REVIEW ARTICLE

Surface plasmon resonance: physics and technology

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Over the last few decades, surface plasmon resonance (SPR) technique has been very promising for sensing applications. It involves light-matter interaction at the interface of the metal and dielectric. This technology is employed in physical, chemical and biological sensing applications. In this review, we present the principle of SPR, different configurations used for excitation of SPR, performance characteristics of a sensor and commercialization of the biosensors technology. A few applications of SPR as biosensors have also been reviewed in this article.

Keywords: Biosensor, optical fibre, surface plasmon resonance.

IT is well known that the surface plasmon resonance (SPR) has been a promising technique for the last few decades. It has many applications in physical, chemical and biological fields[1,2] and also in food safety[3,4]. The first observation of SPR was made by Wood[5] in 1902 on diffraction grating. When a polarized light is shone on a mirror with grating, a dark and bright pattern was observed. This was popularly referred to as Wood anomaly. In 1941, Fano[6] concluded that the anomalies observed by Wood were due to the excitation of surface wave on the surface of grating. Ritchie[7], in 1957, theoretically demonstrated the excitation of surface plasmons (SPs) on metal surface. In 1958, Turbadar[8], while illuminating thin metal films on a substrate, observed a large drop in reflectivity. However, this effect was not linked to SPs. Later Otto[9] in 1968 pointed out that this drop was due to excitation of SPs by attenuated total internal reflection. Kretschmann[10], in 1971, modified Otto's configuration by placing the metal layer below the prism. In the 1970s, SPs were employed for thin film characterization[11,12] and as a probe for electrochemical interface[13]. Subsequently, the SPR phenomenon led to the development of sensors, especially aimed for gas detection and bio-sensing. A formal theoretical study was then proposed for SPR biosensor by Liedberg *et al.*[1] in 1983. It may be emphasized that the SPR biosensor's principle, development and applications have been reviewed by several groups[14–19].

Besides being bulky, the prism-based SPR sensors have the limitation of being manufactured on a larger scale as they involve the use of both optical and mechanical parts. Moreover, these sensors cannot be used for remote sensing. After the introduction of Kretschmann configuration for SPR sensing, efforts were made to develop optical fibres for sensing applications[20,21]. The advantage of using optical fibres is that they have smaller core diameter which allows them to be deployed in small areas and hence used for remote sensing. Optical fibre is highly preferred for sensing applications because of salient features such as high sensitivity, high precision, compactness, flexibility, etc. In recent times, SPR sensors made of photonic crystal fibre (PCF) have attracted considerable attention. Owing to the small size and design flexibility of PCFs, it is possible to control the evanescent field propagating into the core to excite SP. It is also possible to obtain a wide sensing range by optimizing the structural parameters of PCF[22–27]. In general, transfer matrix method (TMM) and finite element method (FEM) are adopted for computing the optical properties of PCF[28,29].

It is well known that SPR is observed only in conductive materials such as gold, silver, etc. Of these, gold (Au) is most widely used for SPR applications as it exhibits plasma resonance in the visible region of the electromagnetic spectrum. It is to be noted that silver (Ag) is less preferred as it is chemically unstable besides being prone to oxidation. To protect silver from oxidation, the silver films are covered with self-assembled monolayers (SAMs) of *n*-alkanethiol[30]. Zynio *et al.*[31] proposed the idea of using Au–Ag as bimetallic layers for prism-based SPR sensors. This bimetallic configuration was later used for fibre optic SPR sensor[32]. Most recently SPR has attracted considerable attention in negative index material (NIM) which is a sub-class of metamaterials. NIMs have a composite structure whose real part of the effective refractive index is less than zero. They are artificial materials engineered to fulfil certain electromagnetic properties[33]. Instead of conventional metals, SPR sensors have also been recently realized by exploiting exotic electromagnetic properties of metamaterials for chemical and biological sensing applications[34,35].

