



UNIVERSITI PUTRA MALAYSIA

**DEVELOPMENT OF EFFICIENT PROCESSING METHOD FOR THE
PRODUCTION OF CELLULOSE NANOFIBRILS FROM OIL PALM
BIOMASS**

LIANA BINTI NOOR MEGASHAH

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By

LIANA BINTI NOOR MEGASHAH

**Thesis Submitted to the School of Graduate Studies, Universiti Putra
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Philosophy**

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

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Chair : Hidayah Ariffin, PhD
Faculty : Biotechnology and Biomolecular Sciences

Cellulose nanofibrils (CNFs) is an emerging, versatile nanomaterial with vast applications such as plastics, papers, composites, thickener agents, healthcare, coatings etc. Nevertheless, there are several issues in the sustainable production of CNFs pertaining to the non-ecofriendly pretreatment method for cellulose isolation related to the use of chlorinated solution, as well as high energy intensity and throughput limitation which limit the productivity during nanofibrillation process by wet disc mill (WDM). The natural cellulose high degree of polymerization (DP) caused the formation of a highly viscous cellulose suspension during processing and was hypothesized to contribute to the high energy requirement and low productivity of the nanofibrillation process. In this research, cellulose isolation from oil palm biomass was conducted by using a totally chlorine free (TCF) bleaching for lignin removal. A multi-step pretreatment method consisting of a sequence of pretreatment, *i.e.*, superheated steam (SHS), enzymatic hydrolysis and 5% NaOH was evaluated for its effectiveness in hemicellulose removal and its effect on the environmental loads. The multi-step pretreatment method was compared with the conventional soda pulping method at 14% NaOH under elevated pressure. After cellulose isolation step, the cellulose was treated by SHS at 150°C for 1 and 2h (SHS1 and SHS2) aimed at depolymerization for DP reduction. This method was compared with the enzymatic hydrolysis. It was shown that the multi-step pretreatment method produced lesser purity cellulose from oil palm biomass (83-88%) as compared to soda pulping method (89-95%). Its environmental load based on qualitative analysis was however similar to that of soda pulping. The TCF bleaching successfully removed the lignin almost completely, showing the effectiveness of

TCF bleaching as an alternative to chlorinated bleaching. In the subsequent experiment for cellulose DP reduction, it was demonstrated that SHS treatment caused cellulose DP reduction up to 43% after 2h of SHS treatment (SHS2). As

a comparison, enzymatic hydrolysis contributed to almost similar percentage reduction after 6h and 12h of hydrolysis using 20 FPU/g and 10 FPU/g cellulase, respectively. The SHS treated cellulose was used in nanofibrillation process, and it was interesting to note that SHS2 cellulose (DP - 820) contributed to lower viscosity CNFs suspension (60 cP), shorter processing duration (4.0 h/kg), and smoother processing without clogging even at 4 wt% solid content processing; compared to the untreated (UT) cellulose (DP - 1,440). All these contributed to higher CNFs productivity by 86% from 0.044 kg/h to 0.320 kg/h, and lower energy consumption by 90% from 42.3 kWh/kg to 4.2 kWh/kg, compared to the untreated (UT) cellulose. The results obtained confirmed the hypothesis that CNFs productivity and energy consumption were related to the original characteristic of cellulose, *i.e.*, high DP. The SHS treatment also contributed to versatile properties of CNFs produced, as exhibited by the characteristics of the CNFs as well as the CNF films. CNF-SHS films were thinner, stiffer and had smoother surface compared to CNF-UT film. Higher light transmittance by 22.5% and greater water-repellent property by 15.1% were also recorded for CNF-SHS2 film as compared to the CNF-UT film. SHS treatment also promotes the production of versatile mechanical properties of CNF films, to meet vast applications of nanofilms. Feasibility analysis conducted showed that the pretreatment and production methods proposed are technical feasible, which can attribute to the lower environmental loads compared to the conventional process, the simplicity of the process, as well as the availability of the steam energy should the plant be located near the palm oil mill. It is also economically feasible with the NPV of USD 714,031 for 10 years and IRR of 45%. CNFs from OPEFB is also potentially marketable based on its comparable characteristics to those of commercial CNFs, as well as its market acceptability based on the survey. Overall, the proposed processing methods provided herewith will contribute significantly towards a more sustainable CNFs production from oil palm biomass in the near future.

