UNIVERSITI PUTRA MALAYSIA

RESIDUAL OIL REMOVAL FROM OIL PALM EMPTY FRUIT BUNCHES USING HIGH PRESSURE WATER SPRAY SYSTEM

NOOR SERIBAINUN HIDAYAH BT MD YUNOS

FK 2015 45

RESIDUAL OIL REMOVAL FROM OIL PALM EMPTY FRUIT BUNCHES USING HIGH PRESSURE WATER SPRAY SYSTEM

By

NOOR SERIBAINUN HIDAYAH BT MD YUNOS

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

June 2015

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

RESIDUAL OIL REMOVAL FROM OIL PALM EMPTY FRUIT BUNCHES USING HIGH PRESSURE WATER SPRAY SYSTEM

By

NOOR SERIBAINUN HIDAYAH BT MD YUNOS

June 2015

Chair: Azhari Samsu Baharuddin, PhD
Faculty: Engineering

Oil extraction rate (OER) is one of the key factor in evaluating the performance and efficiency of the palm oil mill by comparing the ratio of oil palm produced per day with the total fresh fruit bunches (FFB) processed per day. Under Palm Oil National Key Economic Area, Malaysia is expected to achieve OER value up to 23% in the year 2020. One of the strategies to achieve this goal is to study the potential of recovering the residual oil from oil palm biomass which is oil palm empty fruit bunches (OPEFB). Hence, this study is aimed to develop a baseline study of the characterisation and localisation of the residual oil on the fiber and also to investigate the effects of the hydrothermal method on the yield and quality of the oil as well as structures of the fibers. A new method called High Pressure Water Spray System (HPWSS) was introduced and performed by spraying pressurized water (500 psi) at 30°C, 60°C, and 90°C and combination of water-steam at 120°C and 150°C onto the surface of the OPEFB. Soxhlet extraction and palm oil tester were used to recover the residual oil on the different parts of fiber (spikelet, stalk and press-shredded) and to determine the qualities of the residual oil. The baselines studies show that the highest residual oil found was on the spikelet (7.39±3.45%) followed by press-shredded OPEFB (3.61±0.34%) and lastly stalk fiber (2.04±2.31%). This result was in agreement with the Sudan dye analyses. The initial residual oil of OPEFB contains a higher amount of valuable carotenes (409.02 ppm), comparable values of Deterioration of Bleach ability Index (DOBI) (2.42), Peroxide Value (PV) (1.84 meq/kg), and fatty acid compositions in comparison to the raw crude palm oil (CPO) from the mill. The experimental results of HPWSS indicated that, the highest oil yield was achieved at 150°C (94.41±0.02%) followed by 120°C (86.14±0.03%), 90°C (80.54±0.02%), 60°C (70.37±0.21%) and 30°C (63.24±0.58%), respectively. The residual oil exhibit better quality of Free Fatty Acid (FFA) (6.21±0.35%), DOBI (2.42±0.23), PV (4.89±0.27meq/kg), phosphorus content (6.87±0.71 ppm) and iron content (3.43±0.35 ppm) at 30°C compared to 150°C where the values of FFA, DOBI, PV, phosphorus, and iron content were (7.79±0.77%), (1.63±0.38), (5.85±0.60 meq/kg), (11.70±0.61 ppm), and (7.17±0.15 ppm), respectively. From the gas chromatography-flame ion detector (GC-FID) analyses, similar fatty acid composition of the residual oil at each treatment temperature was observed. This result was in agreement with the spectroscopy graph obtained from Fourier transform infrared (FTIR). Lignocellulosic content analyses revealed some increment of cellulose content in the OPEFB (39.1%) while major decrement of hemicellulose content (19.80%) and lignin content (22.43%) was discovered after HPWSS treatment at 150°C. Scanning electron microscopy (SEM) and nitrogen adsorption analysis (BET) analyses showed smoother surface, higher surface areas, higher total pore volume and lower pores diameter as the temperature of HPWSS increased. Based on the results obtained, the residual oil was considered as low quality oil and cannot be consumed as edible oil. Nevertheless, this residual oil still can be used for non-edible applications such as soap, cosmetics, bio-lubricant and bio-diesel. The residual OPEFB fiber can be used as a feedstock for bio-conversion applications. Therefore, the HPWSS is a promising technique believed to be useful for the removal of residual oil from OPEFB.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Sarjana Sains

PENYINGKIRAN SISA MINYAK DARI TANDAN KOSONG KELAPA SAWIT MENGGUNAKAN SISTEM TEKANAN TINGGI SEMBUR AIR

Oleh

NOOR SERIBAINUN HIDAYAH BT MD YUNOS

Jun 2015

Pengerusi: Azhari Samsu Baharuddin, PhD
Fakulti: Kejuruteraan

Kadar perahan minyak (OER) adalah salah satu kunci dalam menentukan prestasi dan kecekapan kilang kelapa sawit dengan membandingkan nisbah minyak kelapa sawit yang terhasil dengan jumlah tandan kelapa sawit segar yang diproses untuk sehari. Dibawah Bidang Ekonomi Minyak Kelapa Sawit Utama Negara, Malaysia dijangka akan mencapai nilai OER sehingga 23% dalam tahun 2020. Salah satu strategi untuk mencapai sasaran ini adalah kajian tentang potensi mendapatkan semula sisa minyak dari sisa kelapa sawit iaitu tandan kosong kelapa sawit. Oleh itu, kajian ini bertujuan untuk membangunkan kajian dasar tentang pencirian dan penyetempatan lebihan minyak pada serat dan juga menyiasat kesan kaedah hidroterma pada hasil dan kualiti minyak serta struktur tandan kosong kelapa sawit. Kaedah baru yang dipanggil sistem tekanan tinggi sembur air (HPWSS) telah diperkenalkan dengan menyembur permukaan tandan kosong kelapa sawit pada tekanan tinggi (500 psi) di suhu 30°C, 60°C, dan 90°C dan kombinasi air-stim (500 psi pada 120°C dan 150°C). Pengekstrakan soxhlet dan penguji minyak sawit telah digunakan untuk mendapatkan sisa minyak pada bahagian-bahagian serat (spikelet, empulur dan tekan-dicincang) dan untuk menentukan kualiti sisa minyak. Kajian dasar menunjukkan sisa minyak yang tertinggi telah dijumpai pada serat spikelet (7.39±3.45%) diikuti serat tekan-dicincang tandan kosong kelapa sawit (3.61±0.34%) dan akhir sekali serat empulur (2.04±2.31%). Keputusan ini adalah dalam persetujuan dengan analisis pewarna sudan. Sisa minyak tandan kosong kelapa sawit yang awal mengandungi kandungan karotena bernilai yang tinggi (409.02 ppm), nilai kemerosotan keupayaan peluntur indeks (DOBI) (2.42), nilai peroksida (PV) (1.84 meq/kg) dan, kandungan asid lemak yang setara dalam perbandingan dengan minyak kelapa sawit mentah dari kilang. Hasil uji kaji HPWSS menunjukkan kandungan hasil minyak yang tertinggi telah dicapai di suhu 150°C (94.41±0.02%) diikuti 120°C (86.14±0.03%), 90°C (80.54±0.02%), 60°C (70.37±0.21%) dan 30°C (63.24±0.58%), masing-masing. Sisa minyak mempamerkan kualiti asid lemak bebas (FFA) (6.21±0.35%), DOBI (2.42±0.23), PV (4.89±0.27 meq/kg), fosforus (6.87±0.71 ppm) dan besi (3.43±0.35 ppm) yang lebih baik di suhu 30°C berbanding dengan suhu 150°C dimana nilai FFA, DOBI, PV, kandungan fosforus, dan kandungan besi ialah (7.79±0.77%), (1.63±0.38), (5.85±0.60 meq/kg), (11.70±0.61 ppm), dan (7.17±0.15 ppm), masing-masing. Dari analisis gas kromatografi-api pengesan ion (GC-FID), diperhatikan kandungan lemak asid sisa minyak dari setiap rawatan suhu adalah sama.

