Novel Microfiber Sensor and Its Biosensing Application for Detection of hCG Based on a Singlemode-Tapered Hollow Core-Singlemode Fiber Structure

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***Abstract*— A novel microfiber sensor is proposed and demonstrated based on a singlemode-tapered hollow core -singlemode (STHS) fiber structure. Experimentally a STHS with taper waist diameter of 26.5 μm has been fabricated and RI sensitivity of 816, 1601.86, and 4775.5 nm/RIU has been achieved with RI ranges from 1.3335 to 1.3395 , from 1.369 to 1.378, and from 1.409 to 1.4175 respectively, which agrees very well with simulated RI sensitivity of 885, 1517, and 4540 nm/RIU at RI ranges from 1.3335 to 1.337, from 1.37 to 1.374, and from 1.41 to 1.414 . The taper waist diameter has impact on both temperature and strain sensitivity of the sensor structure: (1) the smaller the waist diameter, the higher the temperature sensitivity, and experimentally 26.82 pm/°C has been achieved with a taper waist diameter of 21.4 μm; (2) as waist diameter decrease, strain sensitivity increase and 7.62 pm/με has been achieved with a taper diameter of 20.3 μm. The developed sensor was then functionalized for human chorionic gonadotropin (hCG) detection as an example for biosensing application. Experimentally for hCG concentration of 5 mIU/ml, the sensor has 0.5 nm wavelength shift, equivalent to limit of detection (LOD) of 0.6 mIU/ml by defining 3 times of the wavelength variation (0.06 nm) as measurement limit. The biosensor demonstrated relatively good reproducibility and specificity, which has potential for real medical diagnostics and other applications.**

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***Index Terms*— Hollow core fiber, refractive index, strain, temperature, biosensor.**

I. Introduction

OPTICAL fiber sensors are widely applied in various aspects such as physical, biomolecule, chemical and food-borne pathogenic bacteria detection because of their advantages such as high sensitivity, immunity to electromagnetic interference, and ease of fabrication [1-3]. There are different optical fiber sensor structures such as singlemode-multimode-singlemode (SMS) fiber structure [4], surface plasmon resonance (SPR) [5-7], fiber Bragg gratings (FBG) [8-11], long-period gratings (LPG) [12, 13] and optical fiber coupler [14].

Hollow core fiber (HCF) is a simple cylindrical light guiding structure consisting of a hollow air part in the center and a quartz tube wall, which has attracted extensive attention and research interest due to its unique property of air core compared with traditional solid core fiber [15]. The principles of HCF based sensor are mainly divided into three types, namely anti-resonance, Fabry–Perot and multimode interference [16-17]. For example, Liu *et al* proposed a high-temperature sensor based on the combination of single mode fiber (SMF) and HCF, namely SMF-HCF-SMF anti-resonance structure which also can be employed in sub-micrometer resolution liquid level [18-19]. Zhang *et al* utilized SMF- short HCF-SMF to form extrinsic Fabry-Perot interferometer for strain sensing [20]. Duan *et al* proposed in-fiber Fabry–Perot and Mach-Zehnder interferometers based on HCF fabricated by arc fusion splicing with small lateral offsets for temperature measurement, but the fatal drawback of this structure is fragile and poor reproducibility because of center offset splicing [21]. Zhang *et al* compared three RI interferometers with different inner diameters (5 μm, 15 μm, 25 μm) based on SMF-HCF-SMF interference structure [22], which has relatively low RI sensitivity.

Human chorionic gonadotropin (hCG) is a multi-functional molecule that plays an important role in pregnancy, fetal growth, pituitary secretion and cancer cell biology [23]. It has been reported that hCG also plays a role in promoting uterine angiogenesis during pregnancy in order to ensure maximum blood supply to the invading placenta which is an important function during pregnancy [24]. It is a cancer promoter in all human malignancies and is a supplement to pituitary LH during the maternal menstrual cycle [25]. In 2019, kumar et al reported a novel hCG detection technology based on magnetic microspheres enhanced microfiber interferometer [26]. Therefore, it is extremely necessary to accurately detect the concentration of hCG in the human body.

