UNIVERSITI PUTRA MALAYSIA

MONITORING CHEMICAL CHANGES OF BINDING CARBOHYDRATES AND SIMULATION OF TEMPERATURE PROFILES DURING STERILIZATION OF OIL PALM FRUIT BUNCHES

THANG YIN MEE

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By

THANG YIN MEE

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Doctor of Philosophy

February 2021

For my wonderful papa and mama, keong, moon, fong, ping, bryan, lisa, minnie
with love….

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

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February 2021

Chairman : Professor Robiah binti Yunus, PhD
Faculty : Institute of Advanced Technology

In a typical palm oil milling process, 30 tons to 60 tons of fresh fruit bunches are processed together and this may cause difficulties for heat to penetrate especially to those fruitlets located at the centre of a large and compact bunch. A complete detachment of the fruitlets is possible only if the moist heat can reach the abscission zone of the fruits to the stalk. A greater understanding of the fundamentals of fruit detachment is necessary as it is shown that the strippability of the sterilized fruitlets would ultimately affect the oil extraction rate.

The study began with the characterization of the binding carbohydrates at the abscission zone of fruitlets before and after sterilization. Response surface methodology (RSM) was applied to model the sugar compositions in sterilized fruits by varying the parameters of pressures, sterilization time, and ripeness of FFBs. Finally, a heat transfer modelling using sophisticated gPROMS software was conducted to provide insights into the heat transfer mechanism inside the sterilizer and FFBs.

The FFBs were collected according to the number of empty fruit sockets: (i) nil; (ii) 1-9; (iii) $\geq$10 as unripe, under-ripe, and ripe bunches, respectively. The PCA plots revealed that the oligomers are the chemical markers that are responsible for differentiating the ripe samples from the others. Both non-structural and structural sugars are the factors that differentiate under-ripe samples from the rest.

All experiments showed some level of the detachment of sterilized fruits after the sterilization. Low sterilization pressure or short holding time resulted in insufficient cell

wall rupture, thus a high percentage of unstripped bunches. HPLC analysis showed that large percentages of oligomers were observed in sterilized fruits, ranging from 2.05-11.44 w/v%. Only about 0.15-1.19 w/v% of simple sugars were detected in sterilized fruits. RSM optimization confirmed that oligomers could be used as a biomarker to relate to fruits strippability after sterilization.

In addition, gPROMS has successfully predicted the temperature profiles in the sterilization experiments. The simulation proposed that the sterilized fruits could only be 100% stripped if the temperature of FFBs is kept above 48^{o}C for at least 53.5 mins. In addition, the oligomers content of sterilized fruits must be more than 3.8 w/v%. Under this optimum condition, the rate of oil loss will be lower and the OER will ultimately be improved.

Abstrak tesis yang dikemukakkan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

PEMANTAUAN PERUBAHAN KIMIA KARBOHIDRAT TERIKAT DAN SIMULASI REKOD SUHU SEMASA PENSTERILAN TANDAN KELAPA SAWIT

Oleh

THANG YIN MEE

Februari 2021

Pengerusi : Profesor Robiah binti Yunus, PhD
Fakulti : Institut Teknologi Maju

Kajian ini menjelaskan pemprosesan ke atas tandan kelapa sawit sesama tangkainya di dalam kilang minyak kelapa sawit. Langkah pemprosesan membebankan kesusahan haba penebusan terutamanya ke bahagian biji kelapa sawit pada tangkainya khasnya saiz tandan kelapa sawit yang besar dan padat. Hasilnya, haba yang lembap dan mencukupi amat diperlukan untuk menebus ke bahagian dalaman tangkai. Pemprosesan memecahkan buah kelapa sawit daripada tangkai memerlukan haba penebusan yang mencukupi. Daripada pemecahan, pengkaji dapat mengurangkan kehilangan minyak.

Kajian ini melibatkan pensterilan ke atas bahagian “abscission” buah kelapa sawit bagi tempoh sebelum dan selepas. Gula composisi buah kelapa sawit digunakan untuk mendapatkan model yang sesuai melalui metodologi permukaan tindak balas (RSM). Model pemindahan haba dilakukan dengan gPROMS dalam kajian ini. Suhu optima untuk keturunan karbohidrat terikat untuk mencapai 100% kadar pemecahan dapat dicadangkan.

