UNIVERSITI PUTRA MALAYSIA

FIBER OPTIC TECHNIQUE FOR DETERMINATION OF MOISTURE CONTENT IN HONEY

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By

NURSAKINAH MOHAMAD IBRAHIM

Thesis Submitted to the School of Graduate Studies, Unversiti Putra Malaysia, in Fulfillment of Requirements for the Degree of Master of Science
June 2015

DEDICATION

Special thanks:
To my beloved Father, Mother, Sisters
and Friends.

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

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June 2015

Chairperson: Associate Professor Dr. Zulkifly Abbas, PhD.
Faculty: Science.

This thesis presents a critical study on the application of fiber optic technique as a fast and accurate method to determine moisture content in honey based on reflection, transmission and absorption properties in the wavelength between 300nm and 800nm. Measurements were conducted using an Ocean Optics USB4000 UV-VIS-NIR spectrometer. The honey samples used in this work were from various different honey producers. The actual moisture content was found from standard oven drying method. Moisture content of fresh honey was approximately 14.3%. All the fibre optic measurements suggest strong presence of NADH and Flavins at approximately 340 nm and 480 nm, respectively. However, reflection measurement was imprecise due to the translucency of diluted honey. In contrast absorption measurement results for all honey samples of various percentages of moisture content were distinctively unique. Maximum absorbance was found for samples with moisture content 47.5% at all wavelengths due to high concentration of sugar, crystallization. A finite element method (FEM) was also used to calculate the absorbance in fresh honey. The FEM modeling was implemented using COMSOL Multiphysics version 3.5 software. The electric field distribution trough out fiber and sample were visualized to study the electric field pattern at different wavelengths and a comparison of energy levels trough honey and air were studied. Good agreement between measured and calculated results was obtained for fresh honey. Hence, the accuracy of predicting moisture content using fiber optic technique of honey was obtained. Good linear relationship was found between absorbance and moisture content of honey within the range of 430nm until 495nm where the board band excitation of Flavin occurs. However, for all other wavelengths, only polynomial second order relationships were found to give good regression coefficients due to crystallization of honey. Calibration equations have been established to predict moisture content in honey based on absorption measurement. The most accurate calibration equation was found at 460nm where the mean relative error between the true and predicted moisture content was 0.08.

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

FIBER OPTIK TEKNIK UNTUK PENENTUAN KANDUNGAN AIR DALAM MADU

Oleh

NURSAKINAH MOHAMAD IBRAHIM

June 2015

Pengerusi: Professor Madya Dr. Zulkifly Abbas, PhD.
Fakulti : Sains.

Tesis ini membentangkan kajian kritikal atas aplikasi teknik gentian optik sebagai satu kaedah yang cepat dan tepat untuk menentukan kandungan air dalam madu berdasarkan ciri-ciri refleksi, penghantaran dan penyerapan dalam panjang gelombang antara hinga 300 nm dan 800 nm. Pengukuran telah dijalankan menggunakan spektrometer Ocean Optics USB4000 UV-VIS-NIR. Sampel madu yang digunakan dalam kerja ini adalah dari pengeluar madu yang berbeza. Kandungan air sebenar didapati daripada kaedah pengeringan ketuhar standard. Kandungan air madu segar adalah lebih kurang 14.3%. Semua ukuran gentian optik mencadangkan kehadiran kukuh NADH dan Flavins pada kira-kira 340 nm dan 480 nm, masing-masing. Walau bagaimanapun, pengukuran refleksi adalah tidak tepat kerana kelutsinaran madu yang dicairkan. Sebaliknya keputusan pengukuran penyerapan untuk semua sampel madu daripada pelbagai peratusan kandungan air adalah unik tersendiri. Penyerapan maksimum telah dijumpai untuk sampel dengan kandungan air 47.5% pada semua jarak gelombang disebabkan kepekatan gula yang tinggi, penghabluran. Satu kaedah unsur terhingga (FEM) juga digunakan untuk mengira peratusan penyerapan dalam madu segar. Permodelan FEM telah dilaksanakan menggunakan versi COMSOL Multiphysics perisian 3.5. Pengagihan medan elektik melalui kabel serat dan sampel telah digambarkan untuk mengkaji corak medan elektrik dengan panjnag gelombang yang berbeza dan perbandingan tenaga antara madu dan udara telah dikaji. Perjanjian yang baik antara keputusan diukur dan dikira telah diperolehi bagi madu segar. Dengan itu, ketepatan meramal kandungan air menggunakan teknik gentian optic dalam madu telah diperolehi. Hubungan linear yang baik didapati antara penyerapan dan kandungan air madu dalam julat 430 nm hingga 495 nm di mana pengujaan boardband bagi flavin berlaku. Walau bagaimanapun, bagi semua jarak gelombang lain, hanya polinomial hubungan peringkat kedua didapati memberikan pekali regresi baik kerana penghabluran madu. Persamaan penentukuran telah dijalinkan untuk meramalkan kandungan air dalam madu berdasarkan pengukuran penyerapan. Persamaan penentukuran paling tepat didapati pada 460 nm di mana ralat relatif min antara kandungan air yang benar dan kandungan air yang diramalkan adalah 0.08.

