UNIVERSITI PUTRA MALAYSIA

DIELECTRIC AND FLUORESCENCE SPECTROSCOPY TECHNIQUES FOR DETERMINATION OF PALM OLEIN DEGRADATION DURING BATCH DEEP FAT FRYING

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By

NUR UL ATIKAH BINTI IBRAHIM

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

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January 2017

Chairman : Associate Professor Samsuzana Abd Aziz, PhD
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Degradation of frying oil by series of complex chemical reaction during frying process will lead to the formation of undesirable compounds which affecting the quality of frying oil. Conventional chemical methods for frying oil quality assessment are well known to be time consuming, require skilled operator and involve significant amount of harmful solvents.

In this study, dielectric and fluorescence spectroscopy techniques were used to monitor the degradation of refined, bleached, and deodorized palm olein (RBDPO) in batch deep-fat frying. Intermittent frying experiment was conducted for five consecutive days. In total, 30 batches of French fries were fried at the temperature of 185±5ºC in each day. The dielectric constant, fluorescence intensity, total polar compounds (TPC) and free fatty acids (FFA) were measured for analysis.

The results showed that there were generally significant differences in the mean of dielectric constant and intensity of fluorescence emission and excitation spectra of RBDPO over five days of frying ($P<0.0001$). The mean dielectric constant of RBDPO increased from 3.09 to 3.17 as the frying cycle increased. Meanwhile, the mean measured fluorescence intensity of RBDPO for emission and excitation spectra were generally decreased for all prominent peaks observed. For example, mean measured fluorescence intensity decreased from 201.83 to 20.59 arbitrary unit (a.u) and from 240.04 to 24.73 a.u at emission peak of 476 nm and excitation peak of 396 nm, respectively.

PLS model developed using dielectric spectroscopy data showed good potential to predict the degradation of RBDPO with the application of GA selector with R^2 of 0.91 and 0.95 for TPC and FFA, respectively and RMSECV of 1.06 %. Whereas, PLS model constructed using fluorescence emission and excitation spectra data showed substantially good potential to predict the degradation of RBDPO even without the application of GA. For example, PLS model developed by correlating fluorescence emission spectra data to TPC and FFA without GA selector had R^2 of 0.98 and 0.99 for TPC and FFA, respectively with RMSECV of 0.52 % and 0.01 %, respectively.

The result from this study indicated that both spectroscopy data were significantly sufficient to be used for monitoring the degradation of RBDPO during batch deep fat frying. This study could help effort on investigating reliable techniques for in-situ determination of physicochemical properties of palm olein.

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

TEKNIK DIELEKTRIK DAN PENDARFLUOR SPEKTROSKOPI UNTUK MENILAI KEROSAKAN OLEIN SAWIT YANG DIGUNAKAN UNTUK MENGGORENG

Oleh

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Degradasi minyak masak disebabkan oleh siri tindak balas kimia yang kompleks semasa proses menggoreng akan membawa kepada pembentukan sebatian yang tidak diingini yang menjejaskan kualiti minyak masak. Kaedah konvensional untuk menilai kualiti minyak masak adalah proses yang memakan masa yang lama, memerlukan tenaga kerja yang mahir dan melibatkan sejumlah besar bahan kimia yang berbahaya.

Justeru itu, di dalam kajian ini teknik spektroskopi dielektrik dan spektroskopi pendarfluor digunakan untuk memantau degradasi minyak olein sawit yang telah ditapis, diluntur dan dinyahbau (RBDPO) dalam proses menggoreng secara berkelompok. Eksperimen menggoreng telah dijalankan selama lima hari berturut-turut. Secara keseluruhan, 30 kelompok kentang telah digoreng pada suhu 185±5°C setiap hari. Pemalar dielektrik, keamatan pendarfluor, jumlah sebatian kutub (TPC) dan asid lemak bebas (FFA) diukur untuk analisis.

Hasil kajian menunjukkan bahawa, secara umumnya terdapat perbezaan yang signifikan dalam nilai purata pemalar dielektrik dan intensiti pelepasan dan pengujaan spektrum pendarfluor RBDPO sepanjang tempoh lima hari menggoreng ($P<0.0001$). Nilai purata pemalar dielektrik RBDPO telah meningkat daripada 3.09 kepada 3.17 apabila kitaran menggoreng meningkat. Sementara itu, nilai purata intensiti pendarfluor RBDPO untuk pelepasan dan pengujaan spektrum umumnya menurun untuk semua puncak ketara yang diperhatikan. Sebagai contoh, nilai purata keamatan pendarfluor yang diukur menurun daripada 201.83 kepada 20.59 unit arbitrary (a.u) dan daripada 240.04 kepada 24.73 a.u pada puncak pelepasan 476 nm dan puncak pengujaan 396 nm, masing-masing.