Sampel kajian iaitu kelapa sawit terdiri daripada (i) kosong (tidak masak); (ii) 1-9 (kalat); (iii) ≥10 (matang). Plot analisa komponen utama menunjukkan oligomer merupakan penanda kimia yang bertanggungjawab untuk membezakan buah yang matang daripada golongan sampel yang lain. Gula berstruktur dan bukan berstruktur adalah faktor yang membezakan buah kalat daripada golongan sampel yang lain.

Keturunan karbohidrat terikat di dalam sabut dan pemecahan pada kadar yang berbeza didapati di dalam eksperimen pensterilan. Malangnya, kadar pemecahan ke atas dinding

cel yang rendah disebabkan oleh pensterilan dengan tekanan yang rendah dilakukan pada masa yang singkat. Analisa HPLC menunjukkan kandungan oligomer di dalam buah mencapai kadar antara 2.05-11.44 w/v%. Hanya 0.15-1.19 w/v% gula didapat di dalam buah selepas pensterilan. Model RSM dapat memastikan bahawa oligomer boleh digunakan sebagai bertanda kimia untuk kadar pemecahan selepas pensterilan.

Eksperimen pensterilan dengan model gPROMS diterima atas keputusan hampir tepat untuk meramalkan profil suhu. Simulasi mencadangkan untuk mencapai 100% pemecahan buah kelapa sawit memerlukan haba penebusan pada suhu melebihi 48^{o}C selama sekurang-kurangnya 53.5 minit. Tambahan pula, oligomer buah mesti mencapai sekurang-kurangnya 3.8 w/v%. Oligomer boleh digunakan sebagai kawalan untuk menentukan kecekapan proses pensterilan di dalam kilang memproses minyak kelapa sawit. Dengan keadaan optima ini, kadar kehilangan minyak dapat dikurangkan, justeru itu, hasilan minyak dapat dinaikkan.

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Additionally, I would like to thank my family members and friends for their unflagging moral support. Moreover, I would like to thank many others who have provided special research materials and other forms of assistance in the completion of this work.

I certify that a Thesis Examination Committee has met on _______ to conduct the final examination of Thang Yin Mee on her thesis entitled "Monitoring Chemical Changes of Binding Carbohydrates and Simulation of Temperature Profiles during Sterilization of Oil Palm Fruit Bunches" in accordance with Universities and University Colleges Act 1971 and Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the relevant degree of Doctor of Philosophy.

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University of Campania Luigi Vanvitelli
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School of Graduate Studies
Universiti Putra Malaysia

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Robiah binti Yunus, PhD
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Universiti Putra Malaysia
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Associate Professor, Ing
Faculty of Engineering
Universiti Putra Malaysia
(Member)

Abdul Azis bin Ariffin, PhD
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Institute of Advanced Technology
Univerisit Putra Malaysia
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David Ross Appleton, Ph.D.
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Biotechnology and Breeding
Sime Darby Plantation Technology Centre Sdn. Bhd.
(Member)

Ahmad Jaril Asis, PhD
Senior Vice President II
Sime Darby Research
Sime Darby Plantation Research Sdn. Bhd.
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Name of Member of Supervisory Committee: Mohd Noriznan Mokhtar

Signature: ____________________
Name of Member of Supervisory Committee: Abdul Azis Ariffin.

Signature: ____________________
Name of Member of Supervisory Committee: David Ross Appleton

Signature: ____________________
Name of Member of Supervisory Committee:

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Term	Definition
a	Air
a, b	Van der Waals
ADD	Absolute average deviation
ADF	Acid detergent fiber
ADS	Acid detergent soluble
amb	ambiance
ANOVA	Analysis of variance
as	Ash
atm	Atmosphere
BA_s	Total bottom surface area of sterilizer
c	Carbohydrate
C5	5-carbon
C6	6-carbon
CA_s	Cord length area, surface between liquid and gas
CCRD	Central composite rotatable design
cond	Condensation
Cp	Specific heat capacity
CPO	Crude palm oil
dew	Dew point
DOBI	Deterioration of bleachability index
EDTA	Ethylenediaminetetraacetic acid