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I certify that a Thesis Examination Committee has met on 19 June 2015 to conduct the final examination of Nursakinah Mohamad Ibrahim on her thesis entitled "Fiber Optic Technique for Determination of Moisture Content in Honey" in accordance with Univerisiti Putra Malaysia [P.U.(A)106] 14 March 1998. The Committee recommends that the candidate be awarded the degree of 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 ABBREVATIONS

UV	Ultraviolet
VIS	Visible light
NIR	Near-infrared
E	Electric field
H	Magnetic field
FEM	Finite Element Method
COMSOL	COMSOL Multiphysics®
HMF	Hydroxymethylfurfural
NADH/NAD+	Nicotinamide Adenine Dinucleotide
NM	Nicotinamide
FAD	Flavin Adenne Dinucleotide
PDD	Photodynamic
FD	Fluorescence detection
FDM	Finite Difference Method
MoM	Method of Moment
CSM	Charge Simulation Method
h	Plank's constant
v	The frequency of the photon
λ	Wavelength
c	Speed of light
I_o	Incident light
I_t	Transmitted light
T	Transmission
E	Energy
A	Absorbance
l	Cell length
R	Reflectance
I_r	Reflected light
Γ	Reflection coefficient
E_i	Electric field of incident waves
E_r	Electric field of the reflected waves at the interface
E_t	Electric field of the transmitted waves at the interface
η_1	Complex impedance of the medium 1
η_2	Complex impedance of the medium 2
σ	Conductivity
H_r	Reflected magnetic field
H_t	Transmitted magnetic field
j	Imaginary unit
ω	Angular frequency
μ	Permeability
ε	Complex permittivity
N	Number of elements (NOEs)
V_e	Element potential typically approximated with polynomial equation
n	The number of nodes
α_i	The element shape function
V_{ei}	Potential at node i
$C^{(e)}$	Element coefficient matrix.

f	Node with free potentials
p	Node with predetermined potentials
$[K]$	Sparse matrix
$[b]$	Excitation matrix
S_λ	Sample intensity at wavelength λ
D_λ	Dark intensity at wavelength λ
R_λ	Reference intensity at wavelength λ
m_{wet}	Weight of the sample before oven dry
m_{dry}	Weight of the sample after oven dry
RF	Radio frequency
CRISP	Center for remote imaging, sensing and processing
PTFE	Polytetrafluoroethylene
P1	Port 1
P2	Port 2

CHAPTER 1

INTRODUCTION

The Codex Alimentarius define honey as the unfermented, natural sweet substance produced by honeybees from nectar of blossoms or from secretions of living parts of plants, which honeybees collect, transform and combine with specific substance of their own, store and leave in the honey comb to ripen and mature. Honey shall not have any objectionable flavor, aroma or taint absorbed from foreign matter during its production, harvesting, processing and storage and shall not contain natural plant toxins in an amount that may constitute hazard to health (CBI MARKET SURVEY, 2006). Honey has been widely use since ancient time. It is used as food, and medicine such as dressing wound and inflammations. Honey is known as a natural remedy for both internal and external bodies. It is also found that honey is an antibacterial agent that rapidly clears infections and promotes healing (Davis, 2005). The quality of honey has become more vital as demands and prices are getting higher. One of the possible abuses is to increase the volume by adding water. The production of honey is similar to organic production that has to follow the rules that has been setup by European Union. The rules are as follows (CBI MARKET SURVEY, 2006):