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

**PEMBANGUNAN KAEDAH PEMROSESAN TENAGA YANG EFISIEN
DALAM PENGHASILAN NANOFIBRIL SELULOSA DARIPADA SISA
BIOMAS KELAPA SAWIT**

Oleh

LIANA BINTI NOOR MEGASHAH

Februari 2021

Pengerusi : Hidayah Ariffin, PhD
Fakulti : Bioteknologi dan Sains Biomolekul

Nanofibril selulosa (CNFs) merupakan nanomaterial baru serta serba boleh dengan aplikasi yang luas seperti plastik, kertas, komposit, agen pemekat, produk kesihatan, pelapis permukaan dan sebagainya. Walaupun begitu, terdapat beberapa masalah dalam penghasilan CNFs yang berkesinambungan dengan kaedah pra-rawatan tidak mesra iaitu pengekstrakan selulosa menggunakan peluntur klorat, serta intensiti tenaga yang tinggi dan daya pemrosesan yang membatasi produktiviti semasa proses nanofibrilasi oleh penggilingan cakera basah (WDM). Darjah pempolimeran (DP) tahap tinggi oleh selulosa semula jadi menyebabkan penghasilan larutan selulosa yang berkelikatan tinggi semasa pemrosesan, dan dihipotesis akan mempengaruhi kepada penggunaan tenaga tinggi dan penurunan produktiviti bagi proses nanofibrilasi. Dalam penyelidikan ini, pengekstrakan selulosa dari biomas kelapa sawit dijalankan dengan menggunakan peluntur bebas klorin (TCF) untuk penyingkiran lignin. Kaedah pra-rawatan pelbagai langkah yang terdiri daripada susunan pra-rawatan, iaitu rawatan stim panas lampau (SHS), hidrolisis enzim dan rawatan berkali berkepekatan 5% telah dinilai memberi keberkesanan dalam penyingkiran hemiselulosa dan memberi impak terhadap beban persekitaran. Kaedah pra-rawatan pelbagai langkah dibandingkan dengan rawatan konvensional pulpa-soda berkepekatan alkali 14% melalui tekanan tinggi. Setelah langkah pengasingan selulosa, selulosa tersebut dirawat menggunakan SHS pada suhu 150°C selama 1 dan 2 jam (SHS1 dan SHS2), bertujuan depolimerisasi untuk mengurangkan DP. Kaedah ini dibandingkan dengan hidrolisis enzim. Kaedah pelbagai langkah memberi hasil nilai selulosa yang lebih rendah daripada biomas kelapa sawit (83-88%) jika dibandingkan dengan kaedah pulpa-soda (89-95%). Walau bagaimanapun, beban persekitaran berdasarkan analisis kualitatif adalah setanding dengan pulpa-

