Chemical and Biological Applications Based on Plasmonic Optical Fiber Sensors

Chiara Perri, Francesco Arcadio, Girolamo D'Agostino, Nunzio Cennamo, Giovanni Porto, and Luigi Zeni

he use of optical fibers has evolved from an optical transmission waveguide to a more intricated device for bio-chemical sensing. The possibility of measuring target substances with rapid and low-cost tools is still a main challenge in different application fields, from environmental monitoring to the medical-diagnostic sector. In recent decades, plasmonic fiber-optic sensors have gained great interest due to their excellent sensitivity and compactness, allowing the possibility of remote sensing for different target analytes that is real-time and label-free. Such sensors have constantly evolved over time. In this work, a short overview on plasmonic optical fiber sensors is presented, including illustrations that focus on the applications and achievements so far.

Introduction to Plasmonic Sensors

Surface Plasmon Resonance (SPR) is one of the fastest growing research fields which investigates the interaction at the nanoscale between photons (light) and surface electrons of a metallic layer [1]. In SPR sensors, the metallic element is in contact with a Molecular Recognition Element (MRE), which locally changes its refractive index (RI) when interacting with the target analyte. This change affects the electromagnetic wave propagating along the metal–dielectric interface in a highly sensitive manner [2], [3].

By exploiting different types of MRE, it has been possible to produce several applications up to now, linking scientists from diverse backgrounds such as chemistry and physics with medical sciences. Although the most common type of plasmonic sensor is typically prism-based (Kretschmann configuration) [4], this kind of sensor needs bulky setups that do not allow field measurements and are challenging to the development of small and low-cost Point-Of-Care (POC) devices (medical diagnostic tests which can be performed at or near the point of care of patients, for instance at bedside). By replacing the prism with an optical fiber, in which light propagates through the phenomenon of Total Internal Reflection (TIR), all limitations due to the Kretschmann configuration can be easily overcome. In recent decades, plasmonic sensors based on optical fibers have constantly evolved, from

the first silica glass fibers-based to the more recent polymeric ones [5]–[7].

In this work, we report on plasmonic optical fiber sensors based on silica optical fibers, then on those based on plastic optical fibers. After these sections on optical fiber sensor platforms, the work recalls a simple and highly sensitive sensing approach to realize optical chemical sensors: plastic optical fibers (POFs) combined with molecularly imprinted polymers (MIPs).

Surface Plasmon Resonance Sensors Based on Silica Optical Fibers

Various designs and geometries have been proposed for the realization of surface plasmon resonance sensors based on optical fibers [8]. The simplest configuration is based on unclad fibers, where a segment of the core is exposed by removing the cladding and covered with a metallic layer or metallic nanoparticles (for instance, gold). Unclad sensors have been developed in recent years, from very large core fibers in the range of 200 μm and 600 μm, instead of small single-mode core fibers. P. Bhatia and B.D. Gupta realized a SPR fiber-optic sensor for urea detection in biomedical applications [9]. In this work, the authors prepared the probe using a silica fiber of 600 µm core diameter of 20 cm length, from which 1 cm length of the cladding was removed from the middle. The unclad portion of the fiber was first coated with a 40 nm layer of silver and then an 8 nm layer of silicon to enhance the sensitivity, through the evaporation technique. Finally, the urease enzyme was immobilized on the silicon layer. The probe was characterized by spectral interrogation and was able to measure solutions with concentrations from 1 mM to 160 mM, which are close to physiological blood concentrations.

In another work carried out by A.M. Shrivastav *et al.* [10], an SPR fiber-optic sensor based on molecular imprinting was fabricated for the detection of profenofos, a type of organophosphorus pesticide (OPPs) used for controlling pests in farming, which can cause injurious effects to the nervous system of humans even at low concentrations. The sensing probe was realized by coating the unclad core of a multimode fiber

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with a 40 nm silver layer and a film of MIP specific for profenofos. The realized sensor detected profenofos in concentrations ranging from 10^{-4} – 10^{-1} µg/L, with a Limit Of Detection (LOD) of 2.5×10^{-6} µg/L.

Plasmonic sensors can be realized also using side-polished (or D-shaped) fibers, which have a planar segment exposing the core used as the sensing region [11], tapered fibers or U-shaped fibers to enhance the optical performances.

