

**THE TIME OF FLIGHT TECHNIQUE TO DETERMINE
THE EFFECT OF TEMPERATURE ON PURE EDIBLE
OLIVE OIL AND CORN OIL**

BY

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**A thesis submitted in fulfilment of the requirement for the
degree of Master of Science (Mechatronics Engineering)**

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ABSTRACT

Authentic and unadulterated edible vegetable oils, for example, olive oil, canola oil, sunflower oil, virgin coconut, corn oil, and other types of oil extracted from seeds, or any other parts of fruits are referred as high graded oil which sold with higher prices at Malaysia market. They provide benefits in anti-inflammatory antioxidants, high immune system and prevent heart disease, however, due to higher prices, the problem is these types of oils tend to be adulterated with other lower graded oils. The research in the study will help in preventing the adulterated oils widely commercialized, the consumers or factory or any second related party should be able to recognize the purity of the oils has been processed or sold. The research study of the ultrasound wave triggered by TDC1000- TDC7200EVM module to calculate the time of flight when the ultrasound wave travel through the different types of high graded edible oils. The time-of-flight data was converted to speed of sound and compare with the data from the literatures to maximize the accuracy of the results. The time-of-flight in frequency domain data on both pure types of olive oil and corn oil were compared to observe the differences in the oil characteristics. It can be concluded from the study that the method of using ultrasound wave shows high accuracy comparable to the literature, and the ultrasound wave can be used to differentiate the different types of high graded edible oils.



خلاصة البحث

يُشار إلى الزيوت النباتية الصالحة للأكل الأصلية وغير المغشوشة ، على سبيل المثال ، زيت الزيتون ، وزيت الكانولا، وزيت عباد الشمس ، وجوز الهند البكر ، وزيت الذرة ، وأنواع أخرى من الزيوت المستخرجة من البذور ، أو أي أجزاء أخرى من الفاكهة على أنها زيوت عالية الجودة تباع بأعلى الأسعار في الأسواق بماليزيا. أنها توفر فوائد في مضادات الأكسدة المضادة للالتهابات ، والجهاز المناعي العالي والوقاية من أمراض القلب ، ولكن بسبب ارتفاع الأسعار ، فإن المشكلة هي أن هذه الأنواع من الزيوت تميل إلى أن تكون مغشوشة بزيوت أخرى ذات تصنيف منخفض. سيساعد البحث في الدراسة في منع الزيوت المغشوشة التي يتم تسويقها على نطاق واسع ، وينبغي أن يكون المستهلكون أو المصنع أو أي طرف آخر ذي صلة قادرين على التعرف على نقاء الزيوت التي تمت معالجتها أو بيعها. الدراسة البحثية لموجة لحساب TDC1000-TDC7200EVM الموجات فوق الصوتية الناتجة عن وحدة زمن الرحلة عندما تنتقل الموجات فوق الصوتية عبر أنواع مختلفة من زيوت الطعام عالية الجودة تم تحويل بيانات وقت الرحلة إلى سرعة الصوت لمقارنة نتائج النتائج على السرعة القياسية للصوت في الأدبيات لزيادة دقة نتائج النتائج. تم تحويل بيانات وقت الرحلة على كلا النوعين النقيين من زيت الزيتون وزيت الذرة إلى مجال التردد ومقارنتها بالاختلافات الملحوظة في خصائص الزيت. يمكن الاستنتاج من الدراسة أن طريقة استخدام الموجات فوق الصوتية تظهر دقة عالية عند مقارنتها بالأدبيات ويمكن استخدام الموجات فوق الصوتية للتمييز بين الأنواع المختلفة من زيوت الطعام عالية الجودة. يوصى بإجراء مزيد من الدراسات لغش زيوت الطعام النقية مع ملاحظة المقارنة مع زيوت الطعام النقية.

APPROVAL PAGE

I certify that I have supervised and read this study and that in my opinion; it conforms to acceptable standards of scholarly presentation and is fully adequate, in scope and quality, as a thesis for the degree of Master of Science (Mechatronics Engineering).



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DECLARATION

I hereby declare that this thesis is the result of my own investigations, except where otherwise stated. I also declare that it has not been previously or concurrently submitted as a whole for any other degrees at IIUM or other institutions.

