



UNIVERSITI PUTRA MALAYSIA

***MULTIFREQUENCY DIELECTRIC SENSING OF MOISTURE AND
SLUDGE CONTAMINATION IN CRUDE PALM OIL***

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**MULTIFREQUENCY DIELECTRIC SENSING OF MOISTURE AND
SLUDGE CONTAMINATION IN CRUDE PALM OIL**

By

KHAIRUNNISA HAMDAN

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia,
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JULY 2012

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Deterioration of Crude Palm Oil (CPO) quality caused by contamination during and after milling stages is a serious problem in palm oil processing. These include contamination pick-up during milling such as sterilizer condensate introduced during sterilizing stage and badly oxidized sludge oil during purification stage. Meanwhile, contamination after milling stages occurred due to illegal activities such as siphoning off CPO while being transported from mill to refineries. The amount that was siphoned off was then replaced by liquid such as water, sludge, diesel or used oil which resulted in deterioration of the CPO being processed at the refineries.

The conventional testing methods to measure CPO quality mostly depending on the determination of parameters such as iodine value (IV), peroxide value (PV), Deterioration of Bleacheability Index (DOBI), free fatty acid (FFA) and moisture content. The procedures to obtain these parameters are laborious, time consuming

and require skilled operators. In-situ monitoring and early detection of CPO contamination could lessen oil degradation, thus enhance the value of the final palm oil products. Therefore, an efficient technique for CPO quality monitoring is needed to enhance the CPO quality and production process.

This study used dielectric spectroscopy technique to detect possible contamination in homogeneous mixture of CPO. Initially, dielectric properties of pure CPO were determined. After that, the dielectric properties of CPO which were artificially contaminated with water and sludge were measured at different temperatures and contamination levels. For water contamination test, dielectric properties of CPO was measured using a 16452A Agilent liquid dielectric test fixture which was connected to a 4263B Agilent LCR meter over six frequencies, ranged from 100 Hz to 100 kHz. For sludge contamination test, the liquid dielectric test fixture was connected to a 4294A Agilent precision impedance analyzer ranging from 3 MHz to 30 MHz. Both tests were replicated three times with a randomized order of temperature and contaminant levels.

The variations of dielectric properties of pure and homogeneous mixture of contaminated CPO at different temperature levels were observed and analyzed using ANOVA and Duncan's multiple range test (DMRT). The principal component regression (PCR) and partial least squares (PLS) analysis were used to develop model for contamination prediction. The results showed that, there was generally

significant different in the value of the mean measured dielectric constant as the temperature increased from 28°C to 55°C ($p < 0.0001$). This study also found that when water was introduced into the CPO, the value of dielectric constant (measured at frequency ranged from 100Hz to 100 kHz) increased from 3.01 to 4.73 with increasing contamination levels. When sludge was introduced into the CPO, the value of dielectric constant (measured at frequency ranged from 3MHz to 30MHz) increased from 3.01 to 63.53 with increasing contamination levels. Generally for both test, there were significant differences between the dielectric constant of pure and contaminated CPO ($p < 0.0001$).

The PCR and PLS calibration models showed a good prediction capability for different temperature with sludge and water contamination levels. The classification of water contamination yielded very substantial correlation with r^2 value ranged from 0.96 to 0.99. The best result was obtained at 55°C with the lowest value of SECV of 0.58%. The classification of sludge contamination yielded very substantial correlation with r^2 value ranged from 0.91 to 0.98. The best result was obtained at 28°C with the lowest value of SECV of 1.04%.

The result from this study could provide the foundation for studies on probing the physiochemical properties of CPO for in-situ monitoring of CPO quality. This study found that the frequency range substantial for contamination detection is generally low (<12 MHz). This is very attractive for industrial application because

instrumentation required for such measurements is relatively inexpensive and does not have signal integrity issues associated with high frequency instrumentation.



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**PENGESAN DIELEKTRIK PELBAGAI FREKUENSI UNTUK
KELEMBAPAN DAN ENAPCEMAR DALAM MINYAK SAWIT MENTAH**

Oleh

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Kemerosotan kualiti minyak sawit mentah (CPO) yang disebabkan oleh pencemaran semasa dan selepas peringkat pengilangan adalah satu masalah serius dalam pemprosesan minyak sawit. Ini termasuk pencemaran yang terjadi semasa pengilangan seperti kondensasi pensteril di peringkat pensterilan dan enapcemar yang teroksida teruk semasa peringkat penulenan. Sementara itu, pencemaran selepas peringkat pengilangan berlaku disebabkan oleh aktiviti yang menyalahi undang-undang seperti penyaluran keluar CPO ketika sedang diangkut dari kilang ke kilang penapis. Jumlah yang telah disalurkan kemudian digantikan dengan cecair seperti air, enapcemar, diesel atau minyak yang telah digunakan mengakibatkan kemerosotan kualiti CPO yang akan diproses di kilang penapisan.

