UNIVERSITI PUTRA MALAYSIA

DIELECTRIC PROPERTIES FOR RAPID ON-SITE DETECTION AND SCREENING OF HALAL AND NON-HALAL FOODS

FATIN NORDALILA BT OMAR

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By

FATIN NORDALILA BT OMAR

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

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

DIELECTRIC PROPERTIES FOR RAPID ON-SITE DETECTION AND SCREENING OF HALAL AND NON-HALAL FOODS

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FATIN NORDALILA BT OMAR

February 2014

Chair : Assoc. Prof. Zurina bt Zainal Abidin, PhD

Faculty : Engineering

Halal verification has become increasingly important especially to the Muslim communities and very challenging nowadays due to the unavailability of a robust and rapid method to detect non-halal products. A potential method for detection and discrimination of products for halal authentication using dielectric properties has been investigated in this study. With regards to the interests of consumers particularly on food intake, this study focuses on several types of primary alcohol, commercial beverages and meat. The aim of this work is to characterize the dielectric response of alcohol and meat samples by using the dielectric properties, which is as potentially rapid on-site determination for halal and non-halal products. Measurements were made with frequency domain technique from 500 MHz to 50 GHz by means of an open-ended coaxial probe connected to Agilent Professional Network Analyzer (PNA-X) N5245A.

For alcohol, the results show dielectric properties of alcohol can be detected until lowest concentration of 0.1% since standardization of alcohol content allowed by JAKIM is < 0.5 %. Frequency 10 GHz – 25 GHz was found to give good separation of graph for different concentration of ethanol solution. Data evaluation of several alcoholic and non-alcoholic beverages were supported with Gas Chromatography Mass Spectrometry (GC-MS) method in order to relate the polarity of volatile compounds and polarization effects in dielectric signal. Calculation of the parameter ε'_{∞}, ε'_{s}, $\varepsilon'_{s} - \varepsilon'_{\infty}$ and τ_H in the proposed Debye model combined with Higasi's equation of relaxation time provided good agreement to the experimental data. Among the parameters, $\varepsilon'_{s} - \varepsilon'_{\infty}$ (R^2=0.9964) and τ_H (R^2=0.9857) gave the best regression line to be used as concentration detection.

Raw beef, pork and chicken meat were characterized by dielectric over the same frequency range and two prominent peaks identified as Peak A (at 7 GHz) and Peak B (at 30 GHz) were found in pork and mix meat samples. Next, parameter of penetration depth, d_p also showed good effects between heated pork and other meats since the correlation between moisture content and d_p is higher (R^2=0.98). The emergence of

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Peak B was continuous in sterilized pork, heated pork, porcine gelatin and pork in can. The consistency of this peak in all pork-based samples in this study shows it can be the potential marker to discriminate non-halal (pork) and halal meat since it was not seen in beef and chicken. It is believed there are some constituent species in pork sample does not destroyed even high temperature were subjected on it. This finding proves dielectric technique could be utilized for rapid on-site verification for any meat samples by detecting the significance Peak B at frequency of ~30 GHz. This technique also can reduce the analysis time and provide high accuracy result for initial screening purpose.

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

SIFAT DIELEKTRIK UNTUK PENGESANAN DAN SARINGAN PANTAS UNTUK MAKANAN HALAL DAN TIDAK HALAL

Oleh

FATIN NORDALILA BT OMAR

Februari 2014

Pengerusi : Prof. Madya Zurina bt Zainal Abidin, PhD

Fakulti ` : Kejuruteraan

Pengesanan halal telah menjadi semakin penting terutamanya kepada masyarakat komuniti Muslim dan amat mencabar pada masa kini kerana ketiadaan kaedah yang mantap dan pantas untuk mengesan produk yang tidak halal. Satu kaedah yang berpontensi untuk pengesanan dan pembezaan produk bagi pengesahan halal dengan menggunakan sifat dielektrik telah diselidik dalam kajian ini. Dengan mengambil kira kepentingan pengguna terutama pada pengambilan makanan, kajian ini memberi tumpuan pada beberapa jenis alkohol utama, minuman komersial dan daging. Tujuan kajian ini adalah untuk mencirikan respon dielektrik sampel alkohol dan daging dengan menggunakan sifat dielektrik sebagai potensi penentuan pantas bagi produk halal dan tidak halal. Pengukuran telah dibuat dengan teknik domain frekuensi daripada 500 MHz hingga 50 GHz melalui cara prob sepaksi terbuka-akhir disambung dengan Rangkaian Profesional Penganalisis Agilent (PNA-X) N5245A.