Keputusan ini adalah dalam persetujuan dengan spektroskopi graf diperoleh dari fourier mengubah inframerah (FTIR). Analisis kandungan lignoselulosa mendedahkan beberapa kenaikan kandungan selulosa di dalam OPEFB (39.1%) sementara susutan utama kandungan hemisellulosa(19.80%) dan kandungan lignin (22.43%) telah ditemui selepas rawatan HPWSS di suhu 150°C. Analisis pengimbasan elektron mikroskopi (SEM) dan penyerapan nitrogen (BET) menunjukkan permukaan yang lebih licin, kawasan permukaan yang lebih tinggi, jumlah isi padu liang yang lebih tinggi, dan liang diameter yang lebih rendah apabila suhu HPWSS meningkat. Berdasarkan keputusan yang diperoleh, sisa minyak dianggap sebagai minyak berkualiti rendah dan tidak boleh digunakan sebagai minyak yang boleh dimakan. Walau bagaimanapun, sisa minyak ini masih boleh digunakan untuk kegunaan yang tidak boleh dimakan seperti sabun, kosmetik, bio-pelincir, dan bio-diesel. Sisa tandan kosong kelapa sawit boleh digunakan sebagai bahan mentah untuk kegunaan bio-pertukaran. Kesimpulanny, HPWSS menjanjikan teknik yang dipercayai berguna untuk penyingkiran sisa minyak daripada OPEFB.

ACKNOWLEDGEMENTS

In The Name of Allah, The Most Gracious and Merciful.

Alhamdulillah, praise to The Almighty Allah SWT who gave me the strength to complete this research project. First and fore most, I would like to express my deepest gratitude to my main supervisor, Dr. Azhari Samsu Baharuddin and my co-supervisors, Dr. Mohd Noriznan Mokhtar, Dr. Alawi Sulaiman for their support, guidance, constructive supervision, brilliant ideas and guidance throughout the completion of this thesis. May the precious knowledge that I gain from them will be rewarded by Allah SWT. I would like to express my sincere gratitude to Universiti Putra Malaysia (UPM) and Ministry of High Education (MOHE) for the sponsorship under Graduate Research Fellowship Scheme (GRF) and MYBRAIN scheme throughout this study.

I would also like to express my sincere appreciation to all lecturers at the Department of Food and Process Engineering, Faculty of Engineering, University Putra Malaysia. My sincere thanks also go to non-academic staff of Department of Food and Process Engineering especially En. Chairil Anuar, En. Zahir and En. Shahrul for their invaluable assistance in various aspects and also Mr. Zainuri Busu and Felda Besout Palm Oil Mill for providing the facilities at the mill throughout this project.

Thank you and best wishes to all my friends especially my best friend, Matun, Nadia, Suhaiza, Hasmillah, Loo, Hakimah, Boon, CJ and Javier for always there to share the problems and opinions during the project. Their understanding and readiness to lend their helping hands throughout the completion of this project are very much appreciated.

Last but not least, I would like to express my endless gratitude to my beloved father, Md. Yunos Bin Daim for his encouragement, for my lovely mother, Zahriah Bt Md. Nor for her loves and sacrifices, my mother and father in law, Ngatini Bt Jimakon, Shariff Bin Abdullah and all my family members for their supports, love and understanding. My special thanks goes to the most special person in my life, my beloved husband Mohd Saiful Idlan Bin Shariff for always be by my side to share the problems, concern, attention and loves. My research completion would not have been possible without their love and support.

Thank you. May Allah bless upon all of you.

I certify that an Examination Committee has met on 8 June 2015 to conduct the final examination of 1RRU6HUEQQUWKHVLWOHG Residual Oil Removal from Oil Palm Empty Fruit Bunches using High Pressure Water Spray SystemQFRUGQH with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A)106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

Members of the Examination Committee were as follows:

Rosnah Shamsudin, PhD
Associate Professor
Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Siti Mazlina Mustapa Kamal, PhD
Associate Professor
Faculty of Engineering
Universiti Putra Malaysia
(Internal Examiner)

Ahmad Faris Bin Mohd Adnan, PhD
Faculty of Science
Universiti Malaya
(External Examiner)

ZULKARNAIN ZAINAL, PhD
Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 12 August 2015

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

Azhari Samsu Baharuddin, PhD
Senior Lecturer
Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Noriznan Mokhtar, PhD
Senior Lecturer, Ing
Faculty of Engineering
Universiti Putra Malaysia
(Member)

Alawi Sulaiman, PhD
Senior Lecturer
Faculty of Engineering
Universiti Teknologi MARA
(Member)

BUJANG BIN KIM HUAT, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

Declaration by graduate student

I hereby confirm that:

- this thesis is my original work;
- quotations, illustrations and citations have been duly referenced;
- this thesis has not been submitted previously or concurrently for any other degree at any other institutions;
- intellectual property from the thesis and copyright of thesis are fully-owned by Universiti Putra Malaysia, as according to the Universiti Putra Malaysia (Research) Rules 2012;
- written permission must be obtained from supervisor and the office of Deputy Vice-Chancellor (Research and Innovation) before thesis is published (in the form of written, printed or in electronic form) including books, journals, modules, proceedings, popular writings, seminar papers, manuscripts, posters, reports, lecture notes, learning modules or any other materials as stated in the Universiti Putra Malaysia (Research) Rules 2012;
- there is no plagiarism or data falsification/fabrication in the thesis, and scholarly integrity is upheld as according to the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) and the Universiti Putra Malaysia (Research) Rules 2012. The thesis has undergone plagiarism detection software.

Signature: ________________________ Date: ______________________

Name and Matric No: Noor Seribainun Hidayah BT Md Yunos GS34189

Declaration by Members of Supervisory Committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) are adhered to.

Signature: ________________________________
Chairman of Supervisory Committee: DR. AZHARI SAMSU BAHARUDDIN

Signature: ________________________________
Member of Supervisory Committee: DR. MOHD NORIZNAN MOKHTAR

Signature: ________________________________
Member of Supervisory Committee: DR. ALAWI SULAIMAN

TABLE OF CONTENTS

		Page
ABSTRACT		i
ABSTRAK		iii
ACKNOWLEDGEMENTS		v
APPROVAL		vi
DECLARATION		viii
LIST OF TABLES		xiii
LIST OF FIGURES		xiv
LIST OF APPENDICES		xvi
LIST OF ABBREVIATIONS		xvii
CHAPTER		
1	**INTRODUCTION**	
1.1	Overview of the Study	1
1.2	Problem Statement	2
1.3	Objectives	2
1.4	Scope of the Study	2
1.5	Significance of the Study	3
2	**LITERATURE REVIEW**	
2.1	The oil palm	4
2.2	Oil palm empty fruit bunches (OPEFB)	9
2.2.1	Characteristics of OPEFB fiber	10
2.3	Oil extraction and recovery process	10
2.3.1	Soxhlet extraction	11
2.3.2	Mechanical extraction	12
2.3.3	Supercritical carbon dioxide	13
2.3.4	Ultrasound extraction	13
2.3.5	High pressure water spray system	14
2.4	Crude palm oil characteristics	14
2.4.1	Triglycerides	15
2.4.2	Fatty Acids	15
2.4.3	Minor Components	16
2.5	Oil analysis (Quality characteristics of palm oil)	18
2.5.1	Free fatty acids (FFA)	18
2.5.2	Peroxide value (PV)	19
2.5.3	Deterioration of bleach ability index (DOBI)	20
2.5.4	Phosphorus content	21
2.5.5	Trace metal	21

