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Fundamental characteristics of a dual-colour fibre optic SPR sensor

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Abstract

In this paper, we present the fundamental characteristics of a novel dual-colour optical fibre surface plasmon resonance (SPR) sensor for a portable low-cost sensing system. The principle of the proposed SPR sensor is based on the differential reflectance method. Light from two light-emitting diodes (LEDs), which are flashing alternately with different wavelengths, is fed to a sensor via two optical couplers. The reflected light is detected by a photodiode. Changes of reflectance at two wavelengths are proportional to the refractive index change of the medium of interest. Taking the difference in reflectance at two wavelengths improves the sensitivity almost twofold. Measuring ethanol solutions with different refractive indices reveals that the sensor has a linear response to the refractive index change from 1.333 to 1.3616. By measuring the stability in the time response we estimate that the limit of detection (LOD) of the refractive index is 5.2×10^{-4}.

Keywords: surface plasmon, optical fibre, refractive index

1. Introduction

The surface plasmon wave (SPW) is an oscillation of free electrons propagating at the interface of a metal layer and a dielectric layer. When the wave vectors for the incident photons and plasmons are equal in magnitude and direction at the same frequency, the metal layer absorbs the incident photons and plasmons are excited. This is called surface plasmon resonance (SPR). Because this matching condition is very sensitive to variations in the refractive index of the dielectric layer adjacent to the metal layer, SPR-based sensors have been used as biosensors and chemical sensors [1–3] to detect the adsorption of biomolecules or concentration of solutions.

In the last two decades, the SPR-based sensors have been intensively developed. Like Biacore AB [4] in Sweden, there have been many companies that have been successful in commercializing SPR-based biosensor systems. Because most such systems employ the well-known Kretschmann–Raether configuration [5] with a metal layer on a dielectric prism and a heavy rotating mechanism, they are bulky and expensive; hence their application is limited to laboratory use. On the other hand, SPR sensors employing an optical fibre as a sensing probe have also been developed, which has allowed the miniaturization of SPR sensors and reduction of production cost. Although optical fibre SPR sensors have such merits, sensors proposed up to now seem to be insufficiently portable or cost effective. The first optical fibre-based SPR sensor was reported by Jorgenson and Yee [6], who used the wavelength interrogation technique. In this technique, a step-indexed multimode fibre with a sensing part, which is formed by partial removal of the cladding followed by the deposition of a metal layer on the exposed core, is used. A white light source is used, and reflected or transmitted light is detected by a spectrograph. In the angular interrogation technique, a monochromatic light source such as a laser diode is also used, and its beam is launched into a step-indexed multimode fibre at an incident angle adjusted by a precision rotator [7]. The transmitted light power versus the angle of incidence is measured. The SPR sensor with a single-mode optical fibre

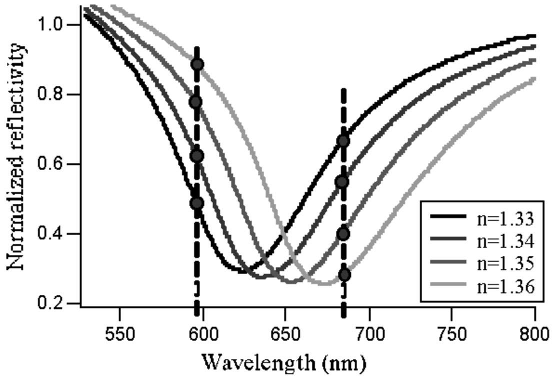

Figure 1. Principle of the dual-colour optical fibre SPR sensor.

was also presented [8]. The shortcoming of this sensor is that it requires precise polarization control.

We report a novel optical fibre SPR sensor with portability and lower production cost. Employing two light-emitting diodes (LEDs), two optical couplers, a photodiode, a sensor and a laptop computer leads to these advantages.

2. Principle of proposed sensor

The principle of the dual-colour optical fibre SPR sensor is shown in figure 1. The reflectance SPR spectrum from a multimode optical SPR sensor [6] with a mirror at its end can be obtained using a spectrograph and a white light source. The four different SPR spectra shown correspond to different refractive indices of samples. The reflectance SPR spectrum shifts towards the longer wavelength region as the refractive index of the solution increases. Turning our attention to two wavelengths across the dips, it can be seen that the reflectance for shorter wavelength increases and that for longer wavelength decreases. The reflectance varies almost linearly with the change of the refractive index if two wavelengths are properly selected. By taking the difference between the reflectance at two wavelengths, almost twofold improvement of sensitivity can be expected. This principle is essentially the same as the one reported by Suzuki *et al* [9]. They, however, used a multimode optical waveguide instead of the multimode optical fibre that we use here. With the multimode optical fibre, the number of reflections in the sensing part is much larger than that in the waveguide. Therefore, the expansion of the measurable refractive index range can be expected for this sensor.

