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A D-shaped fiber SPR sensor with a composite nanostructure of MoS_2-graphene for glucose detection

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ABSTRACT

Fiber-based techniques make it possible to implant a miniaturized and flexible surface plasmon resonance (SPR) sensor into the human body for glucose detection. However, the miniaturization of fiber SPR sensors results in low sensitivity compared with traditional prism-type SPR sensors due to limited sensing area. In this paper, we proposed a D-shaped fiber SPR sensor with a composite nanostructure of molybdenum disulfide (MoS_2)-graphene to improve the sensor sensitivity. Compared with the traditional cylindrical fiber, the planar sensing area on the side-polished fiber makes it easier to modify two-dimensional materials. Chemical vapor deposition (CVD) graphene and CVD MoS_2 were modified on the sensor surface to obtain the MoS_2-graphene composite nanostructure. π-π stacking interactions were used to modify pyrene-1-boronic acid (PBA) on the graphene. The excellent photoelectric properties of the MoS_2-graphene composite nanostructure and the ability of PBA to specifically bind glucose molecules improved the glucose detection performance of the SPR sensor. The results show that specific detection of glucose was realized and that the highest sensitivity was achieved with three-layer MoS_2 and monolayer graphene.

1. Introduction

Continuous glucose monitoring is important for the detection and treatment of diabetes. At present, implantable enzyme electrode sensors are mainly used in clinical environments to achieve continuous glucose monitoring. However, this method is susceptible to human bioelectricity interference, resulting in signal drift during measurement [1]. The surface plasmon resonance (SPR) sensor can overcome this problem by detecting the glucose concentration using an optical signal. Furthermore, glucose molecules are not consumed in the detection of the refractive index of a glucose solution near the sensor surface by using a glucose-specific receptor, which remains a great challenge for hypoglycemia detection using an enzyme electrode sensor [2]. However, current commercial SPR instruments are mainly based on prism structures. Although this structure has high sensitivity and accuracy, it is not suitable for implantation because of its large size and complicated structure. SPR sensors fabricated on optical fibers are small and flexible, but their sensitivity is relatively low compared to conventional prismatic SPR sensors. Two-dimensional materials have been used to modify prism-type SPR sensors to increase sensitivity [3–5], but it is still a significant challenge to construct nanostructures on the micron-scale cylindrical surface of a fiber SPR sensor [6]. Therefore, a D-shaped fiber with a flattened surface that is close to a prism structure is preferred rather than a cylindrical fiber for easier modification of the two-dimensional materials.

Graphene is widely used due to its high carrier mobility and large surface area to volume ratio [7,8]. A prismatic SPR sensor model based on a molybdenum disulfide (MoS_2)-graphene structure was proposed [9]. Compared with a SPR sensor without modification or only modified with graphene, the phase sensitivity was improved by more than 500 times. However, only simulation and theoretical analysis of the structure were performed in that paper. A U-shaped fiber SPR sensor based on graphene and silver nanoparticle modification, which achieved a wavelength sensitivity of 1198 nm/RIU, was proposed [10]. In a certain range, more graphene layers obtain higher sensor sensitivity. However, graphene cannot absorb enough energy to maintain high electron mobility between graphene and metal films due to the low light absorption coefficient of graphene (2.3%) [11], while the number of graphene layers exceeds a certain value. A multimode fiber (core diameter: 600 μm) SPR sensor with a surface structure of metal film--graphene-MoS_2, which uses the highly hydrophobic surface of MoS_2 to fix the biological targets on the sensor surface, was proposed [5]. The

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sensor had a high sensitivity of 6.2 μm/RIU with a metal film made of copper or aluminum that included 16 or 27 graphene layers, respectively, together with monolayer MoS_2. However, the number of graphene layers as so large that the sensor's resonance depth was small. When the metal film was gold, the sensitivity decreased to 5.0 μm/RIU, although it had a larger resonance depth. A larger and sharper resonance peak is preferred to determine the resonance wavelength with high accuracy. Therefore, it is desirable to obtain high detection sensitivity as well as a large and sharp resonance peak in designing a SPR sensor. In this paper, gold film is used to excite the SPR effect, and monolayer graphene and multilayer MoS_2 are modified on the gold surface to obtain a fiber SPR sensor with high sensitivity and large resonance depth.

