UNIVERSITI PUTRA MALAYSIA

INTERDIGITATED ELECTRODE (IDE) SENSOR FOR FRYING OIL DEGRADATION ASSESSMENT DURING HEATING

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FK 2015 2

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By

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

June 2015

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirements for the Degree of Master of Science

INTERDIGITATED ELECTRODE (IDE) SENSOR FOR FRYING OIL DEGRADATION ASSESSMENT DURING HEATING

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June 2015

Chairman : Samsuzana Abd Aziz, PhD
Faculty : Engineering

The repeated usage of frying oil had been proven hazardous to human health due to degradation process by thermal reactions that contribute to the formation of polar compounds in the oil, which in turn changes the quality of the oil. The oxidized products and lipid polymers formed in repeatedly-used frying oil would also pose health risks.

Many technical approaches are being used to evaluate the degradation of frying oil. These processing include column chromatography to determine the free fatty acids (FFAs), image analyses to measure the total polar compounds (TPC), and Fourier transform infrared spectroscopy (FTIR) to measure the peroxide value (PV). However, these methods are commonly time consuming, laborious, need substantial amounts of solvents, and require skilled operators. Consequently the development of a simple and fast method is essential to assess the degradation of frying oil.

In this study, interdigitated electrodes (IDE) sensor was designed to appraise the frying oil degradation at several heating time intervals by measuring the changes on its electrical characteristics. The probe was designed using IDE structure and the photolithography was performed to fabricate pattern for metal and insulation layers of the sensor.

Frying oil samples a set of 30 each containing 130 ml of palm oil were heated at 180^{o} C up to 30 hours. For each one hour increment, one sample was taken out of the oven and cooled at room temperature before further analyses. Then the impedance and capacitance measurement were carried out using the IDE sensor connected to a inductance (L), capacitance (C), and resistance (R) meter (4263B, Agilent Technologies, Japan) using Kelvin clip leads (16089E, Agilent, Japan) over five frequencies (100 Hz, 1000 Hz, 10 KHz, 20 KHz, and 100 KHz). The percentage of TPC of each heated sample was measured using a frying oil tester (Testo 270, InstruMartlnc, Germany) whereas the viscosity was tested utilizing a viscometer (Sv-10 Vibro Viscometer, A&D Company

Limited, Japan). The experiments were repeated three times and the average of them were taken.

The results were analyzed to find the correlation between electrical properties of oil (impedance and capacitance)with oil degradation parameters (TPC and viscosity). The results showed that a significant correlation was found between oil impedance and capacitance with TPC and viscosity. For example, at 100 Hz the correlation between impedance and TPC has coefficient of determination (R^2) of 0.92 while the correlation between impedance and viscosity has R^2 of 0.85. When the regression equations used to predict TPC and viscosity with impedance measurements were validated using the validation data set, the lowest RMSE of 4.03% and 3.48% respectively was found while the highest RMSE was 4.61%. In addition, the principle component analysis (PCA) model to predict TPC and viscosity using impedance measurements yielded a significant performance with $R^2$of 0.88 and 0.86, respectively.

Similarly, the capacitance measurements have significant correlation with TPC and viscosity. For example, at 100 KHz, which is the best correlation given, the correlation between capacitance and TPC has coefficient (R^2) of 0.90 while the correlation between capacitance and viscosity has R^2 of 0.87. When the regression equations used to predict TPC and viscosity with capacitance measurements were validated using the validation data set, the lowest RMSE of 3.88% and 3.64% respectively was found, where the highest RMSE was 5.04%. In addition, the PCA model to predict TPC and viscosity using capacitance measurements yielded a significant performance with R^2 of 0.86 and 0.84, respectively.

This study found that the designed IDE sensor has good potential for a simple and inexpensive way of monitoring frying oil degradation. The result from this study could provide the foundation for building a portable sensor to be used in assessing the degradation properties of frying oil.

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

SENSOR ELEKTROD SALING DIGIT (IDE) UNTUK PENILAIAN DEGRADASI MINYAK MENGGORENG SEMASA PEMANASAN

Oleh

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Penggunaan semula minyak menggoreng telah terbukti sangat berbahaya kepada kesihatan pengguna akibat proses degradasi oleh tindak balas haba yang menyumbang pada pembentukan kompaun kutub di dalam minyak tersebut dan menyebabkan perubahan tahap kualiti minyak. Bahan yang teroksida dan pembentukan folimer lipid dalam minyak menggoreng yang telah diguna berulang kali, akan menyebabkan risiko terhadap kesihatan pengguna.