In this paper, we proposed to taper the HCF fiber section to improve RI sensitivity. Theoretical study of the mode interferometer properties and spectral responses of RI are provided by using the beam propagation method (BPM). Furthermore, the influence of waist diameter on temperature and strain responses is experimentally investigated. Finally, highly sensitive and specific detections of hCG are analyzed in detail based on the proposed fiber structure.

II. Theoretical Analysis

Figure 1 shows a schematic diagram of the STHS fiber structure. A short section of HCF is fusion spliced between two traditional SMFs and the center of the HCF section is tapered to a small diameter. The HCF has a core diameter of 10 μm, which is very close to that of the SMF (9.2 μm). The fusion splice will introduce slightly air core collapse when connecting the HCF to SMF. As light injected from the input SMF to HCF, part of light will be transmitted as guided mode within cladding of the tapered HCF (between the air core and surrounding material). These modes will interference each other, resulting in wavelength shift of interference dip once the surrounding RI changes. The rest light will be transmitted into the air core of HCF, which will act as leaky mode and disappear after transmitting to the output SMF. Hence, for the STHS fiber structure, the principle of transmission is the guided cladding modes within tapered HCF section, where the silica cladding of the tapered HCF acts as a core of the waveguide and both the air core and surroundings act as cladding of the waveguide.

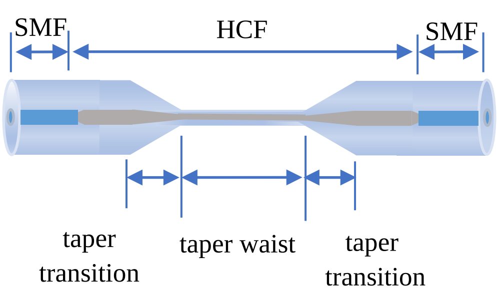
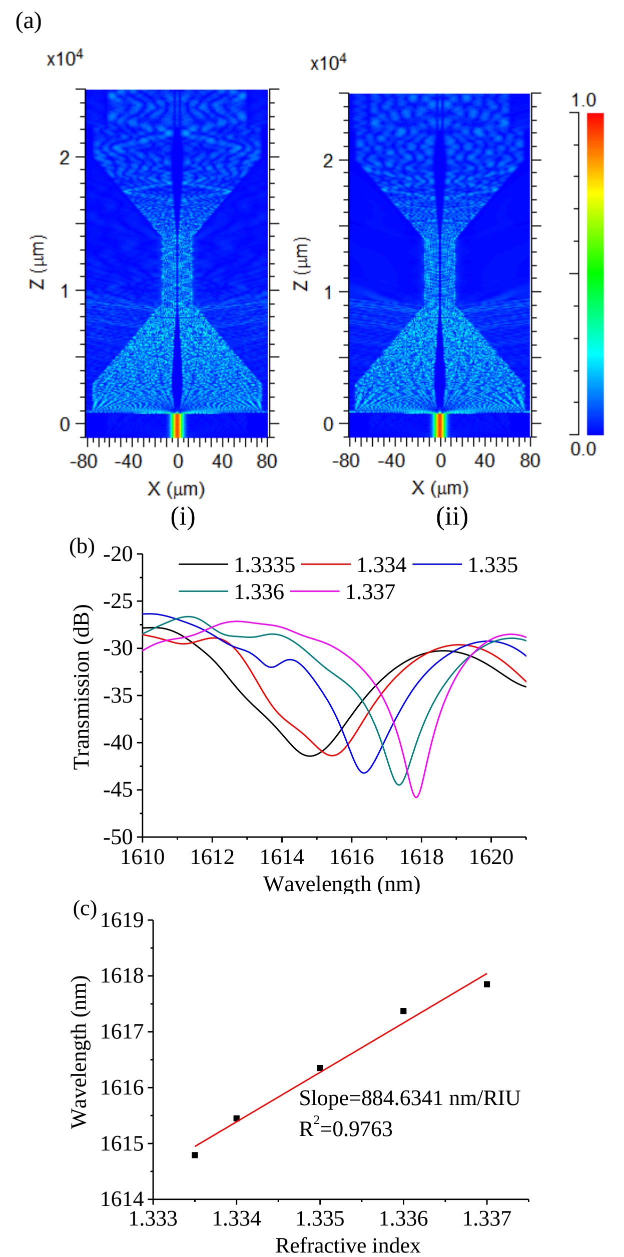