Model PLS menunjukkan potensi yang baik untuk meramalkan kerosakan minyak masak dengan penggunaan GA terhadap data spektroskopi dielektrik dengan R^2 sebanyak 0.91 dan 0.95 untuk TPC dan FFA, masing-masing dan nilai RMSECV sebanyak 1.06%. Manakala, model PLS yang dibina menggunakan data pelepasan dan pengujaan spektrum pendarfluor menunjukkan potensi yang sangat baik untuk meramalkan kerosakan minyak masak walaupun tanpa penggunaan GA. Model ramalan tanpa penggunaan GA mempunyai R^2 sebanyak 0.98 dan 0.99, untuk TPC dan FFA, masing-masing dengan nilai RMSECV sebanyak 0.52% dan 0.01%, masing-masing.

Hasil daripada kajian ini menunjukkan bahawa maklumat daripada kedua-dua data spektroskopi adalah mencukupi untuk digunakan bagi memantau degradasi RBDPO semasa proses menggoreng secara berkelompok. Kajian ini dapat membantu dalam usaha mengkaji teknik yang boleh digunakan untuk penentuan sifat-sifat *physicochemical* olein sawit secara *in-situ*.

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I certify that a Thesis Examination Committee has met on 17 January 2017 to conduct the final examination of Nur Ul Atikah bt Ibrahim on her thesis entitled "Dielectric and Fluorescence Spectroscopy Techniques for Determination of Palm Olein Degradation during Batch Deep Fat Frying" 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 of Science.

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I hereby confirm that:

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Declaration by Members of Supervisory Committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
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LIST OF ABBREVIATIONS

ANOVA	Analysis of variance
AOCS	American oil chemists' society
CC	Column chromatography
CPO	Crude palm oil
DMRT	Duncan's multiple range tests
DF	Double fractionated
DGF	German society for fat research
DSC	Differential scanning calorimetry
EVOO	Extra virgin olive oil
FFA	Free fatty acids
FTIR	Fourier transform infrared
GA	Genetic algorithm
IV	Iodine values
LVs	Latent variables
NEM	Nonelution material
NIR	Near infrared reflectance
NMR	Nuclear magnetic resonance
OFA	Oxidized fatty acids
p-AV	p-anisidine value
PCA	Principle component analysis
PCR	Principle component regression
POo	Standard palm olein
POPs	Phytosterol oxidation products
PLS	Partial least square

PV	Peroxide values
RBDCO	Refined, bleached and deodorized coconut oil
RBDPO	Refined, bleached and deodorized palm olein
RFO	Red fruit oil
RMSECV	Root mean square error cross validation
RMSPE	Root mean square prediction error
SBO	Soy bean oil
SFO	Sunflower oil
SQ	Special quality
TA	Thermal analysis
TPC	Total polar compounds
UFO	Used frying oil
UV	Ultraviolet

LIST OF NOMENCLATURES

ε'	dielectric constant
ε''	dielectric loss factor
α	corrective coefficient
C_p	parallel capacitance
C_o	air capacitance
L_s	series inductance
R_s	series resistance
R_p	parallel resistance
R^2	coefficient of determination

CHAPTER 1

INTRODUCTION

1.1 General introduction

Frying is the oldest cooking method and it is existed around 2500 BC in the Ancient Egyptian kitchen (Tannahill, 1995). There are different types of frying process were used which are dry frying, shallow frying and deep frying. Among these types of frying, deep frying is the common unit operation which broadly used in the food processing industries like fast food and snack food (Blumenthal and Stier, 1991). This unit operation produces flavorful fried food product with crispy texture, attractive aroma and tempting appearance (Mallikarjunan et al., 2010). These characteristics make deep fried food product such as French fries and potato chips continues to be the major part of the food market that generating billions of dollars annually.