Tapered fibers are formed by heating and softly stretching along the propagation axis. This method makes optical fibers thinner, typically a few millimeters or centimeters over a certain length. The fiber core is also thinner by the same factor as the overall fiber, and the evanescent wave from the core ultimately hits the outer surface and is exposed to the medium around it. The SPR sensor results as a metallic layer is placed over the tapered region of the fiber [12].

Similarly, with the help of a heat source, U-shaped plasmonic fiber-optic sensors are realized by bending. The obtained U-bent region is de-cladded, and a metallic film or nanoparticles are deposited on the surface of the probe. A U-shaped fiber optic probe coated with glucose-capped silver nanoparticles (Ag NPs) for the detection of mercury ions in aqueous solution was presented by Shukla *et al.* [13]. The authors prepared the probe by cutting 1 cm of the length of the cladding in the middle of the fiber and bending the optical fiber using a butane flame. After a washing step of the U-bent region based on an acid-alkali procedure, the probe was dipped in glucose-capped Ag NPs solution. The glucose-capped Ag NPs-coated optical fiber probe was then dipped in mercury solutions of different concentrations, reporting a limit of detection for mercury of 2 ppb [13].

Plasmonic Sensors Based on Plastic Optical Fibers

Due to their excellent flexibility, simple handling, wide numerical aperture, large diameter, and the fact that plastic is capable of withstanding smaller bend radii than glass, POFs are particularly advantageous over silica fibers. Thanks to such properties, which increased their popularity and competitiveness in telecommunications, they are preferred in the development of optical sensors as well, since they have easier manufacturing and handling processes [14].

Recently, Boruah *et al.* developed a portable optical U-shaped fiber sensor for heavy metals detection in aqueous medium [15]. The authors manufactured the sensing probe from a 10 cm length plastic optical fiber (core diameter 600 µm), from which 2 cm cladding was removed from the middle using a surgical blade. The unclad portion was inserted in a glass capillary tube of diameter 1 cm and heated to develop U-shaped probe of 1 cm radius. Next, the sensing probe was coated with oxalic acid-functionalized gold nanoparticles (Au NPs) for the selective binding of Pb2+ ions. Sensing operations were performed using a setup consisting of a white LED as light source and an optical detector. The use of a LED instead of a spectrometer allowed the sensing setup to be compact and eased the handling process. The sensing region of the prepared

probe was then placed in contact with liquid samples of Pb²⁺ and the obtained detection limit was 2.1 ppb, which is below the WHO guidelines value of 10 ppb [15].

Plasmonic sensors can be implemented in plastic optical fibers using different geometries, such as D-shaped fibers as well. In 2011 Cennamo et al. designed a SPR sensor configuration based on a D-shaped POF [16], aiming at producing a very highly sensitive, robust, low-cost, and reliable sensor. The authors realized the SPR sensor as a 10 mm long D-shaped POF that can be monitored and exploits only two components: a white light source and a spectrometer. First, a POF was embedded in a resin block with a specific trench. Then, the D-shaped was obtained by removing the cladding of a 980 µm core plastic optical fiber along half the circumference by using two different polishing papers (5 µm and 1 µm grit). A buffer of Microposit S1813 photoresist was spun on the exposed core, and finally a thin gold film was deposited by sputtering technique [16]. Since then, several applications were developed with this optical platform in the environmental, industrial and medical sectors by coupling the D-shaped POF sensor with biological receptors (antibodies, aptamers, etc.) or synthetic receptors such as MIPs [17]-[20].

Other SPR sensors implemented in D-shaped POF have been realized. Recently, Junxia Sun et al. realized a sensitive and selective SPR sensor that employs gold-supported graphene composite film/D-shaped fiber to detect dopamine, an important neurotransmitter in the human body [21]. To obtain the D-shaped sensor, the authors first exposed part of the core in the middle of a 20 cm long POF to form a 15 mm long D-shape area. Then, the D-shape region was polished with sandpaper, cleaned and modified with a hybrid gold/ graphene layer. In particular, the sensitive layer was produced using a one-step procedure where the gold film was used as a supportive layer for graphene transferring, so that the sensitive layer could be fixed directly in the D-POF. Finally, a dopamine binding aptamer (DBA) was immobilized on the Au film/graphene D-POF sensor. The proposed sensor demonstrated good sensitivity and selectivity by detecting dopamine in the range of concentration from 10⁻¹⁰M to $10^{-6}M$ [21].