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LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
MRM	Multiple Reflection Method
RRM	Reference-Reflection Method
TRM	Transmission Reflection Method
ARM	Angular Reflection Method
SMEs	Standard Definitions of Small and Medium Enterprise
MNCs	Multinational Corporations
FTIR	Fourier Transform Infrared
DSC	Differential Scanning Calorimetry
VCO	Virgin Coconut Oil
NMR	Nuclear Magnetic Resonance Spectroscopy
PLS	Partial Least Square Methods
PCA	Principal Component Analysis
LIBRA	Matlab Library for Robust Analysis
SLSM	Sequential Least Square Method
ARX	Auto-Regressive Exogenous Method
TM	Transition Method
SNR	Signal to Noise Ratio
TOF	Time of Flight Signal
MATLAB	Matrix Laboratory
TI	Texas Instruments
EVM	Evaluation Module

LIST OF SYMBOLS

v	Velocity
d	Width
t	Time
hpa	hectopascal
$^{\circ}\text{C}$	Celsius
tof	Time of Flight
C	Sound Velocity
Z	Impedance
$N(L)$	count rate density
L	pulse height output
$R(E_n, L)$	detector response matrix
$\varphi(E_n)$	energy distribution of the incident neutrons
ρ	density
K_s	Isotopic (adiabatic) compressibility
N_i	Count rate
X_j	Incident neutron

CHAPTER ONE

INTRODUCTION

1.1 BACKGROUND OF THE STUDY

Malaysian products have been exported worldwide and expanding years by years and has received much admiration and respect from importing countries due to its high quality and reliability (MITI, 2019). However, the economy is growing faster and there are many ways to increase value and trust of the Malaysian Product. One of the ways is by reducing fraud from the processed food. Processed food are foods that require little processing or production and packaged to stay fresh. A few examples of processed foods include canned and frozen fruits and vegetables, packaged foods labeled “natural” or “organic,” such as cereals, fresh meat, and poultry, and jarred baby foods. Also, the foods with health and nutrition claimed on the label, such as “may reduce risk of heart disease”, “low in fat” or “high in calcium” and foods fortified with nutrients such as fiber, vitamin D, and omega-3 fatty acids (International Food Information Council Foundation, 2010). An example of the food fortified with fatty acids is vegetable oil, which also comes with the terms triglyceride extracted from a plant. Vegetable oil is important in the daily diet. Virgin coconut oil is made from fresh coconuts via cold pressing to produce an oil that has an ideal balance between the medium-chain fatty acids and more sensitive polyphenols and vitamins. A case study from PerkinElmer and Universiti Tunku Abdul Rahman (Perkin Elmer & Universiti Tunku Abdul Rahman, n.d.), has stated that everywhere in the global food supply chain, incidents of food-borne illnesses due to adulteration or food mishandling are on the rise in the Southeast Asia countries. There are cases where the virgin olive oils sold in the market, were actually lower graded olive oil that has been blended with some good extra virgin olive oil. Some

cases the virgin olive oils were just deodorized oil. The oils were blended with other type of oil that has same characteristic to give the virgin olive oil a little color, a little flavor and then sold as extra virgin. Some suppliers take a vial of colorless, odorless sunflower oil, and added a few drops of chlorophyll, and it eventually becomes the color of olive oil (Deborah Bogle and Tom Mueller, 2011).

Hence, one way to reduce the fraud in the pure liquid material is by being able to determine the type of liquid before being sent to consumers. A standard development organization that serves as an open forum for the development of international standard control value called ASTM International (American Society for Testing and Materials) is useful for the characterization of materials depends on the materials speed of sound, density, velocity, melting points and boiling points. The changes in the quality of liquid are premeditated from the changes in the parameters such as viscosity, density, ultrasonic velocity, acoustic impedance, and intermolecular free length (Fasina & Colley, 2008; Vol & Press, 2006). Each oil type has their own physical properties divided by density, viscosity, and saturated acid. Pressure-viscosity coefficient increases exponentially with the adiabatic bulk modulus (Ohno et al., 2014). The relationship demonstrates that the pressure-viscosity coefficient can be predicted from the adiabatic bulk modulus (Ohno et al., 2014). Based on the relationship on the adiabatic bulk modulus, the value for the speed of sound for the different type of oil can be calculated.