Principle of surface plasmon resonance

Electric field, when applied at a point on a metal, causes movement of free electrons. These free electrons get

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attracted by a background positive ion and the restoring force pushes the electrons in the opposite direction. This collective oscillation of electrons is plasmons. SPR refers to the excitation of SP at the interface of two media with dielectric constants of opposite polarity, say, a metal and a dielectric. In a dielectric medium the SPs have greater propagation constant than the light wave due to which they cannot be excited by direct light. However, they can be excited by a TM polarized light. The electric field intensity of SP decays exponentially at the interface of the metal and dielectric as shown in Figure 1 (ref. 16). When the SPs are excited there exists an energy transfer from incident light to SPs due to which the intensity of the reflected light is reduced.

Methods of SP excitation

For exciting SP by light, the incident light propagation constant and SPR propagation constant should be equal. Since a metal has negative dielectric constant and a dielectric medium has positive dielectric constant, at a given frequency, the propagation constant of SP is greater than the incident light. Thus direct light cannot excite SPs. Therefore, many techniques have been proposed to excite SPR such as prism coupling[10], grating coupling[36], waveguide coupling and recently by optical fibre[20]. The most common technique is the prism coupling in which there are two different configurations, namely, Otto configuration and Kretschmann configuration.

Otto configuration

In 1968, Otto explained the excitation of SPR by prism coupling. The concept here lies in the coupling of the evanescent wave and the surface plasmon wave. When a light beam is incident at an angle, greater than the critical angle, the evanescent wave arises at the bottom of the prism due to attenuated total internal reflection[9]. For the excitation of SPR, a metal layer is placed below the prism, leaving a gap between them as in Figure 2. A sample liquid of lower refractive index than that of the prism is filled in the gap. Then, the evanescent wave at the interface of prism and liquid excites the SPR at the interface of the metal and dielectric, if the wave vectors of both match.

Kretschmann–Raether configuration

In Otto configuration, it is quite difficult to maintain the gap between the metal and prism. This glitch was resolved by Kretschmann–Raether in their new configuration[10] which is shown in Figure 3. In this configuration, a metal layer is coated below a high refractive index prism and is then kept in contact with the sensing medium of lower refractive index.

When a plane polarized light is incident on the interface of the prism and metal, a part of the light gets reflected into the prism and a part gets transmitted into the metal as an inhomogeneous electromagnetic wave[37]. This is referred to as evanescent wave which decays exponentially. The evanescent wave gets coupled with the SP

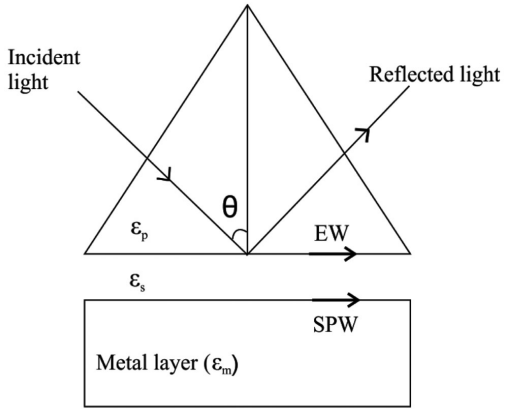

Figure 2. Excitation of SPR using Otto configuration.

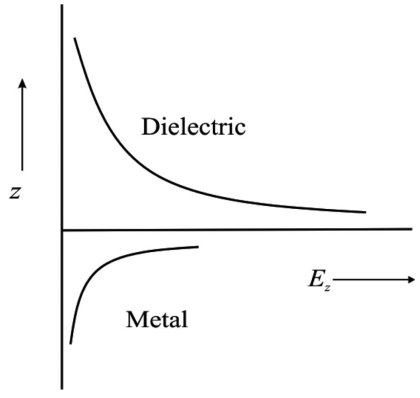

Figure 1. Variation of electric field intensity across the transverse direction at the metal-dielectric interface.