- Crops on which the bees has feed may not been chemically treated
- Bees should be able to survive harsh times (winter) on self-produced honey and therefore may not be fed sugar to increase honey production
- There may not be any airports or main road near bee hives
- Diseases may not be treated with veterinary medicine, but only with a limited number of organic substances
- Bees may not be pacified during the harvest of the honey

Figure 1.1 and figure 1.2 show the world's major exporters and importers. Honey has become a demanding product internationally due to its uses and benefits in areas such as food and medicine and the authenticity, a natural product with no chemicals and preservatives. Economically, the honey industry faced serious issue on its long term viability and there is a high competition in honey industry globally. Honey industry has been experiencing difficulties in poor profitability and declining since 1970's (Commonwealth of Australia, 2008).

Top Exporters	2009 Value	Share
China	$ 284,064,882	11.8%
Germany	$ 212,519,898	8.9%
Mexico	$ 164,486,793	6.9%
Brazil	$ 134,944,059	5.6%
New Zealand	$ 117,387,647	4.9%
Spain	$ 116,734,881	4.9%
Hungary	$ 114,880,765	4.8%
India	$ 87,560,291	3.7%
Canada	$ 81,491,036	3.4%
Vietnam	$ 74,327,554	3.1%

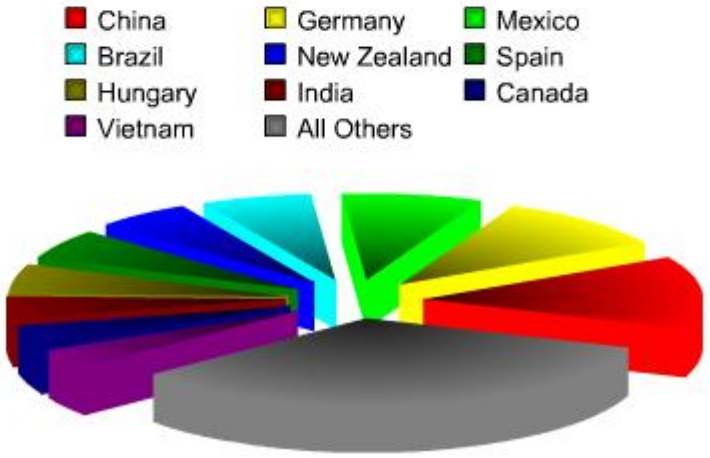

Figure 1.1: World's major exporter of honey
(Source: Thompson, 2012)

Top Importers	2009 Value	Share
Germany	$ 566,730,846	23.6%
United States	$ 383,484,376	16.0%
United Kingdom	$ 209,248,442	8.7%
France	$ 164,804,186	6.9%
Japan	$ 143,605,160	6.0%
Italy	$ 107,259,552	4.5%
Belgium	$ 82,810,198	3.5%
Saudi Arabia	$ 63,562,950	2.7%
Netherlands	$ 60,432,528	2.5%
Spain	$ 55,354,508	2.3%

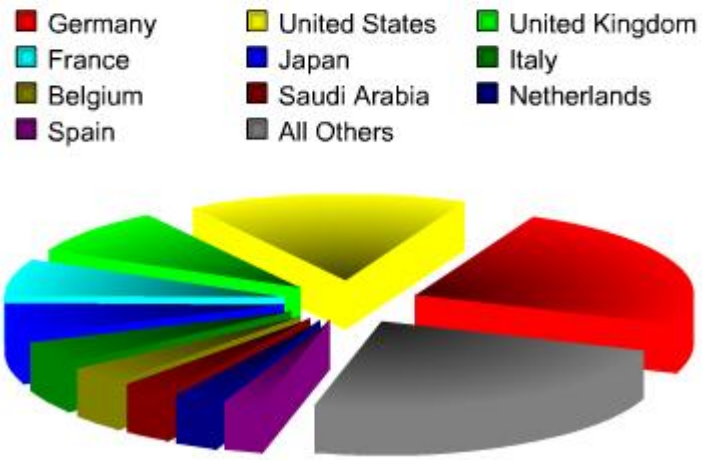

Figure 1.2: World's Major Importers of Honey
(Source: Thompson, 2012)

