

Optical Detection of Volatile Organic Compounds: A Review of Methods and Functionalized Sensing Materials

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With growing awareness of the detrimental effects of volatile organic compounds (VOCs) on human health and their potential in disease screening, the demand for effective detection methods has surged. Optical detectors are highly desirable because of their excellent versatility, fast response time, and suitability for remote detection. This review aims to analyze various optical sensors for VOC detection, focusing on the functionalizing materials used in different optical transducers. The review is organized into six sections, each covering a different sensor category based on the type of optical phenomenon utilized for VOC detection: diffraction, interference, polarization change, optical resonance, absorption, and evanescent wave propagation. Achievable sensitivities, limitations, and advantages of each approach are discussed, providing valuable insights for the development of advanced VOC detection technologies.

1. Introduction

1.1. Volatile Organic Compounds: Sources and Impact on Human Health

Volatile organic compounds (VOCs) are organic air pollutants with boiling points between 50 and 260 °C.^[1] Under typical indoor temperature and humidity conditions, VOCs primarily exist in the gas phase. They cause a wide range of negative health effects, including respiratory, cardiovascular, neurological, and carcinogenic diseases.^[2] Specifically, VOCs are known to cause pulmonary issues such as asthma, wheezing, and throat irritation.^[3] The most common VOC exposure-related cancer is lung cancer, followed by breast, gastric, skin, thyroid, and prostate cancers.^[4] Some

of the most harmful and carcinogenic VOCs include acetaldehyde, benzene, ethylbenzene, formaldehyde, naphthalene, styrene, and tetrachloroethylene.^[2] Aromatic hydrocarbons have been linked to skin and nasal irritation, while benzene poses significant health risks, such as preterm birth and leukaemia.^[5]

Sixty-five of the most abundant individual compounds were identified by reviewing sources, concentrations, and health effects in European residences in 2022.^[2] The list of sources of the most health-concerning analytes is highly ubiquitous, including, among others, composite boards, ceiling tile, glues, wallpapers, paints, fireplaces and heaters, candles, cosmetics, sprays, air fresheners, cleaning products, and floor coverings. Overall, 52% of identified VOCs were sourced from construction and building materials, 41% from consumer products, and the rest from space heating. Predominantly in summer and always in winter, concentrations of VOCs inside houses exceed those outside.^[6] VOCs most often found were n-butane, ethanol, acetone, and propane. Studies in China have shown significantly higher levels of aromatic hydrocarbons compared to residences in other countries.^[5] Further investigations into indoor air quality during the interior finishing stages of two residences revealed median VOC concentrations of 50.71 and 57.81 $\mu\text{g m}^{-3}$, notably higher than outdoor levels of 31.83 and 39.89 $\mu\text{g m}^{-3}$, respectively.^[7] The highest contributions came from BTEX (Benzene, Toluene, Ethylbenzene, Xylenes)^[8] compounds, notably toluene, followed by aliphatic hydrocarbons. Additionally, separate measurements showed a median formaldehyde concentration of 40 $\mu\text{g m}^{-3}$.

Apart from indoor air monitoring, a major VOC sensor application is breath analysis. VOCs are present in everyone,

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Table 1. List of selected VOCs with their concentrations and biomarkers use.

VOC ^{a)}	Chemical Family	Concentration (Indoor, PPB)	Osha Permissible Exposure Limits (PPM)	Concentration (Breath, PPB) ^[10]	Biomarker Use ^[11]
Acetone	Ketone	3 – 30 ^[2] 18 (median) ^[6]	500	42–753	Asthma Covid-19 Diabetes Gastric cancer Lung cancer Malaria Sleep apnoea
Benzene	Aromatic hydrocarbon	0.03 – 3 ^[2] 0.16 (median) ^[6]	1	4–12	COPD Lung cancer Malaria
Ethanol	Alcohol	49 (mean) ^[2] 21 (median) ^[6]	1000	5-480	Breast cancer Covid-19 Diabetes Lung cancer
Isopropanol	Alcohol	0.4 (mean) ^[2]	400	13.3 (average)	Breast cancer COPD Diabetes Lung cancer
Methanol	Alcohol	0.2 (median) ^[6]	200	450 ^[18]	Breast cancer Covid-19 Diabetes Lung cancer
Toluene	Aromatic hydrocarbon	0.5 – 13 ^[2] 0.4 (median) ^[6]	10	10–179	Breast cancer COPD
Xylenes	Aromatic hydrocarbon	0.1 – 2.5 ^[2] 0.3 (median) ^[6]	100	5-59	Asthma Breast cancer Lung cancer Sleep apnoea

^{a)} Values represent real-world target concentrations for sensor applications. While some proof-of-concept devices described in this review have demonstrated detection at these levels under controlled laboratory conditions, they are not representative of the current standard performance across technologies.

including lung cancer patients, smokers, and healthy individuals, but their concentrations can vary between these groups.^[9,10] For example, exhaled formaldehyde concentration is significantly lower in smokers compared with healthy and cancer patients. Conversely, cancer patients have higher concentrations of pentanal, hexanal, and octanal.^[9] Recent research in breath VOC biomarkers is predominantly related to lung cancer (52% of research articles), followed by asthma, diabetes, chronic pulmonary disease, tuberculosis, sleep apnoea, and breast, gastric, prostate, and colorectal cancers.^[11] Y. Saalberg et al. reviewed possible biomarkers for lung cancer, listing 77 of them, among the most promising being 2-butanone and 1-propanol.^[12] However, reliable diagnosis requires detecting multiple VOCs, as individual compounds lack specificity. While breath analysis is a major focus, VOCs are not limited to exhaled air. Costello et al. identified 1840 VOCs in the human body, with 872 specifically found in breath.^[13] VOCs can also be detected in other biological fluids, such as saliva and blood, 44% of them being hydrocarbons.

Table 1 compiles some of the most common VOCs, indicating their typical concentration ranges. Comprehensive air quality monitoring systems and breath analysers often require high sensitivity, sometimes reaching detection limits in a low ppb range. In certain diseases or when following a diet, sub-ppm and higher concentrations may be present in exhaled air, such as increased acetone levels during the ketone diet.^[14] Limits of exposure to VOC in the indoor environment are typically prescribed per 8-hour period^[15] and often refer to total VOC exposure (TVOC),^[16] which would mean cumulative exposure in the few ppm range. In industrial applications, where VOC concentrations can exceed those typically found indoors, a detector should be able to detect hundreds of ppm of the analyte. An example of fields of interest includes explosives markers, chemical and petrochemical industries, manufacturing, food packaging and storage, greenhouse

and farm monitoring.^[17] Notably, highly diverse VOC sources and a plethora of substances in the breath demand not only sensitivity of detection but also high selectivity to precisely determine the composition of air samples, which is often even more challenging to achieve.