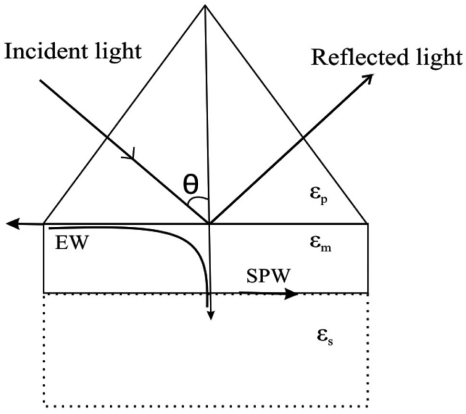

Figure 3. Excitation of SPR using Kretschmann configuration.

wave when the propagation constants of both waves are equal. The first commercialized SPR biosensors were bulky in nature, but today the biosensors based on Kretschmann configuration are compact, portable and cost-effective.

Excitation of SPR in grating structures

Excitation of SPR can also be done by diffraction of light with diffraction grating. When light from a dielectric medium is made to fall on a metal grating, a series of diffracted waves are produced. These diffracted waves get coupled with SP waves if the propagation constants of both the waves are equal. The excitation of SPR at the grating surface is shown in Figure 4. The modelling of grating-based SPR sensing device is complex and hence analysis of these devices is quite challenging.

Excitation of SPR in waveguides

The principle of excitation of SPR in a waveguide structure is similar to that of Kretschmann configuration. In this case, the modes of dielectric waveguide excite the SPs. As shown in Figure 5, when a waveguide mode reaches the metal layer, it evanescently penetrates into the metal layer and then gets coupled with SP at the external surface of the metal. This coupling occurs when the propagation constant of both modes is equal. The deployment of waveguide in SPR sensors facilitates effective controlling of the properties of light. The strength of coupling depends upon the thickness of the metal layer and the interaction length. Thus, one can obtain miniaturized and easily controllable sensors using the waveguides.

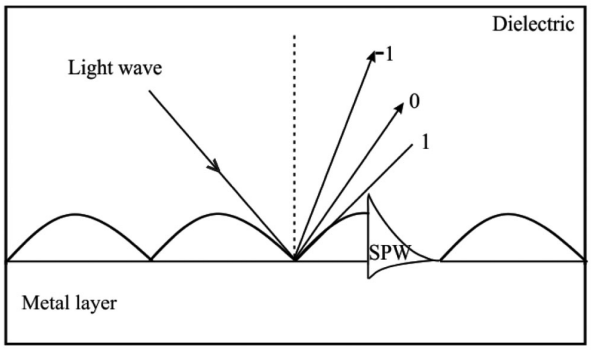

Figure 4. The diffraction of light at the surface of grating.

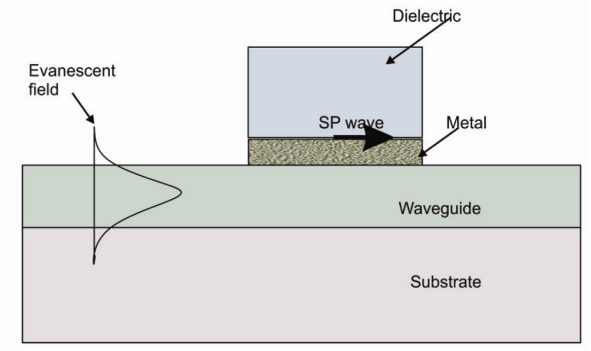

Figure 5. The excitation of SPR using waveguide modes

Excitation of SPR in optical fibres

Apart from communication, optical fibres have also been exploited for realizing sensors. As the diameter of an optical fibre is in the micro-metre range, it is quite possible to fabricate ultra-miniaturized SPR sensors using optical fibres. In optical fibre SPR sensor, a small region of cladding is removed and is coated with a metal layer. As shown in Figure 6, the metal layer is placed in contact with the medium to be sensed. In an optical fibre, the light is guided by total internal reflection at the boundary of the core and cladding. Here, the evanescent wave that decays exponentially is generated at the cladding region.

