FIBER OPTIC SENSOR FUNCTIONALIZED WITH GLUCOSE OXIDASE AND GOLD NANOPARTICLES FOR GLUCOSE AND SUCROSE DETECTIONS IN *DAUCUS CAROTA*

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DEDICATION

This thesis specially dedicated to both of my parents, Multar Masngud and Maznah Samad for their encouragements and endless supports. It is also dedicated to my three siblings, who support me financially and emotionally. Without their enormous sacrifices, I would have never completing this thesis and become the individuals as I am today.

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ABSTRACT

Tapered single mode fiber (SMF) immobilized with glucose oxidase enzyme (GOD) and gold nanoparticles (AuNP) for recognition of glucose and sucrose, respectively have been proposed in this work. Tapered fiber is fabricated using flame heating technique and is coated to improve the sensitivity of fiber based sensor. 3-(aminopropyl) triethoxysilane (APTES), GOD and AuNP are functionalized onto the tapered region of SMF through covalent interaction. The purpose of this study is to develop coated tapered fiber for determining the sensitivity and selectivity of glucose and sucrose in different concentration of glucose and sucrose solutions and in different types of *Daucus carota*. Solution concentration of 0.1 g/ml, 0.2 g/ml and 0.3 g/ml were used to differentiate the spectrum intensity of the fiber towards different amount of glucose and sucrose in the solutions. Baby carrots, imported carrots and organic carrots were used as an indicator to prove the existence of glucose and sucrose in these different types of *Daucus carota*. The experimental results demonstrate the sensitivities of GOD-immobilized fiber were 0.01045 $a.u/gml^{-1}$, 0.11415 $a.u/ml$, 0.12689 $a.u/ml$ and 0.16807 $a.u/ml$ towards glucose solution, baby carrots, organic carrots and imported carrots, respectively. Meanwhile, the sensitivities of AuNP-immobilized fiber towards sucrose solution, baby carrots, imported carrots and organic carrots were 0.00840 $a.u/gml^{-1}$, 0.000483 $a.u/ml$, 0.000698 $a.u/ml$ and 0.000777 $a.u/ml$, respectively. The slopes obtained from the graph represent the sensitivities of the fibers towards different types of testing samples.

ABSTRAK

Serat mod tunggal tirus (SMF) yang diimobilisasi dengan enzim glukosa oksidase (GOD) dan nanopartikel emas (AuNP) untuk pengiktirafan glukosa dan sukrosa, masing-masing telah dicadangkan dalam karya ini. Serat tirus dibuat menggunakan teknik pemanas api dan dilapisi untuk meningkatkan kepekaan sensor berasaskan serat. 3- (aminopropil) triethoxysilane (APTES), GOD dan AuNP difungsikan ke kawasan SMF yang meruncing melalui interaksi kovalen. Tujuan kajian ini adalah untuk mengembangkan serat tirus bersalut untuk menentukan kepekaan dan selektivitas glukosa dan sukrosa dalam kepekatan larutan glukosa dan sukrosa yang berlainan dan dalam pelbagai jenis *Daucus carota*. Kepekatan larutan 0.1 $\mathrm{g/ml}$, 0.2 $\mathrm{g/ml}$ dan 0.3 $\mathrm{g/ml}$ digunakan untuk membezakan intensiti spektrum serat terhadap jumlah glukosa dan sukrosa yang berlainan dalam larutan. Lobak bayi, wortel yang diimport dan wortel organik digunakan sebagai petunjuk untuk membuktikan kewujudan glukosa dan sukrosa dalam berbagai jenis karot Daucus. Hasil eksperimen menunjukkan kepekaan serat yang tidak bergerak GOD adalah 0.01045 $\mathrm{a.u/gml^{-1}}$, 0.11415 $\mathrm{a.u/ml}$, 0.12689 $\mathrm{a.u/ml}$ dan 0.16807 $\mathrm{a.u/ml}$ terhadap larutan glukosa, wortel bayi, wortel organik dan wortel import, masing-masing. Sementara itu, sensitiviti serat yang tidak bergerak AuNP terhadap larutan sukrosa, wortel bayi, wortel import dan wortel organik adalah 0.00840 $\mathrm{a.u/gml^{-1}}$, 0.000483 $\mathrm{a.u/ml}$, 0.000698 $\mathrm{a.u/ml}$ dan 0.000777 $\mathrm{a.u/ml}$, masing-masing. Lereng yang diperoleh dari grafik mewakili kepekaan serat terhadap pelbagai jenis sampel ujian.

