UNIVERSITI PUTRA MALAYSIA

UV-VIS SPECTROSCOPY CHARACTERISTIC OF CRUDE PALM OIL AT 55ºC - 120ºC

NOR AZRINA BINTI ANUAR

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By

NOR AZRINA BINTI ANUAR

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

January 2018

DEDICATION

Special thanks:

My beloved husband and son

My parents, brothers, sisters and friends.

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

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Chairperson : Associate Professor Zulkifly Abbas, PhD
Faculty : Science

This thesis describes an application of fibre optics technique to determine properties of heated crude palm oil (CPO) at 300 to 895 nm in the ultraviolet-visible (UV-VIS) spectrum based on reflection, transmission and absorption measurement. The CPO was heated on the hotplate at 55, 80, 105 and 120 ˚C for 60 min. The heated CPO was sample out for every 10 min and let cool to room temperature before performing the measurements using an Ocean Optics USB4000 UV-VIS-NIR spectrometer. Colour of the heated CPO changed from orange-red to almost colourless particularly after 60 min suggesting that it was heat bleached. All the measurements indicated presence of tocotrienol and tocopherol, carotenoid and chlorophyll b elements at approximately 350, 460 and 650 nm, respectively. However, the measurements trend for the elements could not be determined since their variations were very small and overlapping. It was found that the reflection measurement was imprecised due to the translucency of the CPO. Conversely, the transmission and absorbance measurement were found to increase and decrease, respectively at 400 to 765 nm mainly for CPO with heating times of 50 and 60 min regardless of heating temperatures. This was probably because of change in CPO properties and its electromagnetic interaction as it had been heated for longer times. However, the transmission and absorbance for 10 to 40 min heated CPO from 55 to 105 ˚C were similar to the non-heated CPO since their colours were not much differ indicating that some of their properties and electromagnetic interaction were maintained. Region from 525 to 565 nm in the VIS spectrum was chosen for further analysis since the measurements showed many changes at these wavelengths. The cut off wavelengths from 525 to 565 nm decreased as the heating times and temperatures increased except for 105˚C which was slightly off the trend. It was possibly that the CPO was heat bleached and there was also a measurement error at 105˚C. It was also observed that the decreasing trend in the cut offs for 60 min heated CPO was different and this was probably due to change in its properties as it appeared almost colourless and had higher transparency among the CPO at other heating times. The sensitivity and critical heating time showed decreasing trends from 525 to 540 nm at all heating temperatures except for 105˚C. In addition, it was noted that the measurements were highly sensitive at low wavelengths from 525 to 540 nm and also

at lower heating temperatures from 55 and 80 ˚C. The mean relative errors of the transmission and absorbance indicated that wavelengths from 540 to 555 nm and 535 to 550 nm respectively were within 10% errors for all heating temperatures excluding 105˚C. It was concluded 540 nm might be useful for the prediction of the heating times at 55, 80 and 120˚C since both measurements were highly sensitive and had mean relative error which was less than 10 % at this wavelength.

