CAPABILITY OF AgSiN AND SU-8 FILM IN PROTECTING SILVER-BASED SURFACE PLASMONS RESONANCE (SPR) TECHNIQUE FOR HONEY WATER CONTENT DETECTION

MARLIANA BINTI JAAFAR

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DEDICATION

Special for my beloved family especially my father and mother,
Jaafar bin Mohd Nor and Raedah binti Md Dehan
And to my supportive husband,
Muhamad Asyraf bin Mohd Hamin.

Also, to my encouraging supervisor,
Assoc. Prof. Dr. Maisara binti Othman

Thanks a lot for their patient, kindness, and cooperation.
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May God bless all of them.

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ABSTRACT

Stingless bee honey is a natural sweetener product that has greater nutritional and medicinal values compared to the other honey bees. Due to the higher market demand and limited production levels of honey, stingless bee honey becomes a high-value commercial food product targeted for adulteration, which can cause a loss of natural therapeutic value in honey. Besides, the higher water content in stingless bee honey will promote the presence of yeast during the fermentation process, which can degrade the quality and shorten the shelf life of honey. Therefore, an optical water content detection is employed using a silver-based of prism-coupled SPR technique. Four models of SPR sensing structures are proposed in this research. The behaviour and effect of SU-8 photoresist film and the silver-silicon nitride (AgSiN) as an additional sensing layer are investigated to protect the silver film from erosion and minimize the oxygen element on the sensing surface. Based on the results, the Cr – Ag SPR structure effectively detects the adulterated honey in terms of water content percentage. The lamination of SU-8 film on the Cr – Ag layer can protect the silver surface from degradation and improve the performance of SNR, detection accuracy, and figure of merit at the range of 0.81140 to 5.13352, 1.24953 (1/°) to 1.78571 (1/°), and 0.30549 (1/%) to 0.43657 (1/%), respectively. The smallest full width at half maximum (FWHM) value is produced also at Cr – Ag – (SU-8) structure between 0.8003° to 0.5600°. In the meantime, the existence of the AgSiN layer in the Cr – Ag – AgSiN – (SU-8) sensing structure matches the resonance angle and altered the minimum reflectivity values by only 5.26% after 24 hours of testing. Moreover, the deposition of the AgSiN layer has the potential to reduce the formation of silver oxide with the lowest atomic oxygen percentage of 9.04% and the smallest bandgap size of 3.868 eV. It indicates that the AgSiN layer is practicable to increase the absorption in the sensing samples.

ABSTRAK

Madu lebah kelulut adalah produk pemanis semulajadi yang mempunyai nilai perubatan dan pemakanan lebih baik berbanding madu lebah yang lain. Oleh kerana permintaan pasaran yang tinggi dan tahap pengeluaran madu yang terhad, madu lebah kelulut telah menjadi produk makanan komersial bernilai tinggi yang disasarkan untuk pemalsuan dan boleh menyebabkan kehilangan nilai terapeutik semulajadi dalam madu. Selain itu, kandungan air yang tinggi dalam madu lebah kelulut akan menggalakkan kehadiran yis semasa proses penapaian, sekali gus merendahkan kualiti dan memendekkan jangka hayat madu. Oleh itu, pengesanan optik terhadap kandungan air di dalam madu dilaksanakan dengan gabungan prisma berasaskan perak menggunakan teknik SPR. Empat model struktur pengesanan SPR dicadangkan dalam penyelidikan ini. Ciri-ciri dan kesan filem fotoresist SU-8 dan perak-silikon nitrida (AgSiN) sebagai lapisan pengesanan tambahan diuji untuk melindungi filem perak daripada terhakis dan meminimumkan unsur oksigen. Berdasarkan hasil kajian, struktur SPR Cr – Ag berjaya mengesan madu yang dipalsukan berdasarkan peratusan kandungan air. Laminasi filem SU-8 pada lapisan Cr – Ag telah melindungi permukaan perak daripada degradasi dan meningkatkan prestasi SNR, ketepatan pengesanan, dan angka merit pada julat masing-masing 0.81140 hingga 5.13352, 1.24953 (1/°) hingga 1.78571 (1/°), dan 116.043 RIU^{-1} hingga 165.838 RIU^{-1}. Nilai terendah *full width at half maximum* (FWHM) juga dihasilkan pada struktur Cr – Ag – (SU-8) antara 0.8003° hingga 0.5600°. Sementara itu, kehadiran lapisan AgSiN dalam struktur Cr – Ag – AgSiN – (SU-8) mempunyai persamaan pada sudut resonans dan mengubah nilai pemantulan minimum sebanyak 5.26% sahaja selepas 24 jam pengujian. Tambahan pula, pemendapan lapisan AgSiN berpotensi mengurangkan pembentukan oksida perak dengan peratusan atom oksigen terendah sebanyak 9.04% dan saiz jurang jalur terkecil iaitu 3.868 eV. Ini menunjukkan bahawa lapisan AgSiN boleh digunakan untuk meningkatkan penyerapan dalam sampel pengesanan.

