CHARACTERIZATION OF OIL PALM EMPTY FRUIT BUNCH FIBRES-MICROCRYSTALLINE CELLULOSE FOR POTENTIAL HIGH MECHANICAL STRENGTH PAPER

FARIS SYAHIRAN BIN ISMAIL

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By

FARIS SYAHIRAN BIN ISMAIL

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

November 2019

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To my beloved parent, family, teachers and friends

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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November 2019

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The abundance of biomass generated from oil palm industry can be utilized by producing value added products such as high durability paper. Therefore, this study aimed to determine the effects of oil palm empty fruit bunch (OPEFB) pulp and paper properties after beating and adding microcrystalline cellulose (MCC) during the papermaking process. The pulps were produced using Kraft pulping condition with the incorporation of 0%, 3%, 6% and 9% of MCC during papermaking process. The pulps were also beaten at 500 and 1,000 revs of beating level. TAPPI Standard Method was used for the papermaking. Pulp characterization involved pulp freeness, pulp viscosity, pulp drainage and morphological observation. Paper testing included physical, optical, mechanical and thermal properties. Chemical composition of EFB fibre showed an amount of 43.66% and 25.74% of alpha cellulose and of lignin respectively which were suitable for pulping process. The removal of lignin about 82.71% and increase in percentage of alpha cellulose after the pulping process indicated that the pulps can be used to produce high strength paper. Increased fibrillation caused by beating resulted in lower freeness value of 1,000 beaten pulps compared to other samples. Micrograph observations of the paper showed that the paper became more flatten with the introduction of beating and consequently, decreased the thickness of paper. Smoothness of the paper increased with higher beating revolution and higher concentration of MCC, but porosity worsened. The result indicated that addition of MCC into the beaten and unbeaten pulps resulted in positive effect to the mechanical strength of the paper. Tensile index and tear index of unbeaten pulps showed increment by 71.4% and 24.5%, respectively with the addition of 9% MCC concentration,

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while 6% MCC to 500 revolutions beaten pulps increased freeness value, tear strength, burst strength, folding endurance and brightness by 1.15%, 39.07%, 18.54%, 0.06% and 4.74%, respectively. Optical properties of the paper showed that unbeaten pulp decreased with the addition of MCC in terms of brightness, whereas the brightness of the beaten pulps increased with increasing concentration of MCC. Thermal analysis showed that MP0a (EPB pulp) had the highest thermal degradation compared to other samples. Addition of MCC resulted in negative effect to thermal stability as it decreased the thermal stability of the unbeaten and beaten pulps. In conclusion, the study discovered that the less than 6% of MCC improved the properties of the 500 revolutions beaten pulps in terms of mechanical strength, while overdose of MCC addition and high beating revolution of EFB fibres degraded all major properties of the paper.

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

PENCIRIAN MIKROKRISTAL SELULOSA-SERAT TANDAN KOSONG KELAPA SAWIT YANG BERPOTENSI SEBAGAI KERTAS BERKEKUATAN MEKANIKAL TINGGI