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

SIFAT SPECTROSKOPI UV-VIS BAGI MINYAK SAWIT MENTAH PADA 55ºC – 120ºC

Oleh

NOR AZRINA BINTI ANUAR

Januari 2018

Pengerusi : Professor Madya Zulkifly Abbas, PhD
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Tesis ini menerangkan aplikasi teknik gentian optic sebagai satu kaedah yang cepat dan tepat untuk menentukan sifat minyak sawit mentah yang dipanaskan pada gelombang ultraungu-cahaya Nampak berdasarkan pengukuran refleksi, penghantaran dan penyerapan. Pengukuran telah dijalankan menggunakan spektrometer Ocean Optics USB4000 UV-VIS-NIR dari 300 sehingga 895 nm. Minyak sawit mentah telah dipanaskan di atas alat pemanas makmal pada 55, 80, 105 dan 120ºC selama 60 min. Sampel minyak sawit mentah yang dipanaskan itu diasingkan untuk setiap 10 min dan dibiarkan sejuk sehingga suhu bilik sebelum pengukuran dijalankan. Warna minyak sawit mentah yang dipanaskan telah berubah warna daripada jingga kemerahan ke hampir tidak berwarna (jernih) pada penghujung masa pemanasan. Semua ukuran gentian optic menunjukkan kehadiran tokotrienol dan tokoferol, karotenoid dan klorofil b masing-masing pada kira-kira 350, 460 dan 650 nm. Walau bagaimanapun, pengukuran refleksi adalah tidak tepat disebabkan oleh kelutcahayaan minyak sawit mentah. Penghantaran dan penyerapan masing-masing meningkat dan menurun pada cahaya nampak terutamany a untuk masa pemanasan selama 50 dan 60 mini ttanpa mengira suhu pemanasan. Ini mungkin disebabkan oleh perubahan sifat minyak sawit mentah dan interaksielektromagnetik kerana ianya telah dipanaskan untuk jangka masa yang lama. Sebaliknya, penghantaran dan penyerapan pada masa pemanasan yang lain adalah hamper sama dengan minyak sawi tmentah yang tidak dipanaskan terutamanya pada 55 sehingga 105ºC. Cut-off gelombang dari 525 hingga 565 nm menurun apabila masa dan suhu pemanasan meningkat kecuali untuk 105 ºC yang tidak mengikut trend. Ini mungkin kerana minyak sawit mentah mengalami pelunturan secara pemanasan dan juga terdapat ralat dalam pengukuran pada 105 ºC. Trend penurunan dalam cut-off minyak sawit mentah bagi pemanasan selama 60 min juga didapati berbeza dan ini mungkin merupakan petunjuk pada perubahan sifat minyak tersebut secara menyeluruh kerana warnanya telah menjadi hamper tidak berwarna dan mempunyai kelutsinaran yang paling tinggi berbanding masa pemanasan yang lain. Kepekaan dan masa pemanasan kritikal menunjukkan trend penurunandari 525 sehingga 540 nm bagi semua suhu pemanasan kecuali 105ºC. Walau bagaimanapun, pengukuran ini diperhatikan mempunyai kepekaan yang tinggi pada gelombang yang rendah iaitu dari

525 sehingga 540 nm dan juga pada suhu pemanasan yang rendah. Purata ralat relative bagi penghantaran dan penyerapan masing-masing menunjukkan pengukuran dari 540 sehingga 555 nm dan 535 sehingga 550 nm adalah dalam 10% ralat bagi semua suhu pemanasan kecuali 105 ºC. Oleh itu, disimpulkan bahawa pengukuran pada 540 nm kemungkinan berguna untuk ramalan masa pemanasan bagi suhu pemanasan 55, 80 dan 120ºC.

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

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

UV	Ultraviolet
VIS	Visible light
CPO	Crude palm oil
NIR	Near-infrared
min	Minute
MPOB	Malaysian Palm Oil Board
RBD	Refined, bleached and deodorized
FFA	Free fatty acid
PV	Peroxide value
AV	Anisidine value
DOBI	Deterioration of bleachability index
DSC	Differential scanning calorimeter
FTIR	Fourier-transform infrared
GC-MS	Gas chromatography-mass spectrometry
HPLC	High performance liquid chromatography
IR	Infrared
EEM	Excitation emission fluorescence spectroscopy
FELDA	Federal Land Development Authority
PTFE	Polytetrafluoroethylene
OD	Optical density

CHAPTER 1

INTRODUCTION

The oil palm (*Elaeis guineensis jacquin*) was formerly originated from South Africa. Later, it was introduced to East Asia as an ornamental in 1871 (Basiron and Chan, 2004) and planted at the Bogor Botanical Garden Java, Indonesia in 1884 (Siew, 2002). The oil palm was then commercially exploited for its oil from 1911 whereby the first oil palm estate established (Basiron and Chan, 2004). Now, the oil palm industry is one of major contributors for Malaysian economic growth. In fact, Malaysia is the second palm oil world exporter after Indonesia (Figure 1.1) and it is largely traded to China (Figure 1.2).The palm oil is not only used for edible purposes but also for non-food products ranging from oleochemicals to biomass-derived materials (Murphy, 2014;Sulaiman et al., 2012). Recently, edible oils including palm oil have been used to produce biodiesel fuel for vehicles as one of the alternative replacements for non-renewable fuels. This palm oil biodiesel is accounted for below 10% of total palm oil production and this industry sector is continuing to grow bigger as research and technologies advanced (Murphy, 2014). Thus, indisputably the international trade of the palm oil will increase in numbers with much diversity and it would be the interest of all parties involved that high quality of palm oil be maintained from the harvesters to the end users.