3 METHODOLOGY

3.1 Flow diagram of Research Work 22
3.2 Materials and reagents 23
3.3 Materials preparation 23
3.4 OPEFB fiber characterization 24
3.4.1 Analysis of lignocellulosic content 24
3.4.2 Determination of neutral detergent fiber (NDF) 25
3.4.3 Determination of acid detergent fiber (ADF) 25
3.4.4 Determination of acid detergent lignin (ADL) 25
3.4.5 Inorganic element analysis 26
3.4.6 Determination of elements 26
3.4.7 Determination of ash content 27
3.5 Analysis of initial residual oil from OPEFB 28
3.5.1 Determination of oil content 28
3.5.2 Residual oil localization using Sudan red dye 28
3.5.3 Contact angle measurement 28
3.6 Characterisation of residual oil 29
3.6.1 Determination of FFA 29
3.6.2 Determination of DOBI and carotene content 30
3.6.3 Determination of PV 30
3.6.4 Determination of fatty acids composition 30
3.6.5 Determination of moisture 31
3.6.6 Determination of impurities 31
3.7 High pressure water spray system (HPWSS) 31
3.7.1 Selection of bunch 31
3.7.2 Residual oil removal from OPEFB using HPWSS 31
3.7.3 Residual oil removal efficiency 32
3.7.4 Separation for the mixture of oil and water 33
3.8 Residual oil analysis 33
3.8.1 Determination of FFA 33
3.8.2 Determination of DOBI and carotene content 33
3.8.3 Determination of PV 33
3.8.4 Determination of phosphorus content 34
3.8.5 Determination of Iron (Fe) content 34
3.8.6 Determination of fatty acids composition 34
3.8.7 Fourier transforms infrared spectroscopy (FTIR) 34
3.8.8 Turbidity measurement 34
3.9 Lignocellulosic composition of clean OPEFB 35
3.9.1 Scanning electron microscopy (SEM) 35
3.9.2 Nitrogen adsorption analysis (BET) 35

4 RESULT AND DISCUSSION
4.1 Properties of oil palm empty fruit bunches (OPEFB) 36
4.2 Comparison of oil content analysis among press-shredded, stalk, and spikelet of the OPEFB 39
4.3 Contact angle measurement 40
4.4 Physicochemical quality parameters of Crude Palm Oil (CPO) 43
4.5 Residual oil removal using high pressure water spray system (HPWSS) 46
4.5.1 Bunch selection for residual oil removal using HPWSS 47
4.6 Residual oil yield after HPWSS 49
4.7 Effect of HPWSS on the quality of residual oil 52
4.7.1 Qualities characteristics of residual oil from OPEFB 53
4.7.2 Characteristics of residual CPO from OPEFB 56
4.7.3 FTIR spectra analysis 59
4.7.4 Turbidity measurement 62
4.8 Production of clean biomass (OPEFB) 63
4.8.1 Lignocellulosic compositions of OPEFB fiber after HPWSS 63
4.8.2 Scanning electron microscopy (SEM) 65
4.8.3 Nitrogen adsorption analysis (BET) 67
5 CONCLUSION AND RECOMMENDATIONS
5.1 Conclusion 69
5.2 Recommendations for future work 70

REFERENCES 71
APPENDICES 84
BIODATA OF STUDENT 87
LIST OF PUBLICATIONS 88

LIST OF TABLES

Table		Page
2.1	Distributions of palm oil mill in Malaysia	5
2.2	Oil extraction rate (%) (National Average)	5
2.3	Oil palm biomass chemical composition	9
2.4	Properties or components of OPEFB	10
2.5	Fatty Acid Composition of CPO	16
2.6	Carotenoid composition of palm oil	17
2.7	Quality of CPO based on DOBI values	20
4.1	Mass Fraction Composition of Raw Press-shredded, Spikelet, and Stalk of the OPEFB	38
4.2	Oil Content in Press-shredded, Spikelet, and Stalk of the OPEFB	39
4.3	Physicochemical quality characteristics of Crude Palm Oil (CPO) from the mill, and residual oil from press-shredded and spikelet	44
4.4	Qualities of residual oil extracted from OPEFB at different temperature.	53
4.5	Characteristic of residual oil at different temperature treatment of HPWSS	58
4.6	Evaluation of FTIR spectrum	61
4.7	BET analysis on OPEFB fiber	67

LIST OF FIGURES

Figure		Page
2.1	Demographic distributions of palm oil plantations in Malaysia.	4
2.2	Schematic diagram of palm oil milling process.	6
2.3	Production of OPEFB from the threshing process.	7
2.4	Condition of mesh mesocarp fiber in the digester.	7
2.5	Oil palm biomass generate (wet basis) from oil palm mill in Malaysia.	8
2.6	Oil palm empty fruit bunches (OPEFB).	9
2.7	Typical schematic diagram for soxhlet extraction apparatus.	11
2.8	Simplified flow chart for residual oil recovery system.	12
2.9	The principal of supercritical CO_2 extraction apparatus.	13
2.10	Crude palm oil with fruitlets.	14
2.11	Typical palm oil triglyceride structure.	15
2.12	The difference structures of saturated and unsaturated fatty acids.	16
2.13	Formation of free fatty acids (FFA).	19
2.14	Reaction mechanism of unsaturated fatty acids with oxygen.	20
3.1	Flow diagram of the research work.	22
3.2	Production of press-shredded fiber in the oil palm mill.	23
3.3	Components of OPEFB; stalk and spikelet.	24
3.4	F i b e r t e c Ê 2 0 1 0 , A u t o	24
3.5	Ground size OPEFB fiber at 1.0 mm.	26
3.6	Furnace.	27
3.7	Soxhlet extraction set up and rotary evaporator.	28
3.8	Contact angle goniometer.	29
3.9	Palm oil tester.	30
3.10	High pressure water spray system components.	32
3.11	Separation of free oil from the mixture of oil, water and dirt.	33
3.12	FTIR.	34
4.1	Morphological pictures under fluorescence light of a) spikelet, b) press- shredded, and c) stalk of the OPEFB.	40

4.2	Image of static contact angle. a) spikelet surface with H_2O, b) Press-shredded surface with H_2O, c) spikelet surface with CPO, and d) press-shredded surface with CPO.	41
4.3	Measured contact angle over time for interaction of H_2O with the surface of spikelet and press-shredded of OPEFB.	42
4.4	Measured contact angle over time for interaction of CPO with the surface of spikelet and press-shredded of OPEFB.	43
4.5	Schematic diagram of the high pressure water spray system.	47
4.6	Determination of OPEFB sizes in three days.	48
4.7	Production of OPEFB at the threshing station.	48
4.8	Oil yield profile using high pressure water sprays system (HPWSS) at different temperature.	49
4.9	Residual oil recovered from OPEFB at different temperature of HPWSS and raw CPO.	51
4.10	Oil attachments on the surfaces of OPEFB spikelet before and after treatment of HPWSS using sudan red dye under fluorescent light.	52
4.11	Oxidation mechanism of unsaturated fatty acids.	55
4.12	FTIR spectra of residual CPO from raw OPEFB and residual oil recovered from HPWSS at different temperature.	60
4.13	Turbidity value for residual oil at different temperature of HPWSS.	62
4.14	Lignocellulosic composition of OPEFB after HPWSS at different temperature.	63
4.15	Condition of residual OPEFB after HPWSS at different temperatures condition. a) OPEFB at temperature 30°C, b) OPEFB at temperature 90°C, and c) OPEFB at temperature 150°C.	65
4.16	SEM micrograph (1000x magnification) of OPEFB surface. a) raw OPEFB fiber, b) OPEFB fiber at 30°C, c) OPEFB fiber at 60°C, d) OPEFB fiber at 90°C, e) OPEFB fiber at 120°C, and f) OPEFB fiber at 150°C.	66

LIST OF APPENDICES

Appendix		Page
A	Poster presented in Palm Oil Milling Technology Colloquium POMTecô15)	84
B	Scanning Electron Microscopy (SEM) of OPEFB fiber after HPWSS	85
C	List of Chemicals	86

LIST OF ABBREVIATIONS

FFB	Fresh fruit bunches
MS	Mesocarp fiber
DC	Decanter cake
MPOB	Malaysian Palm Oil Board
PKO	Palm kernel oil
CPO	Crude palm oil
OPEFB	Oil palm empty fruit bunches
POME	Palm oil mill effluent
PKS	Palm kernel shells
OER	Oil extraction rate
HPWSS	High pressure water spray system
CO_2	Carbon dioxide
UAE	Ultrasound assisted extraction
FFA	Free fatty acids
DOBI	Deterioration of bleach ability index
PV	Peroxide value
FTIR	Fourier transforms infrared spectroscopy
ATR	Attenuated total reflection
ADF	Acid detergent fiber
NDF	Neutral detergent fiber
ADL	Acid detergent lignin
ICP-OES	Inductively coupled plasma-optical emission spectrometer
GC	Gas Chromatography
FAO	Food and Agriculture Organization of the United Nations
WHO	World Health Organization
RO	Residual oil
GC-FID	Gas chromatography ïflame ion detector
H_2O	Water
Tg	Teragram