Oleh

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Abstrak merupakan ringkasan keseluruhan tesis dan wajib diberi perhatian rapi Biojisim yang banyak dihasilkan daripada industri kelapa sawit boleh digunakan untuk penghasilan produk nilai tambah seperti kertas yang mempunyai kekuatan mekanikal yang tinggi. Oleh itu, kajian ini menentukan kesan terhadap serat tandan kosong kelapa sawit (OPEFB) dan sifat kertas selepas pemukulan dan penambahan selulosa mikrokristal (MCC) semasa proses pembuatan kertas. Pulpa yang dihasilkan menggunakan kaedah Kraft yang digabungkan dengan penambahan 0%, 3%, 6% dan 9% MCC semasa proses pembuatan kertas. Pulpa juga dihentak pada tahap pemukulan 500 dan 1,000. Kaedah Piawaian TAPPI digunakan untuk pembuatan kertas. Kaedah pencirian pulpa melibatkan ujian-ujian seperti kebebasan pulpa, kelikatan pulpa, saliran pulpa dan pemerhatian morfologi. Ujian kertas termasuk sifat fizikal, mekanik, optik dan haba. Komposisi kimia serat EFB menunjukkan sejumlah 43.66% dan 25.74% alfa selulosa dan lignin masing-masing yang sesuai untuk proses menghasilkan pulpa. Penyingkiran 82.71% lignin dan peningkatan peratusan alfa selulosa selepas proses penghasilan pulpa menunjukkan bahawa pulpa tersebut boleh digunapakai untuk membuat kertas yang mempunyai kekuatanmekaniakal yang tinggi. Peningkatan pemfibrilan yang disebabkan oleh tahap pemukulan 1,000 mengakibatkan ujian nilai kebebasan pulpa yang lebih rendah berbanding dengan sampel lain. Pemerhatian terhadap mikrograf kertas menunjukkan bahawa kertas menjadi lebih mendatar apabila diperkenalkan dengan pemukulan pulpa yang mengakibatkan pengurangan ketebalan kertas. Kelicinan kertas meningkat dengan revolusi hentakan dan kepekatan MCC yang lebih tinggi tetapi menjadikan keliangan kertas bertambah teruk. Hasilnya menunjukkan bahawa penambahan MCC ke dalam pulpa yang tidak dipukul dan dipukul memberi kesan positif kepada kekuatan mekanik kertas. Indeks ketegangan dan indeks

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siat pulpa yang tidak dipukul masing-masing menunjukkan kenaikan sebanyak 71.4% dan 24.5% dengan penambahan 9% kepekatan MCC manakala penambahan 6% MCC kepada 500 revolusi pulpa dipukul meningkatkan nilai kebebasan, kekuatan koyak, kekuatan letus, ketahanan lipatan dan kecerahan sebanyak 1.15%, 39.07%, 18.54%, 0.06% dan 4.74% masing-masing. Ciri-ciri optik kertas melihatkan bahawa pulpa yang tidak dipukul mengalami penurunan dengan penambahan MCC dari segi tahap kecerahan sementara kecerahan pulpa yang dipukul menunjukkan kenaikan dengan penambahan kepekatan MCC. Analisis haba menunjukkan bahawa MP0a (pulpa EFB) mempunyai kemerosotan termal tertinggi berbanding sampel lain. Penambahan MCC menghasilkan kesan yang negatif terhadap kestabilan termal kerana penambahan MCC menurunkan kestabilan termal pulpa yang tidak dipukul dan dipukul. Kesimpulannya, kajian mendapati bahawa penambahan peratusan MCC kurang daripada 6% manambah baik sifat-sifat pulpa yang dipukul dengan tahap pukulan 500 revolusi dari segi kekuatan mekanikal manakala penambahan MCC yang berlebihan dan revolusi pemukulan yang tinggi kepada serat EFB didapati memberi kesan buruk kepada semua sifat utama kertas.

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LIST OF ABBREVIATIONS

EFB	Empty fruit bunch
MCC	Microcrystalline cellulose
TAPPI	Technical Association for the Pulp, Paper and Converting Industry
NFC	Nanofibrillated cellulose
MFC	Microfibrillated cellulose
CPO	Crude palm oil
OPT	Oil palm trunk
OPF	Oil palm frond
FFB	Fresh fruit bunch
H_2SO_3	Sulphurous acid
$Ca(HSO_3)_2$	Calcium bisulfite
NaOH	Sodium hydroxide
Na_2S	Sodium sulphide
CSF	Canadian standard freeness
SR	Schopper riegler
µm	micro meter
GSC	Ground calcium carbonate
PCC	Precipitate calcium calbonate
TiO_2	Titanium dioxide
CNC	Cellulose nanocrystal
NCC	Nanocrystalline cellulose
nm	nano meter
TEA	Toughness energy absorption
L/d	aspect ratio
kg/mm	kilogram per milimeter
%	Percentage
min	minute
g	gram
ml	millilitre
hr	hour
^oC	degree Celsius
$NaClo_2$	Sodium chlorite
w/w	weight per weight
rpm	revolutions per minute
rev	revolutions
SEM	Scanning electron microscope
FESEM	Field emission scanning electron microscope
cm^2	square centimeters
ml/min	millilitre per minute
kNm/g	kilo newton meter per gram
kV	kilo volt
kNm^2/g	kilonewton square metre per gram
cm^{-1}	wavenumber
±	plus or minus
FTIR	Fourier-transform infrared spectroscopy
TGA	thermogravimetric analysis