A few methods for liquid characterization and identification have been recognized and studied. The methods are differential scanning calorimetry, chromatography, and spectroscopy. One of the studies was using differential scanning calorimetry technique for detecting lard adulteration in butters required sample preparations kept in glass vials under the refrigerated condition with temperature

control, and tends to be laborious since the sample need to undergo the blending process according to the percentage of lard in buffer (Tomaszewska-Gras, 2012).

Instrumental techniques based on chromatography and spectroscopy have received particular attention from researchers worldwide since they are efficient and able to control adulteration practices in food lipids (Spectroscopy, Spectroscopy, & Spectroscopy, n.d.). When compared to chromatographic techniques, infrared spectroscopy is an attractive option for detection of adulterations in oils and fats due to the speed of analysis, and minimal sample preparation (Marikkar, Smirghaniandi, Jaswir, & Nazrimmarikkar, 2016; Rubalya Valantina, Chandiramouli, & Neelamegam, 2013; Spectroscopy et al., n.d.).

Ultrasonic Spectroscopy is the characterization of the ultrasonic response of a material to the propagation of a low energy ultrasonic wave, which can measure the absolute frequency dependent and attenuation of liquids. The ultrasonic spectroscopy characteristics which are non-destructive, non-intrusive and in-situ is one major contribution (Tomaszewska-Gras, 2012). The method of ultrasonic spectroscopy is suitable for determining the properties of liquids.

The frequency dependence of liquids, where the frequency used in the related research controlled from 1MHz, 2MHz, 3MHz, and 5MHz to study the different acoustic impedance value in different liquid showed the acoustic impedance is dependent on the frequency of the ultrasound. Based on the frequency range studied, it was concluded that coconut oil, cottonseed oil, till oil, palm oil, sunflower oil, and groundnut oil responded with a significant increase of ultrasonic velocity at 1 and 2 MHz and there are no significant change in ultrasonic velocity observed in the frequency range of 3 and 5 MHz.(SK Mahammad Ali & Basharith Ali 2014). The characteristic of the suitable transducer is investigated based on the transducer capacity

and reliability to transmit and receive signal through any liquid material. The attenuation occurs with any type of signal, whether digital or analog. Attenuation is a natural consequence of signal transmission over long distances. Sound absorption refers to the process by which a material, structure, or object takes in sound energy when sound waves are encountered, as opposed to reflecting the energy (James A. Zagzebski, 1996). Technique for the real-time density to monitor the ultrasound velocity are further classified techniques into Multiple Reflection Method (MRM), Reference-Reflection Method (RRM), Transmission Reflection Method (TRM), and Angular Reflection Method (ARM). ARM and RRM are proven to be suitable for highly sound absorbing liquids, however they require calibration measurements (Hoche, Hussein, & Becker, 2013).

1.2 PROBLEM STATEMENT

In many applications of liquid identifications, it is necessary to identify the physical properties of the materials and the changes in the physical properties during the entire process (Sadat, Singh, & Dwivedi, 1995). However, the existing methods requires sample preparation where the sample been blended with other chemicals before proceed, resulting the liquid been investigated cannot be defined in terms of the liquids standoff physical properties, also the liquid itself after been mixed with other chemicals cannot be used by consumers, and the methods also requires the experiment to be conducted in the lab (Waskitho, Lukitaningsih, & Rohman, 2016). To make situation worse, some of the techniques showed the results on the changes of physical properties were rather subtle although adequately explained on the chemical transformation, and requires more than a day on the sample preparation due to the crystallization up to -60°C and heating up to 60°C (Marina, Che Man, Nazimah, & Amin, 2009).

Therefore, the new method of using ultrasound wave to read the time of flight signal when the signal travel through the oil medium and received back by the transducer should be much more simple methods and requires less sample preparations. The sample preparation in this research does not require any blending or solidification methods. The TDC1000-TDC7200EVM module is light-weighted and easy carry, which means the experiments can be conducted outside the lab while maintaining the control on barometric pressure and humidity condition. Meanwhile, for the data signal analysis using the MatLab software, the time required is almost in real-time.