soda. Peluntur TCF berjaya menyingkirkan lignin hampir keseluruhannya, membuktikan keberkesanan peluntur TCF sebagai rawatan alternatif bagi menggantikan peluntur klorit. Dalam eksperimen berikutnya untuk pengurangan DP selulosa, rawatan SHS menunjukkan penurunan DP selulosa sehingga 43% dalam rawatan SHS selama 2 jam (SHS2). Sebagai perbandingan, hidrolisis enzim menyumbang kepada pengurangan peratusan yang hampir sama setelah hidrolisis selama 6 dan 12 jam menggunakan enzim "cellulase" pada 20 FPU/g dan 10 FPU/g. Rawatan SHS selulosa telah digunakan dalam proses nanofibrilasi, dan yang menarik perhatian bahawa selulosa SHS2 (DP - 820) menyumbang kepada kelikatan CNFs yang lebih rendah (60 cP), jangka masa pemrosesan yang lebih pendek (4.0 h/kg), dan pemrosesan yang lebih lancar tanpa menyebabkan penyumbatan, walaupun pada pemrosesan dengan kelikatan 4% kandungan pepejal; berbanding dengan selulosa (UT) yang tidak dirawat (DP - 1,440). Kaedah ini menyumbang kepada peningkatan produktiviti CNFs sebanyak 86% dari 0.044 kg/h kepada 0.320 kg/h, dan penggunaan tenaga yang lebih rendah sebanyak 90% daripada 42.3 kWh/kg kepada 4.2 kWh/kg berbanding dengan selulosa yang tidak dirawat (UT). Hasil yang diperoleh mengesahkan secara hipotesis bahawa produktiviti dan penggunaan tenaga CNFs adalah berkait dengan ciri asal selulosa, iaitu DP yang tinggi. Rawatan SHS juga menyumbang kepada sifat serbaguna CNFs yang dihasilkan, seperti yang dipamerkan oleh ciri-ciri CNFs dan juga CNF filem. Filem daripada CNF-SHS adalah lebih nipis, kuat dan mempunyai permukaan yang lebih licin berbanding filem daripada CNF-UT. Transmisi cahaya adalah lebih tinggi iaitu 22.5% dan sifat kalis air yang tinggi sebanyak 15.1% juga dicatatkan untuk filem CNF-SHS2 berbanding dengan filem CNF-UT. Rawatan SHS juga mendorong penghasilan sifat mekanikal serbaguna filem CNF, bagi memenuhi aplikasi nano-filem yang luas. Analisis kebolehlaksanaan yang dilakukan menunjukkan bahawa kaedah pra-rawatan dan proses penghasilan yang dicadangkan secara teknikal adalah fisibel yang tersedia yang mana dapat menyumbang kepada pengurangan beban persekitaran berbanding proses konvensional, kesederhanaan proses, serta ketersediaan tenaga stim sekiranya lokasi kilang berhampiran dengan kilang sawit. Ia juga dapat dilaksanakan secara ekonomi dengan nilai NPV iaitu USD 714,031 bagi tempoh 10 tahun dan IRR sebanyak 45%. CNFs dari OPEFB juga berpotensi untuk dipasarkan berdasarkan ciri kesetandingnya dengan ciri-ciri CNFs komersial, serta penerimaan pasaran berdasarkan tinjauan. Secara keseluruhan, kaedah pemrosesan yang dicadangkan dapat menyumbang secara signifikan terhadap pengeluaran CNFs yang lebih mampan dari biomas kelapa sawit pada masa hadapan.

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“Whoever takes a path for seeking knowledge, Allah will ease for him the path to Paradise”.

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

Hidayah binti Ariffin, PhD

Professor Ts.
Faculty of Biotechnology and Biomolecular Sciences
Universiti Putra Malaysia
(Chairman)

Mohd Rafein bin Zakaria @ Mamat, PhD

Associate Professor
Faculty of Biotechnology and Biomolecular Sciences
Universiti Putra Malaysia
(Member)

Yoshito Ando, PhD

Associate Professor
Graduate School of Life Sciences and System Engineering
Kyushu Institute of Technology, Japan
(Member)

ZALILAH MOHD SHARIFF, PhD

Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

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Name and Matric No.: Liana binti Noor Megashah, GS47569

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Signature: _____
Name of Chairman
of Supervisory
Committee: Hidayah binti Ariffin

Signature: _____
Name of Member of
Supervisory
Committee: Mohd Rafein bin Zakaria @ Mamat

Signature: _____
Name of Member of
Supervisory
Committee: Yoshito Ando

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LIST OF ABBREVIATIONS

| | |
|---------------------|---|
| α | Alpha |
| β | Beta |
| θ | Theta |
| $^{\circ}\text{C}$ | Degree Celsius |
| η | intrinsic viscosity |
| η_{inh} | inherent viscosity |
| AGU | Anhydroglucose unit |
| AFM | Atomic force microscopy |
| AQ | Anthraquinone |
| AXU | Active xylanase unit |
| ATR | Attenuated total reflectance |
| BNC | Bacterial nanocellulose |
| CED | Cupriethylenediamine |
| CNF | Cellulose nanofibril |
| CNC | Cellulose nanocrystal |
| cP | Centipoise |
| CrI | Crystallinity index |
| DP | Degree of polymerization |
| ECF | Elemental chlorine free |
| GPa | Giga-pascal |
| FE-SEM | Field emission-scanning electron microscope |
| FPU | Filter Paper Unit |
| FTIR | Fourier-transform infrared spectroscopy |