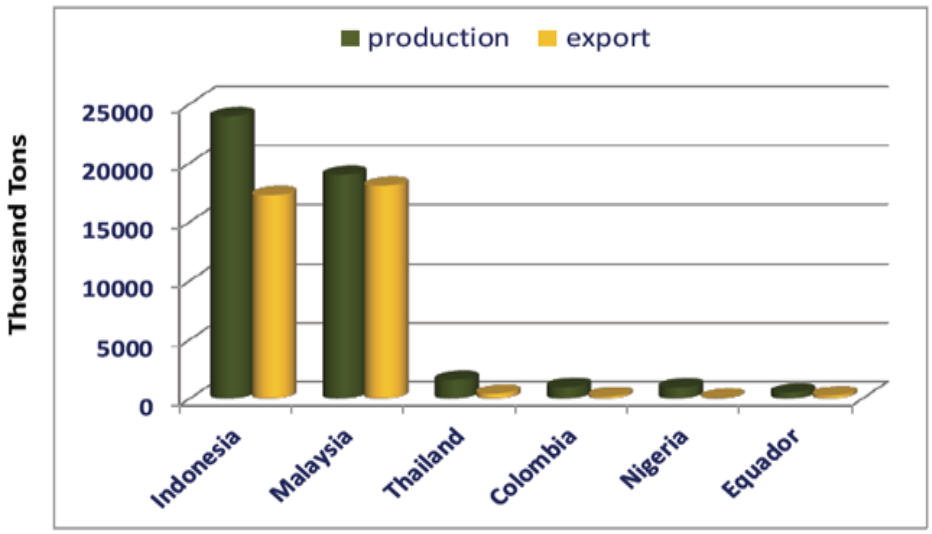

Figure 1.1: Major palm oil producers and exporters (Source: MPOB, 2011)

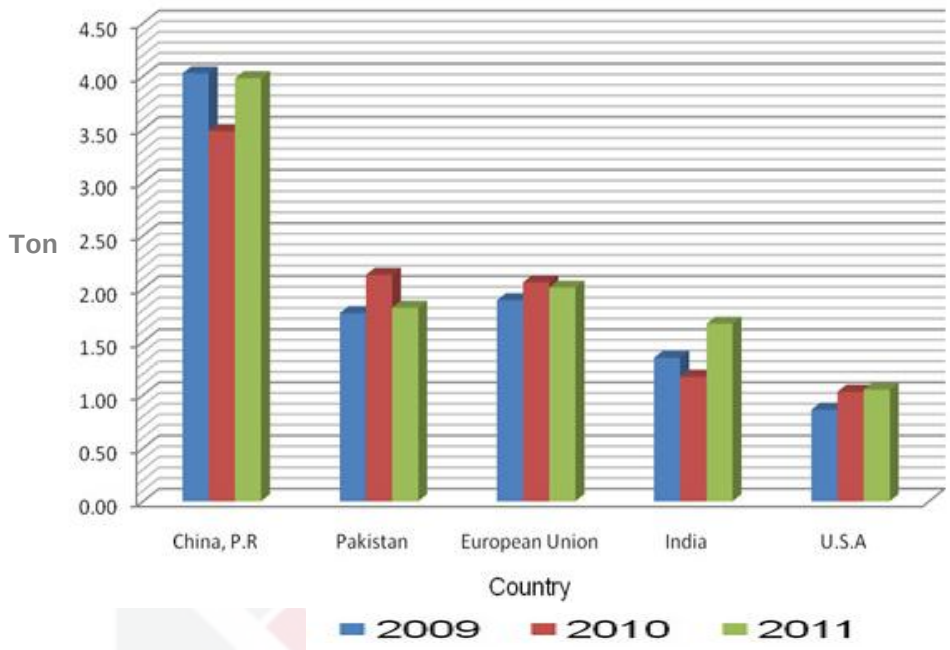

Figure 1.2: Major importers of Malaysian palm oil from 2009 to 2011 (Source: MPOB, 2011)

1.1 Overview of Palm Oil

The oil palm is known for its efficiency to produce oil among the other oil-producing plants. The palm is usually bearing fruits after three years of planting and continues to do so until 25 years (Siew, 2002). There are two types of oil that can be obtained from the palm fruit which are crude palm oil (CPO) and palm kernel oil (PKO). CPO is derived from the mesocarp of the fruit whereas PKO is from kernel inside the nut. The general processes of CPO include mechanical pressing extraction, physical refining and fractionation in order to produce refined palm oil. CPO is greatly favourable than other vegetable oils since it is rich in vitamin A and E due to high content of carotenoids and tocotrienols, respectively (Mba et al., 2015).These carotenoids and tocotrienols are also antioxidants that help stabilize the oil against oxidation (Siew, 2002). CPO is largely used as cooking oil because of its almost equal amount of saturation and unsaturation fatty acids, and its high smoke point which is about 230 °C that corresponding to the oxidative stability and also it is not easily smoke when first used (Mba et al., 2015).