In general, the optical fibre SPR devices exhibit high sensitivity and they may also be used for chemical and biological sensing applications. Further, these sensors are used for measuring the surface roughness, refractive index of a liquid, temperature, glucose level and different kinds of gases.

Performance parameters

To enumerate the performance of any sensor, some vital parameters such as sensitivity, detection accuracy, resolution, repeatability, reproducibility, detection limit, drift, robustness, selectivity, etc are to be defined.

Sensitivity

The ratio of change in sensor output (say, wavelength, intensity, phase, etc.) to the change in quantity to be measured (say, analyte concentration, refractive index, etc.) is defined as the sensitivity of sensors. For instance, the

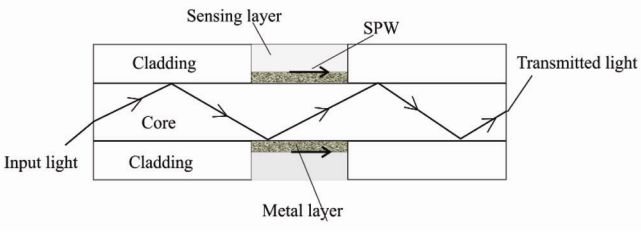

Figure 6. The fibre optic based excitation of SPR.

sensitivity of a refractive index based SPR sensor is given by

$$S = \Delta\lambda/\Delta n,$$

where $\Delta\lambda$ is the change in resonant wavelength and Δn is the change in refractive index.

Detection accuracy

The detection accuracy of a SPR sensor means how exactly a sensor can detect the refractive index and the resonance angle of the sensing layer.

Repeatability

The sensor's capacity to reproduce the output value over a short interval of time under the same measurement conditions is known as repeatability. The stability and lifetime of the biosensor head leads to good repeatability.

Resolution

The resolution of a sensor is the smallest change in quantity to be measured that produces a detectable change in the sensor output. The resolution is related to precision with which the measurement is made. Resolution is the characteristics of the detector but not of the sensing probe.

Reproducibility

Reproducibility literally means the ability of multiple sensors with the same underlying principle to provide a similar response to the analyte used. Further, the sensors should produce the same output even when the experiments are carried out at different places by different operators.

Detection limit

It is the lowest concentration of measurand that a sensor can detect. This value not only depends on the sensor, but also on the noise of the system. It is related to the sensitivity and resolution by the relation

Detection limit (DL) = resolution/sensitivity.

Robustness

Although optical microfibres are easy to fabricate, they are mechanically fragile, especially, in liquid environment for bio-sensing applications. To enhance the robustness of the microfibre in liquid environment, an additional mechanical support is provided.

Selectivity/Specificity

It is the capability of a sensor to specifically determine the analyte in a complex medium. Specificity does not involve any quantitative measurement, but it is just a property used in real sensing environment.

Drift

Drift is a small undesirable change in the characteristics curve of the sensor with time. If the drift decreases with time, it means that the sensor has lost its capability to produce output and has become old.

Sensitivity, accuracy, repeatability, detection limit, etc. are those parameters for characterizing the performance of any SPR biosensor.

Commercialization of SPR biosensor technology

The world's foremost commercialized analytical instrument based on SPR for studying biomolecular interactions was launched by BIAcore® in 1990. BIAcore, which is a part of GE Healthcare, launched many models of SPR biosensors, namely, Biacore® 1000, Biacore® 2000, Biacore® 3000, Biacore® X, Biacore® T100 (ref. 38). Later, Texas Instruments developed another SPR biosensor system called Spreeta™ evaluation kit (formerly called TISPR–1 experimenter's kit) which is the smallest SPR biosensor[39]. Further, BioTuL Bio Instruments GmbH developed Kinetic Instrument 1 (ref. 40), an SPR biosensor system. Both Biacore and Texas Instruments depend on the angular interrogation of SP which is excited by prism couplers. On the other hand, Quantech (USA) offers an SPR biosensor instrument that depends on the wavelength interrogation of SP in grating-based structures. It has been demonstrated recently that the BI 2500 SPR sensors are compatible for electrochemical and gas sensing[41]. GenOptics provides SPRi–Plex™ instrument for SPR imaging technology[42]. Of late, HORIBA Scientific has come up with the SPRi-Biochips™ and the SPRi-Slides™ for SPR imaging and for label-free analysis[43]. Biosuplar-6, a surface plasmon resonance spectrometer developed by Analytical μ Systems, has a unique advantage in SPR measurement in liquids and gases[44]. Figure 7 shows the various companies in the commercialization of SPR biosensor technology.