| | |
|-------|---|
| GNI | Gross National Income |
| KBr | Potassium bromide |
| kHz | Kilohertz |
| kN | Kilo-newton |
| nm | nanometer |
| MFC | Microfibrillated cellulose |
| MPa | Mega-pascal |
| MPOB | Malaysian Palm Oil Board |
| NaOH | Sodium hydroxide |
| NREL | National Renewable Energy Laboratory |
| OPEFB | Oil palm empty fruit bunch |
| OPF | Oil palm frond |
| OPMF | Oil palm mesocarp fiber |
| OPT | Oil palm trunk |
| PA | Peracetic acid |
| PHA | Polyhydroxyalkanoates |
| PKS | Palm kernel shell |
| POME | Palm oil mill effluent |
| PP | Polypropylene |
| PSD | Power spectral density |
| SEM | Scanning electron microscopy |
| Ra | Arithmetic mean |
| SDGs | Standard development goals |
| SiC | Silicon carbide |
| SHS | Superheated steam |
| TAPPI | Technical Association Pulp & Paper Industry |

| | |
|-------|--|
| TCF | Totally chlorine free |
| TEMPO | 2,2,6,6-Tetramethylpiperidine-1-oxyl radical |
| TG | Thermal gravimetric |
| WDM | Wet disc mill |
| XRD | X-Ray diffractometer |



CHAPTER 1

INTRODUCTION

1.1 Overview

About 80 million tons of oil palm biomass is being generated in Malaysia annually (Aljuboori, 2013; Hassan et al., 2019). Oil palm biomass consists of oil palm mesocarp fiber (OPMF), oil palm empty fruit bunches (OPEFB), oil palm fronds (OPF), oil palm trunks (OPT), leaves and roots. Oil palm biomass are mainly used for fuel generation, composting and mulching (Abas et al., 2011; Hamzah et al., 2019). Other applications include animal feed, organic fertilizer, biosugar, biogas, biochar, and biocomposites (Hassan et al., 2019; Nordin et al., 2017; Samsudin et al., 2019; Zainal et al., 2017). Recently, the use of oil palm biomass for cellulose nanofibrils (CNFs) production has been reported (Norraahim et al., 2018; Yasim-Anuar et al., 2019).

CNFs production from plant cellulose has attracted interest and great deal of research has been widely done (Bardet & Bras, 2014; Foster et al., 2018). CNF's are generally lightweight, has high aspect ratio, gel-like in water, hydrophilic and semi-crystalline (Klemm et al., 2011). Due to these characteristics, CNFs is used to reinforce biocomposites, enhance paper properties, functions as a thickener in paints and coatings, as a low calorie food additive, and could act as a functional material in many other versatile applications (Ariffin et al., 2018; Bardet & Bras, 2014; Kasuga et al., 2018; Missoum et al., 2013; Mohamad Haafiz et al., 2013). At present, commercial scale CNFs production mainly takes place in developed countries such as USA, Canada, Japan, UK, Sweden and Finland, and the CNFs is produced from wood-based resources (Future Markets Inc., 2019). Based on the report, commercial CNFs from non-wood resources is very little, mainly from bamboo. Being a country with abundance of oil palm biomass, Malaysia has great potential to be CNFs producer considering its potential as an emerging material with versatile applications. Therefore, the feasibility of CNFs production from oil palm biomass should be determined.

One of the challenges in CNFs production is related to the isolation of cellulose from lignocellulosic materials since the current pretreatment methods are time-consuming and involve hazardous chemicals. Desizing, scouring and bleaching are the three common pretreatment process to prepare high degree of whiteness of cellulose pulp with minimum degradation (Prabaharan & Rao, 2003). In the conventional process, pulping such as steam explosion and kraft pulping are used to rupture the fiber cell wall and results in removal of hemicellulose (Nechyporchuk et al., 2016; Suzuki et al., 2017). Besides pulping, other cellulose isolation methods are by using ethanol organosolv, acid, alkali, or ionic liquid. These pretreatment methods are inefficient for complete cellulose isolation (Tian

et al., 2017), mainly due to the presence of residual lignin and recalcitrant hemicellulose. Bleaching agents like sodium chlorite, sodium hypochlorite, chlorine dioxide, and chlorate which are strong oxidizing agent are commonly used to remove the lignin. However, these bleaching agents are toxic and could affect human health if handled carelessly; and at the same time, the disposal of these chemicals into the water stream could have a negative impact on aquatic life and other organisms. Thus, it is important to consider the type of pretreatment process in cellulose isolation towards sustainable production of CNFs.