1.2. Non-Optical Methods for VOC Detection

One of the most powerful techniques for gas sensing is mass spectrometry (MS), typically coupled with other methods, such as gas chromatography, providing high sensitivity and specificity for identifying thousands of VOCs.^[19,20] Those techniques were listed among the typically used methods for determining the air composition in residences.^[2] Moreover, MS was implemented as a powerful tool for breath analysis.^[20,21] While powerful in laboratory settings, MS is unsuitable for portable VOC sensing due to its bulky and expensive equipment and time-intensive operation, requiring experienced users.^[22] In contrast, commercially available sensors, such as photoionisation detectors, nondispersive infrared sensors, metal-oxide semiconductors, and electrochemical detectors, offer compact, cost-effective solutions.^[23]

VOC sensor research typically falls into three categories: electrical, optical, and gravimetric.^[19] Electrical sensors, relying on resistance or capacitance changes, benefit from simple readouts and integration with electronics. Gravimetric sensors measure mass shifts by detecting changes in resonant frequency when VOCs are adsorbed.^[24] Thus, they are extremely susceptible to other mass-changing factors, especially temperature, pressure, and humidity. Many of the proposed VOC sensors face significant challenges hindering market viability. Key issues include sensitivity to atmospheric factors, baseline drift, slow response times, and high working temperatures. Additionally, these sensors often

require complex signal processing, suffer from large measurement noise, and face difficulties in fabrication, reproducibility, and short lifespans. These drawbacks make it challenging to develop robust, reliable, and low-cost VOC sensors.^[23]

While finding the optimal sensing method is crucial, equally important is selecting the right material to enhance sensitivity and selectivity. A single sensor typically lacks the ability to differentiate between various VOCs. Therefore, sensor arrays are often developed to achieve a more comprehensive analysis. These arrays consist of multiple transducers, each functionalized to respond differently to specific VOCs, allowing for the creation of a unique fingerprint for the gas mixture. One of the commonly implemented materials for VOC sensing is metal oxides, such as ZnO and NiO, often commercialized in chemiresistors.^[19] These materials detect gases through redox reactions with oxygen species on their surfaces, but often require high operating temperatures. Carbon materials, like carbon nanotubes (CNTs) and graphene, offer a high surface area and excellent electrical conductivity, making them effective for VOC detection. Polymers, known for their flexibility and ease of processing, can be chemically modified for enhanced performance, though they tend to have slower response times. A relatively new class of materials, metal-organic frameworks (MOFs), is crystalline and porous with tunable pore sizes, allowing for high selectivity and sensitivity.^[25,26] MOFs have found applications across all types of sensors, though current challenges include stability and efficient integration into existing technologies. Another class of materials for VOC detection is zeolites.^[27,28] They show high stability and tunable pore sizes and can be synthesized in the form of nanoparticles, thus can be suitable for optical detection.

1.3. Optical Approaches in VOC Detection

An ideal sensor must offer high sensitivity with low detection limits and high selectivity while combining several key advantages, such as stability, high signal-to-noise ratio, reliability, low cost, compact size, reproducibility, reusability, fast response time, and ease of operation. Among these features, selectivity remains one of the greatest challenges in sensor development. Optical sensors operate based on the interaction between light and matter, detecting changes in light intensity, wavelength, or polarization to measure various physical properties. Optical sensors for VOC detection normally consist of three main components: a light source, a transducer functionalized with a VOC-sensitive material, and a light detector. Different probe beam modulation techniques can be implemented—intensity modulation, phase modulation, or wavelength modulation, depending on the underlying optical phenomena utilized. For instance, if the transducer is based on a diffraction structure, the presence of the VOC can be detected as a variation of the diffracted light intensity. Interferometers are utilized to detect changes in the interference patterns caused by the presence of VOCs, introducing difference in the phase of the two interfering beams. Polarization-based sensors alter the state of polarization of light as it passes through the VOC-affected transducer, while other optical sensors rely on VOCs affecting evanescent wave propagation, often utilized in optical fibers, or make use of plasmonic structures. Other commonly used types of optical sensors are based on luminescent or

fluorescent materials that change their properties upon interaction with VOCs, and they normally provide a visual, colorimetric response.

In setups involving color changes, visual detection, colorimetric devices, or optical spectrum analysers may be employed to monitor shifts in peak wavelength. Many optical sensors provide fast, real-time measurements, which are crucial for continuous monitoring applications, such as industrial safety and environmental control. Optical sensors, particularly those utilizing photonic structures or fiber optics, also offer significant potential for miniaturisation, making them ideal for portable or wearable devices. In addition, the sensing material in an optical transducer can often be readily customized. For instance, fibers can be coated with different materials; photonic structures can be fabricated from various substances. This flexibility facilitates the development of sensor arrays with individual elements made of different functionalising materials, thus potentially capable of detecting multiple VOCs simultaneously. Moreover, optical methods that use spectroscopic techniques (such as surface-enhanced Raman spectroscopy or non-dispersive infrared sensors) can provide intrinsic selectivity, characteristics of the transduction method.

Since optical methods do not rely on electrical fields or high-power inputs, they are generally safe to use and operate at room temperature. Furthermore, as long as the sensitive material is either resistant to or calibrated for the potentially varying atmospheric conditions, the other components of the sensor are typically unaffected by external factors such as environmental temperature and humidity. Many optical sensors, for example, diffraction and fiber-based sensors, are immune to electromagnetic interference and thus can be implemented in industrial processes associated with strong electromagnetic fields. Moreover, fibers facilitate remote sensing capability, critical in explosive environments where VOCs are often found. A considerable disadvantage of many optical sensors is the relatively costly demodulation, resulting from their slow adaptation in real-time applications.

Commercial optical sensors are predominantly nondispersive infrared (NDIR) sensors.^[23] These sensors operate by detecting the unique absorption of radiation at specific wavelengths by VOCs, which results in a reduction of the radiation reaching the detector.^[29] Unlike spectrometers, NDIR sensors utilise colour filters and may also incorporate a reference gas chamber for improved accuracy. Although other absorption spectroscopy methods exist—some involving complex technologies such as coupling with gas chromatography—since they typically do not involve sensing materials and are already extensively described elsewhere,^[30] they will not be further discussed in this review.

Figure 1 depicts the table of contents of this review, organized by the main optical phenomena involved in the operation of the sensor and subcategories representing different VOC sensing techniques.