Untuk alkohol, keputusan menunjukkan sifat dielektrik alkohol dapat dikesan sehingga kepekatan serendah 0.1% di mana piawaian kandungan alkohol yang dibenarkan oleh JAKIM ialah < 0.5%. Frekuensi 10 GHz – 25 GHz didapati memberikan pemisahan yang baik untuk graf kepekatan larutan etanol yang berbeza. Penilaian data terhadap beberapa minuman beralkohol dan tidak beralkohol disokong dengan kaedah Kromatografi Gas-Spektrometri Jisim untuk mengaitkan kekutuban sebatian meruap dengan kesan polarisasi dalam isyarat dielektrik. Pengiraan parameter ε'_{∞}, ε'_{s}, ε'_{s} - ε'_{∞} and τ_H dalam cadangan model Debye yang digabungkan dengan persamaan kelonggaran Higasi member persetujuan yang baik kepada data eksperimen. Antara parameter-parameter tersebut, ε'_{s} - ε'_{∞} (R^2=0.9964) dan τ_H (R^2=0.9857) memberi garis regresi yang terbaik untuk digunakan sebagai pengesanan kepekatan.

Daging lembu mentah, daging babi dan daging ayam telah dicirikan oleh dielektrik pada julat frekuensi yang sama dan dua puncak yang menonjol dikenali sebagai Puncak A (pada 7 GHz) dan Puncak B (pada 30 GHz) telah ditemui dalam sampel daging babi dan daging campuran. Seterusnya, parameter kedalaman tembusan, d_p menunjukkan kesan yang baik antara daging babi yang telah dipanaskan dan daging lain di mana kolerasi di antara kandungan kelembapan dan d_p adalah tinggi (R^2=0.98). Kemunculan

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Puncak B adalah berterusan dalam sampel daging babi yang telah disterilkan, daging babi yang telah dipanaskan, gelatin babi dan daging babi dalam tin. Konsistensi puncak ini dalam semua sampel berasaskan daging babi dalam kajian ini menunjukkan ia boleh menjadi penanda potensi untuk membezakan daging yang tidak halal (daging babi) dan yang halal kerana ia tidak ditemui pada daging yang lain. Adalah dipercayai bahawa terdapat beberapa spesies konsistuen dalam sampel daging babi yang tidak musnah walaupun suhu tinggi telah dikenakan padanya. Penemuan ini membuktikan teknik dielektrik boleh digunakan untuk pengesahan pantas bagi apa sahaja sampel daging dengan mengesan Puncak B yang signifikan pada frekuensi ~30 GHz. Teknik ini juga dapat mengurangkan masa analisis dan memberi ketepatan keputusan yang tinggi untuk tujuan saringan awal.

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Fatin Nordalila bt Omar
February 2014

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I certify that a Thesis Examination Committee has met on 14 February 2014 to conduct the final examination of Fatin Nordalila binti Omar on her thesis entitled "Dielectric Properties for Rapid On-Site Detection and Screening of Halal and Non-Halal Foods" 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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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

NA	Network Analyzer
PCR	Polymerase Chain Reaction
FTIR	Fourier Transform Infrared
HPLC	High-Performance Liquid Chromatographic
MW	Microwave
RF	Radio frequency
GC-MS	Gas Chromatography-Mass Spectrometry
JAKIM	Department of Islamic Development Malaysia

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CHAPTER 1

INTRODUCTION

1.1 General introduction on halal concept

Identification and discrimination of halal and non-halal product has becoming increasingly important especially to the Muslim communities. It has become very challenging nowadays for 2 billion Muslims out of more than 6.5 million world populations to ensure that all matters related to halal and *haram* is taken care in their daily lives.