On top of cellulose isolation, the major obstacles in CNFs production are the high energy consumption for the disintegration of the cellulose fibers into CNFs, and the low productivity of the CNFs production process. CNFs is produced through multiple cycle mechanical process of cellulose suspension in order to provide shearing forces to allow fibrillation of the cellulose into nanocellulose. This generally results in high energy consumption. The CNFs suspension eventually becomes thicker throughout the process due to the nanofibrillation which causes the cellulose suspension to increase in viscosity.

Dissertations by Norrrahim (2018) and Yasim-Anuar (2018) reported that mechanical processing of oil palm biomass into CNFs using a wet disc mill (WDM) has several advantages compared to other processes such as high pressure homogenization and ultrasonication. CNFs produced by WDM had smaller diameter size (<50 nm), and the productivity is much higher compared to the other two methods due to the ability to process cellulose suspension with higher cellulose content (wt%) in WDM. Moreover, WDM does not involve any chemicals such as strong acid to disintegrate the fibers (Hideno et al., 2009; Xiao-zheng Sun et al., 2017; Zakaria et al., 2015). WDM is a promising method for large scale processing of CNFs.

1.2 Problem statements

There are three main issues related to the sustainable production of CNFs as outlined earlier:

- i) a need for efficient and sustainable cellulose isolation methods
- ii) a major cost to producing CNF relate to high energy consumption
- iii) the long cellulose fibers resulting in the low productivity of CNF

A further goal is to prepare CNFs without chemical derivatization of the cellulose. For instance, there are large numbers of publications in which TEMPO mediated oxidation is commonly used. However, the process been considered too expensive. In order to produce CNFs, lignocellulosic material including oil palm biomass need to be pretreated for cellulose isolation, with the aim to remove lignin and hemicellulose which can interfere the nanofibrillation process. Regardless, current pretreatment is not environmentally friendly and is highly

dependent on chemicals usage, which may lead to environmental issues. The common pretreatment used to fractionate the lignocellulosic component requiring active chlorine or halogenated chemicals which is unfavorable to the environment. It has been demonstrated that complete removal of hemicellulose is not needed as the presence of some amount of hemicellulose could promote the nanofibrillation of cellulose (Norrahim et al., 2018). It is hence in this research the pretreatment is designed in such a way to use a more environmentally friendly pretreatment method and yet efficient for the removal of hemicellulose and lignin.

The high energy consumption during nanofibrillation is postulated due to the increase in cellulose suspension viscosity when the number of cycles is increased, as a result of nanofibrils formation. The fibrillation causes cellulose surface area to increase and exposes abundance of hydroxyl groups, which promotes the interaction with water through hydrogen bonds. This effect is related to the cellulose degree of polymerization (DP). Cellulose with higher chain length results in higher viscosity and eventually requires more energy for the microfibrils disintegration into nanofibrils. In this study, controlled reduction of cellulose DP is designed to lower the energy consumption. The effect of the DP reduction on the nanocellulose characteristics is portrayed by characterizing the nanofilm produced from the nanocellulose with reduced DP.

Productivity of the CNFs relates to the cellulose throughput during WDM processing. In the conventional processing using WDM, cellulose throughput is between 1 – 2 wt% of the total cellulose volume suspension processed (Nair et al., 2014; Qin et al., 2016; Wang & Zhu, 2016). Higher cellulose suspension concentration used leads to clogging, and high energy consumption due to the increased in viscosity. This cellulose feature (long fiber length) is the main drawback of produce CNFs at high concentration using WDM. It is postulated that the productivity can be increased when the viscosity issue is solved while achieves at high solid content (in kg) of CNFs product.

The research was designed to tackle the above-mentioned issues as it is important to offer a platform for sustainable CNFs processing, especially for the production of CNFs from the local biomass. Techno-economic feasibility of the CNFs was also conducted.

1.3 Objectives

The general objective of this research was to develop a sustainable processing method for CNFs production from oil palm biomass. The specific objectives of this study were:

1. To determine the feasibility of reducing the use of hazardous chemicals for cellulose isolation from oil palm biomass by using non-halogenated chemical pretreatment method.
2. To investigate the effect of superheated steam and cellulase enzyme treatments on controlled reduction of cellulose degree of polymerization.
3. To determine the influence of cellulose degree of polymerization on the productivity of cellulose nanofibrils production and energy consumption in wet disc milling process.
4. To characterize nanocellulose films developed from various degree of polymerization cellulose nanofibrils for their physical, mechanical, and thermal characteristics.
5. To evaluate the techno-economic feasibility of cellulose nanofibrils production from oil palm biomass based on multiplier to an economic factor.