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LIST OF SYMBOLS AND ABBREVIATIONS

%	-	Symbol Percentage
Λ	-	Grating period
μ	-	Relative permeability
μ_0	-	Magnetic permeability of the vacuum
µm	-	micrometer
µW/%	-	Microwatt per percentage
2D	-	Two-Dimensional
3D	-	Three-Dimensional
A	-	Ampere
a.u.	-	Unitless
ADC	-	Analog-to-digital converter
ADDR	-	Address
AFM	-	Atomic force microscopy
Ag	-	Silver
AgSiN	-	Silver-silicon nitride
A_i	-	Amplitude of incident light
AlN	-	Aluminium nitride
A_p	-	Angle of the prism
ATR	-	Attenuated total reflectance
B	-	Magnetic flux density
b	-	Symbol medium
c	-	Speed of electromagnetic wave in free space
C3	-	Dicotyledonous
C4	-	Monocotyledonous
cm	-	centimeters
Cr	-	Chromium

d	-	Dielectric region
D	-	Electric displacement
D6	-	Digital six
DA	-	Detection accuracy
DI	-	Deionized
DLC	-	diamond-like carbon
DNA	-	Deoxyribonucleic acid
d_o	-	Diffraction order
E	-	Electric field
e	-	Electron charge
E_0	-	Amplitude of the electric field
EDS	-	Energy dispersive spectroscopy
eV cm^{-1}	-	electron volt per centimeter
eV.s	-	electron volt in second
ε	-	Dielectric function of medium
ε_0	-	Permittivity of vacuum
ε_b	-	Dielectric constant of metal or sensing layer
ε_d	-	Dielectric constant of dielectric region
ε_m	-	Dielectric function of metal region
FBG	-	Fiber Bragg grating
FESEM	-	Field-Emission Scanning Electron Microscope
FODS	-	Fiber optic displacement sensor
FOM	-	Figure of merit
FTIR	-	Fourier transform infrared
FWHM	-	Full-width at half maximum
GaN	-	Gallium nitride
GC	-	Gas chromatography
GC-MS	-	Gas chromatography coupled with mass spectrometry
Ge	-	Germanium
GND	-	Ground
H	-	Magnetic field
h	-	Planck’s constant

H_0	-	Amplitude of the magnetic field
HFCS	-	High-fructose corn syrup
HMF	-	Hydroxymethyl furfural
HPAEC-PAD	-	High-performance anion-exchange chromatography with Pulsed Amperometric Detection
HPLC	-	High-Performance Liquid Chromatography
I^2C	-	Inter-integrated circuit
InN	-	Indium nitride
IPA	-	Isopropyl ethanol
j	-	Symbol medium
J_{ext}	-	External current
k	-	Wave number
$\mathbf{k}$	-	Wave vector
KFT	-	Karl Fischer Titration
LC	-	Liquid chromatography
m	-	Metal region or metal film
m/s	-	meter per second
m_e	-	Electron mass
MEKC	-	Micellar Electro Kinetic Capillary Chromatography
mm	-	millimeters
MoS_2	-	Molybdenum disulfide
$MoSe_2$	-	Molybdenum diselenide
mV	-	millivolt
mW	-	milliwatt
N	-	Electron density
N	-	Nitrogen
n	-	Refractive index value
NF	-	Noise Figure
NGA	-	Next Generation Access
NGWSPR	-	Nearly guided wave SPR
nm	-	nanometer