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CHAPTER 1

INTRODUCTION

1.1 Background

Increase demand for paper can be associated with rising population growth and industrialization in developing countries causing intensive usage of wood for papermaking source in pulp and paper industry. This phenomenon could lead to negative impact to the environment such as over exploitation of timberland. Therefore, production of pulp fibres from agricultural biomass deserves a consideration (Puitel et al., 2015). The availability of these agricultural biomass renders the cost cheaper as they are not fully utilized (Mahale & Goswami-giri, 2015).

The palm oil industry creates enormous quantity of empty fruit bunch waste after the crude oil was processed (EFB) (Singh et al.,2013). Common method used to manage this waste is by burning openly. However, this causes uncontrollable apprehensions to the environment. Traditional approach will take longer time because the wastes are left and take time to biodegrade. These biomasses also can be converted into other beneficial products that give potential income for the palm oil manufacturing countries (Hashim et al., 2011). The oil palm tree originated from tropical rain forest in West Africa. It is known as *Elaies guineesis* (Kaliwon et al., 2010). Oil palm is a type of crop commonly found in rainy tropical wetlands. The tree grows in a deep soil with continuous moisture throughout the year (Verheye, 2010). Oil palm is brought to Malaysia as a decorative plant in the 1800s. Later, under the government's agricultural diversification program, it is cultivated for commercial planting to minimize the dependence on the exports of tin and rubber to the country's economy (Mahlia et al., 2012).

Empty fruit bunch (EFB) shared similarities to wood in in their chemical composition, physical, and mechanical attributes. It can be used as raw materials in making wood-based product such as pulp and paper (Rushdan et al., 2007 in Abdul Khalil et al., 2010).

Beating is a mechanical action experienced by the fibre in the presence of water which produces the best maximum conditions for fibres to intertwine in the web and bonding between the fibres by increasing the contact area between the fibres. As a result, the fibres become more flexible as fibrillation on the surface increased (Adel et al., 2016).

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Beating affects, the sheet quality by improving flexibility, enhancing swelling ability and thus, improves the bonding ability which increases the tensile strength. However, too much beating also can affect negatively as it deteriorates the tensile strength (Harsono et al., 2016; Osong et al., 2016). Excess refining adversely affect the bending stiffness of paper that lead to paper densification (Osong et al., 2016).

Cellulose served as the principal strengthening part in plant components and is one of the most copious biopolymers on earth as it can be found in all wood and other plant-based constituents (Siró & Plackett, 2010). According to Osong et al., (2016), nanocellulose can be categorized as cellulose nanofibres and cellulose nanostructured materials. Cellulose nanofibres are subdivided into nanocrystalline cellulose or cellulose and nanofibrillar cellulose (NFC) whereas cellulose nanostructured materials are sub-grouped into microcrystalline cellulose and microfibrillar cellulose (MFC) based on TAPPI WI 3021.

Microcrystalline cellulose (MCC) is a distinguished term used for commercially available manufactured goods. The sizes of highly crystalline MCC are micrometer and have very high purity cellulose (Berglund 2005; Siró and Plackett 2010).