Kaedah konvensional untuk mengukur kualiti CPO kebanyakannya bergantung kepada penentuan parameter seperti nilai iodin (IV), nilai peroksida (PV),

kemerosotan indeks pelunturan (DOBI), asid lemak bebas (FFA) dan kandungan kelembapan. Prosedur bagi mendapatkan parameter ini agak merumitkan, memakan masa dan memerlukan operator yang mahir. Pemantauan terus dan pengesanan awal pencemaran CPO boleh mengurangkan degradasi minyak, dengan itu meningkatkan nilai akhir produk minyak sawit. Oleh itu, teknik yang cekap untuk pemantauan kualiti CPO diperlukan untuk meningkatkan kualiti dan proses penghasilan CPO.

Kajian ini menggunakan teknik spektroskopi dielektrik untuk mengesan pencemaran yang mungkin terdapat dalam campuran homogen CPO. Pada mulanya, sifat dielektrik CPO tulen ditentukan. Selepas itu, sifat-sifat dielektrik CPO yang tercemar dengan air dan enapcemar secara buatan diukur pada suhu dan tahap pencemaran yang berbeza. Untuk ujian pencemaran air, sifat dielektrik CPO telah diukur menggunakan alat pengujian dielektrik bendalir Agilent 16452A yang disambungkan kepada alat pengukur LCR Agilent 4263B terhadap enam frekuensi, berjulat dari 100 Hz hingga 100kHz. Untuk ujian pencemaran enapcemar, alat pengujian dielektrik bendalir tersebut disambungkan kepada alat penganalisa impedance Agilent 4294A yang berjulat antara 3 MHz hingga 30 MHz. Kedua-dua ujian telah diulang tiga kali dengan susunan suhu dan tahap pencemaran secara rawak.

Variasi sifat dielektrik CPO tulen dan campuran homogen tercemar di peringkat suhu yang berbeza telah dikaji dan dianalisis menggunakan ANOVA dan DMRT. Regresi komponen utama (PCR) dan analisis perbahagian persegi terkurang (PLS)

telah digunakan untuk membangunkan model ramalan pencemaran. Hasil kajian menunjukkan bahawa, secara umumnya terdapat perbezaan yang signifikan dalam nilai purata pemalar dielektrik apabila suhu meningkat daripada 28°C hingga 55°C ($p < 0.0001$). Kajian ini juga mendapati bahawa apabila air telah dimasukkan ke dalam CPO, nilai dielektrik malar (diukur pada frekuensi antara 100Hz hingga 100 kHz) meningkat dari 3.01 kepada 4.73 dengan pertambahan tahap pencemaran. Apabila enapcemar dimasukkan ke dalam CPO, nilai dielektrik malar (diukur pada frekuensi antara 3MHz hingga 30MHz) meningkat dari 3.01 kepada 63.53 apabila tahap pencemaran semakin meningkat. Amnya bagi kedua-dua ujian, terdapat perbezaan yang signifikan antara pemalar dielektrik CPO tulen dengan CPO tercemar ($p < 0.0001$).

Model PCR dan penentukuran PLS menunjukkan keupayaan ramalan yang baik untuk suhu yang berbeza dengan tahap pencemaran enapcemar dan air. Klasifikasi pencemaran air menghasilkan korelasi yang sangat signifikan dengan nilai r^2 yang berjulat dari 0.96 hingga 0.99. Hasil yang terbaik telah diperolehi pada suhu 55°C dengan nilai SECV yang paling rendah iaitu sebanyak 0.58%. Klasifikasi pencemaran enapcemar menghasilkan korelasi yang sangat signifikan dengan nilai r^2 yang berjulat dari 0.91 hingga 0.98. Hasil yang terbaik telah diperolehi pada 28°C dengan nilai SECV yang paling rendah iaitu sebanyak 1.04%.