Fig. 1. A schematic diagram of the proposed STHS fiber structure

Numerical simulations based on the STHS fiber structure was carried out by using BPM. The simulation conditions are based on the mesh size of the 2D model in the X and Z directions, the grid size is set to 0.1 and 1 μm, respectively. The model boundary conditions are based on the perfectly matched layer condition. The core and cladding diameters of the SMF is set to 9 and 125 μm, and the corresponding RI are 1.4507 and 1.4428, respectively. The HCF has RI of 1.0 and 1.4428, and diameter of 10 μm and 150 μm, for core and cladding respectively. The lengths of taper transition and taper waist section of the HCF (21 mm) are set to 6 and 5 mm, respectively. The taper waist diameter is 26.5 μm. The air hole become collapsed from 10 μm to 5 μm at the interface between SMF and HCF. Figure 2(a) (i) and (ii) show the distribution of the optical field propagating along the STHS fiber structure when free space wavelengths are 1614.79 nm (dip) and 1618.63 nm (peak) at the surrounding RI of 1.3335, which indicate an obviously mode interference within the tapered HCF section. As light incident from SMF to air hole collapsed HCF, most light coupled into the cladding layer of HCF as guided mode and transmitted within the tapered HCF section. However, at the interface between HCF and output SMF, very limited light coupled back to the SMF. Figures 2(b), (c) and (f) show that the simulated spectra red shift the as the external RI increases at three different external RI ranges from 1.3335 to 1.337, from 1.37 to 1.374, and from 1.41 to 1.414. The RI sensitivities are calculated as 885, 1517, and 4540 nm/RIU [shown in Figs. 2(c), (e), and (g)], respectively.



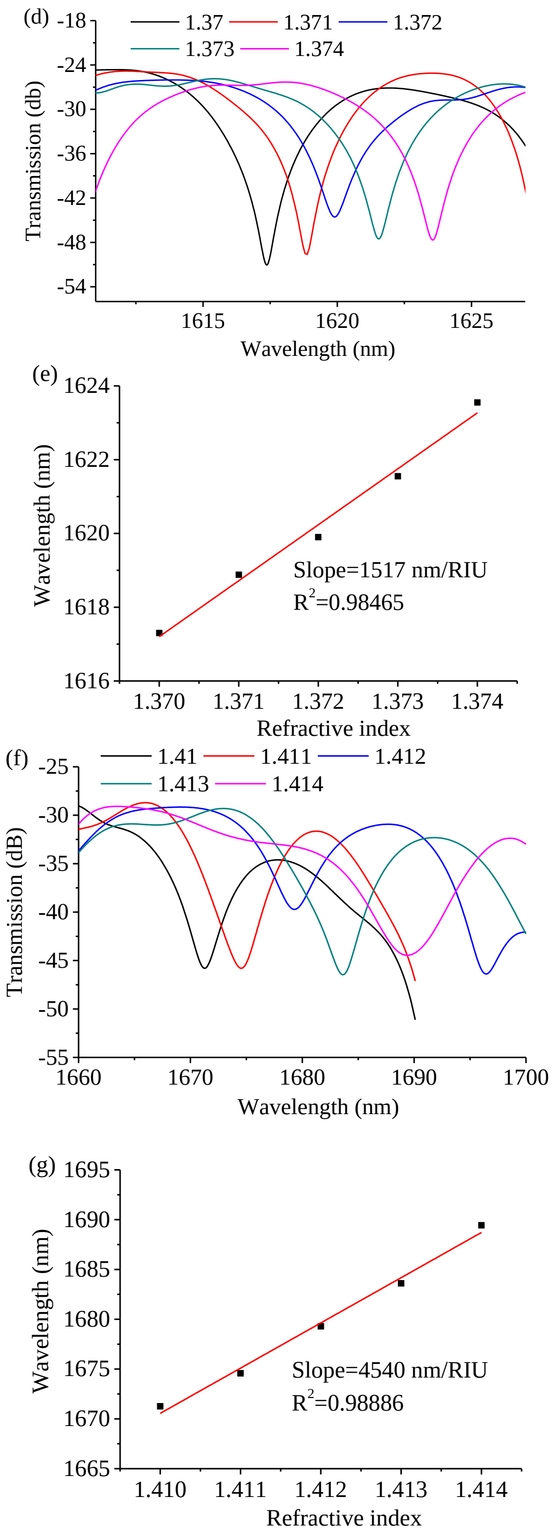


Fig. 2. Simulated results of the STHS fiber structure with taper waist diameter of 26.5 μm: (a) Optical field; (b) spectral response at external RI range of: (b) 1.33; (d) 1.37; (f) 1.41; and calculated RI sensitivity at RI range: (c) 1.33; (e) 1.37; (g) 1.41.