2. Sensors Employing Diffraction from Periodic Structures

Diffraction of a light wave is a change in the direction of wave propagation that occurs at the edges of an obstacle or aperture,

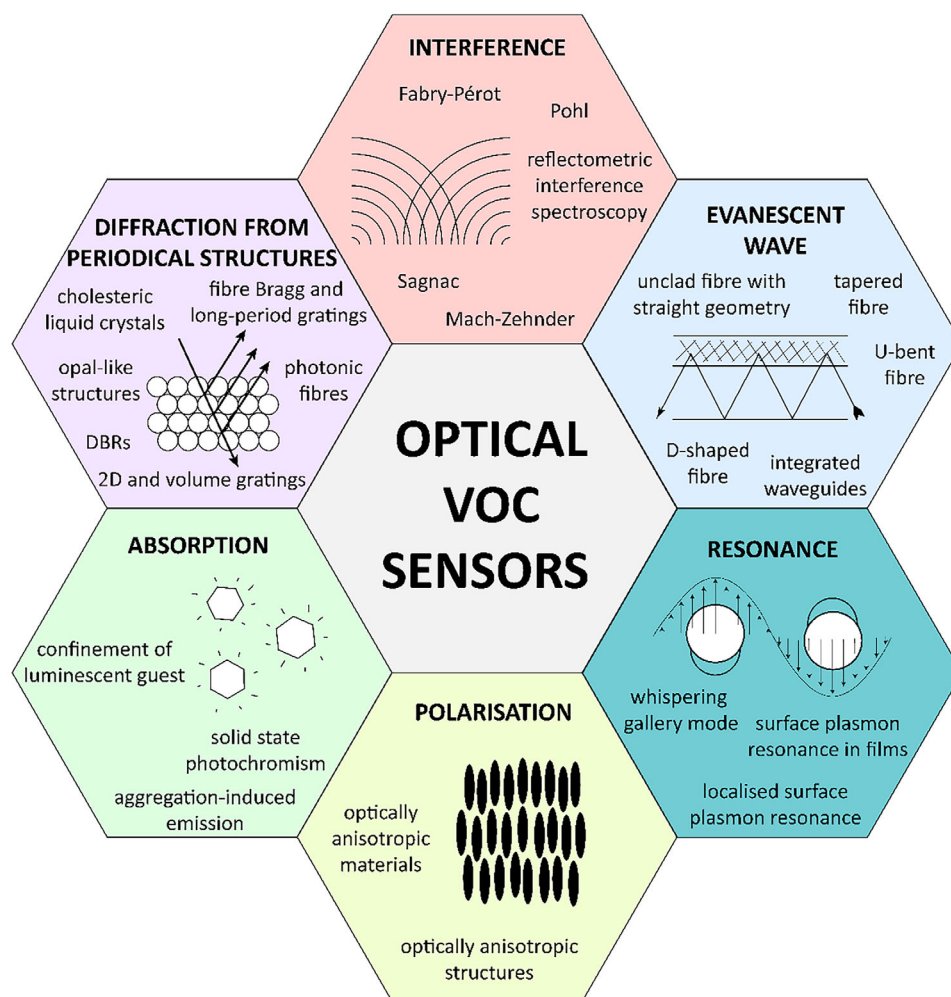


Figure 1. The scope of this review, i.e., the classification of different optical sensors for VOC detection.

or due to changes in the refractive index of the medium in which the wave propagates. Diffraction is readily observed when light is incident on periodic structures, such as diffraction gratings or crystal lattices. The periodicity of the so-called photonic crystal (PhC) results in constructive interference for certain angles or wavelengths (i.e., photonic bandgap) of light scattered by the structure, thus leading to the formation of discrete diffraction orders or Bragg peaks. Periodic structures may play a role as the transducer in various types of sensors, relying on alterations in their diffraction properties. Those alterations are caused by dimensional changes, changes in the average refraction index of the layer, or the refractive index modulation.^[31–33] However, regardless of the optical sensing principle, the amplitude of the change—such as layer swelling or refractive index variation—must be large enough to produce a detectable signal. This highlights the combined role of material responsiveness and detector sensitivity. For example, a detailed analysis of the typical layer thickness changes required to observe a 1% variation of the measured diffraction efficiency of sensors based on surface relief gratings/volume holographic gratings is provided in^[34,35] Diffractive transducers for sensing applications can also be combined

with mechanical parts, i.e., in the form of optical cantilevers and membranes.^[36,37]

This section explores techniques of optical VOCs sensors based on diffractive transducers: opal-like crystals, in-fiber periodic structures, and non-integrated into optical waveguides 1D photonic crystals, surface and volume gratings. A detailed list of materials used in each method, along with achieved sensitivities, is presented in Table S1 (Supporting Information). For further reading, it is worth noting the 2D cavities created by introducing defects into well-organized 2D structures. They may be applied to VOC sensing, benefiting from the ability to confine photons, thus enhancing the interaction between material and optical field, and often combining resonance effects.^[38–40]

2.1. Opal-Like Diffractive Structures

The name opal-like structure refers to a 3D photonic crystal, i.e., microstructured dielectric material appearing in the form of a crystalline colloidal array, characterized by a layered, periodic arrangement of spherical particles. The inverse-opal