1.1 Composition of Honey

Different types of flowers produce different types of honey. Honey bees are the types of bees that work to collect nectar from flowers to their hives and turn it into honey. Thus, different countries or district produce different types of honey. In this research, Australian honey is used. These honeys may vary in mineral quantity and content, water content, colour and etc. (White, Reithof, Subers, & Kushnir, 1962). Although it may vary, there is a general honey composition especially in high

content of sugar. The general composition of honey is as following in Table 1. As can be seen honey has a high percentage of sugar with a low percentage of water. This high content of sugar will result in crystallization. Crystallization is a natural honey phenomenon. High content of sugar with minimum water content spontaneously separates out of the supersaturated honey solution. Glucose becomes glucose monohydrate and crystalize (Berg, 1998). Water content is one of the most important elements in determining the quality of honey.

Table 1: Average Composition of Honey (White, Reithof, Subers, & Kushnir, 1962)

Component	Average	Standard Deviation	Range
Water (%)	17.2	1.5	13.4 – 22.9
Fructose (%)	38.2	2.1	27.2 – 44.3
Glucose (%)	31.3	3.0	22.0-40.7
Sucrose (%)	1.3	0.9	0.2-7.6
Maltose (%)	7.3	2.1	2.7-16.0
Higher Sugars (%)	1.5	1.0	0.1-8.5
Free acid (%)	0.43	0.16	0.13-0.92
Lactone (%)	0.14	0.7	0.0-0.37
Total acid (%)	0.57	0.20	0.17-1.17
Ash (%)	0.169	0.15	0.020-1.028
Nitrogen (%)	0.041	0.026	0.000-0.133
pH	3.91		3.42-6.10
Diastase value	20.8	9.8	2.1-61.2

1.2 Fiber Optics

1.2.1 Electromagnetic Wave propagation

Optical application has grown tremendously in every scientific area which has proven its reliability. Hundreds of analytical method involving optical application can be found in literature. In optical application involves ultraviolet (UV), visible light (VIS), and near infrared (NIR) sections in the electromagnetic spectrum. Electromagnetic radiation consists of electric and magnetic field components that are perpendicular to each other as shown in Figure 1.1. Characterization of electromagnetic radiations is based on its range of wavelengths. Light is a part of the electromagnetic spectrum as shown in Figure 1.2 which consists of microwave, infrared, ultraviolet, x-rays, gamma, visible light and etc. Light can exhibit both a wave theory and a particle theory at the same time. Much of the time, light behaves like a wave. Light waves are also called electromagnetic waves because they are made up of both electric (E) and magnetic (H) fields. Electromagnetic fields oscillate perpendicular to the direction of wave travel, and perpendicular to each other. Light waves are known as transverse waves as they oscillate in the direction traverse to the direction of wave travel.