Specific applications of SPR-based biosensors

Fibre optic SPR sensors ensure wide applications in the detection of biological and chemical species which include

environment monitoring, food safety and diagnostics in the medical field. The sensing principle is based on the change in refractive index of the medium. In what follows, we delineate some of the applications.

Detection of dengue NS1 antigen using LRSPP

The non-structural 1 (NS1) antigen of dengue can be detected using a long range surface plasmon polariton waveguide (LRSPP)-based biosensor. LRSPP excitation is achieved using an optical fibre with a butt coupled to the metal waveguide. Three naturally occurring antibodies for dengue NS1 antigen can be used as a recognition element. The sensor die consists of gold stripes embedded on Cytop cladding, fluoropolymer with low index. The set-up is shown in Figure 8 (ref. 45). A polarization maintaining (PM) fibre is connected to the input of the LRSPP biosensor. The output beam is directed to the infrared camera and to a power metre to visualize the emerging mode and to observe real time changes in the output signal respectively. NS1 antigen is detected in blood plasma as well as in clean fluid.

Figure 7. Commercialization of SPR biosensor technology.

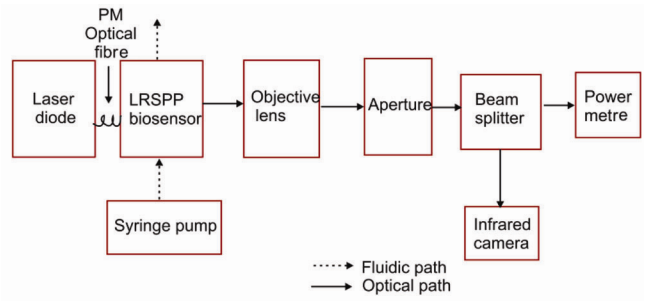

Figure 8. Experimental set-up for detection of dengue NS1 antigen[45].

Detection of pregnancy associated plasma protein

An SPR biosensor was proposed to detect pregnancy associated plasma protein A2 (PAPP-A2) in blood serum samples[46]. PAPP-A2 plays an important role in the development of the foetus and in post-natal growth. The serum samples were obtained from two groups of women: one from pregnant women and the other from healthy non-pregnant women. The detection assay image is shown in Figure 9: the primary antibody Ab1 is immobilized on the SPR sensor surface which captures the PAPP-A2. Later PAPP-A2 binds with the secondary antibody Ab2 followed by the binding of streptavidin coated gold nanoparticles. Single surface referencing (SSR) approach is used in this biosensor to detect PAPP-A2, where the functionalized surface of the SPR chip is split into the detection channel and the reference channel[47]. It was found that, serum obtained from pregnant women showed higher PAPP-A2 concentration.

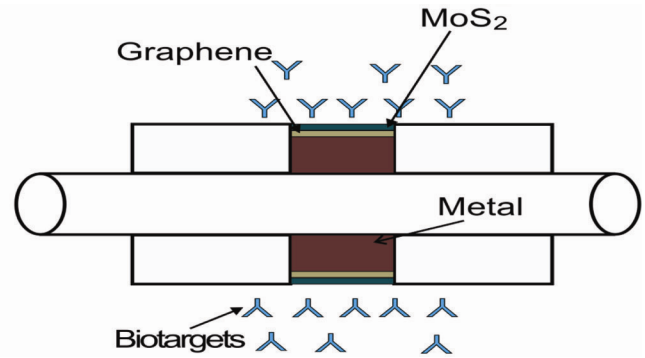

Figure 10. MoS_2 based fibre optic biosensor[48].