Furthermore, the SPR sensor measures the concentration of the analyte based on the variation of the refractive index near the sensor surface. In the detection of glucose in body fluids, there are many disruptors, such as uric acid (UA), ascorbic acid (AA) and dopamine (DA). The sensor's selectivity to glucose can be improved by modifying the glucose-specific receptor on the sensor surface. Concanavalin (Con-A) is a commonly used glucose-specific receptor [12], but it is unsuitable for implantation in humans due to its immunotoxicity. A glucose/galactose-binding (GGB) protein was modified on the gold surface of the prism SPR sensor to achieve glucose-specific detection [13]. The measurement range of the glucose concentration was 0.1–100 mg/dL, and the coefficient of variation was less than 4.5%. However, the stability of the GGB protein is poor, which makes it unable to work reliably for a long time. Phenylboronic acids have emerged as synthetic receptors that can reversibly bind to the *cis*-diols of glucose molecules [14]. A glucose sensor that detected the viscosity variation caused by the affinity binding between glucose and poly (acrylamide-ran-3-acrylamidophenylboronic acid) (PAA-ran-PAAPBA) was proposed [15]. Compared with PAA-ran-PAAPBA, pyrene-1-boronic acid (PBA) has better stability, and the method for modifying PBA is much easier. In this work, PBA is modified on the surface of the fiber SPR sensor to achieve specific detection of glucose.

In this paper, based on D-shaped single-mode fibers, chromium and gold films were coated on the polishing area of the SPR sensor by thermal evaporation deposition. To improve the SPR sensor sensitivity, MoS_2 with a high light absorption coefficient and graphene with high electron mobility and a large surface area to volume ratio were modified on the gold surface by liquid phase transfer. Furthermore, PBA was bonded on the sensor surface to achieve selective detection of glucose molecules. Through experiments, the high sensitivity and selectivity of the fiber SPR sensor for glucose were verified.

2. Design of the sensor structure and surface modification

2.1. Design of the sensor structure

The structure of the D-shaped fiber SPR sensor is shown in Fig. 1a. The single-mode fiber SMF28 (coating diameter of 242 μm, cladding diameter of 125 μm and core diameter of 8.2 μm) is polished on one side to obtain the D-shape. While the polished portion is planar, the transition area is in a sloped state. A photo of the SPR sensor after polishing is shown in Fig. 1b. Gold film with chromium as the adhesion layer is used as the metal layer for exciting the SPR effect. MoS_2, graphene, and PBA were successively modified on the gold surface due to the high light absorption coefficient of MoS_2 to enhance the SPR effect, the large surface area of graphene to increase the adsorbance of molecules, and the high specificity of PBA towards glucose for selective detection. Based on the Kretschmann structure [16], the four main parameters, the length of the sensing area, the thickness of the chromium film, the gold film and the residual cladding, were simulated. The optimal parameters (chromium thickness: 5 nm, gold thickness: 35 nm, residual cladding: 0 μm) were determined according to the shape of the resonance peak and the sensitivity of the SPR sensor. The length of the sensing area, which has little influence on the position of the resonance peak, is selected to be 5 mm for sensor miniaturization.

2.2. Surface modification of MoS_2 on the D-shaped SPR sensor

Since MoS_2 has a high light absorption coefficient (monolayer: 5%) [17], the sensitivity of the SPR sensor can be improved by modifying MoS_2 on the sensor surface. Liquid phase transfer was used in this paper for monolayer chemical vapor deposition (CVD) MoS_2 modification.

Monolayer CVD MoS_2 (custom size: 10 × 10 mm) using a one-sided polished sapphire (thickness: 430 μm) as the substrate and a spin-coated polystyrene (PS) film as the cover was purchased from Nanjing 2D Nano Technology Co., Ltd, Nanjing, China. The monolayer coverage is more than 95%. The process for liquid phase transfer of MoS_2 is shown in Fig. 2.