3. Experiment for parametrization

Among the parameters of the dual-colour optical fibre SPR sensor, the thickness of the gold layer, two working wavelengths and the sensing length are controlled. They affect the performance of the sensor. To determine the optimal parameter values experimentally, a preparatory experiment was carried out using the conventional optical fibre SPR sensor system, as shown in figure 2. Subsequently, the dual-colour optical fibre SPR sensor with optimized parameters was fabricated and tested.

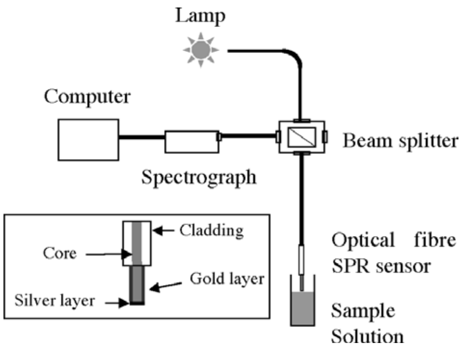

Figure 2. Arrangement for parametrization. The inset shows the detailed structure of the sensing part.

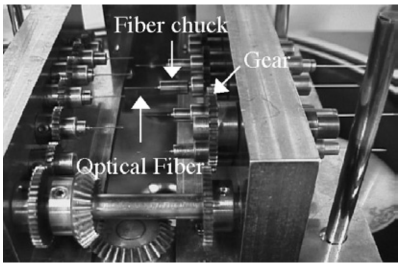

Figure 3. Photograph of the fibre rotating system for uniform metal deposition.

Table 1. Refractive index and concentration of distilled water and ethanol solutions [10].

Refractive index	Ethanol (weight %)
1.333	0.0
1.3395	10.0
1.3432	15.0
1.3469	20.0
1.3505	25.0
1.3535	30.0
1.3583	40.0
1.3616	50.0

At 589.3 nm, 20 °C.

To determine the gold thickness giving the highest sensitivity, multimode optical fibre SPR sensors with gold layers of different thicknesses from 20 nm to 70 nm with 10 nm intervals were fabricated. The optical fibre used was a step-index multimode silica/polymer optical fibre with a core diameter of 400 μm. The cladding of the fibre tip was removed over an area of 20 mm long for a sensing part. The reason for choosing the sensing length of 20 mm is that it gives a sufficiently wide reflectance range. The silver layer was deposited on the end of the fibre to form a mirror. The gold layer was deposited around the core surface by a thermal evaporation technique in a vacuum chamber at a pressure of 3×10^{-6} Torr. A homemade fibre-rotating system was used for homogeneous deposition of gold, as shown in figure 3. By dipping these sensors into distilled water and ethanol solutions with different refractive indices up to 1.3505 (25.0 wt%), SPR spectra were obtained using a spectrograph (Ocean Optics, SD2000). The relationship between the refractive index and concentration of ethanol solutions is summarized in table 1. The sensitivity dependence on the thickness was estimated by