Up to now, several POF-SPR sensors have been realized using biological receptors (antibodies, enzymes, aptamers, etc.) and chemical receptors (nanomaterials, Molecularly Imprinted Polymers, etc.). Chemical receptors present some advantages over the biological ones, thanks to their low cost and resistance in wide ranges of pH (from acids to alkaline values) and temperature.

Low-Cost Optical-Chemical Sensors: Plasmonic POFs Combined with MIPs

Among receptors, MIPs are gaining great interest in low-cost sensor development. MIPs are completely synthetic materials with molecular recognition sites. The synthetic process implies a co-polymerization between functional monomers and a cross-linking reagent with a template molecule (target analyte). The template molecule is coordinated in specific

points by the interaction with the functional monomers, and then the complex is fixed by the cross-linking agent in a highly organized structure. At the end of the process, the template is removed, leaving the molecular recognition sites free [22].

Initially, MIPs were mainly used as sorbents media for stationary phases in chromatography, in solid phase extraction (SPE), carriers in drug delivery and diagnosis, in reactors for catalysis, and for remediation purposes [23]. Although these first applications were very focused, later they have increasingly been exploited also as recognition elements in sensors development, establishing their role as potential substitutes for biological recognition elements [24].

Indeed, compared to their biological counterparts, MIPs demonstrated many advantages: higher stability in thermal, chemical and mechanical conditions; high performance in aqueous/organic mixtures and pure solvents; low-cost in manufacturing processes without animal experimentation phases; template recovery; and the possibility to use template analogues instead of harmful analytes [25].

Although the first applications of MIPs as MRE in sensors were of interest mainly for electrochemical transducers, their use gradually extended to optical sensors, thanks to their immunity to electromagnetic field interferences. Up to now, several MIP-based SPR sensors have been realized in the most varied application fields.

In 2013, Cennamo *et al.* realized an optical chemical sensor based on SPR and MIP for the detection of Trinitrotoluene (TNT) in aqueous solution [26], particularly interesting for the security field. The SPR sensor was prepared as mentioned before [16]. The planar gold layer was employed for depositing an MIP layer used as the specific receptor. A very low volume of 100 μ l of the prepolymeric mixture containing TNT as the template molecule was dropped directly on the D-shaped gold surface of the POF and spun at 1000 rpm for 35 s. The polymerization was then carried out at 70 °C for about 16 h. Then, TNT was extracted by an appropriate washing procedure. The

experimental setup, arranged to measure the transmitted light spectrum, was characterized by a halogen lamp, illuminating the optical sensor system and a spectrum analyzer. The sensor was characterized by dropping aqueous solutions of TNT directly on the D-shaped chemically modified sensing region. The realized sensor demonstrated the ability to detect TNT even at low concentration, down to about 50 µM [26].

In 2015, the same authors realized a different sensor configuration for TNT detection, this time based on LSPR with improved performances. LSPR was excited in five-branched gold nanostars (GNS) suspended in the MIP (GNS-MIP) specific for TNT [27]. To better investigate the sensitivity, the sensing layer was deposited directly on two different POF platforms: tapered and not-tapered. Both sensors showed better performances than the previously proposed, in which the SPR was excited in a thin gold layer at the surface of the POF in contact with the MIP layer (specific for TNT). In particular, the sensor with a GNS-MIP sensing layer on the not-tapered POF exhibited a sensitivity of 8.5×10^4 nm/ M, three times higher than in the gold layer sensor. The sensor with GNS-MIP sensing layer on the tapered POF demonstrated a further sensitivity up to 8.3×10^5 nm/M, thirty times higher than in the gold layer sensor [27].

Recently an interesting application of SPR-POF-MIP sensors has been developed for the detection of a particular class of emerging pollutants, perfluoroalkyl substances (PFAs).