CHAPTER 1

INTRODUCTION

1.1. Overview of the Study

Malaysia, as one of the biggest exporters of palm oil, contributes about 51% of the ZRUOG¶V HGLEOH FUXGH SDOP RLO DQG DFFRXQWV IRU of world exports. In the processing of palm oil, there are two types of products: crude palm oil (CPO) and palm kernel oil (PKO). However, the major problem with this process is the abundance of wastes produced in the form of oil palm empty fruit bunches (OPEFB), mesocarp fiber (MS), decanter cake (DC), and palm oil mill effluent (POME) solids (Baharuddin et al., 2009; Sulaiman et al., 2009; Baharuddin et al., 2010; Baharuddin et al., 2011). From this biomass, the mill generates about 1.5 tons of OPEFB per hectare of oil palm annually (Abd Majid et al., 2012). The OPEFB is generated after the stripping process, in which the sterilized fruits are separated from the bunch stalks. As the major waste of oil palm industry, OPEFB is currently being applied as a wood composite, fiberboard, soil mulching material in the oil palm estate, and as a composting material (Baharuddin et al., 2009; Ibrahim et al., 2009; Kheong et al., 2010). OPEFB is made up of two component parts, which are present in the proportions of 20 to 25% of stalk and 75 to 80% of spikelet (Han and May, 2012). Each of these components can be regarded as lignocellulosic biomass, which is composed of lignin, hemicellulose, and cellulose (Siti Aisyah et al., 2014). During processing, the amount of oil transferred to the OPEFB should be kept to a minimum and also the amount of remaining oil should contain no significant quantity towards the total extracted oil. However, a study conducted by Ngan, (2005) shows that OPEFB does contain a significant amount of oil. Total oil (residual oil) ranged from 0.28 to 1.38% with a mean of 0.75% relative to dry OPEFB. The presence of residual oil on the surface of OPEFB is a result of the stripping and threshing process in the mill where prolonged sterilization and also a lengthy delay between sterilizing and stripping increased the contact time between oil and fiber. According to Abd Majid et al, (2012), the amount of oil entrapped on the fiber depends on the consequence of sterilization of the fruit.

To date, there has been very little research conducted on the removal of residual oil from OPEFB. Detailed study on the exact location of residual oil entrapped is not available in the literature, either on the spikelet or stalk of OPEFB. The oil retained on the OPEFB, which translates as oil losses, has a negative impact on the total OER of palm oil mill industry. The loss of oils from OPEFB is a course of concern for the palm oil industry, as it reflected the overall efficiency of the mill (Sahad et al., 2014). It is believed that the amount of residual oil that could be recovered from the OPEFB could potentially increase the oil extraction rate (OER). Therefore, aimed of this study is to determine the physicochemical characteristics of residual oil and OPEFB fiber for baseline study. Then, a new technology known as high pressure water spray system (HPWSS) will be introduced to remove the residual oil from OPEFB. The evaluation on the effect of HPWSS on the oil yield and physicochemical characteristics of residual oil and OPEFB fiber will be conducted.

1.2. Problem statement

Oil palm empty fruit bunches (OPEFB) is one of the factors that contribute to the oil losses in the mill (Othman and Ng, 2003). OPEFB is made up of lignocellulosic components, such as cellulose, hemicellulose and lignin. This lignocellulosic material has the tendency to adsorb certain amount of oil and therefore reduces the DPRXQW RI ³IUHH´ RLO SURGXFHG $FFRUGLQJ WR 1JDQ (t) in OPEFB is approximately about 8%. The efficiency of crude palm oil extraction is depending on the minimal oil losses during the mill process. Most of the identified source of oil losses in the mill are biomass such as; sterilizer condensate, oil palm empty fruit bunches (OPEFB), mesocarp fiber, nuts, palm oil mill effluent (POME), and spillage in the mill (Menon, 2011). The mills are unable to extract most of the CPO due to the oil losses which leads to the low oil extraction rate (OER).Current oil losses in the OPEFB were poorly recovered and less documented. The oil loss from OPEFB resulted in the reduction of OER in the most oil palm mill. Many scientific reports have been produced concerning the oil contents within the wastes. However limited attempt has been made to recover the oil using green and environmental friendly methods as well as the information on exact localization of remaining residual oil attached on the OPEFB. The current technology applied to recover the remaining oil from OPEFB is using pressing and shredding processes. The OPEFB is transported into the pressing machine, where it is screw pressed, and then shredded for easier handling. The product generated from this process is known as press-shredded OPEFB (Jorgensen, 1985). However, this technology is not efficiently removing the residual oil from OPEFB. From that point, this study propose a new and appropriate technique to enhance the oil removal yield from the OPEFB wastes using the available resources in the mill area with green technology feature and simultaneously achieving the zero waste strategy.

1.3. Objectives

The objectives are:

i) To evaluate the characteristic of the fibers, the initial residual oil, and the localization of residual oil of the oil palm empty fruit bunches (OPEFB).
ii) To investigate the effect of high pressure water spray system (HPWSS) on the yield and the quality of oil, and the physicochemical characteristics of the fiber components (OPEFB).

1.4. Scope of the Study

This study was principally concerned on the strategy to remove the remaining residual oil from OPEFB with high quality and yield of residual oil which leads to the production of clean biomass of OPEFB. This study proposed a new alternative technique which is green, solvent free, and using available facilities on site at the oil palm mill. This study focuses on the investigations of residual oil localization on the OPEFB, the fiber characterization, and the physicochemical characteristics of residual oil. After determination of potential fiber and residual oil from OPEFB, a new technique which is known as high pressure water spray system (HPWSS) for oil removal was performed and the effect of the process was evaluated on the yield and the quality of the oil recovered as well as the physicochemical characteristics of OPEFB fiber. This study aimed to remove the residual oil for edible (high grade CPO) applications or non-edible (low grade CPO) applications such as soaps, resins, candle glycerol, fatty acids, inks, polishing liquids and cosmetics and also to serve the clean OPEFB as a raw material into other valuable product such as bio-sugar and bio-plastic.

1.5. Significance of the Study

This study is proposing a new technique to remove the residual oil entrapped on the surface of the OPEFB. Currently, the most common technique available for oil recovery from OPEFB is using pressed and shredded machine. Based on analysis conducted at the site visit in the palm oil mill utilizing the current method (press-shredded), most of the residual oil located on the surface was transferred into the inner stalk of OPEFB resulted to the loss of oil recovery. However, the proposed technique of this study focused on the removal of the residual oil located on the surface of OPEFB without disrupting other parts of OPEFB. The recovered oil from the OPEFB could either be used to increase the oil extraction rate (OER) of the oil palm mill or as non-edible applications. This process could also increase the market value of OPEFB by the production of clean biomass which might be potential for further applications.

REFERENCES

Ab Rahman, N. N., Al-Rawi, S. S., Ibrahim, A. H., and Nama, M. M. B. (2012). Supercritical carbon dioxide extraction of the residual oil from palm kernel cake. *Journal of Food Engineering*. 108(2012): 166-170.

Abd Majid, R., Mohammad, A. W., and May, C. Y. (2012). Properties of residual palm pressed fibre oil. *Journal of Oil Palm Resource*. 24: 1310-1317.

Abd Wafti, N. S., Yoo, C. K., Lin, S. W., Yaw, T. C. S., and Abdullah, L. C. (2011). Deoiling efficiency for oil extraction from spent bleaching clay and the quality of recovered oil. *Journal of Oil Palm Resource*. 23: 1005-1010.

Abu Bakar, A., Hassan, A., and Mohd Yusof, A. F. (2006). The effect of oil extraction of the oil palm empty fruit bunch on the processability, impact and flexural properties of PVC-U composites. International *Journal of Polymeric Materials and Polymeric Biomaterials*. 55(9): 627-641.