Hasil daripada kajian ini menjadi asas bagi kajian ke atas sifat physiochemical CPO

untuk pemantauan terus CPO yang berkualiti. Kajian ini mendapati bahawa julat frekuensi yang sesuai untuk mengesan pencemaran amnya adalah rendah (<12 MHz). Ini amat menarik untuk kegunaan industri kerana peralatan yang diperlukan untuk pengukuran tersebut adalah murah dan tidak mempunyai isu-isu integriti isyarat yang berkaitan dengan instrumentasi berfrekuensi tinggi.



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I certify that a Thesis Examination Committee has met on 10 July 2012 to conduct the final examination of Khairunnisa Hamdan on her thesis entitled “Multifrequency Dielectric Sensing Of Moisture And Sludge Contamination In Crude Palm Oil” in accordance 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 Degree of Master Science.

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DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.



KHAIRUNNISA HAMDAN

Date: 10 July 2012

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LIST OF ABBREVIATIONS

NKEA	National Key Economic Areas
GNI	Gross National Income
PORIM	Palm Oil Research Institute Malaysia
FFA	Free Fatty Acid
DOBI	Deterioration of Bleacheability Index
FAS	Flame Atomic Absorption Spectroscopy
AAS	Atomic Absorption Spectroscopy
PV	Peroxide Value
FTIR	Fourier Transform Infrared
NIR	Near infrared
IR	Infrared
ANOVA	Analysis of variance
DMRT	Duncan's multiple range test
RBD	Refined, bleached and deodorized

LIST OF NOMENCLATURES

ϵ' dielectric constant

ϵ'' dielectric loss

ω angular frequency

α correction coefficient

C_p parallel capacitance

C_o air capacitance

f frequency

R_p parallel resistance

CHAPTER 1

INTRODUCTION

1.1 Background of Study

The oil palm industry plays an important role in contributing to Malaysia's economic growth. In 2010, exports earnings from oil palm products reached RM 59.77 billion with an increase of 20.4% from RM49.66 billion in 2009 (Choo, 2011). Under the National Key Economic Areas (NKEA), palm oil industry is targeted to raise a total Gross National Income (GNI) contribution of RM125 billion to reach RM128 billion by 2020 (Anon, 2010). In order to achieve this target, only the highest quality of Crude Palm Oil (CPO) that has passed through stringent quality procedures is to be produced. This strict standard operating procedure would prevent any possible contamination introduced during or after milling stages.

The conventional testing methods to measure CPO quality mostly depending on determination of parameters such as iodine value (IV), peroxide value (PV), Deterioration of Bleacheability Index (DOBI), free fatty acid (FFA) and moisture content. The procedures to obtain these parameters are laborious, time consuming and require skilled operators. In order to solve this problem, (Moh *et al.*, 1999) have developed a near infrared (NIR) spectroscopy method to measure PV in CPO. Besides, Man and Moh (1998) had also developed this technique in the determination of FFA in CPO. Results from both studies indicated that NIR spectroscopy can reduced the time taken for sample analysis when compared to the

conventional wet chemical analysis. Besides, the amount of hazardous solvents can be reduced as well as the cost of labor. More recently, Fourier Transform Infrared (FTIR) spectroscopy has been introduced in the determination of FFA, moisture, peroxide and IV of CPO as well as in the analysis of the extra virgin olive oil adulterated with palm oil (Che Man *et al.* 1999a; Che Man and Mirghani, 2000; Moh *et al.*, 1999; Rohman and Man, 2010). Those studies showed very promising results in rapid determination of oil quality. Hence, the development of rapid and non-destructive measuring techniques for CPO quality monitoring has potential to enhance the efficiency of palm oil quality monitoring.

In this study, dielectric spectroscopy technique was introduced as an effort to improve the palm oil quality sensing system in Malaysia. Dielectric spectroscopy, also known as impedance spectroscopy, has been used for process analysis for some time, as it offers the ability to measure bulk physical properties of materials. The advantage of dielectric spectroscopy techniques over existing methods of monitoring materials quality is it offers flexibility in term of design where a custom-bulit system (which lower the cost) can be developed once analytical model of sensory attributes is identified. The penetration depth of dielectric spectroscopy can be adjusted by changing the separation between the sensor electrodes, enabling measurement through other materials to reach the substrate of interest. Because it measures the dielectric properties of materials, it can provide information not attainable from other spectroscopy (Bakeev, 2010).

In agricultural studies, dielectric spectroscopy is one of the established techniques which has been well recognized and developed for automatic monitoring of various agricultural products and food materials such as fruits (Nelson and Trabelsi, 2008; Nelson, 2004; Nelson *et al.*, 2005; Nelson *et al.*, 2007; Canchun and Zichen, 2008) vegetables, meat (Nelson, 2009; Nelson and Trabelsi, 2008; Nelson *et al.*, 2007; Bodakian and Hart, 2002) and cereal products (Nelson and Trabelsi, 2008). It has been used for process monitoring as a substitute method to existing technologies that may not detect all the additives or contaminants in formulating food products (Bakeev, 2010).