NMR	-	Nuclear magnetic resonance
n_p	-	Refractive index of prism
n_s	-	Refractive index of sensing medium
^{o}	-	degree
O	-	Oxygen
^oC	-	Degree Celsius
p	-	Prism
PCA	-	Principle component analysis
PGA	-	Programmable gain amplifier
pH	-	Power of hydrogen
PLS	-	Partial least square
PLX-DAQ	-	Parallax Data Acquisition
POF	-	Plastic optic fiber
p-polarized	-	Parallel polarized
r (x, y, z)	-	Position vector (*x*-axis, *y*-axis, *z*-axis)
R	-	Reflectance
r	-	Reflection coefficient
RF	-	Radio frequency
RI	-	Refractive index
RIU	-	Refractive Index Unit
R_{min}	-	Minimum reflectivity
r_{pms}	-	Amplitude reflection coefficient for prism-metal-dielectric layer
s	-	Sensing layer
S	-	Sensitivity
s/nm	-	second per nanometer
sccm	-	standard cubic centimeters per minute
SCIRA	-	Stable carbon isotope ratio analysis
SCL	-	Clock line
SDA	-	Data line
Si	-	Silicon
SiO_2	-	Silicon dioxide
SMF	-	Single mode fiber

SNR	-	Signal-to-noise ratio
s-polarized	-	Senkrecht polarized
SPPs	-	Surface plasmon polaritons
SPR	-	Surface plasmons resonance
SPs	-	Surface plasmons
SVM	-	Support vector machine
t	-	Transmission coefficient
TE	-	Transverse electric
TiO_2	-	Titanium dioxide
TM	-	Transverse magnetic
UV-Vis	-	Ultraviolet-visible
V	-	Volt
w	-	Thickness of metal film
W	-	Watt
w_c	-	Water content
WS_2	-	Tungsten disulphide
WSe_2	-	Tungsten diselenide
XRD	-	X-ray diffraction
YSI	-	Yellow Spring Instruments
δ_d	-	Skin depth of dielectric
δ_m	-	Skin depth of metal
θ_{ex}	-	External angle
θ_i	-	Incident angle
θ_r	-	Reflected angle
θ_{SPR}	-	Angle of surface plasmons resonance
θ_t	-	Refracted angle
λ	-	Wavelength of incident light
ρ_{ext}	-	External charge
ϕ	-	Phase shift
ω	-	Angular frequency
ω_p	-	Plasma frequency
ω_{sp}	-	Surface plasmons frequency
∇	-	Curl vector

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CHAPTER 1

INTRODUCTION

1.1 Overview

This chapter introduces the relevant background for stingless bee honey and the surface plasmon resonance (SPR). The basic backbone information for the research, which contains the problem statement, objectives, research questions, scope and significance of the research are introduced in this chapter. The organization of the remaining chapters is summarized at the end of this chapter.

1.2 Background of Study

Honey is a natural food sweetener processed by bees from flower nectar or the secretion or excretions of plant-sucking insects on plants' living parts. Honey's physical properties and chemical compositions depend on its environmental factors, climatic condition and geographical region that contribute to its flavor, color, and smell [1]. In addition to this, the treatment of beekeepers and the type of flora and plants from which the bees consume nectar also influence honey's physical and chemical properties [1].

In Malaysia, stingless bee honey also known as Kelulut honey is believed to have higher nutritional and medicinal values than honey from other bees [2]. Stingless bee honey is a "mother of medicine" that can act as anti-inflammatory [2], anti-bacterial [3], [4], anti-oxidant [5] and wound healing activities [6]. The excellent anti-oxidant properties in stingless bee honey make it more valuable and subsequently has higher demand. On the other hand, despite the growing market demand, stingless bee honey lacks of institutional quality standards and has limited industrial production [2].

This is due to the minimal knowledge about the products owing to the limited market distribution of stingless bee honey. Moreover, the lack of comprehensive physico-chemical data has caused the identification of adulteration activities to be more difficult.