EFB	Empty fruit bunches
ELSD	Evaporative Light Scattering Detector
EWL	Equivalent water loss
f	Fruits
fa	Fat
FFA	Free fatty acid
FFB	Fresh fruit bunch
fi	Fiber
g	gram
g	Gas
GHz	Gigahertz
H	Enthalpy
h	Heat transfer coefficient
ha	Hectare
HMF	hydroxymethylfurfural
HPLC	High performance liquid chromatography
hr	Hour
i.d	Internal diameter
J	Joule
k	Kilo
k	Thermal conductivity
K	Kelvin
K	Valve coefficient
kg	Kilogram

kg/m^3	Kilogram per cubic meter
kPa	Kilo pascal
L	Liter
l	Liquid
LC	Liquid chromatography
m	mass
m^2	Square meter
m^2/s	Meter squared per second
m^3	Cubic meter
MARDI	Institute Penyelidikan dan Kemajuan Pertanian Malaysia
min	minute
mL	mililiter
mm	Milimeter
MPa	Megapascal
MPD	Mass pass-through digester
MPOB	Malaysian Palm Oil board
$\dot{m}_s$	Mass flux
MT	Metric ton
MT	Ton
n	Geometric power constant
n.a	Not available
n.d	Not detectable
NDF	Neutral detergent fiber
NDS	Neutral detergent soluble

nm	Nanometer
NREL	National Renewable Energy Laboratory
NS	Non-structural sugar
ºC/s	Degree Celsius per second
°C	Degree Celsius
OER	Oil extraction rate
p	pressure
Pa	Pascal
PCA	Principal component analysis
PG	Polygalacturonase
PPF	Palm pressed fiber
PRESS	Predicted residual error sum of squares
psi	Pound per square inch
Q	Energy balance
$\dot{q}$	Heat flux
R	Universal gas constant
r	Radius
R^2	R-square
R_f	Radius of FFB
RM	Ringgit Malaysia
rpm	Revolutions per minute
RSM	Response surface methodology
S	area
S	Surface area

SA_s	Total surface area of sterilizer
sat	Saturation
$SC\text{-}CO_2$	Supercritical Carbon Dioxide
SS	Structural sugar
t	Time
T	Temperature
TA_s	Total top surface area of sterilizer
th	Thickness
UDR	Under-ripe
µL	Microliter
µm	Micrometer
UPM	Universiti Putra Malaysia
USB	Unstripped bunches
V	Volume
v	Vapor
v/v	Volume per volume
VWD	Variable wavelength detector
W	Watt
w	Wall
W/mK	Watts per meter-Kelvin
w/v	Weight per volume
w/w	Weight per weight
wa	Water
WAP	Weeks after pollination

WAP	Week after anthesis
WS	Water-soluble sugar
%	Percent
x	Mass fraction
α	Thermal diffusivity
γ	Ratio of specific heat
ρ	Density

CHAPTER 1

INTRODUCTION

1.1 Background

The oil palm, *Elaeis guineensis*, belongs to the family of *Arecaceae* (formerly known as the *Palmae*) (Corley and Tinker, 2003). The fruits are borne on spikelets, which are spirally arranged to form a compact fresh fruit bunch (FFB) with the weight in between 10 kg and 25 kg with 1000 to 3000 fruitlets per bunch (Teh *et al.*, 2013; Tan *et al.*, 2010). Each fruitlet consists of a hard kernel (seed) enclosed in a shell (endocarp) which is surrounded by a fleshy mesocarp (Figure 1.1). The crude palm oil (CPO) is obtained by pressing the mesocarp of the oil palm fruit.

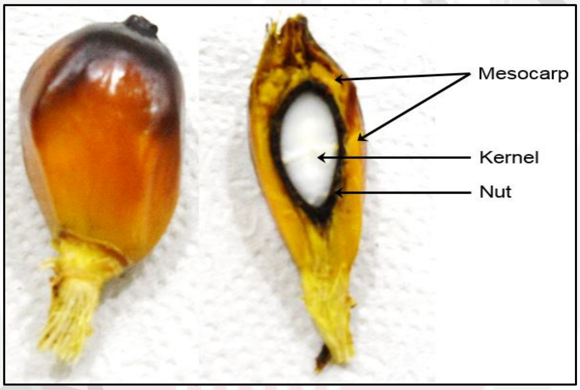

Figure 1.1: Oil palm fruits

The oil palm is the most efficient oil-bearing crop in the world, that can produce about 4 MT to 5 MT of CPO per hectare per year (MPOC, 2021). It is also the most important economic crop for Malaysia. In 2018, the planted area covered 5.85 million ha, it has increased to 5.9 million ha in 2019 (MPOB, 2018; MPOB, 2019). Palm oil contributes to 29% of the global oils and fats. Malaysia is the second largest exporter of palm oil which contributes to 30% of the world total palm oil exports. The demands of palm oil will be triple in the year 2050 exceeding 250 million MT of the total world's production of oils and fats (Corley, 2009; Zulkifli *et al.*, 2017). This is due to the increasing of the world population which is expected to be ten billion in the year 2050 (Barcelos *et al.*, 2015; Kushairi *et al.*, 2017). Thus, the oil palm industry needs to remain competitive by increasing the yield per unit area without the forest expense.