1.2 Problem statement

The total production of crude palm oil (CPO) in year 2017 in Malaysia was approximately 19.92 million tons compared to 17.32 million tons in 2016. The total export of oil palm products in year 2017 also rose by 2.9% to 23.97 miilion tons compared to previous year (MPOB, 2017). With its 6 million hectares of cultivated area, oil palm industry in Malaysia had created 100 million tons of biomass while the empty fruit bunches generated more than 10 million tons from more than 30 million tons of total crop of fresh fruit bunch per year (Abdul Khalil et al., 2010). Some of the EFB are burnt in incinerators and the ash is reused onto the plantation as fertilizer as a means of conventional clearance (Abdullah & Sulaiman, 2013). Though, most are left by the millers as mulch because they are not used for any other means. This is because the bulkiness and wetness of EFB rendered them unsuitable for either handling or transportation purposes (Chiew & Shimada, 2013).

In search of the solutions to commercialize this byproduct, EFB is used in papermaking where there has been important development in recent years in Malaysia. Study conducted by Rushdan et. al, (2007) indicated that EFB has promising prospect to be used for raw materials in making pulp for paper. Production of soda empty fruit bunch (EFB) was suitable for manufacturing of corrugated cartons, printing and writing paper, and other paper based products (Daud & Law, 2011).

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Various type of treatments can be used to make pulp from EFB such as soda pulping, soda antraquinone, alkaline peroxide and acetosolv. Kraft pulping was used in this study as the process yield better quality of pulp in terms of strength. Beating is a mechanical treatment applied to pulp suspension to improve the strength. In this study, the pulps were beaten to different revolutions using mechanical beating process to increase the fibrillation of the fibre for better bonding and flexibility of the paper.

Paper is commonly added with additives such as starch and cellulose to increase their strength. Minerals are also used which can be advantageous to the paper strength or otherwise affect negatively on the mechanical properties. According to Fang, (2019), mineral fillers that are commonly used in papermaking include; precipitated calcium carbonate (PCC), kaolin, talc and titanium dioxide.

Researchers have explored to demonstrate the benefits of nanocellulosic materials as strengthening agent in paper and board. Studies have also shown that incorporations of fibrillated cellulose (Hassan et al., 2015; Taipale et al., 2010; Tarrés et al., 2016) and nanocellulose (Potulski, 2016) improve the mechanical properties of the paper. However, there is little published work related to paper production from EFB incorporated with microcrystalline cellulose and the addition of pulp beating in papermaking.

1.3 Objective study

The general objective of this study was to utilize EFB into potential specialized paper. The product will benefit the industry as it helps to reduce the effect of environmental pollution and help forest conservation. In addition, it also helps towards sustainable future through solving the waste removal crisis in oil palm planting countries. The specific objectives of this research are stated as following:

1. To determine the effects of microcrystalline cellulose addition on the formation of paper from oil palm empty fruit bunch on their pulp characterization, paper physical, mechanical, optical and thermal properties.
2. To determine the effects of different beating level on the oil palm empty fruit bunch pulp with addition of microcrystalline cellulose (MCC) on their pulp characterization, paper physical, mechanical, optical and thermal properties.

3. To determine the effects of different beating level on the strength of oil palm empty fruit bunch paper with addition of microcrystalline cellulose

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(MCC) on their pulp characterization, paper physical, mechanical, optical and thermal properties.

1.4 Hypothesis

1. There is no significant effect of microcrystalline cellulose addition on the formation of paper from oil palm empty fruit bunch on their pulp characterization, paper physical, mechanical, optical and thermal properties.

2. There is no significant effect of pulp beating level on the pulp from oil palm empty fruit bunch with the addition of microcrystalline cellulose (MCC) on their pulp characterization, paper physical, mechanical, optical and thermal properties.

3. There is no significant effect of pulp beating on paper made from oil palm empty fruit bunch with the addition of microcrystalline cellulose (MCC) on their pulp characterization, paper physical, mechanical, optical and thermal properties.

1.5 Scope of Study

This research focused on the influence of different pulp beating level on the pulp characterization, paper physical, mechanical, optical and thermal properties on EFB Kraft pulp incorporated with different percentage of microcrystalline cellulose (MCC).

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