First, MoS_2 with the substrate and cover was cut on four sides and then placed in deionized water. Since the PS film is hydrophobic, the MoS_2 and PS films were gradually separated from the substrate. Second, MoS_2 was salvaged with filter paper and cut into the shape and size as needed. Then, MoS_2 was put into deionized water and floated on top of the fiber. Third, the deionized water between MoS_2 and the fiber was removed by a Pasteur pipette, and MoS_2 lowered and gradually adhered to the fiber surface. Then, the fiber with MoS_2 was placed in an oven at 50 °C for 10 min to remove residual water (to prevent excessive temperature foaming) and dried successively at 90 °C for 10 min. The fiber with MoS_2 was then heated in the oven at 110–120 °C for 5 min to make MoS_2 cling to the gold film due to the van der Waals forces between water molecules. After transferring MoS_2, the fiber was immersed in toluene for 2 h to dissolve the PS film, and the toluene was refreshed once at 1.5 h. Since a Au–S bond is generated between MoS_2 and the gold film at room temperature, MoS_2 will not fall off even when the fiber is immersed in water again. Multilayer MoS_2 can be transferred by repeating this process.

Using liquid phase transfer, different numbers of layers of MoS_2 were modified on the fiber. The Raman spectra of MoS_2 with different layer numbers were detected by a laser micro-Raman spectrometer (DXR) and are shown in Fig. 3.

Two peaks, E^1_{2g} (~385 cm^{-1}) and A_{1g} (~408 cm^{-1}), are shown in

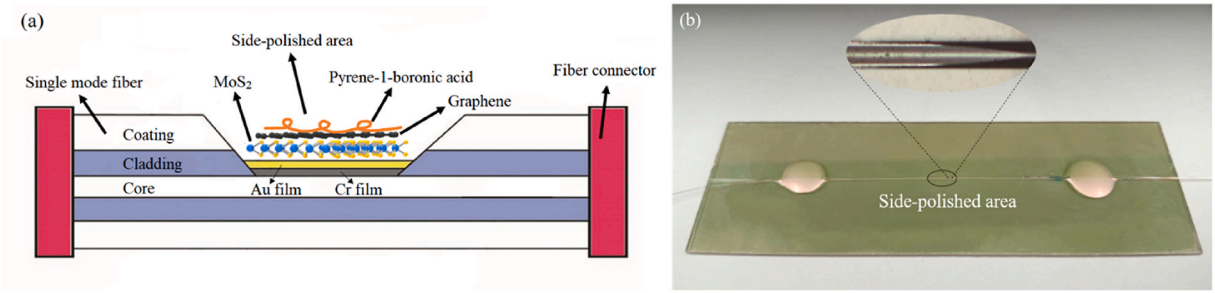

Fig. 1. (a) Schematic of the D-shaped SPR sensor and (b) photo of the D-shaped SPR sensor after polishing.

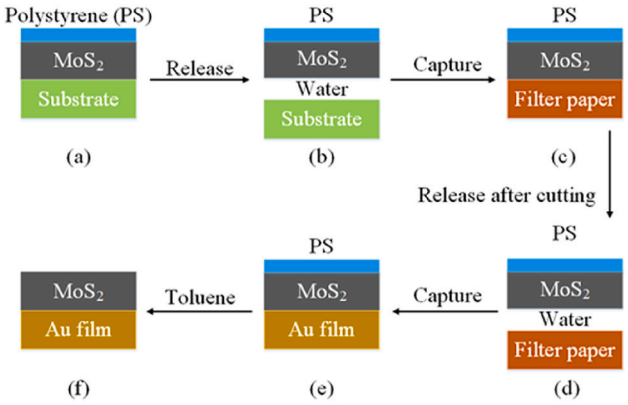

Fig. 2. Schematic of the transferring procedures of monolayer MoS_2: (a) untreated, (b) after being placed in deionized water, (c) after salvaging with filter paper, (d) after being placed in deionized water after cutting, (e) after salvaging to the surface of the gold film and drying, and (f) after toluene treatment. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

Fig. 3. The wavenumber difference between the two peaks increases with the number of MoS_2 layers. The blueshift of the A_{1g} peak was caused by the increased interlayer van der Waals force, which suppressed the atomic vibration. In contrast, the E^1_{2g} peak showed a redshift, as the increased interlayer van der Waals force played a minor role while the stacking-induced structure changed and the long-range Coulombic interlayer interactions in multilayer MoS_2 dominated the change of the atomic vibration [18,19]. These results indicated that the different layers of MoS_2 were successfully modified, and the layer number (less than 5 layers) of MoS_2 was determined by the E^1_{2g} and A_{1g} peaks in the Raman spectra.