III. Experimental Investigation

The STHS Fiber Structure for RI Sensing

A length of 10 mm HCF with 10 μm air core diameter and 150 μm cladding diameter is spliced with two SMFs by using a commercial fusion splicer (Fujikura 80C). The manual splice mode is used to combine the HCF with SMF, and parameters of the manual fusion are listed in Table 1. Due to the surface tension, part of air hole near fusion splice points between the SMF and HCF would collapse. Then, the SMF-HCF-SMF structure is tapered to small diameter by a commercial optical fiber tapering system (OC-2010, JILONG).

Table 1
FUSION SPLICE PARAMETERS USED IN THE EXPERIMENT

Discharge time	Overlapping	Discharge power
100 ms	8 μm	standard +8 bit

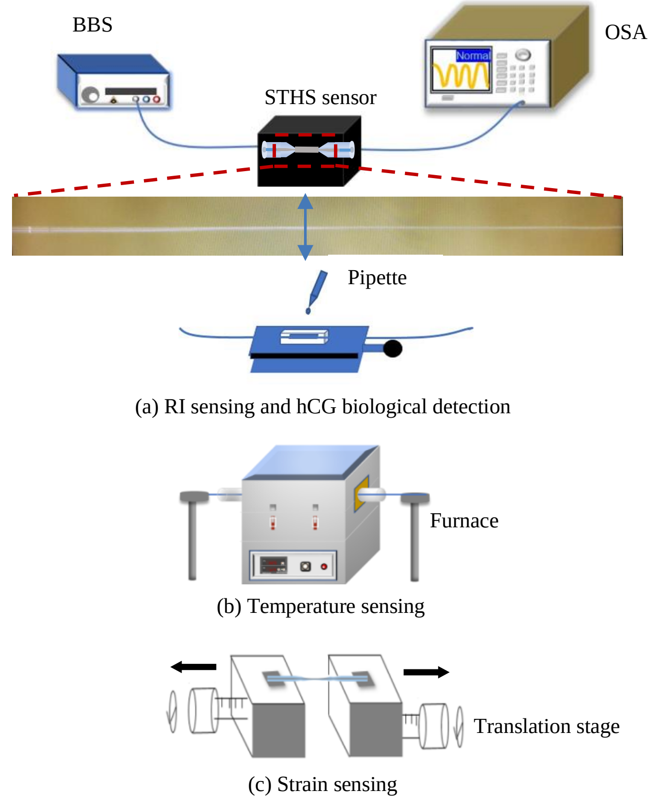
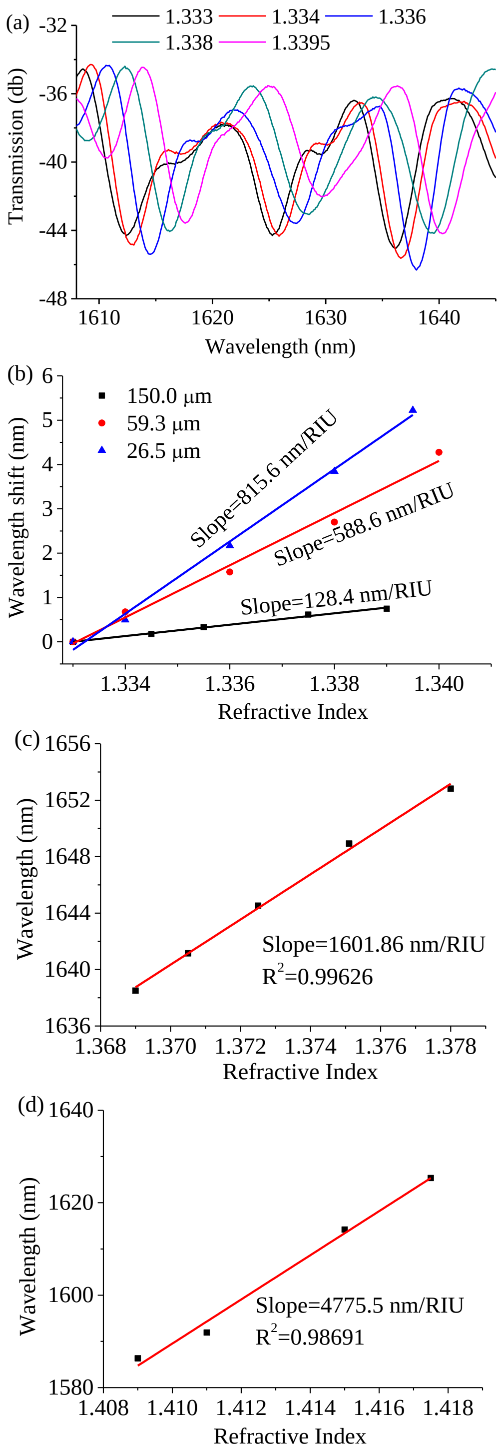