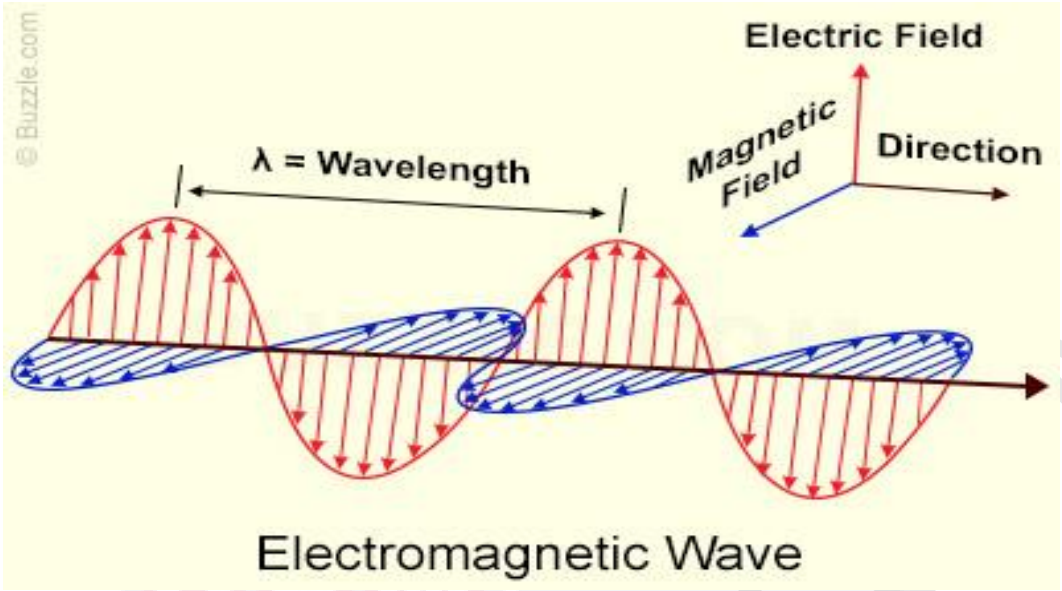

Figure 1.3: An electromagnetic wave showing its electric field and magnetic field propagating.
(Source: Phatak, 2014)

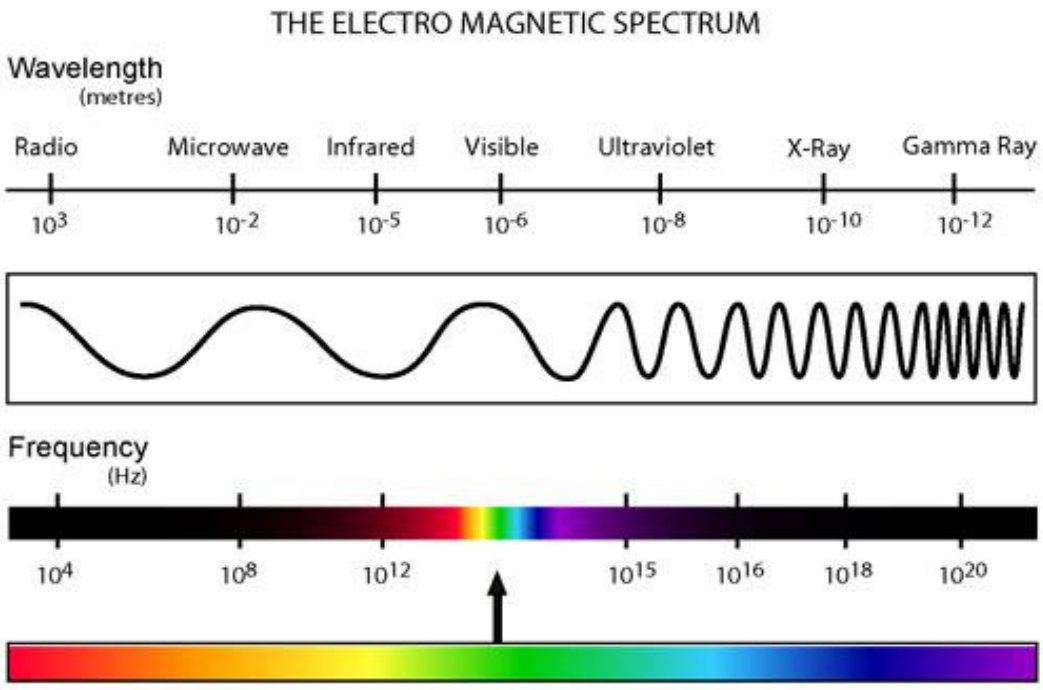

Figure 1.4: The Electromagnetic Spectrum
(Source: Shapley, 2012)

1.2.2 Optical Fiber Sensor

There are many types of sensors that are offered with fiber optics such as to measure pH, temperature, viscosity, humidity, fluorescence, gas and etc. Fiber optics use light waves parameters like light intensity, phase or polarization of light. Fiber optics sensors have grown tremendously for the past few decades due to its benefits and advantages. Optical fiber offers attractive characteristics such as (Omar & MatJafri, 2009; Ghetia, Gajjar, & Trivedi, 2013):

- absolute measurement
- immunity to electromagnetic interference
- excellent resolution and range
- passive operation, intrinsically safe
- water and corrosion resistant
- rugged, small size and light weight
- multiplexed in parallel or in series
- modest cost per channel
- They are easy to implement in any structure due to their small size and cylindrical geometry
- Inability to conduction of current
- Robust to environment
- High sensitivity
- Remote sensing capability

1.3 Problem Statement

This problem has encouraged adulteration in honey production. Adulteration of honey was done to increase the volume of honey in order to gain higher profit. Few common materials are added into honey to increase its volume such as water, glucose, starch and molasses (El-Bialee & Sorourb, 2011). These materials are well blend with honey that it is impossible to see any differences or effect on honey with rough eyes.