Adaganti, S. Y., Kulkarni, B. M., Desai, G. P., and Shanmukhappa, S. (2013). Effect of hdrothermal explosion pretreatment on the composition and structure of calliandra calothyrsus shrub-a lignocellulosic biomass. *International Journal of Renewable and Sustainable Energy*. 3(1): 1-5.

Ahmad, A. L., Chan, C. Y., Abd Shukor, S. R., and Mashitah, M. D. (2008). Recovery of oil and carotenes from palm oil mill effluent (POME). *Chemical Engineering Journal*. 141(1): 383-386.

Aletor, V. A., Ikhena, G. A., and Egharevba, V. (1990). The quality of some locally processed Nigerian palm oil: An estimation of some critical variables. *Food Chemistry*. 36: 311-317.

Ariffin, A. A. (2000). The effect of CPO quality parameters (FFA, M&I, IV, PV, AV, DOBI, and Colour) on refinery production efficiency. Proceedings of the 2000 National Seminar on Palm Oil Milling, Refinery Technology, Quality and Environment, Genting Highlands, Malaysia, pp. 79-88.

Ariffin, A. A. (2007). The effect of specific quality parameters of crude palm oil (CPO) on the recovery and quality of the intended final palm oil products. Presented at MPOB conference- Kuching, Sarawak.

Arora, S., Manjula, S., Gopala Krishna, A. G., and Subramaniam, R. (2006). Membrane processing of crude palm oil. *Desalination*. 191(2006): 454-466.

Baharuddin, A. S., Wakisaka, M., Shirai, Y., Abd-Aziz, S., Abdul Rahman, N. A., and Hassan, M. A. (2009). Co-composting of empty fruit bunches and partially treated palm oil mill effluent in pilot scale. *International Journal of Agricultural Research*. 4(2): 69-78.

Baharuddin, A. S., Lim, S. H., Md. Yusof., M. Z., Abdul Rahman, N. A., Md. Shah, U. K., Hassan, M. A., Wakisaka, M., Sakai, K., and Shirai, Y. (2010). Effect of palm oil mill effluent (POME) anaerobic sludge from 500 m^3 of closed anaerobic methane digestion tank on pressed-shredded empty fruit bunch (OPEFB) composting process. *African Journal of Biotechnology*. 9(16): 2427-2436.

Baharuddin, A. S., Abdul Rahman, N. A., Md. Shah, U. K., Hassan, M. A., Wakisaka, M., and Shirai, Y. (2011). Evaluation of pressed shredded empty fruit bunch (OPEFB)-palm oil mill effluent (POME) anaerobic sludge based compost using Fourier transform infrared (FTIR) and nuclear magnetic resonance (NMR) analysis. *African Journal of Biotechnology*. 10(41): 8082-8089.

Baharuddin, A. S., Sulaiman, A., Kim, D. H., Mokhtar, M. N., Hassan, M. A., Wakisaka, M., Shirai, Y., and Nishida, H. (2013). Selective component degradation of oil palm empty fruit bunches (OPEFB) using high-pressure steam. *Biomass Bioenergy*. 55(2013): 268-275.

Bahrin, E. K., Baharuddin, A. S., Ibrahim, M. F., Abdul Razak, M. N., Sulaiman, A., Abd-Aziz, S., Hassan, M. A., Shirai, Y., and Nishida, H. (2012). Physicochemical property changes and enzymatic hydrolysis enhancement of oil palm empty fruit bunches treated with superheated steam. *BioResources*. 7(2): 1784-1801.

Bandyopadhyay, A. and Bose, S. (2013). Characterization of biomaterials. (1st ed.). Elsevier Publication (2013). San Francisco, CA.

Barret, E. P., Joyner, L. G., and Halenda, P. P. (1951). The determination of pore volume and area distributions in porous substances. I. Computations from nitrogen isotherms. *Journal of the American Oil Chemists Soci*ety. 73(1): 373-380.

Basiron, Y. (2005). Palm oil. Bailey’s industrial oil and fat products, Sixth Edition. John Wiley & Sons, Inc.

Bashi, S. M., Abdullahi, U. U., Yunus, R., and Nordin, A. (2006). Use of natural vegetable oils as alternative dielectric transformer coolants. *Journal-The Institution of Engineers Malaysia*. 67(2): 4-9.

Brundrett, M. C., Kendrick, B., and Peterson, C. A. (1991). Efficient lipid staining in plant material with Sudan Red 7B or Fluoral Yellow 088 in polyethylene glycol-glycerol. *Biotech. Histochem*. 66: 111-116.

Bushman, R. C., and Harris, W. E. Juice extractor equipment washer. U.S. Patent No. 5,657,927.August 19, 1997.

Carmody, O., Frost, R., Xi, Y., and Kokot, S. (2007). Surface characterisation of selected sorbent materials for common hydrocarbon fuels. *Surface Science*. 601(9): 2066-2076.

Carelli, A. A., Baumler, B.B., and Ceci, L. N. (2012). Improved method for the determination of wax esters in vegetable oils. *European Journal of Lipid Science and Technology*. 114 (11): 1312-1319.

Chang, S. H. (2014). An overview of empty fruit bunch from oil palm as feedstock for bio-oil production. *Biomass and Bioenergy*. 62(2014): 174-181.

Che Man, Y. B., Ammawath, W., and Mirghani, M. E. S. (2005). Determining a-tocopherol in refined bleached and deodorized palm olein by fourier transform infrared spectroscopy. *Food Chemistry*. 90(2005): 323-327.

Che Man, Y. B., Moh, M. H., and van de Voort, F. R. (1999). Determination of free fatty acids in crude palm oil and refined-bleached-deodorized palm olein using fourier transform infrared spectroscopy. *Journal of the American Oil Chemists Society*. 76(4): 485-490.

Chiang, Y. C., and Chen, T. C. (2010). Surface characterization and adsorption performance of electrochemically oxidized activated carbon fibers. *Sustainable Environment Research*. 20(6): 387-395.

Choe, E., and Min, D. B. (2006). Mechanisms and factor for edible oil oxidation. *Comprehensive Reviews in Food Science and Food Safety*. 5 (4): 169-186.

Chong, C. L. (1991). An overview of the effect of milling process and storage on the quality of crude palm oil. Paper presented at the Seminar on Developments in Palm Oil Milling Technology and Environmental Management. Genting Highlands, Pahang. pp: 1-3.

Chong, C. L. (2000). Storage, handling, and transportation of palm oil and palm oil products. In: Advances in Oil Palm Research. Basiron, Y., Jalani, B. S., and Chan, K. W (Eds). Palm Oil Research Institute, Kuala Lumpur, Malaysia.

Cravotto, G., Boffa, L., Mantegna, S., Perego, P., Avogadro, M., and Cintas, P. (2008). Improved extraction of vegetable oils under high-intensity ultrasound and/or microwaves. *Ultrasonics Sonochemistry*. 15(2008): 898-902.

Crude palm oil. Retrieved 8 January 2015 from http://babybeanglobal.com.

Cui, X., Zhao, X., Zheng, J., LOh, S. K., Choo, Y. M., and Liu, D. (2014). Robust enzymatic hydrolysis of formiline-pretreated oil palm empty fruit bunches (EFB) for efficient conversion of polysaccharide to sugars and ethanol. *Bioresource Technology*. 166(2014): 584-591.

Cvjetko, M., Jokic, S., Lepojevic, Z., Vidovic, S., Maric, B., and Redovnikovic, I. R. (2012). Optimization of the supercritical CO_2 extraction of oil from rapeseed using response surface methodology. *Food Technology and Biotechnology*. 50(2): 208-215.

Do, T.X., Lim, Y., and Yeo, H. (2014). Techno-economic analysis of biooil production process from palm empty fruit bunches. *Energy Conversion and Management*. 80(2014): 525-534.

Do, T. X., Lim, Y., Jang, S., Chung, H. J., and Lee, Y. W. (2014). Process design and economics for bioethanol production process from palm empty fruit bunch (EFB). *Computer Aided Chemical Engineering*. 33(2014): 1777-1782.