Consequently, stingless bee honey is one of the high-value commercial food products targeted for adulteration. According to the food Codex Alimentarius standard [7], commercial honey is a pure product with no other ingredients and particular constituents being added or removed from it. However, many types of commercial honey have been adulterated with cheaper sweeteners (i.e., glucose syrup, cane sugar and corn syrup), water and other constituents to cater the higher demand and to compensate for the relatively high price of honey. In this situation, the adulteration of honey can undermine its natural therapeutic value.

To overcome these shortcomings, various analytical procedures have been introduced to appraise the authenticity of honey. These procedures include chromatographic methodology [8]–[10], stable carbon isotope analysis [11], [12], spectroscopic [13]–[15], and trace elements technique [12]. These conventional methods are useful and accurate in identifying the honey authenticity, but involving high knowledge to handle the devices, time-consuming and expensive instruments. Hence, several researchers have designed optical sensing devices for adulterated honey detection since it is simple, rapid, and chemical-free approach [16]–[20]. As shown in the graph in Figure 1.1, the optical sensors market is projected to grow at a stable pace over the forecast period. This growth is specifically contributed by the sector of consumer electronics, automotive and transportation, food, and beverage as well as industrial applications.

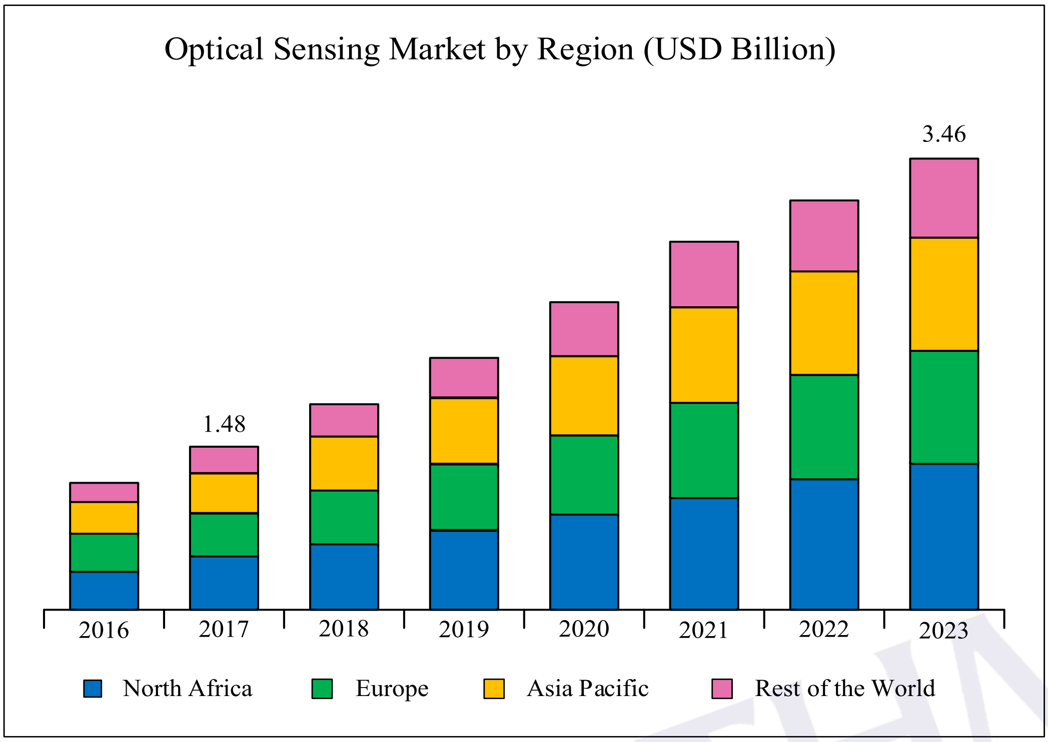

Figure 1.1: Global optical sensors market by region [21].

The optical phenomenon of surface plasmon resonance (SPR) has recently drawn huge attention among the research community due to its high-potential in optical sensing, biomedicine, and electronics. Since SPR is a non-radiative and label-free detection method, it is well-suited for numerous applications in chemical and biological sensing, including the detection of adulterated honey [20]. Surface plasmon resonance refers to the optical excitation of surface plasmons (SPs) at the interface between a metal and a dielectric. The SPR technique is based on the electromagnetic response relying on the variation in refractive index that occurred on the sensing surface due to the adsorption of the target analyte.

