Functionalized Fiber Optics for Glucose Detection

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Abstract. Tapered optical fiber immobilized with glucose oxidase (GOD) has been proposed for glucose recognition. Single mode fiber (SMF) is fabricated by tapering using flame-brushing technique to improve the sensitivity and limit of detection of optical fiber based glucose sensor. Taking the benefits from the free amine groups in 3-aminopropyltriethoxysilane (APTES), the enzyme, GOD are functionalized on the tapered region of SMF through covalent interaction. The pH value of the immobilized enzyme, GOD is the critical parameter for glucose sensing. The lower pH value of buffer solution, which is pH3 shows the highest intensity which is 2949.08 compared with intensity peak of pH5 and pH7 which is 2873.9 and 2700.43 respectively.

1. Introduction

Developing highly sensitive with low limit of detection of sensor recently become favorable for many applications and sensing research studies and industries due to their numbers of advantages. Tapered fiber has attracted considerable interest for implementation of sensor based on optical fiber. Sensors based on tapered fiber optics possess numbers of benefits including light weight, electrically passive operation, low susceptibility to electromagnetic interference, geometrical versatility, low-cost and high sensitivity which enables the detection of biomolecules [1]. These benefits have seen to various potential applications in optical fiber for sensing studies such as for detection of temperature, humidity, refractive index measurement, sugar content, and other biomolecules in various kind of solution [2-5].

Tapered fiber is fabricated by stretching a heated optical fiber to form a structure of reducing core diameter. In order to produce high quality tapered fiber based sensors, tapered fiber must be in low surface roughness, uniform microfiber diameter, high adiabaticity and suitable microfiber diameter with large evanescent field. Tapered fiber is known as microfiber with small diameter of micrometer (µm) and called as nanofiber with smaller diameter within nanometer (nm). Tapered fibers with smaller diameter are highly difficult to handle as thinner tapered fibers are very fragile and lossy [6].

Coating on the fiber surface which acts as the sensing region is required in implementation of optical fiber-based sensor. Functionalized optical fiber based sensors with enzymes have performed selectivity and high sensitivity for biologic substance measurement even in low concentration, which makes it more accurate and effective. Enzyme, GOD immobilized on microfiber will catalyze glucose and generate glucose acid, which will change the ambient refractive index. After the sample is dripped onto the immobilized sensor, glucose that reacts with GOD will be converted into gluconic acid and hydrogen peroxide [7-8]. The chemical reaction shown as follows:
Glucose + $O_2 \xrightarrow{GOD} \text{gluconic acid} + H_2O_2$

In this paper, we proposed a tapered fiber optic based sensor immobilized with enzyme, GOD for glucose detection. Tapered optical fiber using flame brushing technique which acts as sensing region functionalized with enzyme, GOD to enhance the selectivity and sensitivity towards glucose. The immobilized tapered optical fiber been characterized using optical microscope to study the effect of enzyme functionalization onto the surface of optical fiber.

2. Materials and Methods

2.1. Materials
The materials used in this experiment includes single-mode fiber(SMF) with core and cladding diameter of 62.5µm and 125µm respectively. Enzyme, GOD, 3-aminopropyl-triethoxysilane (APTES), buffer solution of pH3, pH5 and pH7, sodium hydroxide (NaOH), de-ionized water and ethanol.

2.2. Fabrication and functionalization of single-mode microfiber
The fabrication of tapered fiber was first done using SMF by removing a part of the buffer using fiber stripper and cleaned with acetone followed by hydrofluoric acid to remove the excess or unwanted buffer and cladding, respectively. The optical fiber then undergoes tapering process using flame brushing technique. Tapering is a process where the optical fiber is stretched and being heated at the same time. This process will make certain portion of the optical fiber become thinner and more sensitive towards the solution. The experimental set up of fiber tapering rig is shown in Fig. 1. It mainly consists of two fiber holders on a linear translation stage, butane burner, and two controller boards which take part in controlling the stages and butane burner position, direction and speed. The fiber was stretched for about 3 cm length and the flame from pistol heat the optical fiber for about 1.50 cm left and right movement while being stretched.

![Figure 1. Schematic diagram of fiber tapering rig.](image)

In order to make the single mode microfiber to only selective for glucose recognition, it should immobilize first with enzyme, GOD. The microfiber was initially cleaned using ethanol and de-ionized water. At this moment, the microfiber was prepared to be functionalized. Firstly, the cleaned microfiber was immersed into 1M NaOH solution for about 1 hour to activate the hydroxyl groups on the microfiber surface. Then, the microfiber was left in APTES solution (10% v/v with ethanol) for about 24 hours and then washed by de-ionized water and ethanol again to remove non-covalently
bonded silane compounds. After that, the silanized microfiber was then immersed in 10 µg/ml of glucose oxidase with buffer solution (pH3, pH5 and pH7) for about 2 hours to bind GOD’s COOH groups with NH₃⁺ together on the surface of microfiber and then dried at room temperature.