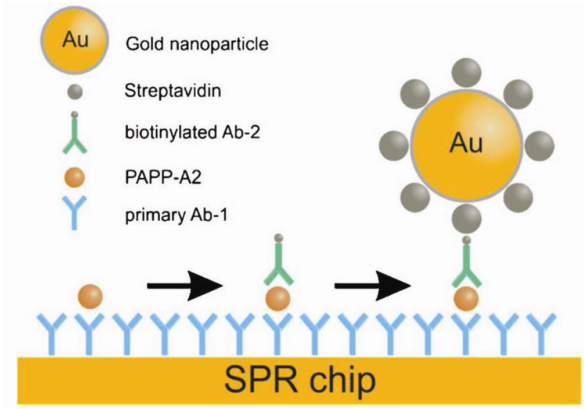

Figure 9. Schematic of detection assay[46].

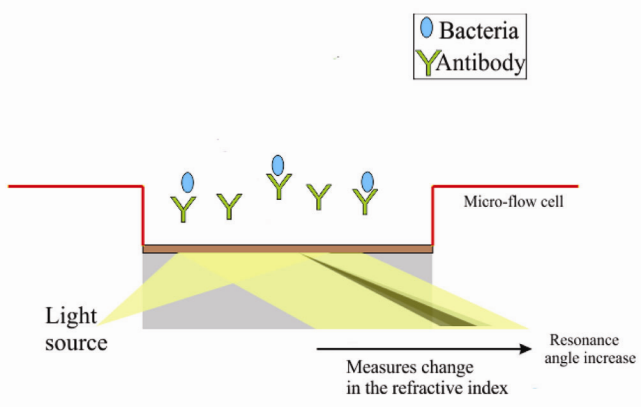

Figure 11. The set-up for food pathogen detection technique[51].

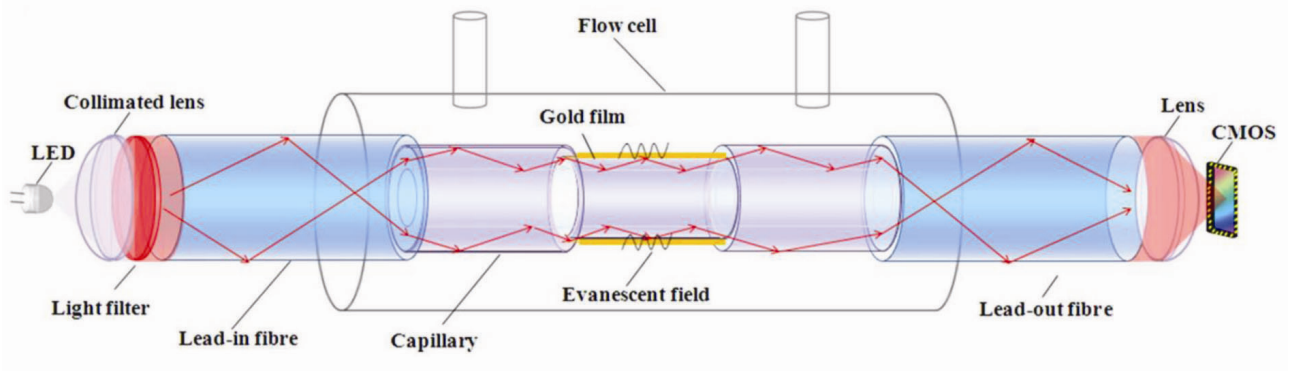

Figure 12. Smartphone based SPR biosensor[52].