2.3. Surface modification of graphene on the D-shaped SPR sensor

Graphene can increase the sensing area, improve electron mobility on the sensor surface, and provide an ideal substrate for PBA immobilization by unique π-π stacking interactions [9,20,21]. Therefore, the SPR sensor with MoS_2 was further modified with graphene. Monolayer CVD graphene (custom size: 5 × 5 cm) using a polymer as the substrate and a very thin poly(methyl methacrylate) (PMMA) film as the cover was purchased from ACS MATERIAL, LLC, USA. When placed in deionized water, the graphene and PMMA film were separated from the substrate. The process for liquid phase-transfer of graphene shown in Fig. 4 was similar to that for MoS_2, but toluene was replaced with acetone to dissolve PMMA [22]. Due to the high surface energy of graphene, it did not fall off even when immersed in water again.

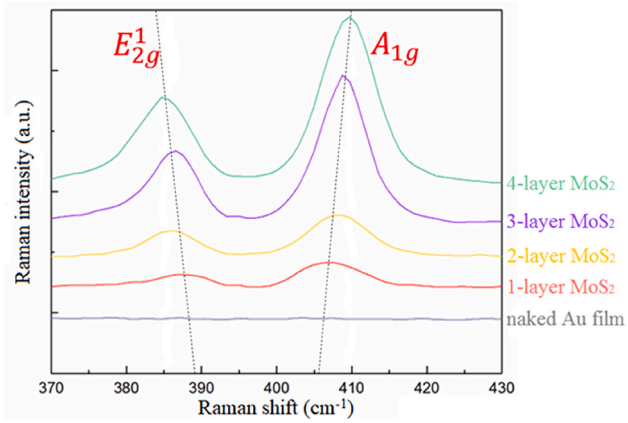

Fig. 3. Raman spectra of SPR sensors with different layers of MoS_2.

The Raman spectra of the SPR sensor surface modified with different numbers of MoS_2 layers and monolayer graphene were detected and are shown in Fig. 5. Both the G (1580 cm^{-1}) and 2D (2700 cm^{-1}) peaks of graphene appeared. These results indicate that graphene was successfully modified on MoS_2.

2.4. Surface modification of PBA on a D-shaped SPR sensor

As shown in Fig. 6, PBA and its derivatives can form reversible covalent complexes with molecules that contain diol units [23], such as glucose, ethylene glycol, dopamine and ascorbic acid. Therefore, the fiber SPR sensor functionalized with PBA can dynamically adsorb glucose molecules and achieve selective detection of glucose.

PBA from Shanghai Aladdin Biotechnology Co., Ltd., was dissolved in acetonitrile and allowed to stand for 30 min. Then, the SPR sensor with monolayer graphene was immersed in PBA (1 mM) for 4 h. PBA was immobilized on graphene sheet with the strong affinity to the graphene via π-π stacking to obtain PBA/graphene composites [24]. Then, the sensor was removed and rinsed with acetonitrile, isopropanol and deionized water in sequence to wash off unbound PBA and residual acetonitrile on the sensor surface. At this point, the functionalized SPR sensor was ready for glucose detection.

3. Results and discussion

3.1. Experimental system

According to the simulation results, the resonance wavelength of the SPR sensor was in the range of 550–600 nm. We selected a tungsten halogen lamp (HL2000, Ocean Optics, Inc.) with a wavelength range of 360–2000 nm. The spectra of the sensor were measured by a spectrometer (USB2000, Ocean Optics, Inc.) with a wavelength range of 300–1100 nm. For continuous glucose detection, the SPR sensor surface was cleaned by deionized water before each test of glucose sample. And each resonance wavelength was measured six times, and the average and standard deviation were calculated to evaluate the measurement results.