Halal is an Arabic term that designated to the meaning of permitted, allowed, approved, licit, lawful or legal. *Haram* or non-halal is an opposite meaning of halal. In religion of Islam, the terms halal and *haram* are applied in many aspects of life in which most major concern is related to daily intake. If the status of an item cannot be ascertained whether lawful or unlawful, it falls in the category of *mashbooh* or *shubhah* (Al-Qaradawi, 1995). This word refers to synonym of questionable or doubtful. When it comes to the halal concept in the daily food intake, it means that it is permitted and fits for consumption by Muslims. However, halal concept not merely deals with food, but it is a comprehensive understanding which the concept known as 'from farm to table' (Che Man, 2006).

A product of food or beverage is said to be illegal when it is involved with one of the following items including pork-based products and its by-products, meat from swine, animals improperly slaughtered and animals killed in the name of anyone other than Allah. In addition, carnivorous animals, amphibious animals, undesirable insects, birds of prey, intoxicants, blood and blood by-products and foods contaminated with any of the above products are also considered as *haram* for consumption (The Malaysian Halal Hub, 2013).

1.1.1 Issues on Halal

Determination of halal consumer products has been a global issue for a long time and remains largely unsolved. The concern of consumer particularly Muslims in Malaysia are more apparent when the halal issues are not limited to food and beverages but also cover non-food products such as cosmetics, toiletries, pharmaceutical, medicine and leather products, which in Malaysia market the producer are mainly by non-Muslims. This includes the imported goods from outside where the verification of halal should be more stringent when brought into our market. These issues are further worsened by the misleading information given by supplier, mislabeling, characterization and general lack of individual awareness regarding this matter.

Consumers are often concerned by a variety of issues mostly on authenticity and adulteration problems. This refers to prohibited ingredients and additives in food or beverage. It can be seen by identifying the source of raw materials whether the origin is

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from animal or plant. The matter also related to slaughtering method (according to *Shariah* law), the processing operation with equipment used, packaging, storage and transportation. The progress in food industry especially in biotechnology had forced consumers to be more concerned with those enzyme application and genetically modified organisms or better known by the terms of GMOs.

Some examples of recent issues in Malaysia are adulteration in bread and the use of pig intestine casings in sausage (Che Man et. al, 2007). In the market, there is confusion over non-halal moon cake (Chinese tradition food) containing porcine ingredients with halal logo. There are also some manufacturers produce bakery ingredients such as margarine and shortening by mixing the blended lard or tallow with other vegetable oils due to the reason of low production cost (Gillies, 1974).

1.1.2 Development of halal research in Malaysia

Presently research and development (R&D) in halal industry is focused on product traceability analysis and verification process development. The integrity of the status of halal product depends on the technology and detection systems which brought to bear on halal determination. These systems must work to facilitate manufacturer obligation to legal requirements for the halal certificate of their products.

In Malaysia, the government has decided to make the country as Global Halal Hub in the region as well as to penetrate the global market internationally (Talib, 2008). Department of Islamic Development Malaysia or JAKIM is a Malaysian government institution, which establishes the requirement of halal logo and implements halal certification system of a product. Among the agencies involved in this field, Universiti Putra Malaysia (UPM) through Halal Product Research Institute (HPRI) has developed several methods to assist in detecting prohibited species commonly in food and beverage products. The techniques include species-specific polymerase chain reaction (PCR) detection (Che Man et al, 2007), Fourier Transform Infrared (FTIR) spectroscopy (Syahariza et al., 2005) and High-Performance Liquid Chromatographic (HPLC) analysis (Marikkar et al., 2005). PCR is a DNA based methods for pork detection while HPLC is an analytical technique based on protein analyses. FTIR is also widely used for halal authentication by providing infrared spectrum of the food sample. Research works to develop existing and new techniques are still continued to achieve more comprehensive and reliable detection.

1.2 Dielectric properties

The interaction of electromagnetic energy with the material describes the dielectric properties of a material. The response of a material to an applied electric field is determined by its permittivity and conductivity. Permittivity is a complex quantity to determine the dielectric properties that influence reflection of electromagnetic waves at interfaces and the attenuation of the wave energy within materials. Conductivity gives a measure of its ability to conduct, i.e. let charge pass through it (Markx and Davey, 1999; Abidin, 2005; Tanaka et al., 2008; Sosa-Morales et al., 2010).

