THE OPTIMIZATION OF CELLULOSE NANOFIBRE (CNF) PRODUCTION FROM EMPTY FRUIT BUNCH (EFB) USING STEAM EXPLOSION PRE-TREATMENT

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We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Master of Science.

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I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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ABSTRAK

Buah tandan kosong (BTK) adalah 24% daripada 168 juta tan biojisim lignoselulosik kelapa sawit yang dihasilkan di Malaysia, ianya mengandungi selulosa yang tinggi (sehingga 65%). Selulosa boleh diekstrak dengan menggunakan proses rawatan termokimia dan proses pra-rawatan seperti letupan wap akan dapat membantu untuk meningkatkan keberkesanan proses rawatan termokimia. Pengisaran adalah rawatan mekanik yang paling berkesan untuk meleraikan selulosa kepada serat nano selulosa (SNS) dan pengoptimuman proses pengisaran dapat membantu untuk menjimatkan penggunaan masa dan tenaga. Tujuan kajian ini adalah untuk mengekstrak selulosa daripada BTK dengan bantuan pra-rawatan letupan wap diikuti dengan rawatan thermakimia dan pengoptimuman menggunakan kaedah gerak balas permukaan (KGBP) untuk rawatan mekanikal bagi penghasilan SNS. Pengekstrakan selulosa bermula dengan pra-rawatan letupan wap pada 20 bar dengan tempoh masa berbeza (3-10 min); rawatan air panas dengan tempoh masa berbeza (15-90 min); rawatan alkali menggunakan natrium hidroksida (NaOH) pada kepekatan yang berbeza (2.5-20%); dan peluntursan menggunakan dua bahan kimia yang berbeza (natrium hipoklorit dan natrium klorit) dengan teknik peluntursan yang berbeza (sistem 1 dan 2). Pengoptimuman penghasilan SNS bermula dengan penyaringan faktor (kelajuan, masa dan konsistensi) menggunakan reka bentuk faktorial penuh (RBFP) dan KGBP dihasilkan menggunakan reka bentuk komposit pusat (RBKP) untuk mengenal pasti kondisi terbaik. Sepanjang pengekstrakan selulosa dan penghasilan SNS, sifat kimia, sifat termal dan kehabluran serat yang dirawat telah dianalisis menggunakan analisis transformasian inframerah Fourier spektroskopi (TIFS), analisis termogravimetri (ATG) dan belauan sinar-x (BSX); pancaran medan mikroskopi elektron pengimbasan (PMMEP) untuk melihat morfologi dan saiz morfologi serat letupan wap, selulosa dan SNS; dan komposisi kimia dan sifat selulosa yang diekstrak dianalisis menggunakan kaedah standard seperti kaedah TAPPI. PMMEP menunjukkan bahawa letupan stim pada 10 min membantu meleraikan struktur serat BTK. TIFS dan ATG menunjukkan bahawa sifat kimia dan terma serat tidak terjejas semasa pengekstrakan selulosa dan penghasilan SNS. BSX menunjukkan kehabluran serat meningkat apabila bilangan rawatan meningkat tetapi, saiz domain hablur serat telah berubah-ubah apabila serat menjalani rawatan. TAPPI menunjukkan bahawa selulosa yang diekstrak mempunyai 85.2% kandungan selulosa yang tinggi dan hampir semua lignin telah dikeluarkan hanya meninggalkan 0.07% daripadanya. Faktor konsistensi serat telah diasingkan dengan bantuan RBFP kerana ia memberikan sumbangan paling rendah semasa rawatan mekanikal dan KGBP menunjukkan keadaan terbaik untuk penghasilan SNS adalah pada 722 rpm, 30 minit, dan 5% konsistensi. FESEM menunjukkan rawatan mekanikal telah mengurangkan saiz selulosa daripada 8.25 μm kepada 17.85 nm bagi SNS. Kesimpulannya, selulosa berjaya diasingkan dari BTK dan penghasilan SNS telah dioptimumkan, di mana ia akan dapat mengurangkan penggunaan tenaga bagi rawatan mekanikal untuk aplikasi perindustrian dan seterusnya, mesra alam.