Fig. 3. The schematic diagram of the experimental setup (a) RI sensing and hCG detection, (b) temperature measurement and (c) strain sensing

Firstly, the influence of the taper waist diameter on RI response was investigated with three different tapered waist diameters of 150 (untampered), 59.3 and 26.5 μm. A schematic diagram of the experimental setup is shown in Fig. 3. The light supplied by a broadband source (BBS, SC-5-FC) is transmitted through the STHS fiber sensor and detected by an optical spectrum analyzer (OSA, YOKOGAWA AQ6370D). Figure 4(a) show the spectral responses of the STHS fiber structure with a taper diameter of 26.5 μm vs. RI at range of 1.33. The comparison of RI sensitivities with different tapered waist diameters is shown in Fig. 4(b). With increase of surrounding RI, wavelengths of dips are monotonically redshift for all the three tapered waist diameters, and the RI sensitivities increase from 128 nm/RIU to 816 nm/RIU as the taper diameters decrease from 150 to 25.6 μm. We also test RI responses at RI ranges from 1.369 to 1.378 and from 1.409 to 1.4175 with a taper waist diameter of 25.6 μm in Figs. 4(c) and (d), RI sensitivities of 1601.86 and 4775.5 nm/RIU were achieved, which agree very well with numerical simulation results [Figs. 2(e) and (g)]. In addition, a stability test has been carried out by immersing the fabricated STHS fiber sensor into phosphate buffered saline (PBS) buffer [shown in Fig. 4(e)]. The results indicate that the STHS sensor has very good stability with wavelength variations of ± 0.02 nm over 35 minutes.



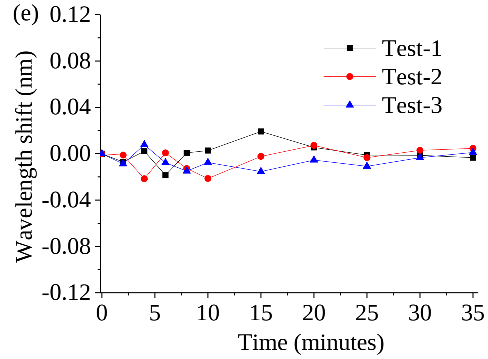
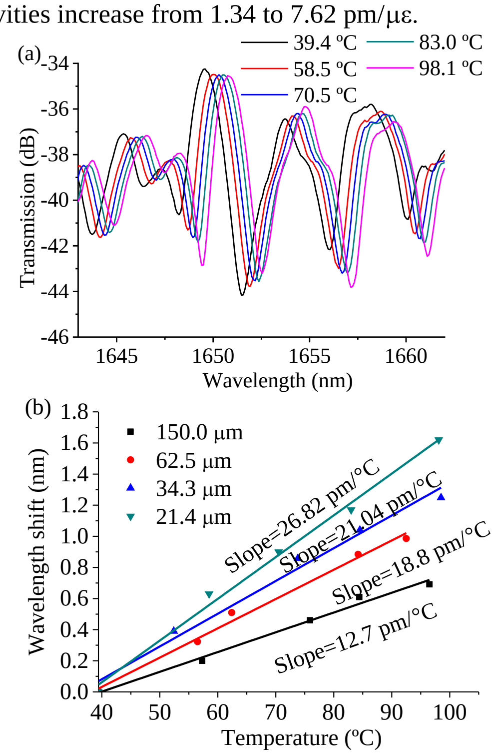


Fig. 4. Measured (a) spectral response of the sensor vs. RI variations at RI range of 1.33; wavelength shift of the sensor at RI range of (b) 1.33 (c) 1.37; (d) 1.41; and (e) stability tests of the sensor in PBS buffer.