1.3 RESEARCH OBJECTIVES

In order to solve the previously outlined problem, this research is aimed at achieving the following objectives:

- i. To identify the time of flight signal on pure types of edible oils.
- ii. To analyze the characteristics of the different ultrasound reflection frequency signal.
- iii. To determine the effect of temperature on the different characteristics of pure olive oil and corn oil to velocity and time of flight signal.

1.4 RESEARCH METHODOLOGY

The general process of the research can be divided into four stages, where the first stage is literature review on the findings, identifying and determination methods available for the adulteration in liquid material. All the research studies were compared and the method which is more suitable for the current modern needs are studied. Second stage, the research material was studied and the different types of oil frequency were analyzing to either can be differentiated from one another. Third stage, the details on the methods

on distinguishing between the different frequencies and the time of flight methods were studied. However, the frequency was studied based on one type of oil with different temperature.

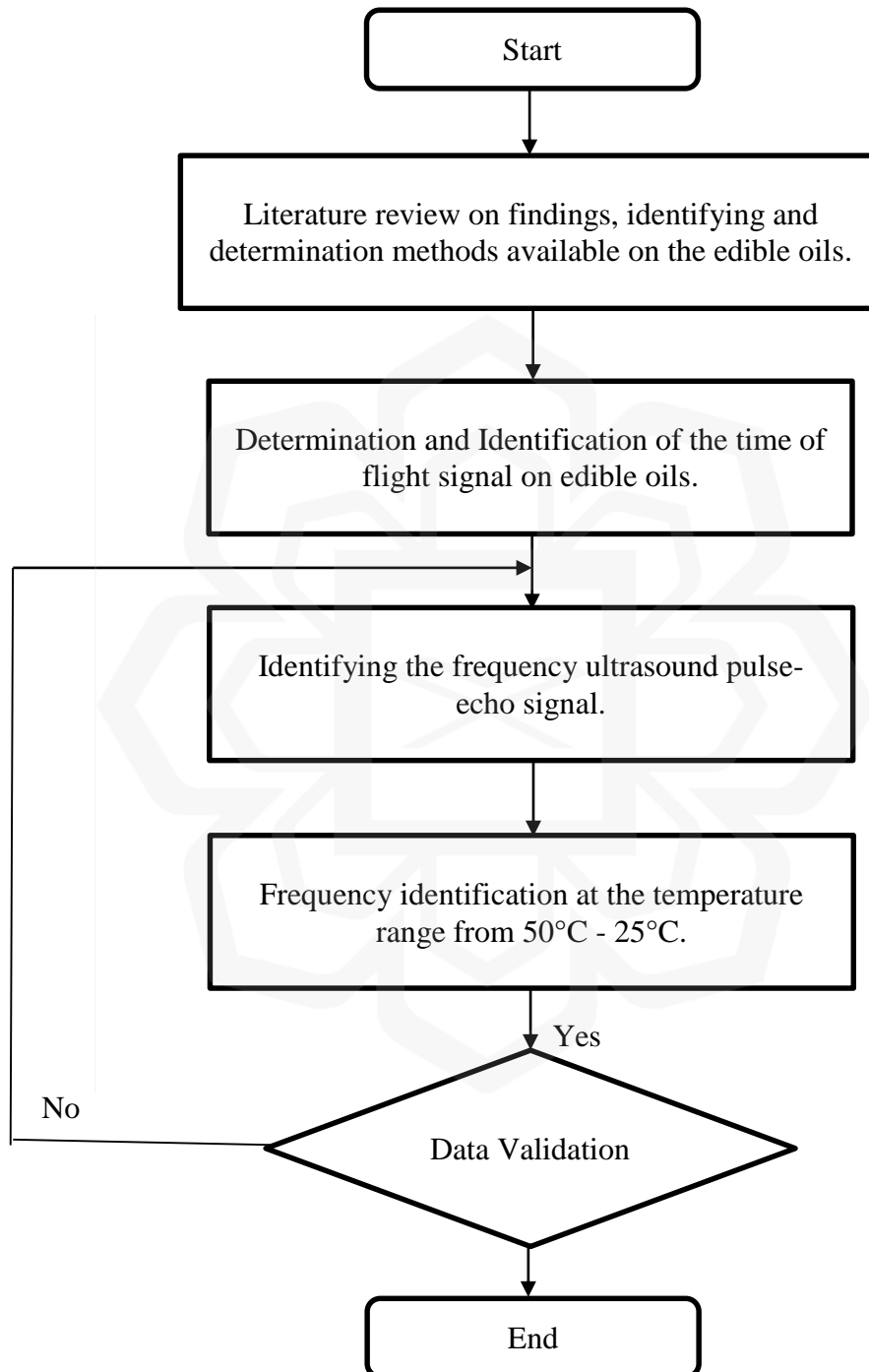


Figure 1.1: Research Flowchart.

Figure 1.1 shows flowchart of the research methodology used in this research from the beginning of the research study until the end of research validation. The findings of research done based on the ultrasound signal as a method to determine the frequency of liquid using the pulse-echo method, was explained further in the research. The last stage was the validation on the entire process flow either proven to differentiate the frequency or failed to do so.

1.5 SIGNIFICANCE OF THE STUDY

This study will contribute on the consumer's awareness based on the findings derived to carefully check on the quality of pure corn oil before making the rush decision on buying them from the market, while the methods studied in the research is practically can be commercialized. The research is useful in detecting the oil with irregularity in the pure corn oil and the irregular frequency can be proven as adulterated corn oil.

1.6 LIMITATIONS OF THE STUDY

The research is limited on pure and unadulterated olive oil and corn oil bought at available market. The study focus on the effect of manipulating the temperature range between 25⁰ C – 50⁰ C, while keeping the humidity and barometric pressure constant on the velocity and ultrasound reflection signal of pure and unadulterated corn oil. There should be wider range of temperature investigated. The various types of other edible oils should be considered, and purchased world-wide.

1.7 THESIS OUTLINE

Chapter 1 is an introduction that describes the direction of the research by providing the background of different types and benefits of high graded oils, intended application,