There are many guidelines available to ensure the palm oil quality. Malaysian Palm Oil Board (MPOB) and Department of Malaysian Standard are among the organizations that provide the guidelines for CPO and refined palm oil in Malaysia (Basiron and Chan, 2004). The guidelines specified the protocols for quality assessments, characteristics of the oil and also recommendation for storage and transport of the oils. The quality assessments include free fatty acid (FFA) content, moisture and impurities, peroxide

value (PV), anisidine value (AV), colour and deterioration of bleachability index (DOBI). Meanwhile, the recommendation for storage and transport give information regarding different type of tanks and their heating installation, type of pipelines and operational temperatures for loading, unloading and storage. This information is in great details since CPO is in semi solid at ambient temperature and would require certain heating conditions to prevent the blockage of the tanks and pipelines. Good refined palm oil is only coming from the good quality CPO thus adherence to the good handling practices of the oil is essential.

1.2 Fibre Optics

1.2.1 Electromagnetic Wave Propagation

Optical application is one of the applications that widely used in scientific areas due to its high reliability. Many analytical methods involving optical application are reported in literature which employ ultraviolet (UV), visible light (VIS) and near-infrared (NIR) sections in the electromagnetic spectrum. Electromagnetic radiation comprises electric and magnetic fields which are perpendicular to each other as shown in Figure 1.3. The electromagnetic radiation is classified and arranged based on its range of wavelengths and frequencies as shown in Figure 1.4. Light is a part of the electromagnetic spectrum that makes up from radio wave, microwave, infrared, visible light, ultraviolet and others. The visible light and ultraviolet will be the spectrum of interest. Light can exhibit as a wave and particle at the same time. However, light behaves live a wave at most of the times. Light waves are one of the electromagnetic waves because they consists both electric and magnetic fields. Electromagnetic fields oscillate perpendicular to the direction of wave travel, and perpendicular to each other. Light waves are known as transverse waves as they oscillate in the direction transverse to the direction of wave travel.

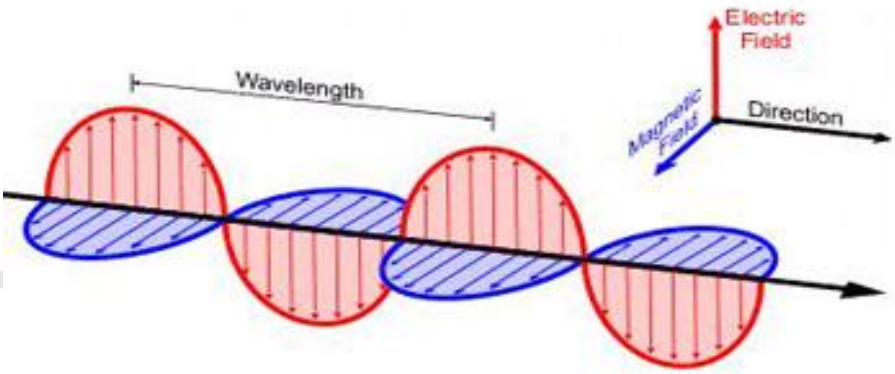

Figure 1.3: Propagation of electric and magnetic field in an electromagnetic wave
(Source: Phatak, 2014)

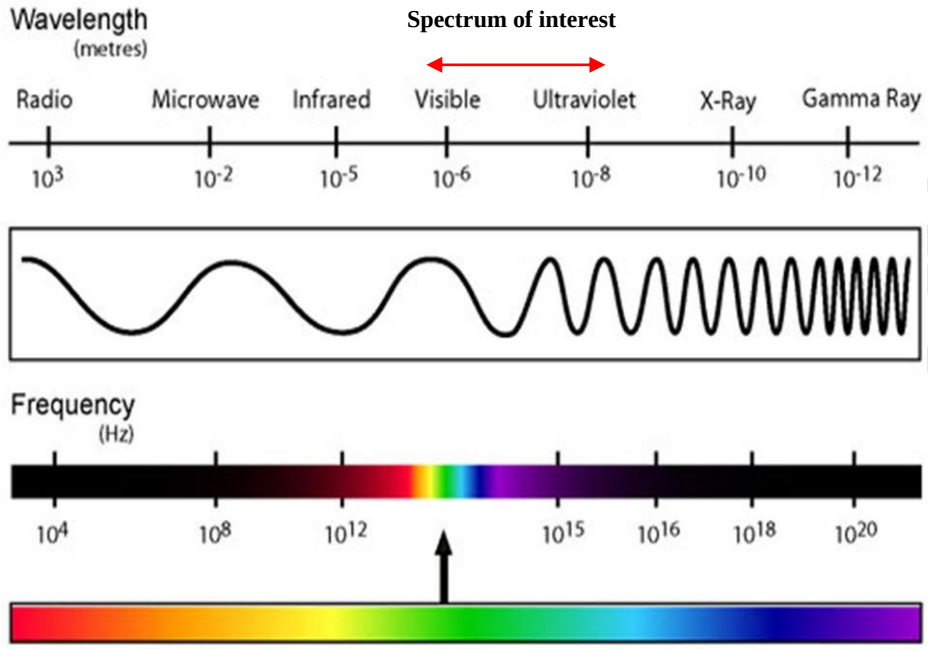

Figure 1.4: Electromagnetic spectrum (Source: Shapley, 2012)