The STHS fiber structure for temperature and strain sensing

In the following, temperature and strain sensing performances of the STHS fiber structure were tested experimentally. The experimental device diagram for temperature and strain sensing are shown in Figs. 3(b) and (c). Figure 5(a) shows the spectral responses with taper waist diameter of 21.4 μm at different temperatures, where wavelength shifts towards longer wavelengths monotonically as temperature increases from 39 to 98 °C. The influence of taper waist diameter on temperature response is shown in Fig. 5(b). As the waist diameters decrease from 150 to 21.4 μm, the temperature sensitivities gradually increase from 12.7 to 26.82 pm/°C. Then, the spectral responses when different strain was applied to the sensor are shown Fig. 5(c). The wavelength undergoes blue shift with growth of strain. Figure 5(d) shows the influence of the taper waist diameter on strain response. As the diameters decrease from 150 to 20.3 μm, the strain sensitivities increase from 1.34 to 7.62 pm/με.



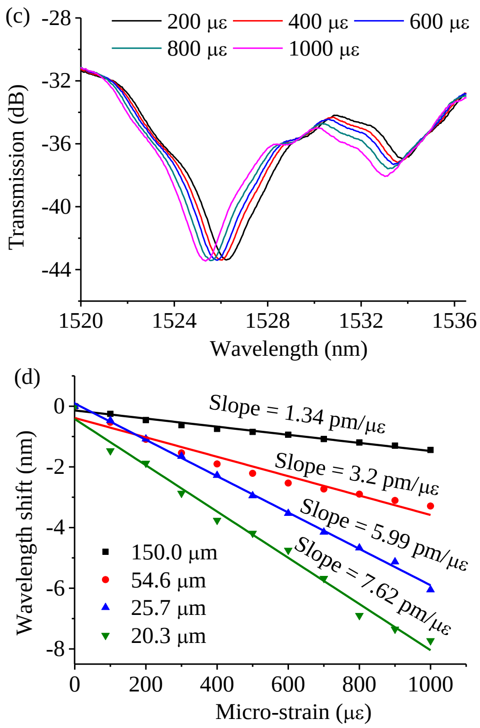


Fig. 5. Measured (a) spectral responses at different temperatures; (b) wavelength shifts vs. temperature with different taper waist diameter; (c) spectral responses at different strain; (d) wavelength shifts vs. strain with different taper waist diameter.

Functionalization of the STHS fiber structure for hCG detection

As studied above, the STHS fiber sensor with a diameter of 26.5 μm has maximum RI sensitivity, hence was selected for biosensing application. Before functionalizing the fiber sensor, the STHS fiber sensor was immersed into potassium hydroxide (KOH)-ethanol standard solution to clean the surface of the fiber for one hour at room temperature. It was then washed several times with deionized water and compared with the pH indicator paper until it was neutral (pH=7.0). The fiber sensor was then functionalized following the four steps below:

i. Immerse the fiber sensor in 5% 3-(3-triethoxysilylpropyl) oxolane-2,5-dione (silanization reagent) for four hours to produce carboxyl group on the fiber surface.

ii. Wash the fiber sensor with pH buffer standards DIN 19266 values (PB, pH=6.0), then immerse into the mixed solution of 1-(3-Dimethylaminopropyl)-3-ethylcar-bodiimide hydrochloride (0.8 mg/ml EDC) and hydroxy-2,5-dioxopyrolidine-3-sulfonicacid sodium salt (1.2 mg/ml NHSS) for one hour to obtain NHS active ester.

iii. The above treated fiber sensor is then immediately immersed into hCG-β-mAb solution with concentration of 15 μg /ml for four hours to immobilize the hCG-β-mAb on the fiber sensor surface.

iv. Wash the above sensor three times with PBS buffer, and then immerse it in 1% bovine serum albumin (BSA) solution to block the remaining unbound sites and then wash it with PBS buffer.