The resonance wavelength of the SPR sensor depends not only on the refractive index of the glucose solution near the sensor surface but also on the thickness of chromium, gold, residual cladding, and the two-dimensional materials. Therefore, the resonance wavelength of each sensor varies from one to another. In glucose detection, differential method was utilized, in which the resonance wavelength of deionized water was used as a reference value, and the resonance wavelength shift measured for each glucose concentration was used to analyze the sensor

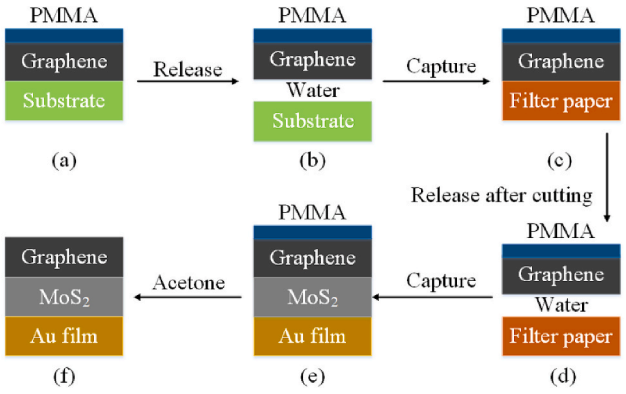

Fig. 4. Schematic of the transferring procedures of monolayer graphene: (a) untreated, (b) after being placed in deionized water, (c) after salvaging with filter paper, (d) after being placed in deionized water after clipping, (e) after salvaging to the surface of MoS_2 and drying, and (f) after acetone treatment.

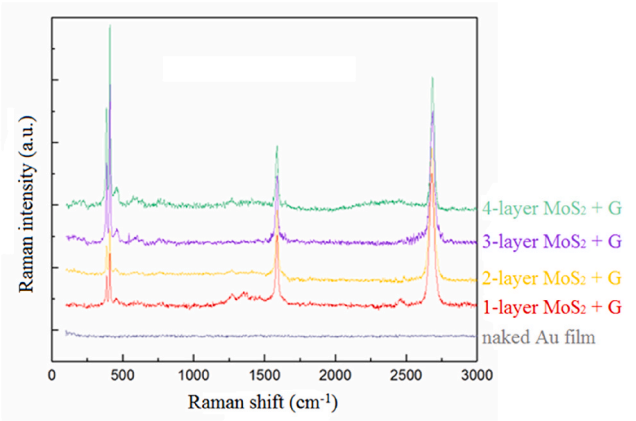

Fig. 5. Raman spectra of SPR sensors with different layers of MoS_2 and monolayer graphene.

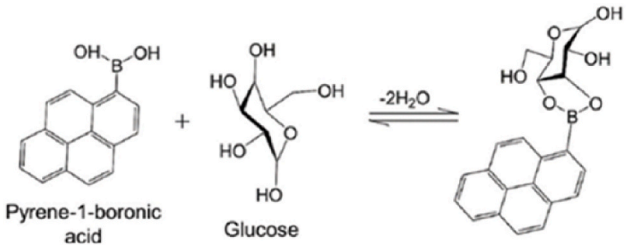

Fig. 6. Reversible binding of glucose to PBA.

performance.

Sensitivity (S) is used as an important indicator for evaluating SPR sensor performance and is calculated through the following relation:

$$S = \frac{\Delta\lambda}{\Delta n} \quad (1)$$

where $\Delta\lambda$ is the resonance wavelength shift between the glucose solution with the highest concentration and deionized water in nm and Δn is the change in the corresponding refractive index by the Abbe refractometer.

3.2. Effect of the MoS_2 layer number on the sensor sensitivity

The SPR sensor modified with different layers of MoS_2 was tested to analyze the effect of the MoS_2 layer number on the sensor sensitivity.