Banyak pendekatan teknikal telah digunakan untuk menilai kualiti degradasi minyak yang telah digoreng seperti kromatografi ruangan dalam penentuan asid bebas lemak (FFA), analisis imej untuk mengukur jumlah kompaun kutub (TPC) dan Fourier Transform Infrared (FTI) untuk mengukur nilai peroksida. Walaubagaimanapun, teknik ini memerlukan masa yang lama, rumit, memerlukan nilai pelarut yang banyak dan juga memerlukan tenaga pakar dalam operasi tersebut.Oleh itu, teknik yang mudah dan cepat diperlukan untuk mengesan degradasi minyak menggoreng.

Di dalam kajian ini, sensor elektrod saling digit (IDE) telah direka untuk menentukan kualiti minyak menggoreng pada beberapa jangka masa pemanasan tertentu dengan mengukur perubahan sifat elektrik minyak tersebut. Alat ini telah direka dengan menggunakan platfom IDE dan teknik fotolitografi untuk memfabrikasi corak pada bahagian metal dan penebat sensor tersebut.

Satu set yang mempunyai 30 sampel minyak menggoreng, di mana setiap sampel mengandungi 130ml minyak kelapa sawit yang telah dipanaskan pada suhu 180°C selama 30 jam. Pada setiap satu jam, satu sampel akan dikeluaran daripada ketuhar dan akan disejukkan pada suhu bilik sebelum analisis seterusnya. Pengukuran impedansdan kapasitan dijalankan dengan menggunakan sensor IDE yang disambung pada mesin meter LCR (4263B, Agilent Technologies, Japan) dan menggunakan klip Kelvin (16089E, Agilent,

Japan) pada lima frekuensi di antara 100Hz sehingga 100 KHz. Peratusan nilai TPC pada setiap sampel diukur dengan menggunakan penguji minyak menggoreng (Testo 270) manakala tahap kelikatan sampel tersebut diukur dengan menggunakan meter kelikatan (Sv-10 Vibro Viscometer, A&D Company Limited, Japan). Eksperimen telah diulang sebanyak tiga kali.

Keputusan telah dianalisis untuk melihat hubungan di antara nilai impedansdan kapasitan dengan parameter degradasi minyak (TPC dan kelikatan).Keputusan analisis menunjukkan kekuatan kolerasi di antara impedansdan kapasitan dengan TPC dan kelikatan.Sebagai contoh, pada 100 Hz kolerasi antara impedansdan TPC mempunyai pekali kolerasi (R^2) 0.92 manakala kolerasi antara impedansdan kelikatan mempunyai pekali kolerasi (R^2) 0.85. Apabila persamaan regresi digunakan untk menentukan nilai TPC dan kelikatan dengan menggunakan pengukuran impedans disahkan menggunakan set data validasi, RMSE terendah diperolehi adalah 4.03% dan 3.48% masing-masing. Selain itu, model PCA bagi menentukan TPC dan kelikatan menggunakan pengukuran impedansmenghasilkan prestasi yang signifikan dengan R^2 bersamaan 0.88 dan 0.86 masing-masing.

Pengukuran kapasitan juga memberikan kolerasi yang signifikan dengan TPC dan kelikatan. Sebagai contoh, pada 100 KHz, kolerasi antara kapasitan dan TPC mempunyai pekali (R^2) bersamaan 0.90 manakala kolerasi antara kapasitan dan kelikatan mempunyai R^2 bersamaan 0.87. Apabila persamaan regrasi digunakan untuk menentukan TPC dan kelikatan dengan pengukuran kapasitan, telah disahkan menggunakan data set validasi, nilai RMSE terendah yang diperolehi adalah 3.88% dan 3.64% masing-masing. Selain itu, model PCA bagi menetukan TPC dan kelikatan menggunakan pengukuran kapasitan menghasilkan prestasi signifikan dengan R^2 bersamaan 0.86 dan 0.84 masing-masing.

Keputusan kajian ini boleh dijadikan asas bagi merekacipta sensor mudah alih untuk digunakan dalam menilai ciri degradasi minyak menggoreng.Kajian ini juga menunjukkan sensor IDE mempunyai potensi yang baik sebagai satu kaedah yang mudah dan murah dalam pemantauan degradasi minyak menggoreng.