A schematic diagram of the fiber sensor surface modification process is shown in Fig. 6(a-d). After functionalization, the fiber sensor can be used for detection of hCG. Once the hCG antigen was specifically captured by the hCG-β-mAb immobilized on the fiber surface as shown in Fig. 6(e), both effective RI and diameter of the fiber sensor increase, resulting in change of the spectral response. The concentration of hCG can be determined significantly by calibrating the wavelength shift of dips.

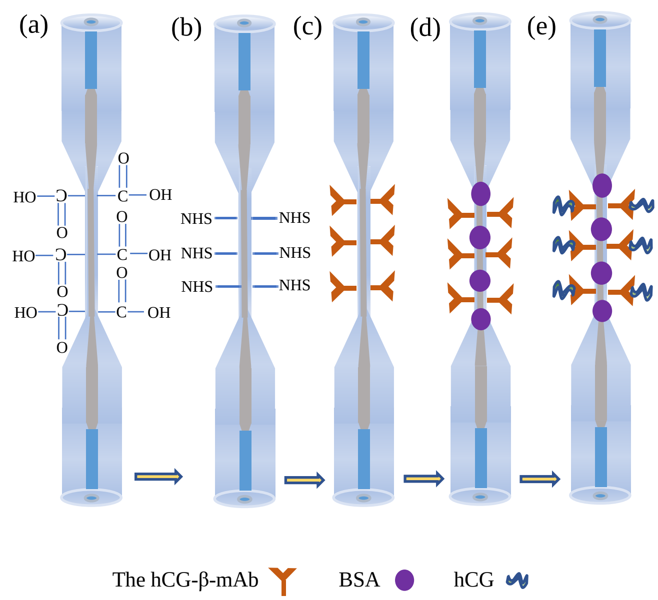


Fig. 6. Schematic diagram of fiber surface modification process: (a) treat with silane reagent to create carboxyl group; (b) generate NHS active ester with EDC/NHSS; (c) immobilize hCG-β-mAb on the fiber sensor surface; (d) block unbound sites with BSA; (e) specific binding with hCG.

Figure 7(a) shows the spectral responses of the fiber sensor (modified with 15 μg/ml hCG-β-mAb, marked as fiber sensor 1) vs. time when it was immersed into hCG solution with concentration of 5 mIU/ml. The spectral response changed rapidly in the first 15 minutes and then stabilized at around 20 minutes, indicating specific binding between hCG-β-mAb and hCG antigen was mainly taken place at the first 15 minutes and then saturated. Figure 7(b) summarized the measured wavelength shifts of the STHS biosensor vs. time when it was immersed in different concentration of hCG solution, where a wavelength shift of 0.5, 0.8, and 1.7 nm has been observed for hCG concentration of 5, 50, and 500 mIU/ml respectively. Assuming the wavelength shift is linear when the hCG concentration is less than 5 mIU/ml, the sensitivity is thus 0.5/5=0.1 nm/(mIU/ml). Since the maximum wavelength variation of the sensor in phosphate buffered saline (PBS) is ±0.02 nm over 35 minutes, the limit of detection (LOD) of hCG can be estimated as 0.6 mIU/ml (0.06/0.1=0.6) assuming 3 times of the maximum wavelength variation (0.06 nm) in PBS is defined as measurement limit. Figure 7(c) shows that as hCG concentration increases, the wavelength shift increases monotonically. The reproducibility of the sensor was investigated by fabricating the indentical STHS fiber structures and functionalize them with the same conditions and use them to do the same test as above. The measurement results were shown in the Fig. 7(c), which indicates relatively good reproducibility. Specificity of the sensor is investigated by immersing the sensors into 7×10^5 CFU/ml *Staphylococcus aureus* (*S. aureus*), 10 mg/ml BSA and 4×10^6 CFU/ml *Escherichia coli* (*E. coli*) solution and the result is shown in Fig. 7(d), which indicates that the developed sensor has good specificity.