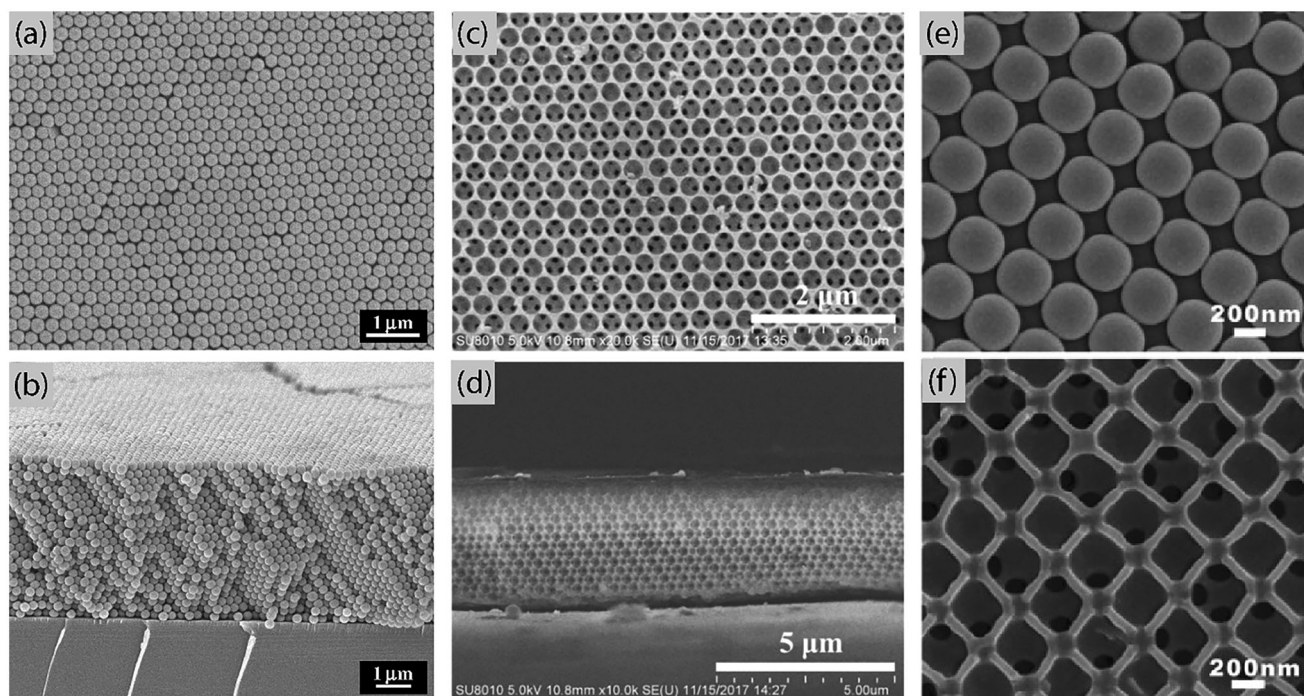


Figure 2. SEM images of exemplary opal-like structures. a) Top and b) side-view of colloidal photonic crystal consisting of 260 nm silica microspheres fabricated by convective self-assembly. Reproduced with permission.^[41] Copyright 2012, Elsevier. Top (c) and (d) cross-sectional view of P(VBC-co-MMA)-SiO₂ inverse-opal photonic crystal. Reproduced with permission.^[42] Copyright 2019, Elsevier. e) The PS colloidal crystal template 100 crystal plane and f) the corresponding inverse-opal HKUST-1 film. Reproduced with permission.^[43] Copyright 2011, Wiley.

structure could be obtained from the colloidal structure placed in a matrix, from which the particles were removed, leaving empty voids in the framework. Examples of such structures representing the two configurations are shown in **Figure 2**. Compared with the opal structure, the inverse configuration is expected to have more accessible diffusion pathways for molecules, which is desirable for sensing applications.

Most VOCs sensors based on opal- and inverse-opal-like structures are perceived as suitable for colourimetric detection because of the transformation of structural colour upon their exposure to the analyte. The detection principle is based on changing the lattice constant of the structure or its refractive index (RI) and, thus, the resultant photonic bandgap. Depending on the specific case, one of the above mechanisms may be dominant, or both mechanisms should be considered. Changes occur due to the interaction of VOCs with PhC, usually through the adsorption of the vapours in the crystal. As a result, the diffraction peak (observed in the reflection or transmission) is shifted, predominantly towards longer wavelengths in the presence of VOCs.

Technology development is based on the fabrication of new materials for PhC structures or the infiltration of the substrate matrix with various types of admixtures. Examples of materials used in opal-like structures for VOCs detection are a composite of polystyrene nanoparticles and polydimethylsiloxane (PDMS),^[44,45] SiO₂,^[42,46–50] often infiltrated with other materials to enhance sensitivity, polystyrene nanospheres,^[51] HKUST-1^[43,52] (type of MOF, **Figure 2f**), and other materials.^[53–56] Cal et al. presented inverse SiO₂ opals doped with gold nanoparticles.^[57] Two peaks in their absorption spectra

were present, each attributed to a different optical mechanism, i.e., Bragg diffraction and localized surface plasmon resonance. Under the influence of changes in RI, both peaks shifted in a different manner, therefore providing more complex sensing information.

Bandgap manipulation via the crystal's lattice constant, refractive index, or introduction of defects increases the versatility of the sensor. The advantage of choosing opal structures is their considerable porosity and relatively large active area, enabling sufficient permeability of volatile compounds inside them. The effect caused by analytes is often visible to the naked eye, such as a colour change, thus providing relatively simple processing information to a non-specialist. Production costs are often low compared to other solid-state sensors, and the structure itself can be readily integrated into lab-on-a-chip devices. However, during observations, attention must also be paid to the probe beam incident angle, as the resultant colour of the structure may strongly depend on it. Opal-like structures are also very susceptible to humidity changes, making them difficult to use as practical devices. Nevertheless, some successful practical applications have been demonstrated, such as the one reported by Yan et al.^[58] The authors reported a flexible silk fibroin/cellulose composite sensor integrated into rubber gloves, with an observable change in colour, indicating VOC presence in the working environment.

H. Zhang et al. demonstrated a sensor array development (**Figure 3**).^[59] A single element was fabricated as an ionic liquid (IL) infiltrated into a multilayer porous silicon chip (Psi). The detection mechanism relied on measuring the diffraction signal in the reflectance spectrum after illumination of the structure with

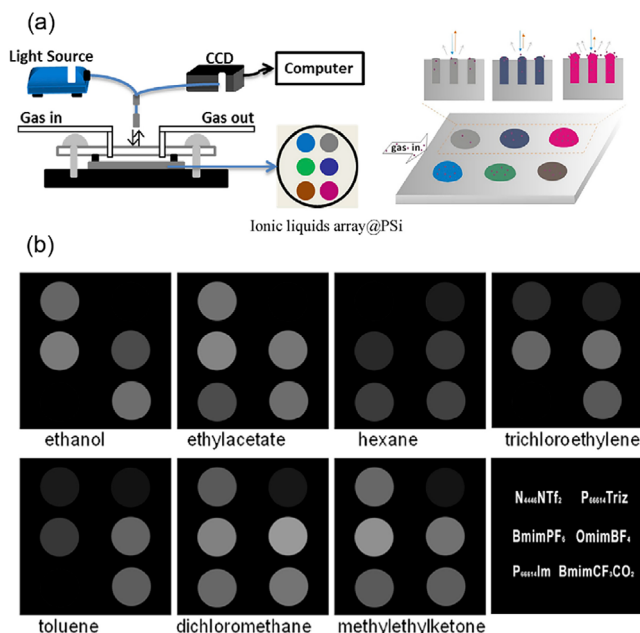


Figure 3. a) Schematic setup of sensing device and sensing array for VOC detection (left). The illustration of the sensing mechanism for the ionic liquids/porous silicon sensor array (right). b) Gray value fingerprint of sensor array upon exposure to VOC. Each sensing spot on the porous silicon sensor array was infiltrated with different ionic liquids. Reproduced with permission.^[59] Copyright 2017, Elsevier.

white light. Liquid density decreased upon exposure to VOC vapor and its condensation. This resulted in liquid swelling and going beyond the silicon channel, forming light-scattering microdroplets on the silicon surface, which significantly decreased the intensity of diffracted light. Complex molecular interactions between VOCs and ionic liquids, causing different sizes and shapes of the microdroplets, enabled the generation of fingerprints unique to different organic compound types.