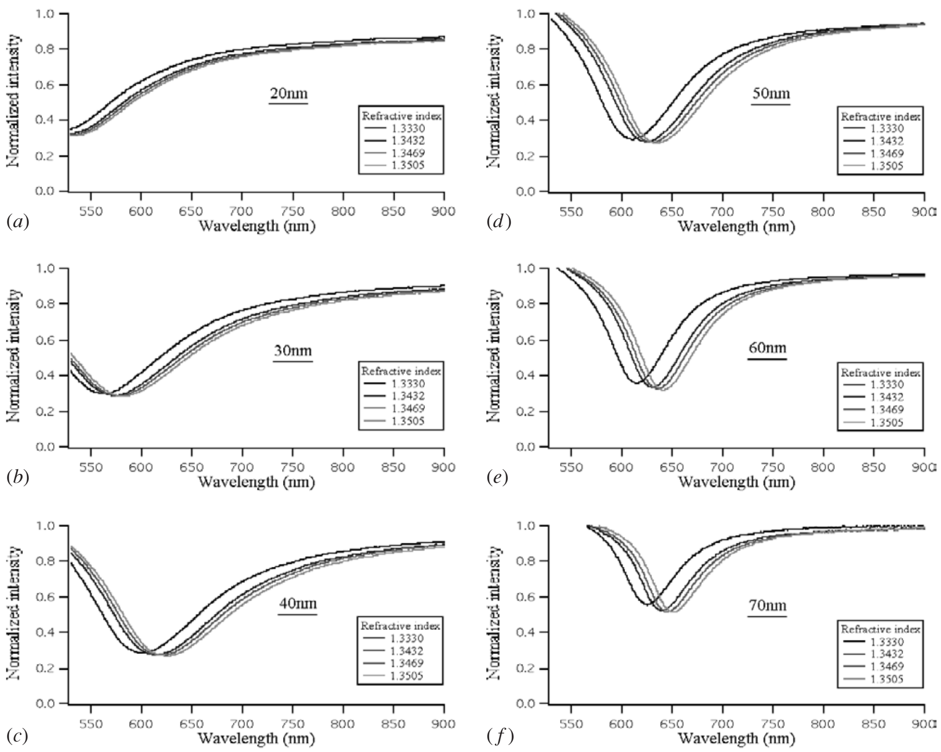

Figure 4. Reflectance SPR spectra for four solutions with different refractive indices. The thickness of the gold layer was changed from 20 nm (*a*) to 70 nm (*f*). All spectra are normalized by air spectrum.

analysing these data. The optimum gold thickness and two working wavelengths were thus found.

Using the optimized SPR sensor, a set of reflectance SPR spectra, which correspond to a series of ethanol solutions with different refractive indices spanning from $n = 1.333$ to 1.3616 (0% to 50% in wt%, see table 1), was obtained to confirm a measurable refractive index range.

Two LEDs were used as light sources of the dual-colour optical fibre SPR sensor. Compared with a laser diode, an LED has a much broader emission spectrum. It is of interest to see how it affects the linearity and the sensitivity of this sensor. Therefore, the effect of the spectral bandwidth of a light source on such characteristics was estimated numerically using the data obtained in this experiment.

4. Optimization of the SPR sensor

4.1. Determination of the gold thickness and two working wavelengths

We gave priority to sensitivity and linearity in the process of parameter determination. Sensitivity is the ratio of the change in reflectance to the change in refractive index. To realize a sensor with the highest sensitivity, its dependence on the gold thickness was investigated. Reflectance SPR spectra for the sensors with different gold thicknesses were obtained. Four reflectance SPR spectra of each sensor for four different solutions are shown in figure 4. Resonance wavelengths, slopes and minimum reflectance for each sensor vary as the gold thickness changes. Watching a set of four SPR spectra for a certain gold thickness, it can also be seen that the resonance wavelength becomes longer as the refractive index of the solution increases. Before determining the sensitivity for each sensor from these four spectra, two working wavelengths should be chosen. The two criteria shown in the inset of figure 5 were used to choose such wavelengths. For the highest sensitivity, the reflectance range ($d - a$ in the inset) should be the widest. Also, for the best linearity, the difference between the two slopes ($(c - a)/(C - A) - (d - b)/(D - B)$ in the inset) should be minimum. These criteria gave almost identical wavelengths for each sensor.

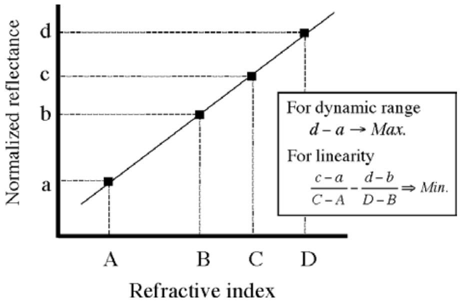

Figure 5. The method of choosing two optimum wavelengths.