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LIST OF ABBREVIATIONS

APTES	-	3-(aminopropyl)triethoxysilane
AuNP	-	Gold nanoparticles
C=O	-	Carbonyl groups
$C_6H_{12}O_6$	-	Glucose
$C_{12}H_{22}O_{11}$	-	Sucrose
ChO_x	-	Cholesterol oxidase
GOD	-	Glucose oxidase enzyme
H_2O_2	-	Hydrogen peroxide
H_2SO_4	-	Sulphuric acid
$HAuCl_4$	-	Chloroauric acid
HF	-	Hydrofluoric acid
HFCVD	-	Hot filament chemical vapour deposition
LOD	-	Limit of detection
LPFG	-	Long period fiber grating
LSPR	-	Localized Surface Plasmon Resonance
MMF	-	Multimode fiber
NaOH	-	Sodium hydroxide
NH	-	Amine groups
NH_4OH	-	Ammonium hydroxide
OH	-	Hydroxyl groups
pH	-	Power of hydrogen
PMM	-	Plastic multimode
PPMU	-	University Laboratory Management Centre
RI	-	Refractive index
SEM	-	Scanning Electron Microscopy
SiN_X	-	Silicon nitride
SMF	-	Single mode fiber
TIR	-	Total internal reflection
UN	-	United Nations
WHO	-	World Health Organization

LIST OF SYMBOLS

a.u	-	Arbitrary unit
cm	-	Centimetre
θ_c	-	Critical angle
℃	-	Degree Celsius
m	-	Gradient/Slope
g	-	Gram
μ	-	Micro
ml	-	Millilitre
mm	-	Millimetre
n	-	Nano
%	-	Percentage
s	-	Second
v/v	-	Volume per volume

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Developing high precision or more accurate sensing devices with low limit of detection (LOD) recently has become favourable in various range of applications and studies due to its advantages. Fiber optic sensors have various benefits including highly sensitive, light weight, small size, resistant to high temperature, multiplexing, and high geometrical abilities [1].

Fiber optic sensor is a device which uses optical fiber as the sensing element, or by means of carrying signals from remote sensor to electronic devices that process the signals. The excellent features of optical fibers such as finest light transmission, easy enhancement of the bio-reaction, long distance signal delivery, economical, and low attenuation allow fiber optic sensor to be widely used in the implementation of numerous application areas [1].

These features are beneficial to different potential applications in optical fiber based sensor for detection of quantities such as temperature, mechanical strain, humidity, pressure, vibration measurements, velocity, displacements, liquid level measurement [2], acceleration calibration, and also bio-chemical measurements such as sugar content and pH value [3].

For insensitive circumstances which include noise, high vibration, extreme heat, wet and ambiguous conditions, the fiber optic sensors can act predominantly. These sensors can easily fit in smaller regions and can be located perfectly wherever flexible fibers are needed. Thus, fiber optic sensors can also be designed to tolerate high temperatures [3].

Optical fiber based sensor has a fiber optic components that connects to a light source to allow detection or sensing in tight spaces or small profiles. The optical fiber typically consists of a silica core surrounded by a transparent layer of silica or plastics cladding material, with a lower refractive index (RI) than the core RI [4]. The difference in RI between the core and cladding enables them to act based on the total internal reflection principle (TIR). TIR states that when the light strikes an interface at a sufficiently oblique angle between two media, it will be totally reflected without energy loss [5].

Tapered fiber using single mode fiber (SMF) provides some excellent characteristics of fiber optic sensor. Smaller core diameter of SMF able to reduce distortion that comes from overlapping light transmission. This reason will result in SMF overcoming high signal attenuation and low transmission speeds to reduce data loss. Multimode fiber (MMF) also often used in sensing technology. However, MMF is suitable for short distance application because the multiple light that propagates along MMF tend to disperse over long distances.

In 2018, a work by Chen demonstrated a glucose sensor of both AuNP and GOD immobilized onto the surface of U-shaped optical fiber probe [6]. The fiber is first fabricated using a flame heating technique and this sensor practically works to measure different concentration of glucose solution and the fiber. From this work, it shows that when the glucose concentration increased, the refractive index of the sensor decreases and thus, spectrum wavelength shifted.