2.2. Distributed Bragg Reflectors

Distributed Bragg reflectors (DBRs), also called dielectric mirrors or Bragg stacks, are one-dimensional photonic crystals made of two alternating layers of dielectric materials with different refractive indices (Figure 4). Periodic modulation of the RI causes photonic bandgap, i.e., frequency regions for which light propagation is forbidden, observed as peaks in the diffracted spectrum observed in reflection. After intercalation of VOCs into DBR, the position of the reflection band may be affected due to the alteration of RI and, for organic DBRs, structure swelling can also be involved. Inorganic DBR will keep physical dimensions constant because of its rigid structure. Due to wavelength shifts in the visible spectrum, akin to opal-like structures, DBRs can provide colorimetric information. Smaller RI layer contrast will narrow the bandgap, which is beneficial for detecting very small spectral variations, thus lowering the detection limit. On the other hand, a larger bandgap or a higher number of alternating interfaces will increase the intensity of the reflected signal.

Because of its 1D structure, DBRs could often be easily integrated with waveguides, such as optical fibers. They also offer low fabrication costs and the possibility of uncomplicated industrial fabrication. A drawback of DBRs is angular dependence on the diffracted spectrum, thus, a fixed angle of incident light needs to be maintained. Furthermore, those structures could be strongly influenced by the temperature and strain, so either uniform external conditions need to be preserved, or compensational measurements must be added.

Materials most commonly used for DBR fabrication are porous silicon layers^[61–69] and alternate multiple layers of mesoporous silica and titania,^[70–73] although many other materials may be found in the literature.^[74–80] An intriguing idea proposed by Guang et al. was to use butterfly wing scales attached to the end of an optical fiber.^[81] Two peaks in the reflectance were visible, indicating VOC presence by their wavelength and intensity changes. Extensive studies on DBR for VOCs detection have been performed by Lova et al.,^[82–85] often implementing cellulose acetate and the Flory–Huggins interactions theory. The authors found that increasing the structure's thickness negatively influences sensing response time, although increasing the amount of DBR interfaces has the opposite effect and reduces response time even more significantly.^[86]

Various MOFs were also implemented in DBR structures. For example, Hinterholzinger et al. presented Bragg stacks based on microporous ZIF-8 combined with a mesoporous titanium dioxide layer (Figure 4).^[60] The combination of these two materials with different morphologies was aimed at obtaining molecular sieving properties. Gao et al. utilized a similar idea, and inspired by vapour-sensitive beetle scales, fabricated ZIF-8/TiO₂@polydopamine Bragg crystal for visual, colorimetric quantification of benzene,^[87] as depicted in Figure 5. The study also shows Bragg stacks composed of ZIF-67 and ZIF-7 for comparison, and, in another work, authors focused on MIL-101(Cr) alternated with photo-crosslinked polymer, further increasing the limit of benzene series vapours detection.^[88]

Using perfluorinated polymers in the DBR layer enabled selective detection of fluorinated species only, remaining insensitive to polar vapours.^[80] The study presented by Megahd et al. revealed the sensing possibility of polar, non-polar, and fluorinated species, reacting to a wide range of analytes with a chemical fingerprint, thus enhancing selectivity.^[89] However, due to the only partial desorption of analytes, the proposed sensor was limited to single-use.

Aiming at the integration of the transducer to a sensor device, Rea et al. proposed etching of Bragg grating on a planar waveguide made of porous silicon.^[68] Later, Wales et al.^[90] fabricated a structure of eight Bragg gratings, each working as an independent refractometer, recorded in a waveguide to detect hydrocarbon vapors. The four central gratings were coated with polysiloxane and used for sensing, although they achieved a relatively high limit of detection in the range of hundreds of ppm. Two uncovered adjacent gratings served to account for environmental influences, mainly temperature. Two remaining gratings on the edges were not considered in measurements because of the occurrence of spectral fringes due to interference effects.

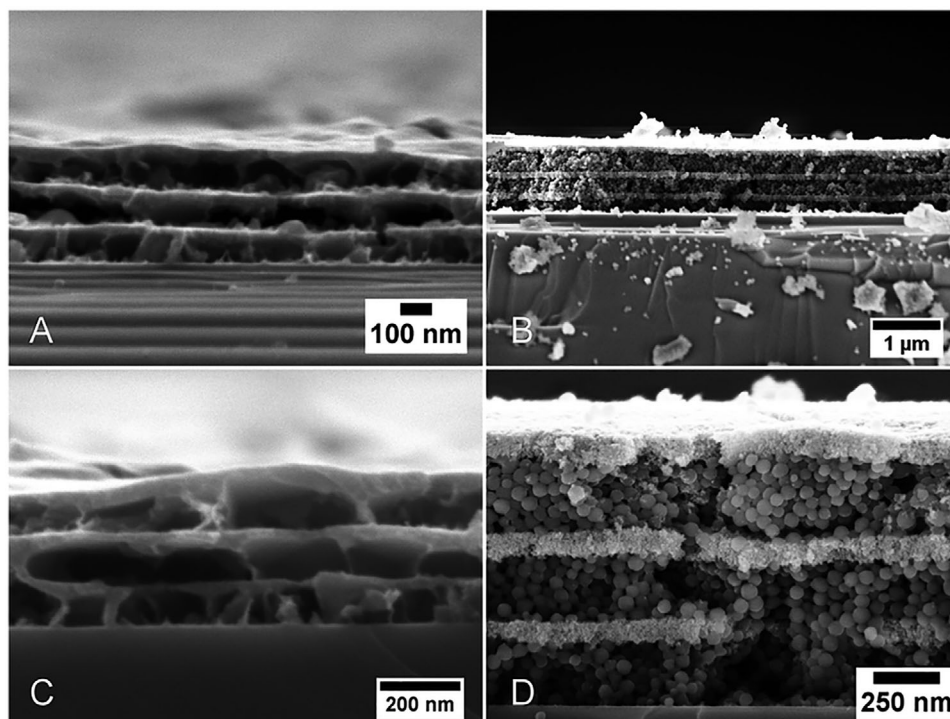


Figure 4. Scanning electron micrographs showing cross-sections of 3-bilayer Bragg stacks with an average film thickness of 50 nm for each titania layer and 70 nm for each ZIF-8 layer for Bragg stack 1 (A and C) as well as for Bragg stack 2 (DMF, B and D) exhibiting a layer thickness of 50 nm for each titania layer and 200 nm for ZIF-8 layers, respectively. Structures are depicted for different magnifications (A:80 000; B:15 000; C:100 000; D:60 000). Reproduced with permission.^[60] Copyright 2012, Royal Society of Chemistry.

