LIGNIN-CONTAINING CELLULOSE NANOFIBRILS IMPROVING THE PAPERMAKING PROCESS AND THE PROPERTIES OF HANDSHEET PAPER

By

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Chair : **Professor Ts. Hidayah Ariffin, PhD**
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The addition of cellulose nanofibrils (CNF) during papermaking is associated with lower productivity processing due to the increase in drainage time, as the CNF addition contributes to the improved inter-fiber network due to hydrophilic interaction. Additionally, its nano-sized enables it to fill the empty space in between the fibers and eventually slows down the water removal. To overcome this issue, partially hydrophilic CNF was prepared by retaining the lignin content in CNF to produce lignin-containing CNF (LCNF). Four types of LCNF samples namely UB, D1, D2 and D3 with various lignin content of 9.7%, 7.7%, 2.8%, and 0.5%, respectively, were prepared from oil palm empty fruit bunch (OPEFB) by manipulating the pulping condition and introducing controlled bleaching process. The effects of LCNF lignin content and LCNF loading (1, 2, 4 and 8 wt%) on drainage time and handsheet characteristics were evaluated. Unbleached OPEFB base pulp was used as control. Overall results showed that lignin content played a significant role in drainage time, whereby the lower the lignin content, the longer the drainage time. For instance, D3 sample which contained almost no lignin gave the longest drainage time at 35.6 s, compared to control sample (8.0 s). LCNF loading also affected the drainage time with 2 wt% of D1 LCNF sample gave the shortest drainage time among all of the LCNF samples. In terms of the mechanical properties of the handsheet, LCNF incorporation seemed to contribute to better results compared to the control sample. For example, the incorporation of 4 wt% D1 LCNF had a tensile index of 36.9 Nm/g, burst index of 5.4 kPa.m^2/g, and folding strength of 152 double folds compared to the control sample which had a tensile index of 31.8 Nm/g, burst index of 4.3 kPa.m^2/g, and folding strength of 62 double folds. Overall results showed that the use of LCNF in papermaking shortened the drainage time as compared to CNF which contributed to better paper processing. Additionally, the LCNF exhibited

incorporation effects with improved physical and mechanical properties as compared to the control sample.

Keywords: Lignin-containing cellulose nanofibrils, papermaking, drainage time, oil palm empty fruit bunch paper, mechanical properties

SDG: GOAL 9: Industry, innovation and infrastructure, GOAL 12: Responsible, consumption and production

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

NANOFIBRIL SELULOSA YANG MENGANDUNGI LIGNIN MENAMBAH BAIK PROSES PEMBUATAN KERTAS DAN SIFAT HELAIAN KERTAS

Oleh

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Penambahan nanofibril selulosa (CNF) semasa pembuatan kertas dapat dikaitkan dengan proses produktiviti yang lebih rendah disebabkan oleh peningkatan dalam masa penyaliran, kerana penambahan CNF menyumbang kepada rangkaian antara gentian yang lebih baik akibat interaksi hidrofilik. Tambahan pula, saiz nanonya membolehkan ia mengisi ruang kosong di antara gentian dan akhirnya melambatkan penyingkiran air. Untuk mengatasi masalah ini, CNF separa hidrofilik telah disediakan dengan mengekalkan kandungan lignin dalam CNF untuk menghasilkan CNF yang mengandungi lignin (LCNF). Empat jenis sampel LCNF iaitu UB, D1, D2,8 dan D3 dengan pelbagai kandungan lignin pada 9.7%, 7.7%, 2.8%, dan 0.5% telah disediakan daripada tandan kosong kelapa sawit (OPEFB) dengan memanipulasi kondisi pemulpaan dan memperkenalkan proses pelunturan terkawal. Kesan kandungan lignin LCNF dan beban LCNF (1, 2, 4 dan 8 wt%) pada masa penyaliran dan sifat helaian kertas telah dinilai. Pulpa asas OPEFB yang tidak diluntur digunakan sebagai kawalan. Keputusan keseluruhan menunjukkan bahawa kandungan lignin memainkan peranan yang penting dalam masa penyaliran, di mana semakin rendah kandungan lignin, semakin lama masa penyaliran. Sebagai contoh, sampel D3 yang hampir tiada lignin memberikan masa penyaliran paling lama pada 35.6 s, berbanding sampel kawalan (8.0 s). Beban LCNF juga mempengaruhi masa penyaliran dengan 2 wt% D1 LCNF memberikan masa penyaliran terpendek antara semua sampel LCNF. Dari segi sifat mekanikal helaian kertas, penambahan LCNF menyumbang kepada keputusan yang lebih baik berbanding dengan sampel kawalan. Sebagai contoh, penambahan 4 wt% D1 LCNF mempunyai indeks tensil 36.9 Nm/g, indeks pecahan 5.4 kPa.m2/g, dan kekuatan lipatan sebanyak 152 lipatan berganda berbanding sampel kawalan yang mempunyai indeks tensil sebanyak 31.8 Nm/g, indeks pecahan 4.3 kPa.m2/g, dan kekuatan lipatan sebanyak 62 lipatan berganda. Keputusan keseluruhan menunjukkan bahawa penggunaan LCNF dalam pembuatan kertas dapat memendekkan masa penyaliran berbanding CNF yang menyumbang kepada pemprosesan kertas yang lebih baik. Selain itu, LCNF mempamerkan kesan penambahan dengan sifat fizikal dan mekanikal yang lebih baik berbanding dengan sampel kawalan.