The oil content in the mesocarp is about 22.5% to 27.5% of the bunch weight (Wood, 1985). However, the current mill practices are unable to achieve these numbers on an average due to inefficient milling process and high oil losses, as well as other factors such as improper harvesting practices, contamination of the *tenera* seed (thin kernel shell) with *dura* material (thick kernel shell) due to faulty pollination, and age of palm trees (Corley and Tinker, 2003). Even the latest technology that offers improved milling efficiency with reduced labour and maintenance cost is still unable to close the gap in the oil extraction rate (OER). The OER in Malaysia remained in the range of 20.49% and 19.95%, respectively for year 2010 and 2018 (MPOB, 2010; Adzmi *et al.*, 2012; MPOB, 2019).

Therefore, the whole oil palm industry in Malaysia has put the hand together to work towards producing premium high yielder (Zamzuri *et al.*, 2007; Kushairi *et al.*, 2018; Parveez *et al.*, 2020), improve the harvesting and milling efficacy and efficiency (Vincent *et al.*, 2014; Sivasothy *et al.*, 2005; Sukaribin and Khalid, 2009), as well as to reduce the oil loss by recovering the residual oil from the solid waste materials (Subramaniam *et al.*, 2013; Jorgensen, 1985). However, some of these approaches require expensive and extensive technology advancement and mostly still in its early stage, furthermore, some approaches are labour intensive. Hence, the mechanism of fruit loosening from the bunch has gained interest among researches especially to minimise oil loss in the milling process, particularly to reduce the unstripped bunches (USB).

A critical and often neglected area where losses can be high is in the recovery of USB. The oil loss due to USB stuck in the bunch stalk accounts for about one-third of the total oil loss in palm oil mills (Babatunde *et al.*, 1988; Zulkifli and Ropandi, 2001; Walat and Ng, 2013). Losses from poor mill efficiency are shown in Table 1.1. Due to ineffective sterilization process, the total oil loss amounts to 0.77% (39.89% of total oil losses). This is equivalents to RM 306.5 million based on an average CPO price of RM 2,000 in 2019 (MPOB, 2019). Thus, any improvement made on the process will contribute significantly to the OER and directly contribute to revenue gain with more CPO produced.

Table 1.1: Oil losses and mill efficiency

Sources of oil loss	**Oil loss on FFB (%)**
Sterilizer condensate	0.16
Empty fruit bunches	0.56
Fruit loss in empty bunches	0.03
Unstripped bunches	0.02
Press cake fiber	0.55
Nuts	0.06
Sludge/ decanter	0.46
Washings/ spillages	0.09
Oil losses	1.93
Oil extraction rate (OER)	20.80
Total oil yield (1.93+20.80)	22.73
Oil milling efficiency (20.80/22.73 x 100)	92

(Adapted from Walat and Ng, 2013)

1.2 Problem Statement

Sterilization is the heart of palm oil milling process. Most of the technology development in the palm oil milling industry so far has focused primarily on the alternative sterilization techniques which aimed at improving the OER (Vincent *et al.*, 2014; Sivasothy *et al.*, 2005; Hadi *et al.*, 2012; *et al.*, 2012; Sukaribin and Khalid, 2009; Omar *et al.*, 2017). The techniques mainly differ in orientations; horizontal, vertical, tilted, spherical, and continuous sterilizers are among the commercially available units. Still, a lot of incidences of USB occur due to inefficient sterilization. This suggests that the reasons underlying the high USB issues are not well understood. A high percentage of USB contributes to oil losses as unstripped fruits contain oil that could have been extracted if the fruits are completely stripped from the bunch.

So far, no attempt was made to assess the fundamental issues that underline the oil palm fruit strippability or separation in these sterilizers. For instance, the effects of sterilization conditions on the degradation of binding carbohydrates at the abscission zone of oil palm fruits are not well known. The conversion of lignocellulosic fibers into simpler sugars affects the strippability as it facilitates the detachment of fruitlets from the bunch. Besides, sterilization condition towards FFBs with different ripeness level yet to be optimized. No study has sought a profiling pattern between the binding carbohydrates characteristics and the manual ripeness determinations of FFBs delivered to the palm oil mill. Furthermore, no study on the suitability of current sterilization parameters in processing the FFBs with different ripeness levels are available. This aspect needs to be explored to provide an in-depth understanding of the sterilization process to maximize the oil yield and quality. As sterilization could facilitate the rupture of the cell wall and release of oil (Owolarafe and Faborode, 2008). In addition, the temperature distribution of FFBs inside the sterilizer are not well established to ensure sufficient moist heat is penetrate to the points of attachement of the fruits to the stalk (abscission zone). The hypothesis here is that better heat penetration efficiency during sterilization process will result in a higher rate of degradation of lignocellulosic fibers to sugars, thus higher percentage of strippability and lower rate of oil loss, and ultimately will improve mill efficiency and OER.