Dollimore, D., and Heal, G. R. (1964). An improved method for the calculation of pore size distribution from adsorption data. *Journal of Applied Chemistry*.14 (3): 109-114.

Ebewele, R. O., Iyayi, A. F., and Hymore, F. K. (2010). Considerations of the extraction process and potential technical applications of Nigerian rubber seed oil. *International Journal of the Physical Sciences*. 5(6): 826-831.

Enhancement in oil quality, Book Chapter 24 of Advances in Oil Palm Research, 2000, Vol 11:935-962, published by MPOB.

Ergonul, P. G., and Nergiz, C. (2013). A research on the turbidity and wax composition of sunflower and corn oils during winterization. *Bulgarian Journal of Agricultural Science, Agricultural Academy*. 19(3): 497-502.

Febrianto, N. A., and Yang, T. A. (2011). Producing high quality edible oil using eco-friendly technology: a review. *Advance Journal of Food Science and Technology*. 3(4): 317-326.

Fofana, I., Bouaicha, A., Farzaneh, M., and Sabau, J. (2008). Ageing behaviour of mineral oil and ester liquids: a comparative study. Annual Report Conference on Electrical Insualtion Dielectric Phenomena.

Formations of free fatty acids (FFA). Retrieved 20 January 2015 from http://www.namrata.com.

Frank, N. E. G., Albert, M. M. E., Laverdure, D. E. E., and Paul, K. (2011). Assessment of the quality of crude palm oil from smallholders in Cameroon. *Journal of Stored Products and Postharvest Research.* 2(3): 52-58.

Gammil, W., and Proctor, A. (2009). A comparative study of high-linoleic acid vegetable oils for the production of conjugated linoleic acid. *Inquiry*. 10: 30-37.

Gammil, W., Proctor, A., and Jain, V. (2010). Comparative study of high-linoleic acid vegetable oil for the production of conjugated linoleic acid. *Journal of Agricultural and Food Chemistry*. 58 (5): 2952-2957.

Gee, P. T. (2007). Analytical characteristics of crude and refined palm oil and fractions. *European Journal of Lipid Science and Technology*. 109 (2007):373-379.

Gibon, V., De Greyt, W., and Kellens, M. (2007). Palm oil refining. *European Journal of Lipid Science and Technology.* 109(2007): 315-335.

Goering, H. K., and Van Soest, P. J. (1970). Forage fiber analysis (apparatus, reagents, procedures, and some application). USDA Handbook 379, U.S. Gov. Print. Office,Washington, DC.

Goh, S. H., Tong, S. L., and Gee, P. T. (1984). Inorganic phosphate in crude palm oil: quantitative analysis and correlations with oil quality parameters. *Journal of the American Oil Chemists Society*. 61(100): 1601-1604.

Goh, S. H., Choo, Y.M., and Ong, S. H. (1985). Minor constituents of palm oil. *Journal of the American Oil Chemists Society*. 62(2): 237-240.

Hai, T. C., (2002). The palm oil industry in Malaysia: from seed to frying pan. Prepared for WWF Switzerland.

Hadi, N. A., Ng, M. H., Choo, Y. M., and Ma, A. N. (2012). Dry heating of palm fruits: effect on selected parameters. *American Journal of Engineering and Applied Sciences*. 5(2): 128-131.

Han, N. M., and May, C. Y. (2012). Determination of antioxidants in oil palm empty fruit bunches. *American Journal of Applied Sciences*. 9(11): 1862-1867.

Han, S. O., and Choi, H. Y. (2010). Morphology and surface properties of natural fiber treated with electron beam. In: Mendez-Vilas A and Diaz J (eds). *Microscopy,Science, Technology, Applications and Education.*4(3): 1880-1887.

Harsono, S. S., Grundman, P., Lau, L. H., Hansen, A., Mohd Salleh, M. A., Mayer-Aurich, A., Idris, A., and Mohd Ghazi, T. I. (2013). Energy balances, greenhouse gas emissions and economics of biochar production from palm oil empty fruit bunches. *Resource, Conservation and Recycling*, 77: 108-115.

Hassan, M. A., Yee, L. N., Yee, P. L., Ariffin, H., Raha, A. R., Shirai, Y., and Sudesh, K. (2013). Sustainable production of polyhydroxylkanoates from renewable oil-palm biomass. *Biomass and Bioenergy*. 50(2013): 1-9.

Hazir, M. H. M., Shariff, A. R. M., Amiruddin, M. D., Ramli, A. R., and Saripan, M. I. (2012). Oil palm bunch ripeness classification using fluorescence technique. *Journal of Food Engineering.* 113(2012): 534-540.

Hendriks, A. T. W. M., and Zeeman, G. (2009). Pretreatments to enhance digestibility of lignocellulosic biomass. *Bioresource Technology*. 100(2009): 10-18.

Henry. O. H. (2011). Monitoring the free fatty acid level of crude palm oil stored under light of different wavelengths. *American Journal of Food Technology*. 6(8): 701-704.

Hepler, P. K. (2005). Calcium: A central regulator of plant growth and developement. *The Plant Cell*. 17: 2142-2155.

Hidayu, A. R., Mohamad, N. F., Matali, S., and Sharifah, A. S. A. K. (2013). Characterization of activated carbon prepared from oil palm empty fruit bunch using BET and FTIR techniques. *Procedia Engineering*. 68(2013): 379-384.

Hj Hassan, H. A. H., Basiron, Y., Sukaimi, J., Hung, L. K., Salleh, H., and Hassan Basri, M. N. (1985). Palm oil factory process handbook part 1, pp. 75-76: Palm Oil Research Institute of Malaysia.

Hock, L. S. (2011). Co-composting of oil palm mesocarp fiber and palm oil mill effluent anaerobic sludge. Masters of Science Thesis, Universiti Putra Malaysia.

Huang, Y. P., and Chang, J. I. (2010). Biodiesel production from residual oils recovered from spent bleaching earth. *Renewable Energy*. 35(2010): 269-274.

Ibrahim, N. A., Ahmad, S. N. A., Yunus, W. M. Z. W., and Dahlan, K. Z. M. (2009). Effect of electron beam irradiation and poly (vinylpyrrolidone) addition on mechanical properties of polycaprolactone with empty fruit bunch fibre (OPEFB) composite. *eXPRESS Polymer Letters*. 3(4): 226-234.

Ijeh, I. I., Nwokohuru, O. W., Ejile, C. E. (2011). Oxidative stability of red palm oil from two palm varities-Elaeis guineensis and Elaeis oleifera: comparative effect of storage temperature and duration. *Continental Journal of Food Sciences and Technology*. 5(2): 18-22.

Iroba, K. L., Tabil, L. G., Sokhansanj, S., and Dumonceaux, T. (2014). Pretereatment and fractionation of barley straw using steam explosion at low severity factor. *Biomass and Bioenergy*. 66(2014): 286-300.

Jorgensen, H. K. (1985). Treatment of empty fruit bunches for recovery residual oil and additional steam production. *Journal of the American Oil Chemists Society*. 62: 282-287.

Jimenez, A., Beltran, G., and Uceda, M. (2007). High-power ultrasound in olive paste pretreatment. Effect on process yield and virgin olive oil characteristics. *Ultrasonics Sonochemistry*. 14(6): 725-731.

Jung, G. W., Kang, H. M., and Chun, B. S. (2012). Characterization of wheat bran oil obtained by supercritical carbon dioxide and hexane extraction. *Journal of Industrial and Engineering Chemistry*. 18(2012): 360-363.

Kamal-Eldin, A. (2006). Effect of fatty acids and tocopherols on the oxidative stability of vegetable oils. *European Journal of Lipid Science and Technology*. 58(2006): 1051-1061.

Karan, C. P., Rengasamy, R. S., and Das, D. (2011). Oil spill cleanup by structured fibre assembly. *Indian Journal of Fibre & Textile Research*. 36(2011): 190-200.

Kelly-Yong, T. L., Lee, K. T., Mohamed, A. R., and Bhatia, S. (2007). Potential of hydrogen from oil palm biomass as a source of renewable energy worldwide. *Energy Policy*. 35(2007): 5692-5701.