Keywords: Nanofibril selulosa yang mengandungi lignin, pembuatan kertas, masa penyaliran, kertas tandan kosong kelapa sawit, sifat mekanikal

SDG: MATLAMAT 9: Industri, inovasi dan infrastruktur, MATLAMAT 12: Bertanggungjawab, penggunaan dan pengeluaran

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LIST OF ABBREVIATIONS

α	Alpha
ς	Zeta
°C	Degree Celsius
%	Percentages
wt.%	Weight Percent
µm	Micrometres
cm	Centimetres
ml	Millilitres
nm	Nanometres
m	Metres
Nm	Newton Metre
w	Weight
g	Grams
kg	Kilograms
mg	Milligrams
kV	Kilovolts
mV	Millivolts
hr	Hours
s	Seconds
min	Minutes
kPa	Kilopascal
mN	Millinewton
Hz	Hertz
rpm	Revolutions per Minute
pH	Potential Hydrogen
C_3H_6O	Acetone
CH_3COOH	Acetic Acid

C_2H_5OH	Ethanol
C_7H_8	Toluene
H_2O_2	Hydrogen Peroxide
H_2SO_4	Sulphuric Acid
$Na_2S_2O_3$	Sodium Thiosulfate
$NaClO_2$	Sodium Chlorite
NaOH	Sodium Hydroxide
$KMnO_4$	Potassium Permanganate
KI	Potassium Iodide
CNF	Cellulose Nanofibrils
LCNF	Lignin-containing Cellulose Nanofibrils
OPEFB	Oil Palm Empty Fruit Bunch
UB	Unbleached
WDM	Wet Disk Milling

CHAPTER 1

INTRODUCTION

1.1 Background

When paper was initially used, which was around 2000 years ago, it was mostly for writing and recording significant drawings, like maps. Paper is used in a variety of ways these days, including the printing of books, magazines, bank notes, tissue, toilet paper, packaging, and construction paper. Woody material is the most often used source of raw materials for making paper. The process of pulping is used to separate cellulose from wood or any other lignocellulosic material in order to create cellulose pulp. Wood resources, such as hardwood and softwood, and non-wood resources can both be used to manufacture pulp (Azeez, 2018). Pulp suspension is the term for pulp diluted with water, and dewatering the pulp suspension produces a paper. The characteristics of the paper that is produced are influenced by both the cellulose fibre network structure and the formulation of pulp suspension. In addition to using lignocellulosic resources to create virgin pulp, recycled paper can also be used to create pulp. The use of recycled paper will require an additional step which involves deinking to ensure that the pulp is safe and suitable to be used (Saxena et al., 2016).

Nowadays, non-wood resources have been the emerging bioresources for the production of paper. Lignocellulose from agricultural residues, such as sugarcane bagasse, rice straw, wheat straw, and oil palm empty fruit bunch (OPEFB) can be used to produce paper. Particularly in Asia, non-wood resources are widely available and this has contributed to their potential use in the production of pulp and paper (Bajpai, 2021). Non-wood resources are defined as any plant material that is not classified as a tree and grows rapidly each year compared to wood, which takes a long time to mature (Kamoga et al., 2013; Liu et al., 2018). Nevertheless, the paper manufactured from several non-wood fibres with short fibres has a low strength (Abd El-Sayed et al., 2020; Otieno et al., 2021). In this case, additives are also commonly added to enhance the quality of the paper.

Various additives can be used in papermaking such as for sizing, dry-strength, wet-strength, dyes, and pigment agents (Hubbe, 2004). These additives are primarily used to enhance the process of papermaking operation as well as the paper properties. Recently, the use of nanocellulose, particularly cellulose nanofibrils (CNF) as an additive in papermaking is getting more popular in the paper industry (Zambrano et al., 2020). Apart from its superior properties as an additive, it offers other great advantages including the ability for the paper mills to produce their own in-house CNF. This is simply because CNF is a nano-sized form of cellulose which can be easily processed at the mill. There have been several reports on the use of CNF in papermaking, which targeted its use as a paper additive and coating material (Das et al., 2020; Shanmugam, 2022). For instance, CNF could serve as a coating material for paper-based packaging by improving the water and oxygen barrier (Yook et al., 2020; Al-Gharrawi et al., 2022), oil barrier (Tayeb et al., 2020; Yi et al., 2022), and grease barrier (Mousavi et al., 2018; Al-Gharrawi et al., 2022) properties. The tiny size of CNF contributes to the formation of a smooth and even surface of the coating material when it is being applied to the paper products (Brodin et al., 2014). Meanwhile, as an additive, CNF incorporated into the paper could increase the bonding between the fibres through the formation of hydrogen bonds (Hu et al., 2020). It was reported that the mechanical properties of papers incorporated with CNF improved, indicating its capability to function as a dry-strength additive (Guan et al., 2018; Kasmani & Samariha, 2019).