Deep frying can be defined as the process of cooking food by completely immersed it into hot oil at temperature typically between 150 to 200°C (Yamsaengsung and Moreira, 2002). During the process of cooking, there is simultaneous heat and mass transfer process between the food product and the oil or fat (Lalas, 2009). Oil plays an important part in the deep frying process because it serves as heat transfer medium (Navas et al., 2007) which continuously or repeatedly heated at high temperature in the presence of air and moisture during frying. This condition exposes oil to the chemical reactions such as hydrolysis, oxidation and polymerization (Abdulkarim et al., 2008). As these reactions continue, the quality of the oil changes and may reach the point where the frying oil should be discarded (Stevenson et al., 1984). The quality of frying oil is very important because the absorption of degraded oil by the food product will affect the quality of final product (Ziaiifar et al., 2008).

Hydrolysis is a chemical reaction which occurred because of the interaction between triglycerides in oil with the water or moisture release from the food in the form of steam. Meanwhile, oxidation occurs because of the reaction between atmospheric oxygen and the oil at the surface. Polymerization is the chemical process that contribute to the formation of higher molecular weight compounds or known as polymers (Mallikarjunan et al., 2010). These chemical reactions produce decomposition compounds which are volatile and nonvolatile (Choe and Min, 2007). The accumulation of decomposition compounds is not only affecting the quality of fried food but could be detrimental to the consumer health.

During frying, food products absorbed large quantities of degraded oil (Blumenthal and Stier, 1991). Consumption of degraded oil was reported unhealthy and may cause chronic diseases (Leong et al., 2012; Adam et al., 2009; Soriguer et al., 2003). Soriguer et al., (2003) investigate the risk of high blood pressure or hypertension on the intake of degraded oil. They found that the intake of polar compounds formed in the degraded oil positively related to the risk of hypertension. Moreover, Adam et al., (2009) study the effect of consuming repeatedly heated palm oil on the development of atherosclerosis. Their results showed that repeatedly heated palm oil eliminates the protective action on aorta which could contribute to the development of atherosclerosis. Recently, Leong et al., (2012) studied the effect of consuming repeatedly heated palm olein on the activity of blood pressure. The results from their study showed that repeatedly heated palm olein increased lipid peroxidation and gave adverse effects on the activity of blood pressure–regulating enzymes. These studies demonstrate how repeated frying oil gives negative impact on the consumer health. Therefore, frying oils need to be discarded after certain duration of use.

There are several official regulations which were adopted in some European countries to limit the usage of frying oil (Table 1.1) with polar compounds as the most accepted parameter for quality evaluation of frying oil in the most countries. Generally, the index of polar compounds or known as total polar compounds (TPC) in used frying oil should not exceed 25%. This guideline was not only adopted in European countries but also in many other countries including Malaysia. Apart from TPC, there are also other parameters which were used to evaluate the quality of frying oil like free fatty acids (FFA), peroxide values (PV) and iodine values (IV).

Table 1.1 : Guideline index in official regulations on used frying oil*

	TPC (%)	FFA (%)	Oxidized Fatty Acids (OFA) (%)	Smoke point (^{o}C)	Polymers (%)
Austria	27	2.5	1	170	-
Belgium	25	2.5	-	170	10
France	25	-	-	-	-
Germany	24	2.0	0.7	170	-
Italy	25	-	-	-	-
Spain	25	-	-	-	-

*(Adapted from Dobarganes and Marquez-Ruiz, 1998)

Various methods were used to evaluate the quality of frying oil based on physical and chemical parameters. Generally, evaluation based on changes in physical properties of frying oil was the most common method used in deep fried food product services (Moreira et al., 1999) and it was done by the experience cook. Usually, the observation are based on the taste of fried food, colour, odour and they also monitor excessive foaming and smoking before making decision to discarded the frying oil (Moreira et al., 1999; Mittal et al., 1998). However, this method is insufficient and inconsistent due to the dependency on the human nature (Mittal et al., 1998). Consequently, over the years, several methods were used and proposed like chemical tests, and instrumental methods. Chemical tests method such as column chromatography (CC) is well known to be time consuming, involve significant amount of harmful solvent and require highly skilled personnel to handle the laboratory analysis.

In order to solve this problem, several attempts were made by researchers to replace the chemical tests using instrumental methods such as Fourier transform infrared (FTIR), near infrared reflectance (NIR), nuclear magnetic resonance (NMR) and differential scanning calorimetry (DSC). Despite, the results from some of these methods showed their potential in determining the frying oil quality, there are still room for improvement and further investigation. For example, Reder et al. (2014) reported that DSC method not sufficiently accurate to predict the quality parameter of edible oil. Hence, the investigation of rapid measurement techniques is essential to enhance the efficiency of frying oil quality monitoring.