1.3 Contribution of the Research

In this project, numerous sterilization experiments were carried out to study the changes in the compositions of the binding carbohydrates at oil palm fruits/ fruits to stalk (abscission zone) before and after sterilization. The binding carbohydrates, namely non-structural, structural and water-soluble sugar compositions of abscission zones were analyzed from unripe, under-ripe and ripe bunches and managed using chemometric techniques for classification purpose according to the ripeness of the FFBs. The sugars composition at the abscission zone could potentially be used as a chemical marker to differentiate those bunches at different ripeness stages.

The relationship between the chemical compositions of the carbohydrates (oligomers and other saccharides, as well as proximate analysis) in the fibers at the abscission zone of FFBs before and after the sterilization process were investigated. The effects of sterilization parameters such as pressure and holding time on the chemical contents of FFBs at different ripeness levels were also evaluated based on the fruitlets strippability. The sugars composition of the sterilized fruit could potentially be used as an indicator to optimize the sterilization parameter to achieve 100% strippability with zero USB. At the same time, to develop a suitable process control monitoring parameter to determine the effectiveness of sterilization and stripability.

The pressure profile during sterilization was modelled using gPROMS software to assess the phenomena of heat transfer in the FFBs. The information was used to relate the degradation of the binding carbohydrates at the abscission zone and concurrently to the strippability of FFBs. Understanding of the temperature profiles within the FFBs during sterilization to provide insights on the impact of pressure and holding time of sterilization on the strippability of FFBs without having the temperature sensor inside the bunch. Having a temperature sensor installed inside the bunch is a costly and challenging process, but this could be solved by using a simulation software. The simulation offers a cheaper way to predict the temperature profiles inside the sterilizer and oil palm bunch. From the profiles, the optimum temperature required for the degradation of the binding carbohydrates at the abscission zone can be calculated for the 100% strippability.

1.4 Research Objectives

The overall objective of this study is to investigate the relationship between the binding carbohydrates compositions at the abscission zone of oil palm fruits/stalk and sterilization efficiency based on the strippability of fruitlets. The specific objectives are:

1. To determine the compositions of the binding carbohydrates in fresh fruits bunches (FFBs) and correlate with its ripeness levels using chemometric method.

2. To evaluate the effects of sterilization conditions on FFBs with different ripeness categories based on the chemical changes in the binding carbohydrates at the abscission zone and relate the effects to the fruitlets strippability.

3. To simulate the temperature distribution of FFB inside the sterilizer and in palm fruit bunches using gPROMS software.

1.5 Scope

1. Characterization at the abscission zone of FFBs before sterilization
   FFBs with different ripeness levels, namely unripe, under-ripe, and ripe were harvested from nearby oil palm plantation. The binding carbohydrates, such as non-structural, structural and water-soluble sugar compositions were determined and subjected to principal component analysis for ripeness classification.

2. Characterization of FFBs after sterilization
   The sterilization trials with double peak steam cycle under various conditions (pressure, temperature, retention time) using FFBs with different ripeness (under-ripe and ripe) were conducted. Then, the physical appearance such as strippability of sterilized fruits was observed. Chemical characteristics such as contents of binding carbohydrates such as oligomers and other saccharides, as well as the secondary degraded products were monitored. The sugar contents in sterilizer condensates obtained were also determined. The sugar compositions and pressure data from the sterilization experiments were modelled using Response Surface Methodology (RSM).

3. Simulation of heat transfer mechanism
   The pressure profiles obtained from sterilization experiment were managed using gPROMS software to predict the temperature profiles within the FFBs throughout the sterilization process.

1.6 Thesis Organization

This thesis is arranged in five chapters. Following the introductory chapter, a review of literature is given in Chapter 2. Materials and methodology including the analytical procedures are included in Chapter 3. Experimental results with discussion are reported in Chapter 4. Finally, Chapter 5 gives a summary for the work described in this thesis and the recommendations for further studies.

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