research-related terms, problem statement of the research, objectives of the research, research methodology and describe the limitations of the study. Chapter 2 gives an extant literature of previous technique and method varied in determines the different types of liquids and related technique using time of flight signals. Chapter 3 describes the research methodology that adopted in this research study. Each stage in the proposed methodology is discussed in details. Chapter 4 discussed the result of proposed method. This thesis concludes with a summary of the research contributions and directions for improvements on future research in Chapter 5.



CHAPTER TWO

LITERATURE REVIEW

2.1 INTRODUCTION

Most applications in the field of detecting adulteration in liquids foods involved the identification of the liquids first, the comparison of either the liquids are pure can determine the purity of the liquids. All the methods known require comparison of the liquids characteristics in terms of either density, velocity or any other measurements with the liquids national standards or literatures. There are various methods that are used for the detection of liquids, for example using the Fourier transform infrared technique, differential scanning calorimetry method, and using ultrasound based method, all these methods will be discussed further in the sub-chapter in Chapter 2.

After obtaining the signal and clarified that the signals are correct, the signal will proceed to be identified using mathematical or algorithm to create the model or just compare between the pure and adulterated liquids. There are a few techniques capable of identify the signal, for example partial least square methods where the system uses mathematical algorithm to create a model signal then used the model signal to compare between other types of liquids or adulterated liquids. However, the methods require input and output signal with the complicated mathematical formulae. Some literatures used the ultrasound velocity to differentiate the types of liquids, which means once the signal is obtained, the use of mathematical formulae, to calculate the difference. Other methods using the time of flight, which can be directly obtained when the ultrasound transmit and receive the signal, directly, the difference in liquids when the signal travelled can be observed.

2.2 FOURIER TRANSFORM INFRARED SPECTROSCOPY METHOD

Based on FTIR stands for Fourier transform infrared method, chemical are commonly used for achieving the above objectives, but these methods are of a destructive nature and the material tested is rendered useless (Waskitho et al., 2016). When IR radiation is passed through a sample, some radiation is absorbed by the sample which causes the sample cannot be used by consumers and some passes through (is transmitted) (Infrared & Ftir, n.d.).