In this study dielectric and fluorescence spectroscopy techniques were explored as an effort to improve the frying oil quality sensing system in Malaysia. According to Nawrocka and Lamorska, (2013), spectroscopic is a promising technique in monitoring the quality agricultural products since it provide rapid and on-line analysis, require minimal or no sample preparation and have the potential to run multiple tests on a single sample.

Dielectric spectroscopy is one of the spectroscopic techniques that provides ability to measure bulk physical properties of materials. It has been recognized as a powerful tool for the electrical characterization of non-conducting or semiconducting materials. This method has been of interest for many years in monitoring quality of agricultural products such as fruits and vegetables (Castro-Giraldez et al., 2010; Nelson et al., 2007; Nelson, 2005). Another spectroscopic technique that was used since the last decades for food analysis is fluorescence spectroscopy. According to Sirkoska et al., (2003), fluorescence spectroscopy offers high sensitivity, simplicity and selectivity which are essential for chemical analysis in monitoring edible oil quality. There are numbers of paper published on the potential of fluorescence spectroscopy in the analysis of oil quality (Guzmán et al., 2015; Sikorska et al., 2012; Cheikhousman et al., 2005; Sirkoska et al., 2004; Engelsen, 1997).

1.2 Problem statement

Frying process involves a complex series of physical changes and chemical reactions that giving rise to numerous volatile and non-volatile compounds (Gertz, 2000). These reactions leads to the degradation of frying oil and up to certain level frying oil should be discarded because it is no longer able to produce high quality fried food and harmful to human consumption (Stevenson et al., 1984). Recently, it was reported in the newspaper that repeatedly used frying oil could give hazardous effect to consumer health (Kosmo, 2013). In that report, two nutritionists advise people to discard frying oil after three times of use as it may turned into a poison (acrolein) that can cause cancer. Determination of time or state for frying oil to be discarded is the most challenging task over the last decade.

Current assessment used for frying oil quality in Malaysia needs intensive laboratory work, which is time consuming and requires high skilled operator, in addition involve significant amount of solvent that could be harmful to the operator. There are some instrumental techniques introduced by researchers over the years but more studies are needed on its feasibility and commercialization.

1.3 Objectives

The overall goal of this study is to evaluate the spectroscopy techniques for monitoring degradation of refined, bleached and deodorized palm olein (RBDPO) in batch deep fat frying process. In order to accomplish this goal, the following objectives were set;

1. To investigate the changes in dielectric properties and fluorescence intensities of RBDPO during frying.
2. To study the relationship between changes in dielectric properties and fluorescence intensities of RBDPO with regards to changes in chemical properties (TPC and FFA) over five consecutive days of frying.
3. To develop model using dielectric and fluorescence spectra data by implementing partial least square (PLS) with genetic algorithm (GA) selector to predict the degradation of RBDPO during batch deep fat frying.

1.4 Scope and limitations

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

1. Degradation parameters measured were TPC and FFA.
2. Frying material used in this study was French fries.
3. Palm olein used in this study was RBDPO from Besetia brand.
4. This study focus on the degradation of frying oil. Hence, the properties of French fries will not be the subject of discussion.
5. Frequency used to measure dielectric constant of RBDPO was from 100 Hz to 10 MHz.

1.5 Thesis organization

This thesis describes a study on the application of dielectric and fluorescence spectroscopy concept in monitoring degradation of RBDPO during batch deep frying. The research conducted in this thesis is expected to give a fundamental input for further development of sensing system in fried food production industry. A review of previous research and studies regarding the uses of dielectric and fluorescence spectroscopy in monitoring food quality are discussed in CHAPTER 2. The concept of dielectric and fluorescence spectroscopy is first explained. Conventional methods and current techniques other than dielectric and fluorescence spectroscopy to determine the frying oil quality are described. A brief introduction of statistical analysis techniques used in this study for predicting the quality of palm olein based on FFA and TPC are also discussed. CHAPTER 3 provides the material, setup and experimental procedures in performing this research. Samples collection from frying experiment is described. The instrumentation setup and measurement procedure for dielectric and fluorescence are described and illustrated by photos. The statistical analysis used is explained in the last section. CHAPTER 4 described the dielectric properties and fluorescence intensity of frying oil samples obtained from frying experiment. The effects frying cycle on dielectric properties and fluorescence intensity of RBDPO samples are also described. The models developed using PLS with GA selector are explained. Finally, CHAPTER 5 which is the final CHAPTER in this thesis outlines the findings of this research and end with some suggestion and recommendation for future work in order to improve the result obtained in this study.

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