ABSTRACT

Empty fruit bunch (EFB) is 24% from the 168 million tonnes of lignocellulosic oil palm biomass generated in Malaysia that has a high cellulose content (up to 65%). Cellulose can be extracted by using thermochemical treatment and pre-treatment processes such as steam explosion that improve the efficiency of further thermochemical treatment process. Grinding is the most effective mechanical treatment to defibrillate the cellulose into cellulose nanofibre (CNF) and optimization of the grinding process can help to reduce the time and energy consumption of the process. The purpose of this study is to extract cellulose from EFB via steam explosion pre-treatment followed by thermochemical treatment and the optimization of mechanical treatment using response surface methodology (RSM) for CNF production. The extraction of cellulose starts with steam explosion pre-treatment at 20 bar at different retention time (3-10 min); hot water treatment at different period of time (15-90 min); alkaline treatment using sodium hydroxide (NaOH) at different concentration (2.5-20%); and bleaching using two different reagents (sodium hypochlorite and sodium chlorite) with different bleaching techniques (system 1 and 2). The optimization of CNF production start with screening of factors (speed, time and consistency) using the full factorial design (FFD) and central composite design (CCD) to generate the RSM for the identification of the optimum condition. Throughout the extraction of cellulose and production of CNF, the chemical properties, thermal characteristic and crystallinity of the treated fibre were analyzed using Fourier transform infrared spectroscopy (FTIR) thermogravimetric analysis (TGA) and x-ray diffraction (XRD); field emission scanning electron microscopy (FESEM) to observe the morphology and size of the steam exploded fibre, cellulose, and CNF. The chemical composition and properties of the extracted cellulose were analyzed using a standard method such as TAPPI method. FESEM shows that the steam explosion at 10 min helped to rupture the structure of the EFB fibre. FTIR and TGA showed that the chemical properties and thermal characteristic of the fibre were not affected throughout the extraction of cellulose and production of CNF. XRD showed that the crystallinity of the fibre increased as the number of treatments increased but the crystal domain size of the fibre had fluctuated as the fibre undergoes all the treatments for cellulose extraction. TAPPI indicated that the extracted cellulose was 85.2% (wt), a high cellulose content, and almost all lignin has been removed, leaving only 0.07% (wt) of it. FFD had helped to screen out the consistency of fibre as it gave the least contribution during the mechanical treatment and the RSM showed that the best condition for CNF production is at 722 rpm, 30 min, and 5% consistency. FESEM shows that the mechanical treatment has decreased the size of cellulose from 8.25 µm to 17.85 nm for CNF. In conclusion, cellulose was successfully isolated from the EFB and the production of CNF is optimized which had greatly reduced the energy consumption of the mechanical treatment. This is great for the industrial application and is environmentally friendly.

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LIST OF SYMBOLS

µm	Micrometer
nm	Nanometer
ºC	Degree Celsius
g	Gram
L	Litre
Kg	Kilogram
mL	Millilitre
L	Litre
kV	Kilovolt
rpm	Revolution per minute
N	Normality
min	Minute
h	Hour

LIST OF ABBREVIATIONS

EFB	Empty fruit bunch
FFB	Fresh Fruit Bunch
AGU	Anhydroglucopyronose unit
CNF	Cellulose nanofibre
NaOH	Sodium hydroxide
NaOCl	Sodium hypochlorite
NaO_2Cl	Sodium chlorite
$K_2Cr_2O_7$	Potassium dichromate
$(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$	Ferrouss ammonium sulfate
FTIR	Fourier transform infrared
IR	Infrared
SEM	Scanning electron microscope
FESEM	Field emission scanning electron microscope
TGA	Thermal gravimetric analysis
XRD	X-Ray diffraction
TAPPI	Technical Association of the Pulp and Paper Industry
POMW	Palm oil mill waste
DOE	Design of experiment
OFAT	One factor at a time
RSM	Response surface methodology
CCD	Central composite design
BBD	Box-Behnken design
DM	Doehlert Matrix design
ISO	International Organization for Standardization
CH	Sodium hypochlorite bleaching
CC	Sodium chlorite bleaching
CHC	First combination bleaching
CCH	Second combination bleaching
DI	Deionized
TG	Thermogravimetric

DTG	Derivative thermogravimetric
LCSB	Lepar Corporation Sdn. Bhd.
T_{on}	Onset of thermal degradation
T_{max}	Temperature of maximum degradation

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