Oil palm empty fruit bunch (OPEFB) is a non-wood fibre resource that can be a good alternative to the primary fibre sources for pulp and paper production, mainly in Malaysia (Ali et al., 2020). Additionally, Malaysia produces up to 22-23 million tonnes of OPEFB as a residue each year (Padzil et al., 2020). The OPEFB is a by-product of the processing of crude palm oil (CPO). It is generated from the empty stalks of the fresh fruit bunch (FFB) after the fruits have been removed. Lignocellulosic fibres of OPEFB are composed of chemical constituents such as cellulose (43-56%) and hemicellulose (17-33%) that are covered in a lignin (13-37%) matrix (Rafidah et al., 2017). Several studies have been conducted to explore the potential of OPEFB in papermaking (Hafizuddin et al., 2017; Daud et al., 2017; Aripin et al., 2020). The paper made from OPEFB has promising potential because it has good printing qualities and a good formation in the papermaking process. Furthermore, OPEFB could produce thin, specialty papers such as security papers, cigarettes as well as photographic papers (Abdullah & Sulaiman, 2013).

1.2 Problem statement

CNF contributes to the enhancement of the mechanical, barrier, and optical properties of the paper. Nevertheless, it is important to note that there is an issue with the addition of CNF during papermaking, whereby the CNF tends to create a stronger inter-fibre network contributing to the difficulty in water drainability. This has caused the dewatering step during papermaking to take a very long time (Demuner et al., 2019; Hu et al., 2020; Sanchez-salvador et al., 2020; Tajik et al., 2021). The deterioration in drainability throughout the papermaking process occurs mostly due to the large surface area and availability of hydroxyl groups in the structure of CNF, which causes it to hold a significant amount of water via hydrogen bonding. Furthermore, because CNF is smaller in size, it might become trapped and plug the pores between the fibres matrix as water is removed (Merayo et al., 2017; Salas et al., 2019).

CNF is mechanically processed cellulose which is commonly made of bleached pulp. In bleached pulp, the lignin portion in the lignocellulose fibre is removed during the bleaching process, leaving cellulose as the primary component of the pulp. Cellulose consists of mainly a large number of hydroxyl groups along its structural chain, which gives CNF a hydrophilic property. It was hypothesized that retaining the lignin content by controlling the bleaching process able to improve the drainage time during the papermaking process since the hydrophilic property of the CNF can be controlled.

Manipulation of the bleaching process by controlling the use of chemicals used and the number of bleaching stages is expected to produce lignin-containing cellulose nanofibrils (LCNF), which is the derivative of nanocellulose with different lignin content. The use of LCNF as an additive in paper manufacturing is expected to improve drainability during papermaking process, and improve the handsheet properties particularly its strength as the presence of lignin is expected to hinder the free hydroxyl groups of CNF from forming hydrogen bonds with water molecules, thereby improving drainability together with handsheet properties. The chemical structure of lignin which is made up of various functional groups gives lignin both hydrophobic and hydrophilic properties (Nair et al., 2018; Zhang et al., 2020; Zhang et al., 2021; Lisý et al., 2022), and thus it is hypothesized that the use of LCNF will improve the above-mentioned properties compared to CNF.

In this study, LCNF was used as additive with the aim to improve the mechanical properties of the handsheet. Effect of LCNF lignin content and LCNF loading were tested on: (i) drainage time during handsheet making, and (ii) the characteristics of the handsheet produced.

1.3 Objectives

The general objective of this study was to determine the effect of LCNF on the drainage time and characteristics of non-wood handsheet derived from OPEFB. The specific objectives of this research were:

1. To characterise the LCNF derived from OPEFB.
2. To evaluate the effect of LCNF lignin content and loading on the drainage time of unbleached OPEFB pulp during handsheet making.

3. To investigate the effect of LCNF incorporation on the OPEFB handsheet properties.

1.4 Scope of study

This research consisted of two parts: (i) production and characterization of LCNF with different lignin content from OPEFB, and (ii) evaluation of the effect of LCNF incorporation on the handsheet making process and the characteristics of handsheet produced.

For the first part, unbleached OPEFB pulp was firstly prepared by soda pulping process at 16% chemical charged for 150°C and 2 hr. Following that, the unbleached OPEFB pulp was bleached by controlling the bleaching process to produce pulp with various lignin content for LCNF production. Each pulp sample was nanofibrillated by using wet disc mill to produce LCNF. The LCNF samples were characterized for its morphological, thermal, nanofibrillation yields and zeta potential properties.

Non-wood handsheet samples were prepared by using unbleached OPEFB pulp incorporated with LCNF of various lignin content and LCNF loading (1, 2, 4, and 8 wt%). Control sample was prepared without LCNF incorporation. All samples were assessed in terms of drainage time during the handsheet making process, as well as handsheet properties such as physical, mechanical, optical, morphological, and permeability properties.

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