2.3 Experimental set-up
The functionalized microfiber been tested for glucose detection in carrot solution. The experimental set up for glucose sensing is shown in the Fig. 2. One end of the immobilized microfiber is connected to halogen light source model HL-2000-LL which provides flexibility for measurements between 360 to 2400 nm respectively. And the other end, it is connected to the spectrometer Ocean Optics USB4000 which provides intensity signal of the functionalized microfiber. The tapered area of the microfiber is immersed with pure carrot solution to achieve the intensity of the immobilized microfiber towards the glucose component in carrots.

![Experimental set-up](image)

Figure 2. Experimental set up for intensity spectra through enzyme, GOD-immobilized tapered fiber probe using spectrometer.

3. Results and Discussions
3.1 Surface characteristics of functionalized single-mode microfiber
The enzyme, GOD immobilized microfiber are characterized using optical microscope. Fig.3 displays the microscope images of surface modification of fabricated tapered fiber. It can be observed that when GOD enzyme are deposited onto the surface of microfiber, the images of the tapered fiber optic becomes blurry and there are small molecules are attached onto it.

![Microscope image](image)
3.2 Glucose detection with GOD-immobilized single-mode microfiber

Different pH value of buffer solution is the critical parameter that been observed in this paper. Fig. 4 shows the intensity spectra of different pH value of GOD-immobilized microfiber which been immersed into pure carrot solution. From the figures, we can observe that there is a peak value at the same wavelength which is at 625.9nm respectively. This wavelength is within the visible spectrum. The lower pH value of buffer solution, which is pH3 shows the highest intensity which is 2949.08 compared with intensity of the other two pH value, pH5 which is 2873.9 and pH7 which is 2700.43.
We can observed that the higher acidity which is pH3 of GOD affected the intensity of microfiber towards glucose component in pure carrot solution.

![Intensity spectrum of different pH value of GOD-immobilized microfiber (a) pH3, (b) pH5 and (c) pH7](image)

**Figure 4.** Intensity spectrum of different pH value of GOD-immobilized microfiber (a) pH3, (b) pH5 and (c) pH7

4. Conclusion
In this paper, we fabricated a tapered optical fiber of SMF using flame brushing technique for glucose selectivity of pure carrot solution. The functionalization of enzyme, GOD onto tapered optical fiber was successfully done by taking the advantages from the free amine groups in 3-aminopropyltriethoxysilane (APTES). The surface of tapered optical fiber after immobilized with
enzyme, GOD also been observed under optical microscopy. Different pH value of enzyme shows
different peak in the intensity spectrum. At the same wavelength which is 625.9 nm, the highest peak
shows for the lower pH value. The lower pH value of buffer solution, which is pH3 shows the highest
intensity which is 2949.08 compared with intensity peak of the other two pH value, pH5 which is
2873.9 and pH7 which is 2700.43.

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References
[1] Razak N. A., Hamida, B. A., Irawati, N., & Habaebi, M. H. 2017 IOP Conference Series: 
Materials Science and Engineering 210 012041
[2] Zhong, N., Chen, M., Wang, Z., Zhong, D., Chang, H., Zhao, M., Li, M 2019 Sensors and 
Actuators B: Chemical, 285 341-349
[3] Luan, X., Yu, R., Zhang, Q., Zhang, S., & Cheng, L. 2019 Surface and Coatings Technology, 363 
203-209
[4] Thomas, P. J. and Hellevang, J. O. 2018 Sensors and Actuators B: Chemical, 270 417-423
[5] Harun, S., Lim, K., Tio, C., Dimyati, K., & Ahmad, H. 2013 Optik, 124(6) 538-543
[6] Li, Y., Ma, H., Gan, L., Liu, Q., Yan, Z., Liu, D., & Sun, Q. 2018 Sensors and Actuators B: 
Chemical, 255 3004-3010
[7] Lin, T., Lu, Y., & Hsu, C. 2010 Optics Express, 18(26) 27560
[8] Chauhan, S. K., Punjabi, N., Sharma, D. K., & Mukherji, S. 2016 Sensors and Actuators B: 
Chemical 222 1240-1250
[9] Kumar, S., Kaushik, B. K., Singh, R., Chen, N., Yang, Q. S., Zhang, X., Zhang B. 2019 
Biomedical Optics Express, 10(5) 2150