1.4 Experimental overview

Chapter 1 of this thesis consists of the general overview, the problem statements, and the objectives of this research. Figure 1.1 shows the overall experimental overview of this research.

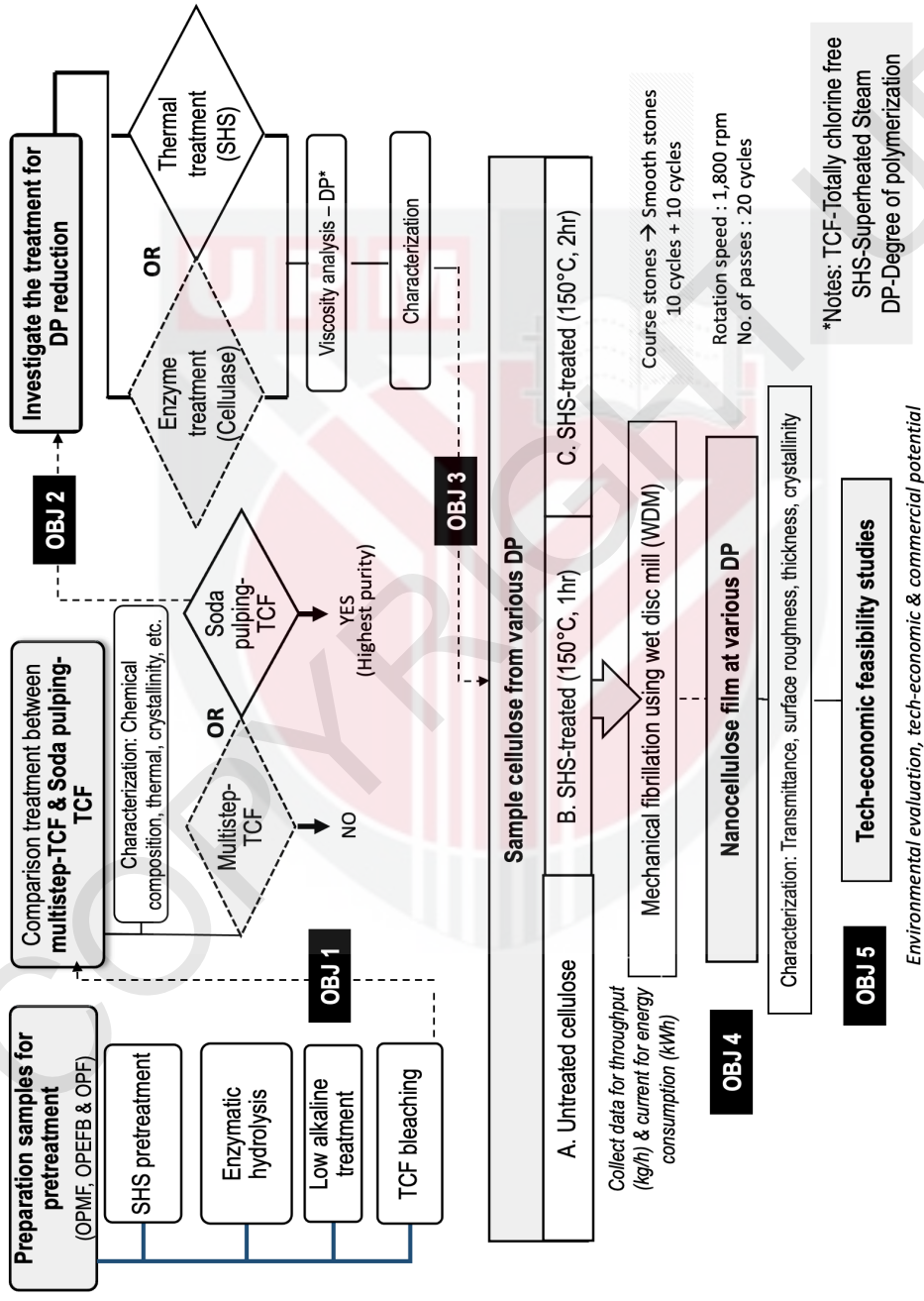


Figure 1.1: Experimental overview

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BIODATA OF STUDENT



Liana Noor Megashah was born on November 5, 1993. She attended her primary school at Sekolah Kebangsaan (SK) Bakri Batu 2 in Muar, Johor for six years. She continued her secondary education at Sekolah Menengah Kebangsaan (SMK) Convent, Muar, Johor (2006 – 2007), SMK Padang Midin, Kuala Terengganu (2007 – 2008), SMK Munshi Abdullah, Melaka (2009) and there she sits for Lower Secondary Evaluation (PMR). At fifth form of secondary school, she went to SMK Puteri, Kota Bharu, Kelantan and completed her Malaysian Certificate of Education (SPM). She got an offer by Majlis Amanah Rakyat (MARA) to pursue a foundation program at Kolej MARA Kuala Nerang (KMKN), Kedah for the year 2011. After completion, she pursued a four-year Bachelor of Chemical Engineering Technology (Hons.) in Bioprocess program at Universiti Kuala Lumpur Malaysian Institute of Chemical and Bio-Engineering Technology (UniKL MICET) in Melaka. Her final year project was on cultivation of soil bacteria and exploitation of its capability for the biodegradation of used engine oil in the environment. She underwent a four-month industrial training program at Melaka Biotechnology Corporation. Her job scope included food testing laboratory focusing on food nutritional fact, antioxidant test and alcohol detection, allowing her to understand on what consumers are concerned about their food products. Later in 2016, she started her Master's degree in the field of Environmental Biotechnology in Universiti Putra Malaysia (UPM), under the supervision of Professor Ts. Dr. Hidayah Ariffin. Her research interests are in developing sustainable methods for commercialize nanocellulose from oil palm waste. During her postgraduate studies, she gets an offer for research attachment at Kyushu Institute of Technology, Japan in May 2017; July – October 