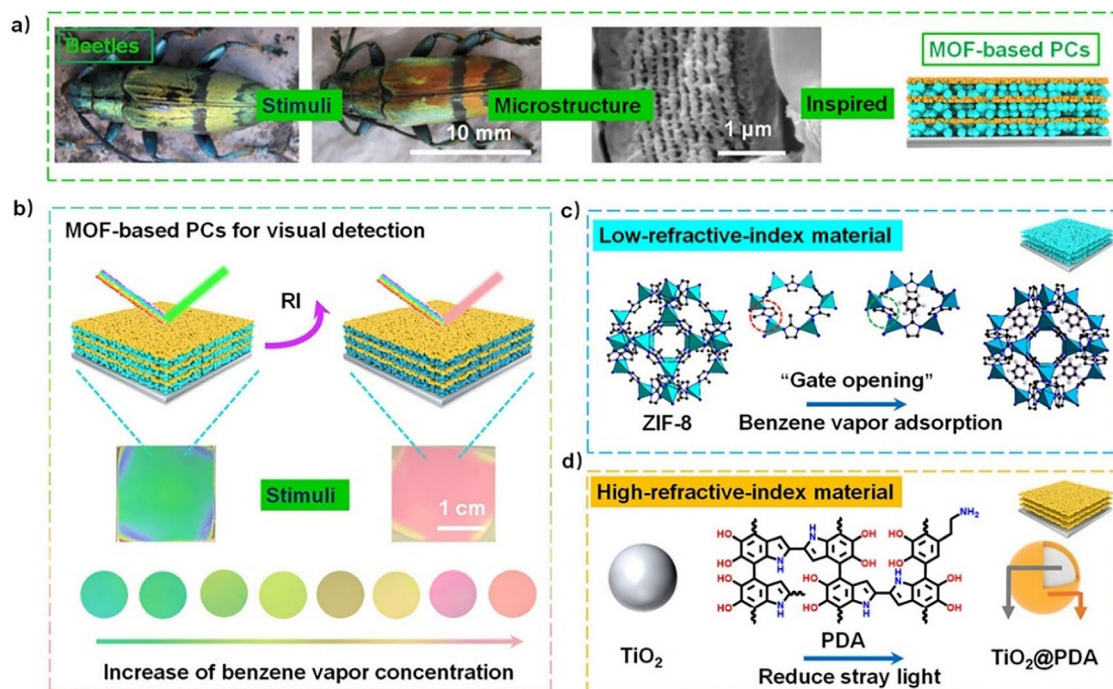


Figure 5. a) Color conversion of *Tmesisternus isabellae* beetle by vapour stimulation and SEM images of its armour microstructure, b) color conversion of MOF-based photonic crystals by benzene vapor stimulation, c) adsorption mechanism of benzene vapour by ZIF-8 with a "gate opening" effect, and d) TiO₂@PDA prepared by TiO₂ and PDA to reduce stray light of MOF-based PCs. Polydopamine was chosen to reduce the stray light and increase colour saturation. Reproduced with permission.^[87] Copyright 2023, American Chemical Society.

2.3. Fiber Bragg Gratings

Fiber Bragg grating (FBG) is inscribed into the core of an optical fiber in the form of periodic refractive index perturbations. To comply with Bragg's conditions, the grating period must be comparable with the operational wavelength of light (a few hundred nanometers). The principle of operation is based on a couple-mode theory, indicating the reflections at the Bragg wavelength and their absence from the transmission spectrum. Bragg wavelength λ_B depends on grating period Λ and effective refractive index n_{eff} of the fiber core, following the equation:

$$\lambda_B = 2\Lambda n_{\text{eff}} \quad (1)$$

Sensing performance is based on the influence of these two parameters through external stimuli or due to evanescent wave interactions. The basic FBG geometry is equidistant areas with alternating values of RI, set perpendicular to the central axis of the fiber. Possible variations of this geometry, influencing the reflection/transmission spectra, are tilted grating, where the plane of the layers is slanted, and chirped grating, with the period varying along the fiber. FBG gratings may be inscribed in single or multi-mode fibers, which determines their operation; they can also be inscribed in photonic fibers. The basic fabrication methods of FBGs utilise UV exposure or femtosecond laser irradiation.

An important advantage of FBGs is their narrow spectral bandwidth, enabling high resolution and lowering the detection limit. As fiber optic sensors, they also feature lightweight, immunity to electromagnetic fields, and could be multiplexed to design sensor arrays. Their sensing response is encoded in wavelength variations; therefore, fluctuations in the power of the source are irrelevant. However, FBGs are highly sensitive to external environmental stimuli such as temperature, strain, and pressure; hence often utilized to detect them. It is evidently a drawback during any other detection, such as gas sensing, and devising a method for compensating for the influence of those factors becomes crucial.

There is a significant number of literature reviews dedicated to optical fiber sensors, covering also FBGs.^[91–96] The first attempts to use FBGs for chemical sensing were based on evanescent wave propagation and considered only measurements of the RI due to environmental RI change.^[97–100] However, while conventional FBGs can detect environmental changes such as temperature, strain, and pressure, they are not sensitive to chemical analytes. They do not typically respond to changes in the surrounding RI as well. Therefore, effective detection required partial cladding removal from the core, usually achieved by fabricating D-shaped fibers^[97] or side-polishing.^[98]

The first selective gas measurements were based on utilising PDMS as a sensing material, allowing the RI changes to be linked to the actual effects of VOCs.^[101,102] Lowder et al. proposed a D-shaped optical fiber with an elliptical core, with the PDMS-covered surface grating etched above the core, to increase the influence of the surrounding medium on the wave propagating in the fiber through the evanescent field.^[101] Park et al. used a bare FBG coated with PDMS, introducing a tensile force during swelling after exposure to a non-polar solvent, hence influencing the Bragg wavelength shift.^[102] Zeolites were also implemented as swelling coating around FBG^[103,104] (Figure 6a–c). Other VOC

sensing-related FBG coatings include polyaniline layers,^[105] ZnO nanolayer (piezotronic mechanism),^[106] and graphene (ammonia gas sensing).^[107,108]

Yan et al. proposed the fabrication of FBG in a microstructured photonic fiber.^[110] The grating was written in a core surrounded by 209 microholes acting as gas cells for acetylene molecules. The analyte interacted with light propagating in the fiber through evanescent waves of guided modes. Another possibility to contrive a Bragg fiber is to surround the fiber core (hollow or solid) with a distributed Bragg reflector, as presented by Malekovic et al.^[109] (Figure 6d). The dielectric mirror around the core responds by swelling after analyte absorption, resulting from physicochemical interaction with the material. Thereby, a change in refractive index influences the interference of light in the DBR fiber cladding and reversibly changes the structural colour of the device.