ACKNOWLEDGEMENT

First and foremost, I would like to express my greatest gratitude to my project supervisor, Dr. Samsuzana Abd Aziz for all her great supervision, supports, advices and guidance that help me a lot in completing this study. Her valuable advice is really useful for me. I also would like to convey my appreciation to my co-supervisors, Professor. Ir. Dr. Wan Ishak b. Wan Ismail and Dr. Fakhrul Zaman Rokhani for their assistance and useful opinions to improve my research and ensure everything was on the right track.

Besides, I would like to acknowledge my groupmate Dyiana, Fizura, Atikah and Munirah for their kind support, cooperation and valuable advice. Also, I would like to thank the Laboratory Technician, En. Sabri who has helped me during the sample preparation.

I would also express my profound gratitude to my wife for her help, encouragement and patience during my study. Also, my deepest thank to my lovely daughter for giving me the inspiration to do well in my study. In addition, my special thanks to my parents, siblings and relatives for their continued love, support and encouragement. They always cheer me up and make my life much easier.

I would like to thank all my friends, especially Yahya, Hussam, Hassan and Mugahed for their assistance and cooperation. They are always by my side through my ups and downs at Universiti Putra Malaysia.

Last but not the least, I would like to express my grateful appreciation to everybody, who has directly and indirectly involved in completing this research. Thank you.

I certify that a Thesis Examination Committee has met on 15 June 2015 to conduct the final examination of Alfadhl Yahya Khaled Al-khaled on his thesis entitled **"Interdigitated Electrode (IDE) Sensor For Frying Oil Degradation Assessment During Heating"** 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 Master degree.

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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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LIST OF ABBREVIATIONS

MASU	Malaysia Association of Standards Users
NITE	National Institute of Technology and Evaluation
JCSS	Japan Calibration Service System
TPC	Total Polar Compound
FFA	Free Fatty Acid
PV	Peroxide Value
IV	Iodine Value
TAG	Triacylglycerol
MAG	Monoacylglycerol
FTIR	Fourier Transform Infrared
NIR	Near infrared
IDE	Interdigitated Electrode
EIS	Electrochemical Impedance Spectroscopy
PCA	Principle Component Analysis
LCR	Inductance, Capacitance, and Resistance meter
CPO	Crude Palm Oil
RPO	Red Palm Oil
DPTG	Dimer and Polymer Triglycerides

LIST OF NOMENCLATURES

ε_o	permittivity of free space
$\varepsilon_{r,oil}$	relative dielectric constant of oil
K_{cell}	cell constant
C_{dl}	double layer capacitance
C	capacitance
f	frequency
Z	impedance
s	distance between electrodes
w	finger width
g	electrode gap
L	finger length
N	number of fingers
R_{sol}	the resistance of solution
σ_{oil}	oil conductivity
$K(k)$	the complete elliptic integral of the first kind with *k* the modulus
$C_{dl,surface}$	the characteristics of double layer capacitance
R_{lead}	the resistance lead

CHAPTER 1

INTRODUCTION

1.1 Background

Frying is the process of submerging food in oil or other types of fat at high temperature in the range of 170-205°C (Moreira *et al.*, 1999). The technique is old and thought to have originated in ancient Egypt circa 2500 BCE. Frying process has been used since antiquity and in most cultures to prepare fishes, breads, meats and vegetables (Tannahill and Reay, 1995). This popularity together with its low cost for large-scale activity, have made the frying technique the best way for preparing most meals in fast food industries, restaurants, cafeterias and canteen. A variety of snack foods were manufactured via frying since the last five decades, consequently causing a dramatic increase of the food industry. Fried foods such as potato chips and fried chicken have gained worldwide popularity (Sayon-Orea *et al.*, 2013).

Frying oil provides an effective medium for heat transfer from heat source to the food (Orthoefer and Cooper, 1996a). In addition, frying oil contributes flavor and crispy texture to foods and often makes up a significant part of the final food product. Moreover, oil differs extensively in its rate of deterioration, functionality and eating quality depending on processing, source, or formulation (Orthoefer and Cooper, 1996b). Many types of edible oils from animal and plant sources are used in frying, depending on availability (Matthäus, 2006). Palm oil is consumed likely in Southeast Asia; olive oil is used commonly in the Mediterranean region while coconut oil is common in the Indian subcontinent (Hu, 2003; Chandrashekar *et al.*, 2010). There are also many other types of oils used for frying in food preparations worldwide such as corn oil, soybean oil, and sunflower oil.