Since dielectric spectroscopy is a rapid, non destructive and less expensive method, the dielectric properties of agricultural products become area of interest for several reasons (Nelson, 1991). These include the sensing of moisture content through its correlation with the dielectric properties of cereal grain and oilseed crops (Nelson *et al.*, 2000), the influence of permittivity on the dielectric heating of products at microwave or lower radio frequencies (Nelson, 1996), and the potential use of permittivities for sensing quality factors other than moisture content (Nelson *et al.*, 1995).

1.2 Problem of Statements

There are several factors that cause CPO contamination as well as deteriorate its fully processed product quality. Gee (2005) reported that the deterioration of CPO in a palm oil mill in Johor, Malaysia was mostly caused by conditions and contamination pick-up during milling. These include contamination with sterilizer condensate introduced during sterilizing stage, contamination with badly oxidized sludge oil during purification stage, and overheating of CPO in storage tank after the extraction process.

Contamination of CPO could also occur with the presence of heavy metal like copper and iron. According to Chooi (1981), these heavy metals promote deterioration by accelerating the process of oxidation of CPO which is shown by the change of color, taste, and flavour. A survey studied by Palm Oil Research Institute Malaysia (PORIM) on the quality of CPO produces by the mills in Malaysia indicated that about 25% of the surveyed mills produced consistently high copper content with average of 0.15 ppm. The copper content level was significantly higher than the maximum level of 0.08 ppm in CPO recommended by the refineries (Rohaya et al., 2003).

Perumal (2009) reported that the CPO contamination also occurred after the milling stages due to the siphoning off CPO while being transported from mill to refineries. The amount that was siphoned off by syndicates was then replaced by liquid such as

water, sludge or used oil which result in contamination of the transported CPO being processed at refineries. A total of 39 cases of siphoning activity were reported in 2010 which involved 252.73 tonne of CPO that worth RM 674000 (Dompok, 2011). This illegal activity causes the industry losses million of ringgit annually and damage Malaysia's image as one of the largest producers of palm oil in the world as well. CPO contamination also occurred in Indonesia. In 1999, Deli Tama Indonesia storage tanks was asked to be cleaned because of diesel-oil contamination of 19 000 metric tons of CPO exported to Rotterdam Port, Netherlands which caused Netherlands to suspend some import contract with them as well as tarnished the image of Indonesian importers in Netherlands (Anon, 1999).

1.3 Objectives

The overall goal of this study is to develop a sensing system for detecting possible contamination in CPO. In order to accomplish this goal, the following specific objectives were set:

- i. to investigate the variation of dielectric properties of homogeneous mixture of CPO across electrical spectrum,
- ii. to investigate the dielectric properties of homogeneous mixture of CPO contaminated with water and sludge contamination at different temperature levels, and
- iii. to develop analytical model for predicting levels of water and sludge contamination in homogeneous mixture of CPO.

1.3 Thesis Organization

This thesis describes a study of application of dielectric spectroscopy concept in detecting possible contamination in palm oil. The research perform in this thesis is hoped will give a fundamental input for further development of sensing system in palm oil processing industry. A review of previous studies regarding the uses of dielectric spectroscopy in monitoring food quality is discussed in Chapter 2. The concept of dielectric spectroscopy is first explained. Conventional and new techniques other than dielectric spectroscopy to determine the oil palm quality are described. Factors influencing dielectric properties of food material such as frequency, temperature, composition and storage time are reviewed. A brief introduction of statistical analysis techniques used in this study for contamination prediction in palm oil were also discussed.

Chapter 3 provides the material, setup and experimental procedures in performing this research. Sample preparations according to the type of contamination as well as its contamination levels are described. The instrumentation setup for dielectric measurements is described and illustrated by photos. The statistical analyses used are explained in the last section.

Chapter 4 described the dielectric properties of pure and contaminated CPO obtained from the experiment. The effects of temperature and contamination levels on dielectric properties of CPO are also described. The classification of the CPO

according to temperature and types of contamination (sludge and water) using PCA and PLS are explained.

Finally, Chapter 5 which is the final chapter in this thesis outlines the findings of this research and ends with some suggestion and recommendation for future work in order to improve the results obtained in this study.



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