The normal fasting blood glucose levels are in the range of 3.9–6.1 mM (70–110 mg/dL) and increase to 7.8–11.1 mM (140–200 mg/dL) within half an hour to an hour after a meal. To cover the normal blood glucose range, a glucose solution with a concentration range of 0–300 mg/dL was prepared. The resonance wavelength of the SPR sensor modified with 1–4 layers of MoS_2 was detected in each glucose solution. The relationship between the absolute change in the resonance wavelength and glucose concentration is shown in Fig. 7. The highest sensor sensitivity was obtained when the number of MoS_2 layers was three. However, the error bar shows that the measurement stability decreased as the number of MoS_2 layers increased. When the number of MoS_2 layers was less than four, the high light absorption coefficient of MoS_2 enhanced the SPR effect and made the sensor more sensitive with more layers of MoS_2. However, the sensor was sensitive not only to the glucose concentration but also to interference factors, which influenced the sensor stability. When the number of MoS_2 layers became too large, the enhancement effect of the MoS_2 layers was overwhelmed by the electron energy loss [9]. In addition, the MoS_2 layers were connected to each other by van der Waals forces, and some MoS_2 may have been blown off when the nitrogen gun was used to remove the liquid from the sensor

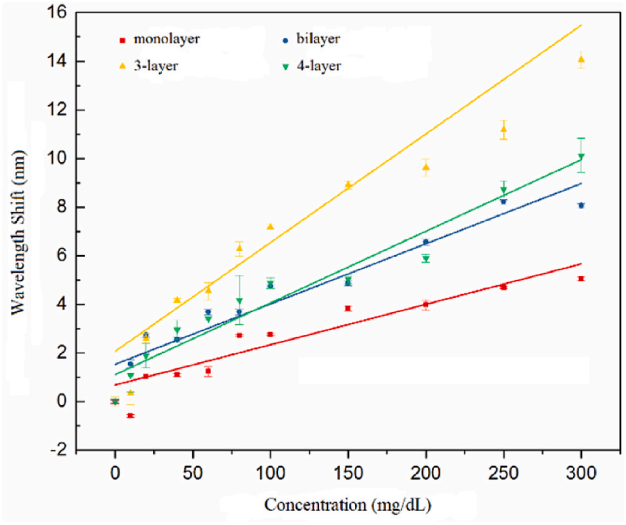

Fig. 7. Resonance wavelength shift versus glucose concentration for SPR sensors with different layers of MoS_2.

surface, which may also influence the stability of the SPR sensor as the number of MoS_2 layers increases.

To further improve the sensitivity of the SPR sensor, graphene was modified on the MoS_2 surface to form a composite nanostructure. According to our previous experimental results, monolayer graphene has the best effect on sensitivity improvement [25], so monolayer graphene was modified. The SPR sensors modified with 1–4 layers of MoS_2 and monolayer graphene were tested, and the relationship between the absolute change in the resonance wavelength and glucose concentration is shown in Fig. 8. Comparing Fig. 7 with Fig. 8, it can be seen the sensitivity of the sensors modified with 1–4 layers of MoS_2 is increased from 930.09 to 2602.73 nm/RIU to 3458.58–6126.25 nm/RIU by monolayer graphene modification. The increased sensitivity could be caused by the high surface-to-volume ratio of graphene, which provides a large surface area for adsorbing biomolecules to increase the local refractive index and sensitivity [26].

The relationship between the sensor sensitivity and the number of MoS_2 layers is shown in Fig. 9. The sensor obtained the highest sensitivity when the number of MoS_2 layers was three (with/without graphene modification). The sensitivity of the sensor with monolayer graphene (6126.25 nm/RIU) was more than twice as high as that of the

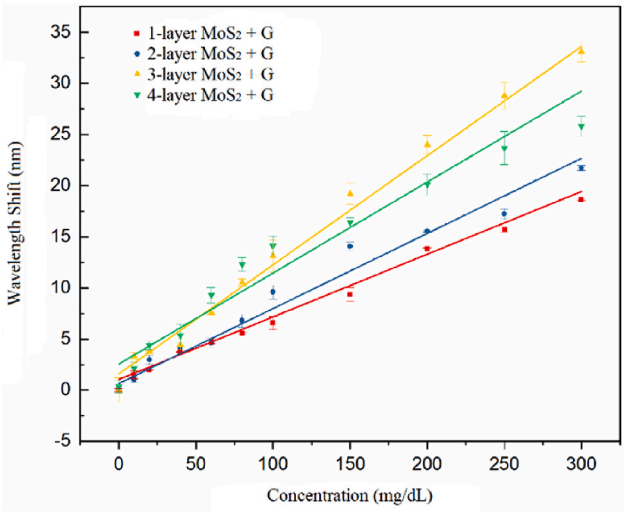

Fig. 8. Resonance wavelength shift versus glucose concentration for SPR sensors with different layers of MoS_2 and monolayer graphene.

sensor without graphene (2602.73 nm/RIU) and more than four times as high as that of the sensor without MoS_2 and graphene (1333.63 nm/RIU) [25], revealing that MoS_2 played an important role in improving the sensor sensitivity.