Using the two wavelengths thus obtained, which are listed in table 2, the differential reflectance at the two wavelengths is calculated for each optical fibre SPR sensor and plotted in figure 6 to compare the sensitivities. It is found that the gold thickness of 60 nm gives the highest sensitivity. The two wavelengths for this sensor are 609.6 nm and 675.9 nm. In figure 4, as the gold thickness increases, the amount of shift of the reflectance SPR spectrum towards longer wavelengths due to the refractive index change of ethanol solution also increases

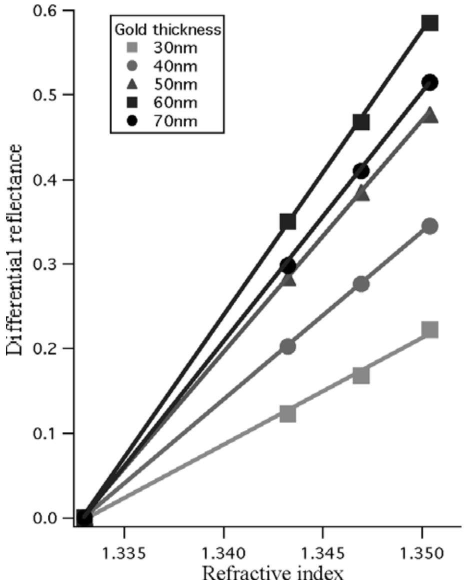

Figure 6. The sensitivity dependence on the gold thickness.

Table 2. Optimum wavelengths found for the sensor for each thickness of gold.

	Optimum wavelength (nm)	
Gold thickness (nm)	Short wavelength	Long wavelength
30	538.1	640.0
40	565.6	661.0
50	587.7	659.2
60	609.6	675.9
70	616.8	664.0

whereas the amplitude of reflectance SPR spectrum decreases. This decrease in the amplitude suppresses the increase in the sensitivity when the gold thickness is greater than 70 nm.

4.2. *Estimation of measurable refractive index range*

To estimate the measurable refractive index range when a dual-colour optical fibre SPR sensor is realized, eight different solutions covering the refractive index range of 1.333 to 1.3616 were prepared and the corresponding reflectance SPR spectra were acquired using the optical fibre SPR sensor with the thickness of 60 nm. Reflectances at 609.6 nm and 675.9 nm for these SPR spectra were read and normalized by those in air and then plotted in figure 7. The differential reflectance, which was obtained by subtracting the reflectance at 675.6 nm from that at 609.6 nm, was also plotted. From figure 7, it was confirmed that the measurable refractive index range of 1.333 to 1.3616 could be achieved with the dual-colour optical fibre SPR sensor.

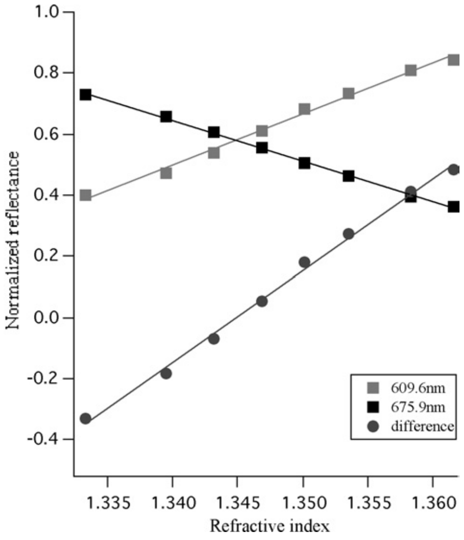

Figure 7. Estimated characteristics of the sensor using SPR spectra.

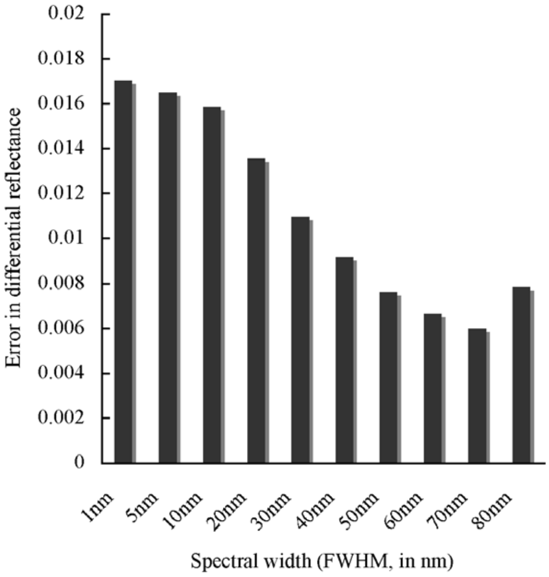

Figure 8. Variation of error with respect to the spectral bandwidth.