MoS_2-based fibre optic biosensor

In an optical fibre, the cladding is removed over a length of 1 cm and the unclad portion is coated with a metal and then with graphene. Finally, a layer of molybdenum disulphide (MoS_2) is deposited[48]. MoS_2 is used in biological sensing applications as its surface is hydrophobic in nature and such surfaces have large affinity for protein-surface adsorption. The biotargets are bonded with the surface of the MoS_2 layer as shown in Figure 10. The metal used here includes aluminium (Al), copper (Cu) and gold (Au). Here, the sensitive detection of biosamples for three different configurations was carried out. It was found that the Cu/graphene/MoS_2 and Al/graphene/MoS_2 configurations exhibited high sensitivity towards the detection of biosamples in the visible region. On the other hand, Au/graphene/MoS_2 configuration was suited when the depth of resonance mattered the most. It is to be noted that MoS_2 serves as an efficient adsorbing layer for biosamples.

Detection of breast cancer antigen

Human epidermal receptor protein-2 (HER2) is a breast cancer associated antigen. It was reported that around 25% of population was diagnosed to have this antigen[49]. A glass slide is covered with a gold layer and a chromium layer is used as an adhesive layer and this arrangement forms the substrate. Here, biotin covalently gets attached to the streptavidin protein by the process of biotinylation. To detect HER2 antigen, the biotinylated antibody AB1 is immoblized on the surface of the substrate. A secondary antibody AB2 is used to characterize the detection mode. It is a challenging task to detect an antigen by spectral monitoring. However, an antigen, when conjugated with huge secondary antibody, produces larger refractive index change which can be detected by a spectral band shift. A shift of 1.2 nm is observed during AB1 immobilization. The extra redshift of 1.3 nm is gained when AB2 is connected to AB1/HER2 layer. A total band displacement of 2.5 nm is obtained. The high signal-to-noise ratio owes to efficient detection of HER2 antigen. This sensor is able to detect the biomarker for a given concentration of 30 ng ml^{-1} (ref. 50). The SPR substrate shows good sensitivity when compared to other plamonic biosensors.

Detection of foodborne pathogens

An SPR-based biosensor has been proposed for selective detection of *Salmonella enteritidis* (SE) in food samples. The set-up is shown in Figure 11 (ref. 51). Antibodies are immobilized on the gold surface and various concentrations of SE solutions are driven into the system. The changes in resonance angle at various concentrations were studied. The immobilization of the anti-*Salmonella* results in a notable change in the resonance angle. A small amount of *Listeria monocytogenes* was attached to a surface coated with SE antibody. It was found that the response achieved by *L. monocytogenes* was less than the response achieved by SE. Furthermore, the change in resonance angle at different dilution levels was less than the detection limit of the target pathogen which, in turn, reflects on the high specificity of the biosensor.

Smartphone based SPR biosensor

In this SPR biosensor, an optical fibre is used to connect the optical components and the sensing element to the phone case[52]. Light from the phone's LED flash enters the lead-in fibres illuminating three channels namely measurement, control and reference channels; while the light from the lead-out fibres is detected by the camera of the phone. A small region of cladding is removed and coated with 50 nm gold film. The experimental set-up is shown in Figure 12. The lead-in fibre provides monochromatic light; when this light reaches the sensing area some of its modes enter into resonance with the gold film due to SPR effect. The binding process of the SPR sensor can be recorded by noting the intensity change of light passing via the sensing region. This system is apt in detecting real time biological interactions.

Biodetection of pathogenic bacteria using bacteriophages

Bacteriophages are used as a bio-recognition element of bacteria. T4 bacteriophage is used to detect *Escherichia coli* (*E. coli*) bacteria and a novel bacteriophage BP14 is used to detect Methicillin-resistant *Staphylococcus aureus* (MRSA) bacteria. A super luminescent light emitting diode (SLED) is used as a light source. A 50 nm gold sensing layer is placed above a coupling prism. A flow cell is developed for real time test. The whole set-up is placed on a goniometer to permit surface plasmon excitation on the gold surface. To record the resonance dip, a CCD camera is used and the phage of the target bacteria is connected to the gold layer of the SPR system. The arrangement is shown in Figure 13 (ref. 53).