3.3. Effect of PBA on the sensor sensitivity

The SPR sensor further modified with PBA was tested to analyze the effect of PBA on the specific adsorption of glucose molecules. The relationship between the absolute change in the resonance wavelength and glucose concentration is shown in Fig. 10. The highest sensitivity was also obtained by the sensor modified with three layers of MoS_2, and the sensitivity reached 6708.87 nm/RIU, which was 9.5% higher than that of the sensor without PBA modification. In comparing Fig. 8 with Fig. 10, it was found that PBA had little effect on sensor sensitivity, but the lower stability of the sensor with more MoS_2 layers was improved by PBA modification.

The selectivity of the sensor modified with PBA was then tested, as shown in Fig. 11. Common interfering molecules, such as uric acid (UA), ascorbic acid (AA) and dopamine (DA), were investigated. The normal blood glucose levels were in the range of 3–8 mM, which was approximately 30 times higher than the concentration (0.1 mM) of interfering molecules in normal human blood [27]. Two glucose concentrations were tested in the experiment: one concentration (0.1 mM) was the same as that of the interfering molecules, and the other concentration of 30 mg/dL was selected. Although the glucose concentration of 0.1 mM (1.8 mg/dL) is much lower than the glycemic threshold of severe neuroglycopenia (30 mg/dL), it still obtained the highest absolute change of resonance wavelength, which shows the possibility for selective detection of glucose. In addition, differential method was used in this work to further reduce the influence of interfering molecules whose concentrations keep at the same level in human body. To further evaluate the sensor selectivity, the glucose solution in a concentration range of 0–300 mg/dL was prepared with the mother liquor containing UA, AA, and DA with a concentration of 0.1 mM, and the sensor modified with PBA was used to measure the glucose concentration. As shown in Fig. 12, the resonance wavelength shift maintained a good linear relationship with the glucose concentration, and little effect of the interfering molecules on the measurement of the glucose concentration indicated that the SPR sensor is suitable for selective detection of glucose.

4. Conclusions

In this work, a novel D-shaped fiber SPR sensor was proposed by using the composite nanostructure of MoS_2-graphene to enhance the sensitivity and functionalized modification of PBA for the selective detection of glucose molecules. A sensitivity of up to 6708.87 nm/RIU was achieved in glucose detection when the sensor was modified with three-layer MoS_2 and monolayer graphene. The stability and selectivity of the glucose sensor was improved by modifying PBA. With interfering molecules, the resonance wavelength shift correlated well with the glucose concentration. These results suggest that the SPR sensor has the potential for continuous glucose monitoring.

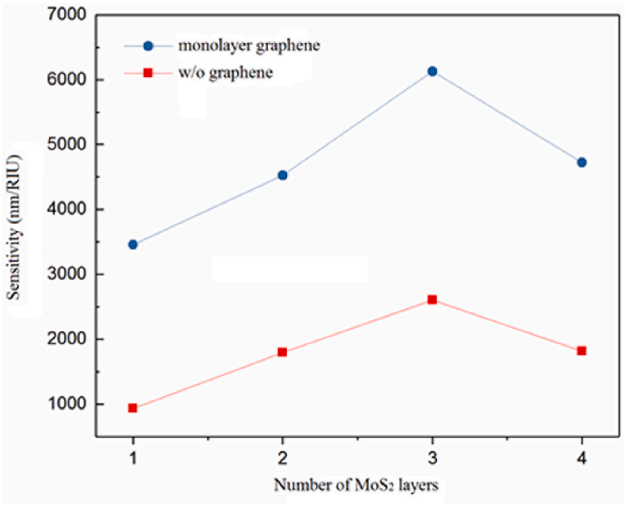

Fig. 9. Sensor sensitivity versus the number of MoS_2 layers.

