, fifth, sixth and seventh chapters will cover results and discussion as well as the information on material, methodology and characterization of the research. From these five chapters, the result will be discussed based on scientific point of view and compared with related works. The last chapter will present the major outcome of the research finding and recommendation for future works.

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LIST OF PUBLICATIONS

Published papers

Lau Kia Kian, Mohammad Jawaid, Hidayah Ariffin, and Othman Y. Alothman. (2017). Isolation and characterization of microcrystalline cellulose from roselle fibers. ***International Journal of Biological Macromolecules***, 103, 931-940. (Published)

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