4.3. *Effect of the spectral bandwidth on the characteristics of dual-colour optical fibre SPR sensor*

Assuming that two light sources emit spectra with a Gaussian distribution whose centre wavelengths are chosen to be the working wavelengths, reflectances at two centre wavelengths were calculated for different spectral bandwidths (FWHMs) ranging from 1 nm to 80 nm. The average error ΔDR in differential reflectance is defined as

$$\Delta DR = \sum \frac{|DR_{mi} - DR_{fiti}|}{N}, \qquad (1)$$

where DR_{mi} is the differential reflectance of the ith sample, DR_{fiti} is the value on the fitted straight line of the ith sample and N is the number of samples. Figure 8 shows how the error varies with respect to the spectral bandwidth. The error becomes smaller as the spectral bandwidth becomes broader, and it is smallest at the spectral bandwidth of 70 nm, which is about three times broader than those of the two LEDs. This means that the linearity can be improved by employing a light source with a broad emission spectrum, to a certain extent, owing to the weighted averaging with respect to wavelength. On the other hand, the sensitivity becomes lower as the spectral bandwidth becomes broader, as shown in figure 9.

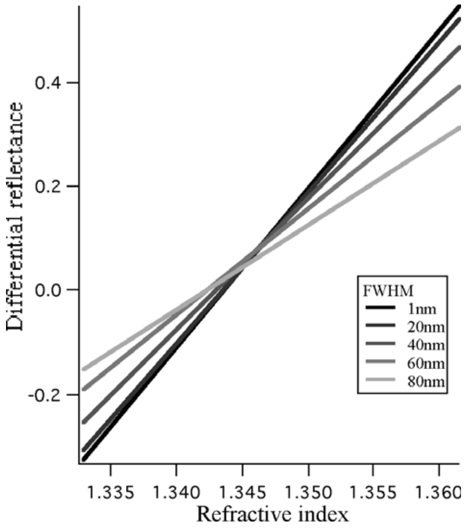

Figure 9. Sensitivity dependence on the spectral bandwidth (FWHM).

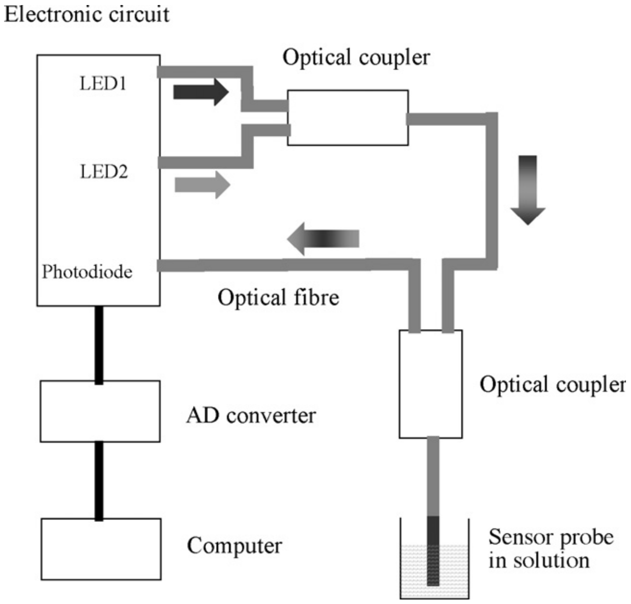

Figure 10. Experimental apparatus for the dual-colour fibre optic SPR sensor.

5. Demonstration of dual-colour optical fibre SPR sensor

We fabricated a dual-colour optical fibre SPR sensor with the sensor parameters determined by the preparatory experiment. The emission centre wavelengths of the two LEDs that we purchased are 612 nm (TOSHIBA, TLOE160A) and 680 nm (EPITEX, L680-02AU) and their bandwidths (FWHMs) are 15 nm and 20 nm, respectively. The used LEDs are of the surface emitting type. Using the experimental apparatus shown in figure 10, we estimated the fundamental characteristics of this sensor. Two LEDs are alternately flashed at a constant rate of 1 kHz and the emitted light is guided to the sensing part via two optical couplers (SUMITOMO, OBC-4002D). When coupling light from an LED to an optical fibre, one must take care of the coupling, since characteristics of SPR are sensitive to mode distribution in the fibre and could be affected by the coupling details. The same eight solutions as used in the preparatory experiment were used as the sensed media.