2017. Later in 2018, she managed to convert from Master's degree course to Doctor of Philosophy. During the course of her PhD study, she managed to publish several publications in journals and books namely *Cellulose*, *Asia-Pacific Journal of Molecular Biology and Biotechnology*, book chapter in Lignocellulose for Future Bioeconomy, and several conference-proceedings.

LIST OF PUBLICATIONS

The following papers is based on the work contained in this dissertation:

Liana Noor Megashah, Hidayah Ariffin, Mohd Rafein Zakaria, Mohd Ali Hassan, Yoshito Ando, Farah Nadia Mohd Padzil (2020). Modification of cellulose degree of polymerization by superheated steam treatment for versatile properties of cellulose nanofibril film. *Cellulose*, 27(13), 7417–7429.

Liana Noor Megashah, Hidayah Ariffin, Mohd Ali Hassan & Farah Nadia Mohd Padzil (2019). Chapter 3 - Multistep, Nonchlorinated Treatment for Cellulose Isolation From Oil Palm Fronds. In H. Ariffin, S. M. Sapuan, & M. A. Hassan (Eds.), *Lignocellulose for Future Bioeconomy* (pp. 31–40). Elsevier.

Liana Noor Megashah, Hidayah Ariffin, Mohd Rafein Zakaria & Mohd Ali Hassan (2018). Multi-step pretreatment as an eco-efficient pretreatment method for the production of cellulose nanofiber from oil palm empty fruit bunch. *Asia-Pacific Journal of Molecular Biology and Biotechnology*, 26(2), 1–8.

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Liana Noor Megashah, Hidayah Ariffin, Mohd Rafein Zakaria & Yoshito Ando (2018). Characteristics of cellulose from oil palm mesocarp fibres extracted by multi-step pretreatment methods Characteristics of cellulose from oil palm mesocarp fibres extracted by multi-step pretreatment methods. *IOP Conf. Ser. Mater. Sci. Eng.*, 368, 012001.

The presentation of papers in conferences/symposiums included in this dissertation was as follows:

Liana Noor Megashah, Hidayah Ariffin, Mohd Rafein Zakaria, Yoshito Andou (2017). Multiple Stage Pretreatment Affecting the Properties of Nanocellulose from Oil Palm Frond. *In International Symposium on Applied Engineering and Sciences (SAES2017)*. Universiti Putra Malaysia.

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