In other works, GOD has been immobilized onto an optical fiber microprobe by modifying the surface of fiber optic with APTES for bio-selective and high-sensitive glucose recognition at different concentrations. This findings proved its potential for label-free sensing capacity, excellent practicality, and higher sensitivity due to its small in size [7,8].

1.2 Problem Statement

Sugar monitoring sensor which have been designed since decades ago by electrochemical method limits the sensing applications because it is highly cost and time consuming during preparation [9]. Electrochemical method is an approach to prepare coatings by controlling voltage, electro deposition time, and concentration of the electrolyte, and monomers [10]. For the reason, this research focused on an easy fabrication and low cost for measurement of corresponding substances.

Standard optical fiber performed zero sensitivity towards its surrounding medium and cannot be utilized for specific measurement of substances because of its optical field completely confined in the fiber core. Hence, tapering and coating with enzyme and metallic nanoparticles provides several benefits including high selectivity, biocompatibility, and good chemical stability [11].

Excessive sugar intake will result in many chronic diseases that deteriorates human health including weight gain, kidney disease, mental health, tooth decay, and so on [12]. Carrots are definitely a preferable vegetable that is claimed to be a healthy food among adults and children due to their number of benefits including low risk of cancer and heart disease, decrease calorie intake, lower cholesterol levels and improved eye health [13].

However, excessive consumption of carrots over a period of time can bring in too much beta-carotene, the molecule which responsible for carrots' bright orange hue and precursor of vitamin A. Carotenemia is a condition which occurs due to carotene, a fat-soluble molecule [13]. High carrots intake tend to accumulate in the outermost layer of skin which resulting in yellow or orange pigmented skin. Therefore, this research helps to identify the sensitivity of the fiber towards different types of *Daucus carota* or carrots in order to determine the amount of sugar composition inside this corresponding substances and so avoid a high consumption of sugar.

1.3 Research Objectives

The main objective of this research is to develop a functionalized tapered SMF for glucose and sucrose detection. The specific objectives of this research includes:

(i) To optimize the tapered fiber structure for better sensing properties.

(ii) To determine and characterize the surface characteristics of immobilized tapered optical fiber.

(iii) To measure the sensitivity and selectivity of sensor towards glucose and sucrose in different type of *Daucus carota*.

(iv) To identify the sensitivity and selectivity of sensor towards different concentration of glucose and sucrose solutions.

1.4 Scopes of Research

This research is focusing on the potential of tapered fiber optic immobilized with glucose oxidase enzyme (GOD) and gold nanoparticles (AuNP) for the recognition of glucose and sucrose sensing. In this study, SMF tapered fiber with waist diameter of 20 µm is fabricated using flame heating technique. One end of the immobilized fiber is connected to halogen light source model HL-2000-LL for measurements between 360 to 2400 nm respectively. The other end is connected to the CCS175/M compact spectrometer from Thor Labs which provides intensity signal of the functionalized fiber in the range of 500 to 1000 nm. The spectrometer is then connected to Thor Labs software which displays various spectrum intensities at different wavelength. Different concentration of glucose and sucrose solutions used in this work are 0.1 g/ml, 0.2 g/ml, and 0.3 g/ml and different types of *Daucus carota* which are baby carrots, imported carrots, and organic carrots.

1.5 Significance of Research

In this research works, a better understanding of glucose and sucrose detection using immobilized tapered fiber was studied for the implementation of high quality sensor based on fiber optic. Fabrication of tapered fiber with flame brushing technique is one of the most economical and versatile method to fabricate fiber with an excellent physical characteristics. Features of tapered fiber were studied deeply for clearer picture in order to produce good fiber based sensor.

In addition, this study will enhance the selectivity and sensitivity of the fiber towards glucose and sucrose by immobilizing the fiber with enzyme and metallic nanoparticles. Different concentration of glucose and sucrose solutions and different types of Daucus carota which used as testing samples were the key factor in determining glucose and sucrose level using immobilized tapered fiber. In this work, fiber coatings using enzyme and metallic nanoparticles which immobilized onto the surface of tapered fiber were also considered for better knowledge.

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