Adamu et al. proposed an optical nose to measure different VOCs simultaneously.^[111] The constructed device comprised six hollow-core Bragg fibers, each of 1–3 μm bandwidth and operating for a different spectral position in a 2–14 μm region. VOCs diffused into the hollow core, causing changes in infrared absorption. The threshold value of intensity changes was determined for each fiber, thus creating a binary code and assigning a digit sequence to each analyte based on the changes caused in individual spectral bands. VOCs with akin molecular structures were characterized by similar codes.

2.4. Long-Period Gratings

Long-period fiber grating (LPFG), like fiber Bragg grating, is characterized by a periodic modulation of the refractive index along the fiber core. The main difference between LPFG and FBG is the much longer period Λ , of a few hundred micrometers and more (see Figure 6a). Guided core mode is coupled with the cladding modes at specified resonance wavelengths λ_i under the phase-matching condition:

$$\lambda_i = (n_{\text{core}} - n_{\text{clad}, i}) \Lambda \quad (2)$$

where n_{core} is the effective refractive index of the core and $n_{\text{clad}, i}$ is the effective refractive index of i th cladding mode. Therefore, a series of attenuation bands, centred at discrete resonance wavelengths λ_i , will occur in the transmission spectrum of the fiber. External factors, such as perturbations in the refractive index of the surrounding medium, could influence cladding modes' refractive indices, hence shifting the resonance wavelengths. For that reason, LPFGs are generally more often used for chemical sensing applications^[91,112] than conventional FBGs, which are insensitive to changes in the refractive index of the surrounding medium unless they are adapted by partial cladding removal. LPFGs are commonly coated with materials able to adsorb the target analytes, thus changing the effective refractive index of the environment surrounding the fiber (Figure 7a). Detection can be performed by measuring the entire attenuation spectrum or by focusing on optical loss recording at a fixed wavelength.

The advantages of LPFGs are similar to those for fiber Bragg gratings and refer to their in-fiber structure, i.e., electromagnetic field immunity, portability, multiplexing possibilities, and

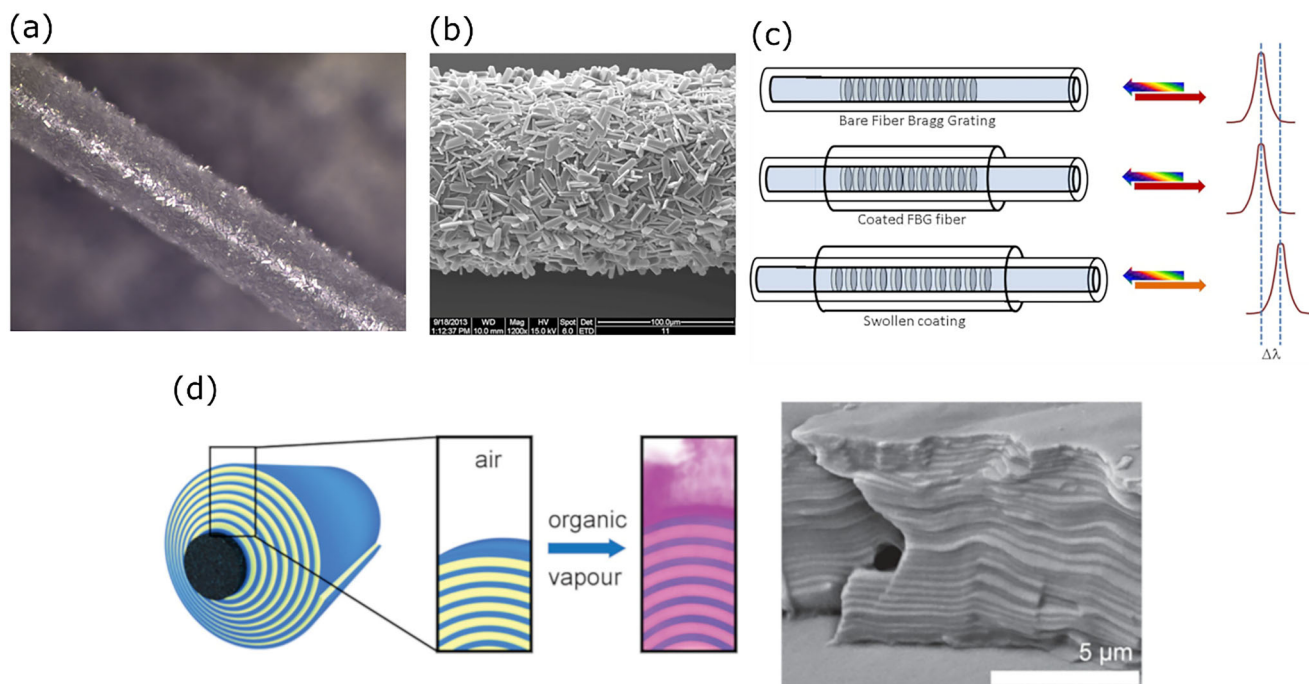


Figure 6. Examples of Bragg gratings implementations with the fiber. a) Optical microscopy image (magnification 20X) of a silicalite-coated fiber. b) SEM image of a fiber coated with 7 μm silicalite coating. c) Working principle of a fiber Bragg grating-based chemical sensor. Swelling of the coating results in a shift in the optical spectrum. Reproduced with permission.^[103] Copyright 2016, Elsevier. d) Schematic cross-section and SEM image of photonic fiber coated with distributed Bragg reflector (rolled PDMS/PSPI multilayered cladding) and color change sensing mechanism based on the adsorption of an organic solvent into polymer material, followed by swelling. Reproduced with permission.^[109] Copyright 2020, Wiley-VCH GmbH.

wavelength-encoded sensing. Since LPFG fabrication does not require as high precision as in the FBG case, there is a larger number of suitable fabrication methods, including UV exposure, ion implantation, CO₂ or femtosecond laser irradiation, and more.^[93] The use of different fiber coatings allows for the fabrication of a device for a specific type of analyte and control over the sensitivity. Critical challenges related to LPFG include broad transmission resonance and multiple resonance peaks, which limit the detection accuracy. Moreover, the relatively long length of the device, where the grating itself is typically about 3 cm long, can impede applications that require high spatial resolution.