1.2.1 Application of dielectric in food research

The study of dielectric properties is closely associated with the storage and dissipation of electric and also magnetic energy in materials. In area of food research, the implementation of the dielectric properties of materials are relatively increasing in its application, processing method, characterization and quality level. It has centred primarily in describing the behaviour of food materials when subjected to high frequency fields in dielectric application (Venkatesh and Raghavan, 2004). Dielectric properties are the most important physical properties of food associated with radio-frequency (RF) and microwave (MW) heating.

1.2.2 Advantages of microwave dielectric method

Microwave (MW) radiation is an electromagnetic wave with frequency, f ranging between 300 MHz and 300 GHz and the wavelength, λ from 1 mm to 1 m. Electromagnetic radiation is a form of energy emitted and absorbed by charged particles, which exhibits wave-like behaviour as it travels through space. MW acts as high frequency electric fields and will generally heats any material containing mobile electric charges such as polar molecules in a solvent or conducting ions in a solid. Figure 1.1 shows all electromagnetic spectrum including the MW region.

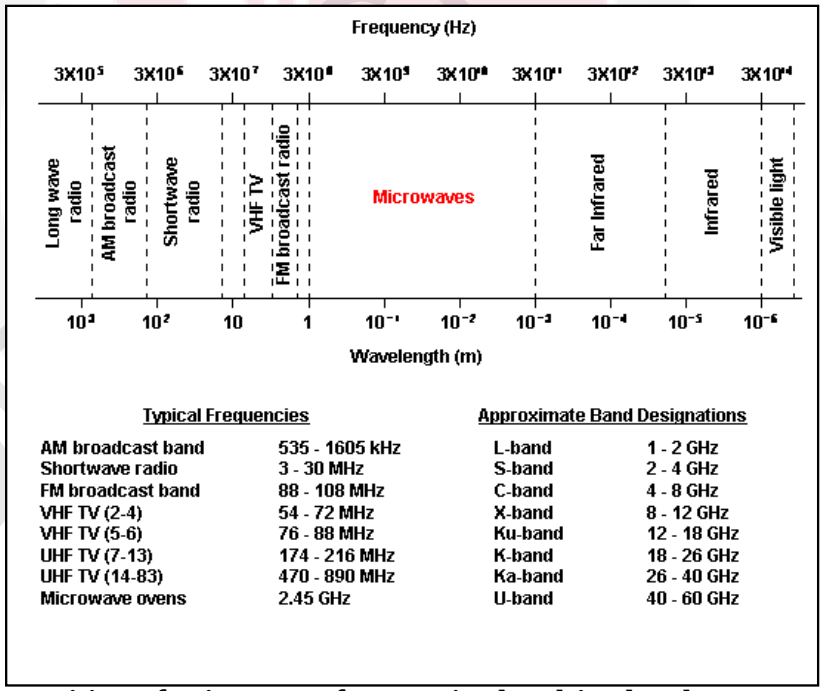

Figure 1.1 The position of microwave frequencies band in the electromagnetic spectrum.

The usage of MW frequencies in detecting dielectric properties of materials is found more comprehensive when the applied dielectric penetrates deeper into a material, which is useful when dealing complex structure that exists in food. This method is very simple, practical, gives an online and real-time signal and the result can be obtained directly (Patel et al., 2008). Nunes et al. (2006) also reported the application of

microwaves range of spectrum in food is attractive because of the ease of use and the rapidity of treatment as well as the measurement. Furthermore, it can be potentially used on foods in optically opaque sealed containers.

1.3 Problem statements

Traditionally, the verification of halal status of products especially food and drinks was made through field monitoring processes that have been dependent on the accuracy of information provided by manufacturer. The processes such as registering the materials/ingredients used, documentation of purchasing of all materials, verification of material documents, identification of critical ingredients, product formulae checking and production observation are costly, time-consuming and cumbersome. Researchers have striven to develop techniques in detecting and identifying species origin in products since last few decades which are very important due to the economic, health and religious issues. For these reasons, efforts to find and develop new detection methods for porcine tracing and alcohol content in products require ongoing and more in-depth research to ensure the integrity of the halal status of the products consumed by Muslims.