Research (Marikkar et al., 2016) compared several reports on the use of modern techniques such as FT-NIR spectroscopy, ^1H and ^{13}C nuclear magnetic resonance (NMR) spectroscopy, FT-Raman spectroscopy, isotope ratio mass spectrometry for the detection of various food adulteration practices. Based on the research, FT-NIR spectroscopy has already been recognized for its uses in the measurement of adulterations in various types of food lipid for example milk, coconut oil, olive oil, etc. The potential applications of FT-Raman spectroscopy in detection of virgin olive oil adulterations by pomace, soybean, and corn oils have also been highlighted in the research. As ^{13}C NMR has been recognized as a valuable tool for analysis of the most abundant fatty acids of various oils, there has been a growing interest among researchers to use it to detect adulterations of various types of edible oils. However, there are disadvantages of the system description on the research. The power of IR spectroscopy to probe the chemical bonds of a sample is largely lost if the sample is atomized during collection with accompanying loss of initial molecular structure. The system is useful on the largely limited to samples that have suffered lesser amounts of alteration.

Moreover, the system of using infra-red spectroscopy requires the samples to be laborious which is opposites of the intention on the research studies which requires the

system to be able to move places. A research study to assess the effectiveness of Fourier transform infrared (FTIR) spectroscopy in detecting adulteration of virgin coconut oil with palm kernel olein as a potential adulterant (Sheikh et al., 2007). The researcher studied on virgin coconut oil showed that the virgin coconut oil has lower lipid levels in serum and tissues, while possesses high potential in protecting low-density lipoprotein against oxidative stress induced by physiological oxidants. Apart from that, coconut oil is also well known for its high content of medium-chain triacylglycerols. Based on the studied, virgin coconut oil with palm kernel olein were investigated using the technique of FTIR spectroscopy combine with the partial least square (PLS) to differentiate and quantify these two type of oils which greatly resembles one another in chemical composition. The virgin coconut oil was blended with palm kernel olein oil with the adulteration ratio of 1:50%, then, the virgin coconut oil was blended with varies type of vegetables oil (canola, extra virgin olive, walnut, sunflower, corn, sesame, soybean and grapeseed).

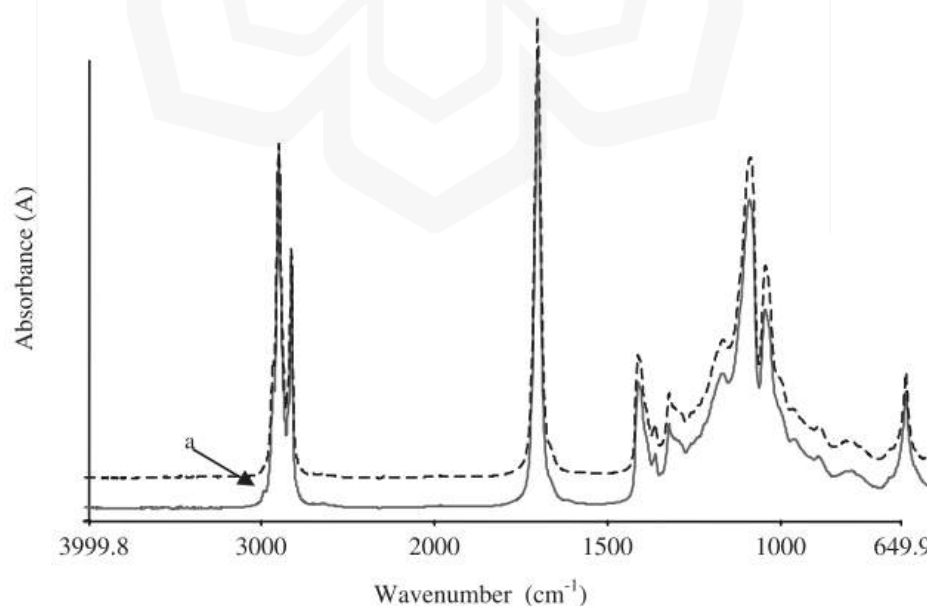


Figure 2.1: Typical spectra of virgin coconut oil and palm kernel olein (Sheikh et al., 2007).