The process of deep-fat frying is primarily a dehydration operation, which means that it is the extraction of water and water-soluble substances from the food being fried and transfer them to the frying oil. At the same time, the food being fried absorbs surrounding fat. As food is placed in hot oil for frying, water from inside the food moves to the outer layer to compensate for evaporation happening on the surface. Therefore, a thin layer of steam is formed between the oil and the food due to difficulty of water moving from the hydrophilic surface of the product to the hydrophobic frying oil (Chen and Moreira, 1997).

Frying oil has different physical and chemical properties. One of the important physical properties is density. Commonly, density of frying oil (approximately 0.8 g/cm^3) is less than that of water; for this reason, the frying oil floats on top of water (Timms, 1985). It also has relatively low melting point and cannot dissolve in water, however, it is soluble in organic solvents such as ether and gasoline. Some chemical important properties of frying oil include hydrolysis, oxidation, and polymerization (Choe and Min, 2006).

During frying, some changes occur in the oil. When the deep-fryer is heated at temperatures between 175 and 205^{o}C, the oil used is subjected to thermal oxidation, hydrolysis and polymerization. Thermal oxidation is the reaction in which change occurs in the number of atoms of oxidation reactants of the oil molecules due to transmission of electrons with each other (Lee *et al.*, 2010).In oxidation, the molecules of triglyceride undergo initial changes into unstable lipids known as “hydroperoxides”, which consequently split to form secondary oxidation products that include volatile and non-volatile compounds such as secondary oxidation products, hydrocarbons, ketones, aldehydes and alcohols (Dana and Saguy, 2001; Velasco*et al*., 2010).

Hydrolysis involves vaporized a bit of moisture from the food being fried while triglycerides (TGs) are hydrolyzed in the frying oil. The hydrolysis reaction produces free fatty acids (FFA), glycerol, diglycerides (DGs)and monoglycerides (MGs) that are called carboxylic acids resulting from fats and oil. The hydrolysis reactant is promoted by bases and acids (Choe and Min, 2007; Pokorny, 1998).

Polymerization is a chemical process which in which small molecules are bound to each other, i.e., the small molecules called monomers are combined together to form repeating units which they are linked to form long molecules known as polymers. In chemical compounds, polymerization occurs via a variety of reaction mechanisms that differ in complexity because of the functional groups present in the reacting compounds and their inherent effects (Wang *et al.,* 2007; Henry and Chapman, 2002).

During frying, several substances are generated due to the degradation of frying oil (Gertz, 2000). This degradation as discussed before, is because of the chemical reactions occurring during frying such as thermal oxidation, hydrolysis and polymerization. And the resulting decomposition yields adverse effects on flavor and color. The increase in polarity, change in the concentration of FFA, variation of PV or viscosity can all be used as indicators for oil deterioration. Consequently, these different criteria are used to assess when the frying oil needs to be discarded (Kress *et al.,* 1990; Susheelamma *et al.,* 2002; Farhoosh and Tavassoli, 2010).

Many technical approaches are being used to evaluate the degradation of frying oil such as, column chromatography to measure the FFA, image analyses to measure the TPC, and Fourier transform infrared (FTIR) to measure the PV (Cert *et al.,* 2000; Gil *et al.,* 2004;Vlachos *et al.,* 2006). However, these methods are commonly time consuming, laborious, need substantial amounts of solvents, and require skilled operators to operate. Consequently the development of a simple and fast method is essential to assess the degradation of frying oil.

More recently, there are many instruments and kits that can be used to determine oil degradation. For example, viscosity meters and electronic-based physical tests such as Vibro Viscometer (Sv-10 Vibro Viscometer, A&D Company Limited, Japan) can be used to observe frying oil at all stages of it's use and determine its viscosity. In addition, instruments such as Testo 270 (InstruMartlnc, Germany), CapSens5000 (C-Cit Ag, Switzerland), and Ebro FOM 310 (ebro®Electronic GmbH, Germany) were developed by companies to measure the quality of frying oil by testing the total polar materials (TPM) based on changes in the dielectric constant of the oil. However, these devices are expensive because they were manufactured by international companies mainly for different or certain type of oil. As Malaysia is the largest producer of palm oil it is important to develop the technology locally. Other than that, there are yet work to be done to overcome the limitation of current devices such as complex calibration requirement, suitability for different type of oil as well as distinct temperature dependencies.