Halal science is a new term means the implementation of science laboratory process to identify the forbidden species and inspect the product for halal authentication. Murugaiah et al. (2009) reported that some of the currently existing methods are expensive, not suitable for routine analysis and complex to perform. The efforts in finding alternative new and improvised methods had been done continuously. Here in this study, another alternative method is explored by using dielectric technique electromagnetic radiation in MW range on food. This technique offers a rapid, easy to use and low cost detection not only for food items but also for non-food materials.

Microwave dielectric is very attractive and powerful method for characterizing food and some of its components in a non-invasive way. The interaction between the materials with electromagnetic energy in the microwave range provides general information known as dielectric constant and dielectric loss (Tanaka et al., 2008). Dielectric measurements as well as the techniques provide simple and non-laborious alternative compared to current lab-based method such as PCR and HPLC analysis. The measurement able to reduce the analysis time and assists efficiently in reliable decision making process.

Although dielectric is not a new technology, however, early works mostly focus on the characterization of a material properties such as aqueous ionic solutions (Hasted, Ritson & Collie, 1948), water solvent (Hasted, 1961), starch (Ndife et al., 1998), pharmaceutical powders (McLoughiin et al., 2003) and meat and ingredients used in meat product (Lyng et al., 2005). However, none of these studies explored the technique for halal purpose. In addition, most of the previous works especially related to food were performed in low frequency range (< 25 GHz). Thus, a study on higher frequency, e.g up to 50 GHz is useful for further characterization of food dielectric properties.

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1.4 Objectives of research

The aim of this research project is to establish the suitability and feasibility of using the dielectric method as potentially rapid on-site determination for some halal and non-halal sample. To achieve this, several specific objectives are outlined as follow:

i) To identify the electromagnetic response in microwave regions (0.5 GHz – 50 GHz) of alcohol and meat samples.

ii) To evaluate the effect of several factors including frequency, temperature, concentration, chemical composition, and manufacturing process on dielectric properties of samples tested.

iii) To characterize, analyze and compare experimental dielectric response from Objective 1 (for alcohol) with Debye and Higasi et al. model and identify the significance characteristic of dielectric response on halal and non-halal meat.

1.5 Scope and limitations of research

An approach to use dielectric method for characterization of samples is proposed. Dielectric properties are obtained through dielectric measurement by using special equipment called Network Analyzer with a method called open-ended coaxial probe. The measurement does not cover all microwave frequencies region but only in the range of 0.5 to 50 GHz with 50 points of frequencies.

Dielectric method used in this study is considered as preliminary work regarding in halal authentication of food and beverage. This method focus on providing basic dielectric data of common foods and drinks in market that have always been highlighted in the issue of halal. The research is limited to meat and alcohol samples. Both samples have different aims where meat samples devoted into detecting the significance meat characteristic and factors affecting raw and processed meat. In addition some of compound analysis such as determination of moisture, fat and protein content are subjected to raw meat samples. Meanwhile, alcohol will emphasize on the analysis of dielectric response, factors affecting on alcohol samples, theoretical analysis of experimental data by using existing model of Debye and Higasi concept and eventually providing the best parameter to be utilized for alcohol content detection.

1.6 Thesis outline

This report consists of 6 main chapters. The first chapter gives general introduction of halal concept and understanding of the dielectric including the statement of problem, objectives and scope of the study. The second chapter provides the detailed overview of the theoretical framework of dielectric, the reviews of model used, analysis used in previous research of dielectric properties on food materials and the existing works in field of halal authentication. The next chapter covers the materials and methodology of the experiment. Both parts describe the type of materials, methods of sample preparation, equipment used for dielectric signal detection and other experimental methodology related to the analysis of this study. In chapter 4, a comprehensive discussion based on the collected results of alcohol samples associated with dielectric constant and dielectric loss factor are elaborated in detail. The discussion is extended to the relevance data by Gas Chromatography-Mass Spectrometry (GC-MS) in assisting better explanation of alcoholic beverages dielectric data and also to apply specific

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theoretical analysis by Debye model and Higasi method on all alcohol samples. Chapter 5 presents dielectric data for all meat samples, the discussion of meat characterization and significant peak finding as well as factors affected the properties. Finally, chapter 6 provides summary and conclusion of the finding in this report and also the prospective further works are recommended in recommendation part. The references, appendices and biography of the researchers are arranged in sequence at the last part of thesis.

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