Kheong, L. V., Zaharah, A. R., Hanafi Musa, M., and Aminudin, H. (2010). Empty fruit bunch application and oil palm root proliferation. *Journal of Oil Palm Research*. 22(2010): 750-757.

Kim, S., and Kim, C. H. (2013). Bioethanol production using the sequential acid/alkali-pretreated empty palm fruit bunch fiber. *Renewable Energy*. 54(2013): 150-155.

Kocaefe, D., Poncsak, S., and Boluk, Y. (2008). Effect of thermal treatment on the chemical composition and mechanical properties of birch and aspen. *BioResources*. 3(2): 517-537.

Kong, S. H., Loh, S. K., Bachmann, R. t., Abdul Rahim, S., and Salimon, J. (2014). Biochar from oil palm biomass: a review of its potential and challenges. *Renewable and Sustainable Energy Reviews*. 39(2014): 729-739.

Lam, M. K., and Lee, K. T. (2011).Renewable and sustainable bioenergies production from palm oil mill effluent (POME): win-win strategies toward better environmental protection. *Biotechnology Advance*. 29: 124-141.

Lau, H. L. N., Choo, Y. M., Ma, A. N., and Chuah, C. H. (2006). Quality of residual oil from palm-pressed mesocarp fiber (Elaeis guineensis) using supercritical CO_2 with and without ethanol. *Journal of the American Oil Chemists Society*. 83: 893-898.

Law, K. N., Wan Daud, W. R., and Ghazali, A. (2007). Morphological and chemical nature of fiber strands of oil palm empty fruit bunch (OPEFB). *BioResources.* 2(3): 351-362.

Lazim, Y., Mohd Salit, S., Zainudin, E. S., Mustapha, M., and Jawaid, M. (2014). Effect of alkali treatment on the physical, mechanical, and morphological properties of waste betel nut (areca catechu) husk fibre. *BioResources*. 9(4): 7721-7736.

Lim, T. T., and Huang, X. (2007). Evaluation of kapok (Ceiba pentandra (L.) Gaertn.) as a natural hollow hydrophobic–oleophilic fibrous sorbent for oil spill cleanup. *Chemosphere*. 66(2007): 955-963.

Lin, S. W. (2004). Deterioration of bleachability index. MPOB Information Series. 253, ISN: 1511-7871.

Liu, W., Zu, Y. G., Tong, M. H., Wu, N., Liu, X. L., and Zhang, S. (2009). Supercritical carbon dioxide extraction of seed oil from Opuntia dillenii Haw and its antioxidant activity. *Food Chemistry*. 114(2009): 334-339.

Loh, S. K., Cheng, S. F., Choo, Y. M., and Ma, A. N. (2006). A study of residuals oil recovered from spent bleaching earth: Their characteristics and applications. *American Journal of Applied Sciences*. 3(10): 2063-2067.

Ma, X., Yang, H., Yu, L., Chen, Y., and Li, Y. (2014). Preparation, surface and pore structure of high surface area activated carbon fibers from bamboo by steam activation. *Materials*. 7: 4431-4441.

Mahlia, T. M. I., Abdulmuin, M. Z., Alamsyah, T. M. I., and Mukhlishien, D. (2001). An alternative enery source from palm wastes industry for Malaysia and Indonesia. *Energy Conversion and Management*. 42(18): 2109-2118.

Mat Som, R., Abdul Aziz, A., Wan Hassan, W. H., and Md Top, A. G. (2012). Conversion of lignocellulose from oil palm biomass into water-soluble cellulose ether. *Journal of Oil Palm Research*. 24: 1412-1420.

Mat Jusoh, J., Abd Rashid, N., and Omar, Z. (2013). Effect of sterilization process on deterioration bleachability index (DOBI) of crude palm oil (CPO) extracted from different degree of oil palm ripeness. *International Journal of Bioscience and Bioinformatics*. 3(4): 322-327.

Mohd Zainudin, M. H., Abd Rahman, N. A., Abd Aziz, S., Funaoka, M., Shinano, T., Shirai, Y., Wakisaka, M., and Hassan, M. A. (2012). Utilization of glucose recovered by phase separation system from acid-hydrolysed oil palm empty fruit bunch for bioethanol production. *Pertanika Journal of Tropical Agriculture Science*. 35(1): 117-126.

Mohammed Ali, A. S., and Abdurrahman, A. M. (2013). Determination of free fatty acids in palm oil samples by non-aqueous flow injection using salicyaldehyde-2, 4-dinitrophenylhydrazone as colorimetric reagent. *Chemical and Materials Engineering*. 1(3): 96-103.

Mohammed, M. A. A., Salmiaton, A., Wan Azlina, W. A. K. G., and Mohamad Amran, M. S. (2012). Gasification of oil palm empty fruit bunches: a characterization and kinetic study. *Bioresource Technology*. 110: 628-636.

MPOB Test Methods. (2004). A compendium of test on palm oil products, palm kernel products, fatty acids, food related products and others. Malaysian Palm Oil Board.

MPOB (2010). Overview of the Malaysian Oil Palm Industry 2010.

MPOB (2013). Economics, and Industry Division, MPOB website. Retrieved October 2013c from http://bepi.mpob.gov.my/index.php/statisyical/oil-extraction-rate/102-oil-extraction-rate2012/481-oil-extraction-rate-of-crude-palm-oil-2012.html.

Mueller, D., Ferrao, M. F., Marder, L., Costa, A. B., and Schneider,R.C.S. (2013). Fourier transforms infrared spectroscopy (ftir) and multivariate analysis for identification of different vegetable oils used in biodiesel production. *Sensors*. 13(4): 4258-4271.

Ngan, M. A. (2005).Oil Content in Empty Fruit Bunch. Palm Oil Engineering Bulletin (Formerly Engineering News) 2005 (075) April - June 21– 23. PALM OIL-Analysis; Palm Oil Mills; Oil Extraction; EFB; MPOB Publications.

Norul Izani, M. A., Paridah, M. T., Anwar, U.M.K., Mohd Nor, M. Y., and H'ng, P. S. (2013). Effects of fiber treatment on morphology, tensile and thermogravimetric analysis of oil palm empty fruit bunches fibers. *Composites: Part B*. 45(2013): 1251-1257.

Ohimain, E. I., Izah, S. C., and Fawari, A. D. (2013). Quality assessment of crude palm oil produced by semi-mechanized processor in Bayelsa state, Nigeria. *Discourse Journal of Agriculture and Food Sciences.* 1 (11): 171-181.

Okechalu, J. N., Dashen, M. M., Lar, P. M., Okechalu, B., and Gushop, T. (2011). Microbiology quality and chemical characteristic of palm oil sold within jos metrolpolis, plateu state, Nigeria. *Journal of Microbiology and Biotechnology Research*. 1(2): 107-112.

Omar, R., Idris, A., Yunus, R., Khalid, K., and Aida Isma, M. I. (2011). Characterization of empty fruit bunch for microwave-assisted pyrolysis. *Fuel*. 90: 1536-1544.

Palm oil mill process flow diagram. Retrieved 20 January 2015 from http://www.palmoilextractionmachine.com/

Palm oil triglyceride structure. Retrieved 20 January 2015 from http://myibbiology.blogspot.com/

Piarpuzan, D., Quintero, J. A., and Cardona, C. A. (2011). Empty fruit bunches from oil palm as a potential raw material for fuel ethanol production. *Biomass Bioenergy*. 35(2011): 1130-1137.

Puah, C. W., Choo, Y. M., Ma, A. N., and Chuah, C. H. (2007). The effect of physical refining on palm vitamin E (Tocopherol, Tocotrienol and Tocomonoenol). *American Journal of Applied Sciences* 4(6): 374-377.

Quitain, A. T., Moriyoshi, T., and Goto. M. (2013). Coupling microwave-assisted drying and supercritical carbon dioxide extraction for coconut oil processing. *Chemical Engineering and Science*. 1(1): 12-16.

Reaction mechanism of unsaturated fatty acids with oxygen. Retrieved 28 January 2015 from http://www.fao.org.

Rong, H., Ryu, Z., Zheng, J., and Zhang, Y. (2003). Influence of heat treatment of rayon-based activated carbon fibers on the adsorption of formaldehyde. *Journal of Colloid and Interface Science*. 261(2003): 207-212.