In this research, adulteration with addition of water in honey is in focused. It is a common agenda in agriculture to review its quality by determining it moisture content. High moisture content can lead to spoilage and overpricing. This alteration will affect the quality of honey in terms of the composition of honey that has its effect especially on healing. This will affect consumers' trust on the benefits of honey because it is impossible for consumers to identify adulteration with naked eye. A standard method to determine moisture content of honey is oven drying method. The disadvantage of this method is that the sample or object tested will be destroyed. Each product varies in moisture content. A common method known widely in honey industry is to determine moisture content of honey brix refractometer. This is primarily because it is inexpensive and readily available. However this method has its disadvantage on its accuracy. Brix refractometer measures the moisture content of honey based on established relationship between sucrose and refractive index. The actual measurement taken by brix is the ratio between weight of sucrose and water solution. The accuracy of the method is questionable with the existence of fructose and glucose which much higher than the composition of sucrose in honey according to Table 1.1. A standard Brix refractometer is not able to give the accurate reading of sucrose since fructose,

glucose and sucrose are dissolve sugar. In addition, the relationship of fructose and glucose with refractive index differs from sucrose (MISCO Digital Honey Refractometer, 2008).

In this project, a fiber optic method is proposed to determine the moisture content of honey via three parameters, reflection, transmission and absorption. One of the reasons this technique is chosen is because the existence of colour amber that will have a reaction with visible light region. Diluted honey has reduced its composition that will have its effect of reflection, transmission and absorption. The values of the parameter react to the quantity of the element in this case the element that gives the colour amber to honey. Different results are expected to be shown between different moisture content.

Fiber optic technique is chosen to conduct the effects on reflection, transmission and absorption on UV/VIS spectrum with different moisture content ranging from 14%-90%. Honey can be perceived by human eyes with the colour amber (Anon, 1985). There is a range of colour that is characterized with types of honey. The range goes by water white to dark amber. The colour of honey is due to the present of flavin. In electromagnetic spectrum, visible wavelength range is the only range that can be perceived by human eyes. Plus, fiber optic is known to have high sensitivity and fast.

A simulation process using Finite Element Method (FEM) via COMSOL Multiphysics is used to compare between measured results and simulated results. FEM is chosen to see the electromagnetic field interaction between different samples. A study on field distribution can be analyzed.

1.4 Objectives

1. To determine the effect of moisture content on the reflection, transmission and absorption properties of honey at visible wavelengths 300 nm to 800 nm.
2. To establish calibration equations to predict moisture content in sample.
3. . To visualize the electric field distribution of the sensor loaded with samples
4. To calculate Finite Element Method in the calculation of absorption of honey in the visible spectra range.

1.5 Scope of the Study

The aim of this study is to choose and analyse the best method among these three parameters of reflection, transmission and absorption in determining the moisture content of honey. Chapter 2 discussed about various technique in determination of moisture content of honey. In addition, an overview of optical application and fibre optic sensors used to analyse the effectiveness of fibre optic technique.

Chapters 3 conversed on the theoretical background of using fibre optic technique. Theory of reflection, transmission and absorption reacting on materials is discussed and the calculations of these parameters were calculated from the raw data of intensity. Technique on calculating finite element method and obtaining the result of absorption via COMSOL Multiphysics were presented in this chapter.

Chapter 4 is the methodology of this research. Chapter 4 expressed the procedures done to run these measurements and simulation via COMSOL Multiphysics. Sample preparations and measurement setup of reflection, transmission and absorption were displayed. Nonetheless, technique on setting proper calibration is discussed. Five

steps of running the simulation of sensor via COMSOL Multiphysics were illustrated and explained.

Chapter 5 begins with the results of reflection measurement, followed by transmission and absorption measurement of honey with different moisture content of honey at wavelength 300 nm to 800 nm. Results were observed and discussed in details. Calibration equations were found and discussed to enhance the accuracy of fiber optic technique. A comparison of data between simulated and measured results was illustrated.

Finally, the contributions and recommendations for future study were described in Chapter 6.

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