Metal is a chemically active and conductive material. Hence, its surface can easily oxidize, causing the plasmonic properties to deteriorate. Noble metals such as gold, silver and copper are the most commonly used for SPR applications. In this regard, even though gold possesses greater resistivity towards oxidation, silver is preferred since it exhibits higher conductivity and a sharper reflectivity spectrum than gold and copper metal. The oxidation and stability issues of plasmonic materials can be solved by protecting the metal surface with a thin and inert layer that is impermeable to water, oxygen, and other corroding agents [22]. For instance, the bimetallic layer (i.e. silver-gold layer) [23] and two-dimensional (2D) structures of graphene [24], [25] and molybdenum disulfide (MoS_2) [26], [27] have been proposed as protective

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APPENDIX A

LIST OF PUBLICATIONS

Journals:

(i) M. B. Jaafar, M. B. Othman, M. Yaacob, B. A. Talip, M. A. Ilyas, N. H. Ngajikin, N. A. M. Fauzi, "A Review on Honey Adulteration and the Available Detection Approaches", *International Journal of Integrated Engineering*, 12(2), 125-131. 2020. *(published)*

(ii) M. B. Jaafar, M. B. Othman, M.M.I. Megat Hasnan, M. Yaacob, H. Haroon, B. A. Talip, "Capability of AgSiN/SU-8 Layer on Silver-based SPR for Adulterated Honey Detection", *Advanced in Nano Research. (review)*

Proceedings:

(i) M. B. Jaafar, M. B. Othman, M. Yaacob, H. Haroon, M. A. Ilyas, A. A. Ayub, "Excitation of Surface Plasmons in Thin Noble Metallic Film of Copper, Silver, and Gold Layer", *2020 IEEE Student Conference on Research and Development (SCORED)*. 27-29 September 2020. Batu Pahat, Johor. (*published*)

(ii) M. B. Jaafar, M. B. Othman, H. Haroon, M. Yaacob, M. A. Ilyas, S. N. A. Zainurin, N. S. Ahmad, "A Simulation of Prism-based Surface Plasmon Resonance Liquid Sensing Device", *2020 IEEE 8th International Conference on Photonics (ICP)*. 12 May-30 June 2020. Kota Bharu, Kelantan. (*published*)

(iii) M. B. Jaafar, M. M. I. Megat Hasnan, M. B. Othman, N. Nayan, Z. Azman, M. Yaacob, R. Mohd Zin, "Investigation of SU-8 as Protection Layer For Prism SPR Sensor Towards Reusable Honey Adulteration Detection", *2021 IEEE International Conference on Sensors and Nanotechnology (SENNANO)*. 22-24 September 2021. Port Dickson, Negeri Sembilan. (*Published*)

(iv) N. S. Ahmad, M. Yaacob, M. B. Othman, N. H. Ngajikin, M. B. Jaafar, M. A. Ilyas, "Simulation of Polymer Multimode Interference Thermo-Optic Switch", *2020 IEEE 8th International Conference on Photonics (ICP). 12 May-30 June 2020*. Kota Bharu, Kelantan. (*published*)

APPENDIX B

LIST OF AWARD

(i) **Silver Medal in International Research & Innovation Symposium & Exposition (RISE) Festival UTHM (24th September 2019):**
M. B. Jaafar, M. B. Othman, M. A. Jabar, M. A. Ilyas, S. N. A. Zainurin. "Monitoring System for Kelulut Honey Quality Using Blynk Application."

APPENDIX C

VITA

The author was born on April 2, 1991, in Kuala Lumpur, Malaysia. She went to Rawang Integrated Boarding School, Rawang, Selangor, Malaysia for her secondary school. Then, she continue her studied at Kedah technical Matriculation College, Pendang, Kedah, Malaysia, before she pursued her degree at the University of Tun Hussein Onn Malaysia, Batu Pahat, Johor. She graduated with the B.Eng. (Hons) in Electronic Engineering in 2014. Upon graduation, she continues her study in master by research for electronic engineering at University of Tun Hussein Onn Malaysia, Batu Pahar, Johor and graduated in 2017. Her research interest during the master studied is in the scope of photonic communication engineering. Thereafter, she further her Ph.D. program at the same university in the background of optical sensing engineering.