The gold chip is coated with bacteriophage. A phosphate-buffered saline (PBS) solution is injected for 10 min to create a baseline followed by injection of PBS solution of *E. coli* for another 20 min. Bacteriophage will lyse the bacteria within 20 min and then the bacteria gets attached to the bacteriophage. This method can be used to detect targets for other bacteria like anthrax.

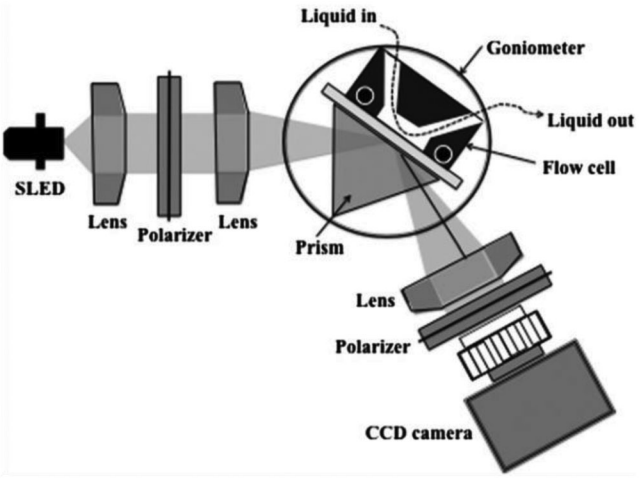

Figure 13. The set-up for detection of bacteria using bacteriophages[53].

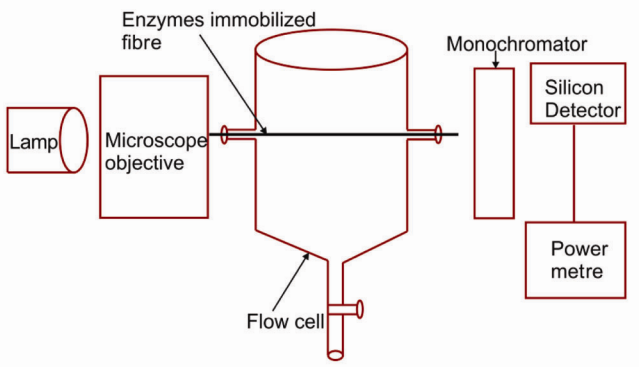

Figure 14. Experimental set-up to detect the presence of naringin in fruit[54].

Naringin content in citrus fruit

Treating citrus fruits has problems in terms of bitterness, which makes it difficult for consumer acceptability. This bitterness is due to presence of naringin in the fruit. An SPR-based fibre optic sensor is used to detect the naringin presence in the fruit[54], whose set-up is shown in Figure 14. Here, the spectral interrogation method is used for detection of naringin. To immobilize the naringinase enzyme, a technique called gel entrapment is used. The light from a tungsten halogen tube is concentrated on one end of the fibre through the circular slit. The probe is mounted in the flow cell and the sample placed across the probe. A silicon detector and power metre detect the output power from the monochromator. The transmitted power from the fibre to the sample is determined at different wavelengths. These powers are divided by the corresponding powers obtained when there is no sample. The SPR spectrum is obtained to study the response characteristics. There is an increase in resonance wavelength as the naringin concentration increases. This occurs when there is an increase in the refractive index of the immobilized layer.

Conclusion

In this review article, we have first discussed the principle of the SPR technique. In the beginning, prism-based SPR sensors were developed using this technique. Later the progress resulted in the development of fibre optic-based SPR sensors. Further, we have discussed various configurations for exciting SP. In order to quantify the performance of the sensors, various characteristic parameters of SPR sensors have been outlined. SPR biosensors used in detecting various chemical and biological analytes have been addressed. Besides, a few applications of such biosensors have also been included. The proliferation of the concept of SPR has paved the way for the ultimate commercialization of SPR biosensors and the same has been dealt with.

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Received 8 September 2017; accepted 30 December 2017

doi: 10.18520/cs/v115/i1/56-63