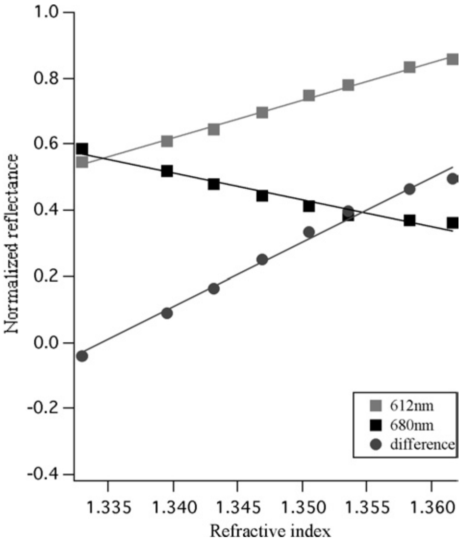

Figure 11. Measured characteristics of the dual-colour optical fibre SPR sensor.

Figure 11 shows sensitivity, linearity and the measurable refractive index range of the sensor. Comparing these characteristics with those shown in figure 7, we see that for the fitted line at 612 nm the reflectance at $n = 1.333$ is higher by about 0.15 and its slope is gentler. In the case of the fitted line at 680 nm, the slope also is gentler and close to flat over $n = 1.36$. Consequently, the sensitivity of this sensor is lower than the estimated one and the linearity decreases in a higher refractive index region. These differences can be explained by the difference between the designed and employed two wavelengths. The employed wavelengths of 612 nm and 680 nm are longer than the designed wavelengths of 609.6 nm and 675.9 nm, respectively. However there is another possibility that accounts for the differences. It is a shift of the resonance wavelength of the sensor towards a longer wavelength. This may be due to the adsorption of some contaminants onto the sensor surface. This is likely to occur when the SPR sensor is exposed to air for a while.

The limit of detection (LOD) of the refractive index, Δn, was also measured. With the sensing part of the sensor immersed in distilled water, the output voltage of the electronics of the sensor was monitored for 10 min. A variance of the normalized reflectance ΔR^2 at any wavelength is expressed as

$$\begin{aligned}\Delta R^2 = {}& \frac{1}{(V_{\mathrm{air}} - V_{\mathrm{BG}})^2}\Delta V_{\mathrm{sig}}^2 + \frac{(V_{\mathrm{sig}} - V_{\mathrm{BG}})^2}{(V_{\mathrm{air}} - V_{\mathrm{BG}})^4}\Delta V_{\mathrm{air}}^2 \\ & + \frac{(V_{\mathrm{sig}} - V_{\mathrm{air}})^2}{(V_{\mathrm{air}} - V_{\mathrm{BG}})^4}\Delta V_{\mathrm{BG}}^2, \qquad (2)\end{aligned}$$

where V_{sig} is the output voltage when the sensor is in a solution, V_{air} that in air and V_{BG} that when the sensor is not connected, and $\Delta V_{\mathrm{sig}}^2$, $\Delta V_{\mathrm{air}}^2$ and ΔV_{BG}^2 are their variances, respectively. Substituting the above measured voltages into (2) and considering the slope of the fitted line of the differential reflectance seen in figure 11, Δn is found to be 5.2×10^{-4}. This value is a bit worse when compared to the traditional Abbe refractometer. Also, another fibre optic technique based on the Fresnel reflection [11] has achieved the refractive index resolution of 2.5×10^{-5}. The coupling efficiency of the LED/optical fibre is currently around 10^{-2}. Also, there is non-negligible leaking of propagating light in the optical couplers. If these two issues are improved, three denominators in (2) will increase and consequently, Δn will be greatly decreased. Adding a temperature controller to the LED driver circuit will also contribute to decreasing Δn.

6. Conclusion

We proposed and demonstrated a dual-colour optical fibre SPR sensor. By analysing reflectance SPR spectra, the optimal gold thickness and two working wavelengths for the sensor to give the highest sensitivity were found. It was also found that the spectral bandwidth of the light source affects the characteristics of the sensor. Based on the results, a dual-colour optical fibre SPR sensor with the gold thickness of 60 nm and working wavelengths of 612 nm and 680 nm was fabricated and its fundamental characteristics were evaluated using distilled water and ethanol solutions. The proposed sensor responds almost linearly to a refractive index change between 1.333 and 1.3616.

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