Rossel, J. B. (1991). Measurement of rancidity. In rancidity in foods. Ed By Allen J. C and Hamilton R. J. UK. Aspen Publisher, pp: 22-51.

Rozman, H. D., Tay, G. S., Kumar, R. N., Abusamah, A., Ismail, H., and Mohd Ishak, Z. A. (2001). The effect of oil extraction of the oil palm empty fruit bunch on the mechanical properties of polypropylene-oil palm empty fruit bunch-glass fibre hybrid composites. *Polymer Plastic Technology and Engineering*. 40 (2): 103-115.

Sabil, K. M., Aziz, M. A., Lal, B., and Uemura, Y. (2013). Effects of torrefaction on the physiochemical properties of oil palm empty fruit bunches, mesocarp fiber and kernel shell. *Biomass and Bioenergy*. 56(2013): 351-360.

Sambanthamurthi, R., Sundram, K., and Tan, Y. A. (2000). Chemistry and biochemistry of palm oil. *Progress in Lipid Research*. 39(2000): 507-558.

Sampaio, K. A., Ceriani, R., Silva, S. M., Taham, T., and Meirelles, J. A. (2011). Steam deacidification of palm oil. *Food and Bioproducts Processing*. 89(4): 383-390.

Sahad, N., Md. Som, A., Baharuddin, A. S., Mokhtar, N., Busu, Z., and Sulaiman, A. (2014). Physicochemical characterization of oil palm decanter cake (OPDC) for residual oil recovery. *BioResources* 9(4): 6361-6372.

Saturated and unsaturated fatty acids. Retrieved 20 January 2015 from http://biology.clc.uc.edu/

Setianto, W. B., Atmaji, P., and Anggoro, D. D. (2012). Recovery of palm kernel oil from palm kernel cake using supercritical carbon dioxide and the solubility examination. Prosiding InSINas 2012.

Shamsudin, S., Md Shah, U. K., Zainudin, H., Abd-Aziz, S., Mustapa Kamal, S. M., Shirai, Y., and Hassan, M. A. (2012). "Effect of steam pretreatment on oil palm empty fruit bunch for the production of sugars," *Biomass and Bioenergy*. 36(2012): 280-288.

Siddique, B. M., Muhamad, I. I., Ahmad, A., Ayob, A., Ibrahim, M. H.and Mohd Omar, A. K. (2013). Effect of frying on the rheological and chemical properties of palm oil and its blends. *Journal of Food Science and Technology*. DOI 10.1007/s13197-013-1124-6.

Siew, W. L. (1987). Phosphorus compunds in palm oil. MSc Thesis. University of Salford, UK.

Siti Aisyah, M. S., Uemura, Y., and Yusup, S. (2014). The effect of alkaline addition in hydrothermal pretreatment of empty fruit bunches on enzymatic hydrolysis efficiencies. International conference and workshop on chemical engineering UNPAR 2013, ICCE UNPAR 2013. *Procedia Chemistry*. 9: 151-157.

Sivasothy, K. Advances in Oil Palm Research, Vol 1, 747, Y. Basiron, B.S. Jalani, and K.W. Chan (eds) , Malaysian Palm Oil Board, Kuala Lumpur) (2000).

Soxhlet extraction apparatus. Retrieved 12 January 2015 from http://mkshelford.blogspot.com/

Subramaniam, V., Menon, N. R., Sin, H., and May, C. Y. (2013). The development of a residual oil recovery system to increase the revenue of a palm oil mill. *Journal of Oil Palm Research*. 25(1): 116-122.

Sulaiman, A., Tabatabaei, M., Hassan, M. A., and Shirai, Y. (2009). The effect of higher sludge recycling rate on anaerobic treatment of palm oil mill effluent in a semi-commercial closed digester for renewable energy. *American Journal of Biochemistry and Biotechnology*. 5(1): 1-6.

Sundram, K., Sambanthamurthi, R., and Tan, Y. A. (2003). Palm fruit chemistry and nutrition. *Asia Pacific Journal of Clinical Nutrition*. 12(3): 355-362.

Syam, A. M., Yunus, R., Ghazi, T. I. M., and Yaw, T. C. S. (2009). Methanolysis of jathropa oil in the presence of potassium hydroxide catalyst. *Journal of Applied Science*. 9: 3161-3165.

Tagoe, S. M. A., Dickinson, M. J., and Apetorgbor, M. M. (2012). Factors influencing quality of palm oil produced at the cottage industry level in Ghana. *International Food Research Journal*. 19(1): 271-278.

Tan, B. L., Sarafis, V., Beattie, G. A. C., White, R., Darley, E. M., and Spooner-Hart, R. (2005). Localization and movement of mineral oil in plants by fluorescence and confocal microscopy. *Journal of Experimental Botany*. 56(420): 2755-2763.

Tan, C. H., Ghazali, H. M., Kuntom, A., Tan, C. P., and Ariffin, A. A. (2009). Extraction and physicochemical properties of low free fatty acid crude palm oil. *Food Chemistry*. 113(2): 645-650.

Tangsathitkulchai, C., Sittichaitaweekul, Y., and Tangsathitkulchai, M. (2004). Temperature effect on the viscosities of palm oil and coconut oil blended with diesel oil. *Journal of the American Oil Chemists Society*. 81(4): 401-405.

Thambirajah, J. J., Zulkali, M. D., and Hashim, M. A. (1995). Microbiological and biochemical changes during the composting of oil palm empty fruit bunches. Effect of nitrogen supplementation on the substrate. *Bioresource Technology*. 52 (1995): 133-144.

Thomsen, A. B., Thygesen, A., Bohn, V., Vad Nielsen. K., Pallesen, B., and Jorgensen, M. S. (2006). Effects of chemical-physical pre-treatment process on hemp fibers for reinforcement of composites and for textiles. *Industrial Crops and Products*. 24(2006): 113-118.

Tunio, S. Q., Tunio, A. H., Ghirano, N. A., and El Adawy, Z. M. (2011). Comparison of different enhanced oil recovery technique for better oil productivity. International *Journal of Applied Sciences and Technology*. 1(5): 143-153.

Umar, M. S., Jennings, P., and Urmee, T. (2013). Strengthening the palm oil biomass renewable energy industry in malaysia. *Renewable Energy*. 60(2013): 107-115.

Uquiche. E., Jerez, M., and Ortiz, J. (2008). Effect of pretreatment with microwaves on mechanical extraction yield and quality of vegetable oil from Chilean hazelnuts (Gevuina avellana Mol). *Innovative Food Science and Emerging Technologies*. 9(2008): 495-500.

Uquiche, E., Romero, V., Ortiz, J., and del Valle, J. M. (2012). Extraction of oil and minor lipids from cold-pressed rapeseed cake with supercritical CO_2. *Brazilian Journal of Chemical Engineering*. 29(3): 585-597.

Vlachos, N., Skopelitis, Y., Psaroudaki, V., Chatzilazarou, A., and Tegou, E. (2006). Applications of fourier transform-infrared spectroscopy to edible oils. *Analytica Chimica Acta*. 573-574(2006): 459-465.

Yahya, A., Sye, C. P., Ishola, T. A., and Suryanto, H. (2010). Effect of adding palm oil mill decanter cake slurry with regular turning operation on the composting process and quality of compost from oil palm empty fruit bunches. *Bioresource Technology.* 101 (2010): 8736-8741.

Yap, S. C., Choo, Y. M., Ooi, C. K., Ong, A. S. H., and Goh, S. H. (1991). Quantitative analysis of carotenes in the oil from different palm species. *Elaeis*. 3: 369-378.

Zaharah, A. R., and Lim, K. C. (2000). Oil palm empty fruit bunch as a source of nutrients and soil ameliorant in oil palm plantations. *Malaysian Journal of Soil Science* 4: 51-66.

Zannen, S., Ghali, L., Halimi, M. T., and Ben Hssen, M. (2014). Effect of chemical extraction on physicochemical and mechanical properties of doum palm fibres. *Advances in Materials Physics and Chemistry*. 4: 203-216.