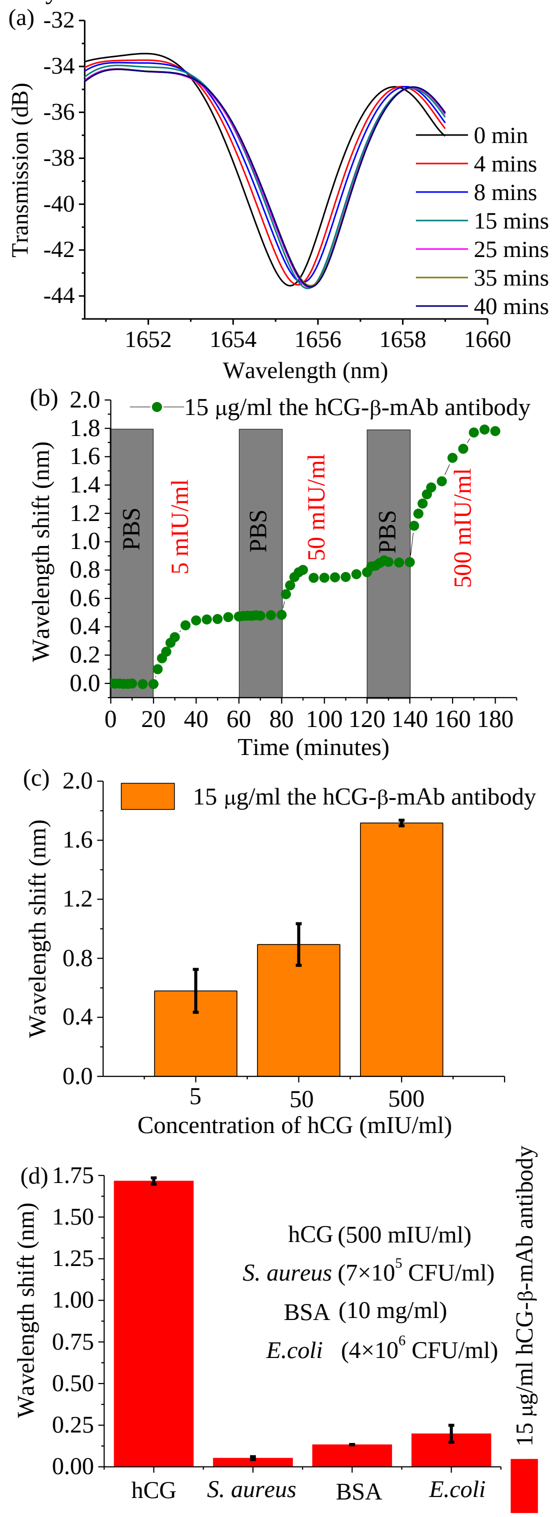


Fig. 7. Measured (a) spectral response variations of the sensor at the hCG concentration of 5 mIU/ml; (b) wavelength shift vs. time in different concentrations of hCG solution; (c) reproducibility: wavelength shift vs. concentration of hCG; (d) specificity results of the biosensor.

IV. Conclusions

In conclusion, a STHS fiber structure based on mode interference was proposed and investigated. Numerical simulations showed that the sensor structure with taper waist diameter of 26.5 μm has RI sensitivity of 885, 1517, and 4540 nm/RIU at RI ranges 1.33, 1.37, and 1.41 respectively, which verified by experimental results of 816, 1601.86, and 4775.5 nm/RIU respectively in the same RI range. The temperature sensitivities depend on taper waist diameters, where smaller diameter has larger temperature sensitivity and experimentally 26.82 pm/°C has been achieved with a taper waist diameter of 21.4 μm. The structure can also be used for strain sensor, where 7.62 pm/με has been achieved with a taper diameter of 20.3 μm. The developed STHS fiber structure is then functionalized with 15 μg/ml hCG-β-mAb for hCG detection, demonstrating its biosensing capability. The biosensor has very good stability with only ± 0.02 nm wavelength variations in PBS over 35 minutes. Experimentally 0.5 nm wavelength shift has been observed for hCG concentration of 5 mIU/ml, which equivalent to LOD of 0.6 mIU/ml. The developed biosensor has relatively good reproducibility and specificity, demonstrating a good potential for real applications such as medical diagnostics.

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