These pollutants are widely distributed in the environment, and it is possible to detect them in various kinds of micro-polluted water, such as river water, lake water and seawater. Due to their chemical stability, they are particularly inert and refractory to different chemical and microbiological treatments. Consequently, they are persistent, bio-accumulative and toxic to mammalian species. In fact, the immune-toxic effects of PFAs are largely demonstrated. Thus, great efforts must be done to identify possible novel approaches for water treatment and/or detection of the PFAs in the environment. The detection of such substances is performed by using

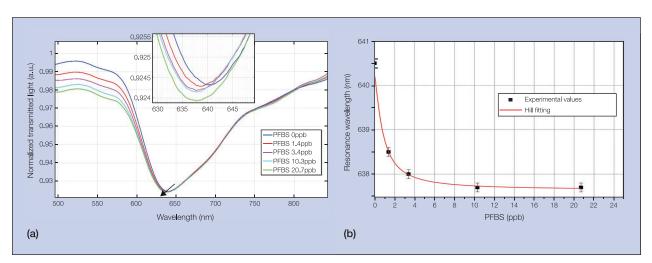


Fig. 1. (a) Resonance spectra obtained at different concentrations of PFBS in water solution (0 – 20.7 ppb) by a plasmonic POF-MIP sensor. Inset: zoom of the resonance wavelengths. Adapted from Cennamo et al. [28]. (b) Dose-response curve (resonance wavelength versus PFBS concentration) along with Hill Fitting of data [28]. (Figures used with permission, ©IEEE, 2019).

conventional methods mainly based on high performance liquid chromatography-mass spectrometry (HPLC-MS), which are expensive and time-consuming.

In this framework, Cennamo et al. realized a novel approach for the detection of perfluorobutanesulfonic acid (PFBS) in water [28], exploiting a low-cost optical chemical sensing strategy that is based on POFs and MIPs. PFBS is a C4 perfluoroalkyl molecule which is difficult to remove with common adsorbent media (for example, active carbons or ion exchange resins). However, using an MIP it has been possible to adsorb this substance, and the relative refractive index variation of the MIP has been detected by SPR-POF technique. The D-shaped POF-SPR platform, prepared as described in [16], was coupled with an MIP able to recognize PFAs [29]. By increasing the concentration of PFBS in water solution, the resonance wavelength is shifted to smaller values, as shown in Fig. 1a, while Fig. 1b presents the resonance wavelength as a function of the PFBS concentrations along with the Hill fitting of the experimental data.

A similar shift, already observed with perfluorooctanoic acid (PFOA) or perfluorooctane sulfonic acid (PFOS) interaction with the same MIP receptor [29], indicates a decrease in the refractive index (RI) value of the MIP layer when the binding occurs. The obtained LOD of PFBS in water solution was lower than 1 ppb [28]. For this specific substance, there is not a maximum residue limit fixed by the European Union regulations yet. The proposed analytical approach demonstrated the feasibility of monitoring pollutants in water, for example PFAs, exploiting this kind of low-cost optical chemical sensor. As many MIP receptors can be deposited on POF platforms to detect various substances, the proposed method can be a promising tool in Water Quality (WQ) monitoring in smart city applications, considering that can be also conveniently linked to the internet [20].

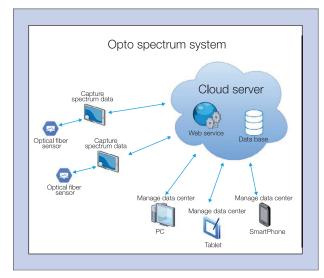


Fig. 2. System architecture of Opto Spectrum System, a low-cost IT system capable of storing and appropriate processing large amounts of data proposed by Cennamo *et al.* [20]. (Adapted from [20], used with permission, ©IEEE, 2020).

Conclusion

A brief overview on plasmonic optical fiber sensors has been presented, focusing on some of the latest applications and achievements related to their implementation. Among surface plasmon resonance sensors, the class based on plastic optical fibers represents the most promising approach for rapid and real time sensing, thanks to the low-cost manufacturing process and their easy handling. Their suitability to be integrated with different kinds of MRE will allow future development of other applications in many fields, addressing topics that have been challenging to date such as environmental water monitoring, on-field measurements and the development of medical-diagnostic Point-Of-Care devices. Furthermore, the possibility of connecting such types of sensors to the internet makes them suitable for the development of several remote sensing applications, opening new research activities, especially towards IoT (Internet of Things) integrated technologies.

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