1.2.2 Optical Fibre Sensor

There are many types of sensors that operate based on fibre optics technique for example pH, temperature, viscosity, humidity, fluorescence and gas sensor. Fibre optics uses behavior and properties of light as its parameters such as light intensity, wavelength and phase or polarization of light. Fibre optics sensors are widely explored for the past few decades due to its benefits and advantages which are (Grattan and Sun, 2000; Culshaw, 2004):

- Absolute measurement
- Immunity to electromagnetic interference
- Excellent resolution and range
- Passive operation, intrinsically safe
- Water and corrosion resistant
- Rugged, small size and light weight
- Multiplexed in parallel or in series
- Modest cost per channel
- They are easy to implement in any structure due to their small size and cylindrical geometry
- Inability to conduction of current
- Robust to environment
- High sensitivity
- Remote sensing capability

1.3 Problem Statement

Crude palm oil (CPO) is in semi-solid form when in room temperature and has to be heated several times before it reaches the refinery to facilitate transportation and its removal from one tank to another. The recommended temperature for heating is not exceeding 55°C which is above its melting point (about 37°C). Nevertheless, in practice, it is often violated to shorten the heating time (Haryati et al., 1997). On the other hand, in some countries, unrefined CPO is domestically used in their cooking and it is heated before or during the cooking. The purpose of heating before cooking is to bleach the oil and carried out by heating the oil at high temperature for short time or at low temperature for long time. Commercially, it is reported that the oil is always been heated at high temperature for long time and often recycled to fry foods (Dongmo et al., 2014).

Repeated or over heating could change the major and minor substances in CPO and hence deteriorate the oil. The deterioration usually occurs via three main processes which are oxidation, hydrolysis and contamination. The oxidation and hydrolysis are rapidly progressed if the oils are in high temperatures. Deep frying usually operates at a temperature range of 150 to 190 °C and the oil will be used for several cycles of frying. Increment in length of frying time could accelerate the deterioration (Mba et al., 2015). This will not only reduce the qualitative and nutritional value of the oil but also formation of hydroperoxides which are dangerous for consumption (Dongmo et al., 2014; Eden, 2002). Therefore, the temperature needs to be cautiously controlled in order to avoid the oils deterioration and also solidification especially during long distance storage and transport.

Simple indication of the oil quality is its colour changes. Normally the colour of repeated heating oil turns darker when compared to the unrepeated heating oil. Therefore, a fibre optic method is proposed to investigate the heating times of CPO via three parameters which are reflection, transmission and absorption on UV-VIS spectrum. The chemical composition and colour changes will affect the parameters and hence could be correlated to the heating times. The method is cheap, fast, portable, easy to operate and accurate when compared to other oil quality assessments such as titration method.

1.4 Objectives

1. To determine the effects of temperatures and heating times on reflection, transmission andabsorption properties at ultraviolet-visible spectrum of CPO samples.
2. To establish calibration equation to predict temperatures and heating times in the samples and identify the appropriate wavelength for the prediction.

1.5 Thesis Outline

The study aims to analyse the reflection, transmission and absorption properties in determining the heating times of CPO. Chapter 2 gives details information in terms of production and composition of CPO and also discusses the effect of CPO repeated

heating. In addition to this, quality assessment of CPO and methods used in the assessment also elaborate particularly on optical methods.

Chapter 3 elaborated on the theoretical background of using fibre optics technique. Theory of reflection, transmission and absorption reacting on materials is discussed and the calculations of these parameters were calculated from the raw data of intensity.

Chapter 4 presented the methodology of this research. Sample preparation and measurement setup of reflection, transmission and absorption were elaborated. Technique on setting proper calibration is also discussed.

Chapter 5 covers the results of reflection measurements, followed by absorption and transmission measurement of CPO with different heating times and temperatures at wavelengths 300 to 895 nm. The results were analysed and discussed in details. The finding of appropriate wavelength to predict the heating times of CPO was also discussed.

Finally, Chapter 6 described the conclusion, main contribution and suggestion for future study.

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