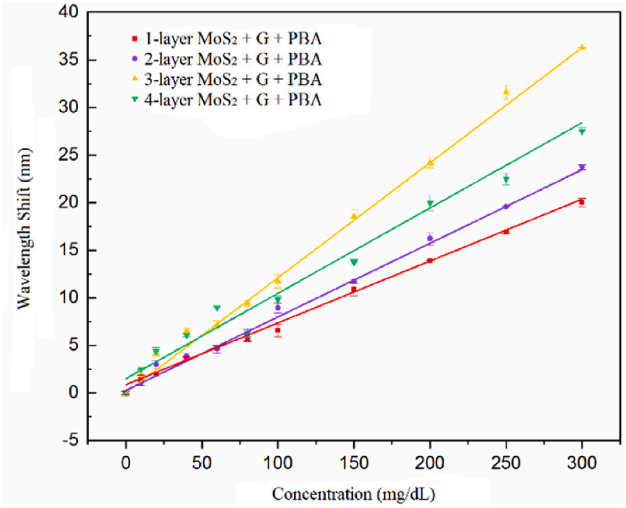

Fig. 10. Resonance wavelength shift versus glucose concentration for SPR sensors with different layers of MoS_2, monolayer graphene and PBA.

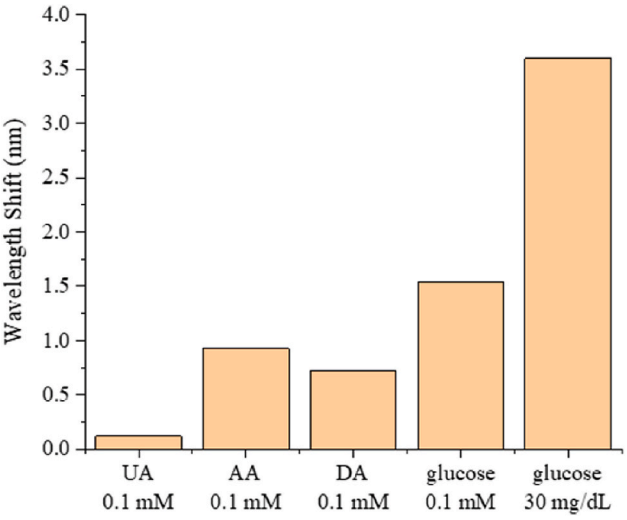

Fig. 11. Resonance wavelength shift for UA, AA, DA, and glucose.

Credit author statement

Haixia Yu: Conceptualization, Methodology, Data curation, Funding acquisition, Writing - original draft, Writing - review & editing. **Yang Chong:** Conceptualization, Methodology, Data curation, Writing - original draft, Writing - review & editing. **Penghao Zhang:** Conceptualization, Methodology, Data curation, Writing - original draft, Writing - review & editing. **Jiaming Ma:** Writing - original draft, Writing - review & editing. **Dachao Li:** Conceptualization, Funding acquisition, Writing - review & editing.

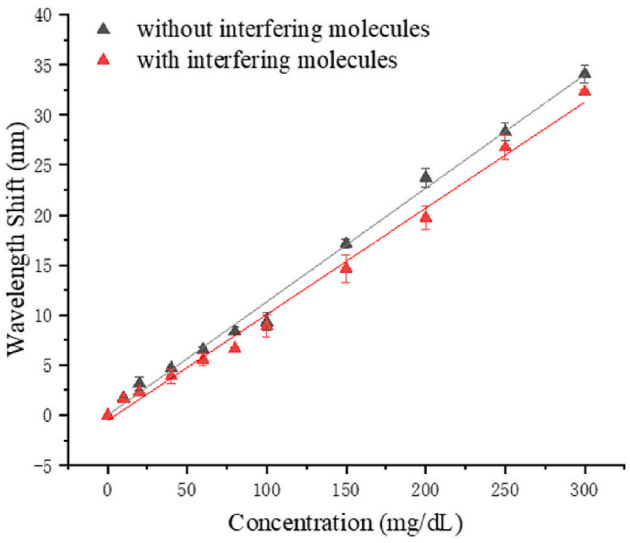

Fig. 12. Effect of the interfering molecules on the relationship between the resonance wavelength shift and glucose concentration.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

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