1.2 Problem Statement

According to the Malaysian Association of Standards Users (MASU), recent lab tests on 19 brands of recycled frying oil that is sold nationwide revealed that repeated exposure to high temperatures had made it unfit for human consumption. Consequently, the Malaysian Health Ministry has reassured Malaysians that it will not condone the use or sale of recycled frying oil for human consumption in the country (The Star newspaper, Tuesday 13 December 2011).

When oil is used continuously in frying process, it leads to degradation which also means the decrease of its quality. This is because during frying, oil is subjected to polymerization, thermal oxidation, and hydrolysis, therefore, some products of the decomposition process negatively affect its color and flavor. Furthermore, repeated use of frying oil produces undesirable constituents which might impose health hazards and cause health problems. Reused oil contains decomposed triglycerides due to the mechanism of hydrolysis and other organic compounds produced by oxidation that are harmful to human health. As a result, repeated monitoring on reused oil is very important to ensure that it is still safe for consumption.

The current frying oil quality assessment in Malaysia requires intensive laboratory work, which is time consuming and very costly for rapid applications. For example, the determination of TPC values involves taking a sample and letting it to cool down before being introduced to a bench top instrument for analysis. However, determining more sophisticated assays such as polymeric triglycerides, fatty acids, carbonyl value, IV, anisidine value and PV requires specialized skill; otherwise the results are not conclusive. For instance, the determination of the content of polymerized and oxidized material (POM) requires two hours of an operator's time.

Currently, there are some devises are using to evaluate the degradation of frying oil such as CapSens5000, Ebro FOM 310, and TPM Veri-Fry. However, there are some limitations with these devices such as complex calibration requirement, suitability for different type of oil as well as distinct temperature dependencies.

Principal component analysis (PCA) is a method of finding patterns to identify the similarities and differences for high dimensional data. In other words, PCA is a mathematical procedure that reduces the dimensionality of the data while retaining most of the variability in the data set, by transforming a large number of related variables into smaller number of variables namely, principal components. Therefore, the need of PCA in this study was to provide one model for electrical properties of frying oil at wide range of frequency.

1.3 Objectives

The overall aim of this study was to develop and evaluate a custom-built sensor to assess the degradation of frying oil. In order to achieve this aim, the following specific objectives were set:

i. To develop an interdigitated electrode (IDE) sensor to assess and monitor frying oil degradation during heating,

ii. To correlate the electrical properties of oil with quality parameters (TPC and viscosity) obtained from standard laboratory analysis, and

iii. To develop an analytical model for predicting TPC and viscosity of palm oil using their electrical proprties measurements.

1.4 Scope and Limitations

The scope and limitations of this study were listed as below:

- The degradation of the oil was subjected only to the effect of heating experiment.
- The oil samples were heated in a laboratory oven without any food frying. Hence, the effect of food frying will not be the subject of discussion.
- The experiment was mimicking the original frying process by heating the oil samples at a temperature of 180^{o} C at different times.
- The frying oil used in this study was olein oil.
- All measurements for impedance, capacitance, TPC, and viscosity were taken at a constant temperature 40^{o} C.

1.5 Thesis Layout

The thesis is divided into five chapters, and each chapter is divided into several sub-sections. Chapter 1 is the introduction, which presents general information about the research topic; whereby problems, motivation, objectives, and scope of the work are briefly discussed. Chapter 2 is the literature review, where some previous studies have been reviewed regarding the properties of palm oil as well as different methods used to assess the quality of frying oil. It further reviews various sensors that are applied to monitor the level of oil degradation. The chapter provided the readers with an understanding into the basic knowledge and background related to this thesis. Chapter 3explainsthe methodology used to appraise the frying oil using the developed sensor. Also in this chapter, samples preparation, laboratory analysis methods and statistical methods are briefly discussed. Meanwhile, Chapter 4 presents the results obtained from the series of tests in this project. In addition, this chapter discusses and explains the principles of the findings. The thesis is concluded in Chapter 5 which summarizes the work done in the project. Recommendations for further improvement are also discussed in this chapter.

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