LPFGs create an especially broad field for implementing different coating layers. Some of the investigated materials are: SnO₂ (surface energy changes),^[116] syndiotactic polystyrene (selective molecular penetration),^[117] silk fibroin,^[118] PDMS (with fiber-loop ring-down spectroscopy technique),^[119,120] graphene and vapochromic dye,^[121] calixarene (analyte molecules temporarily trapped inside the cavity and the voids),^[122] SiO₂ nanoparticles with infused calixarene,^[123] polyelectrolyte^[124] and porphyrin^[125] (ammonia and other amine compounds sensing), zeolites (observed faster response for smaller molecules),^[113,126] and MOFs, mostly ZIF-8.^[127–130] Wang et al. deposited ZIF-8 on the surface of an LPFG and examined how the number of deposition cycles influences the device's sensitivity to acetone vapour, finding that a thicker coating will contribute to higher sensitivity.^[130] While authors set the acetone detection limit at 1.4 ppm (sensitivity of 21.9 nm ppm⁻¹), an even higher sensitivity (26.1 pm ppm⁻¹) was recently demonstrated by Huan et al. with the use of a ZIF-90/graphene oxide-coated LPFG sensor (Figure 7c–e).

Hromadka et al. fabricated an array of three LPGs in a single fiber (Figure 7b).^[114] Each grating was intended to measure different stimuli: temperature-sensitive LPG had no coating, LPG for humidity sensing was covered with SiO₂ nanoparticles, and the VOC-sensitive LPG was coated with silica and infused with a calixarene. A change in central wavelength due to VOCs presence was present only in the resonance band of the third grating, thus not influencing simultaneous temperature and humidity measurement.

2.5. Photonic Fibers

Photonic crystal fibers (PCFs) have been extensively investigated for applications in VOC sensing. The hollow parts in the PCF structure enable enhanced interaction between the gas sample and the evanescent field, making it suitable for high-sensitivity VOC detection.

PCF is a specialized optical fiber with a periodic lattice structure enclosed by a holey glass cladding. Since the first fabrication of PCFs using fused silica,^[131] various fiber designs with different geometries and dimensions have been developed. Some examples of PCF structures are shown in Figure 8a,b. Compared to standard optical fibers, the lattice structure of PCFs can be flexibly designed and tailored by controlling the air holes' distribution and dimensions, enabling selective propagation modes and variations in the PCF's effective refractive index (RI). Light propagation in a PCF is along the fiber length with a total internal reflection between the air holes, confining the evanescent field into

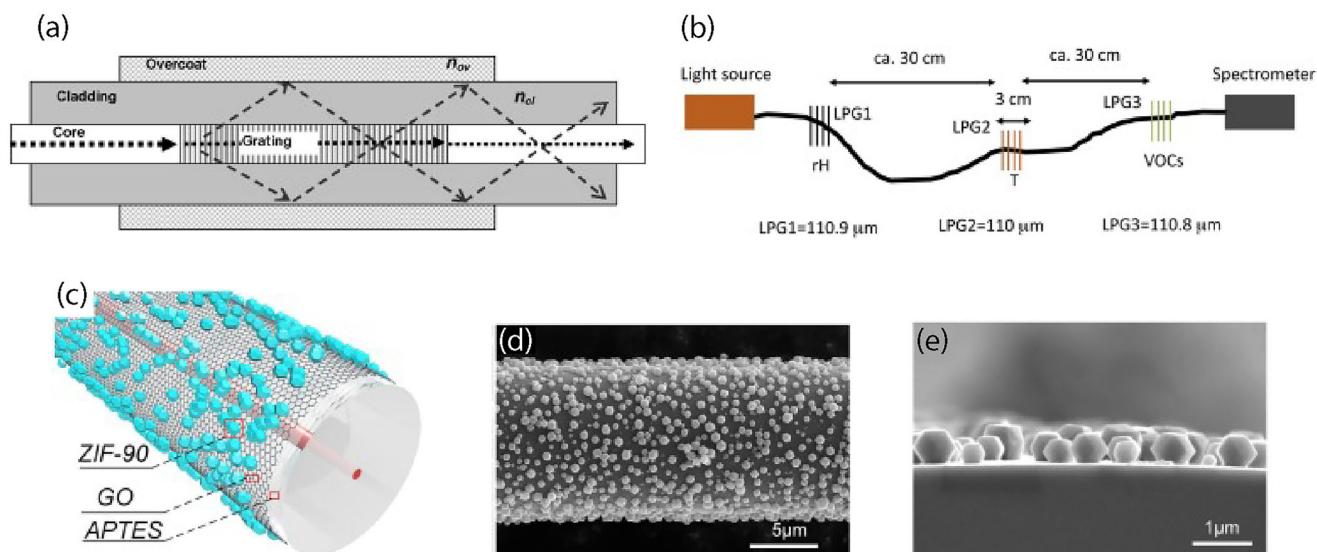


Figure 7. a) Schematic showing a cladding mode of LPFG with a film overcoat. Reproduced with permission.^[113] Copyright 2009, Elsevier. b) Schematic illustration of the LPG sensor array; the individual LPGs were used to measure: LPG1 relative humidity; LPG2 temperature; and LPG3 volatile organic compounds. Reproduced with permission.^[114] Copyright 2017, Elsevier. c) Schematic diagram of the ZIF-90/graphene oxide-coated microfiber LPG sensor. SEM images of the d) fiber and e) the coating film thickness. Reproduced with permission.^[115] Copyright 2023, Elsevier.

the specific parts depending on the distribution of the air holes, as shown in Figure 8c. The air hole structures in PCF provide an increased interaction area, leading to more efficient changes in the effective RI when VOC molecules penetrate the air holes. The sensing mechanism of the PCF-based VOC sensors is thus based on detecting the effective RI change inside the PCF caused by VOC infiltration, which can be sensed by measuring intensity or wavelength shift in the transmission spectrum of the fiber.

PCF-based VOC sensors involve developing unique structural designs to improve sensitivity. The typical implementation involves the construction of fiber-interferometric structures (e.g., a Fabry–Pérot (FP) interferometer, or a Mach–

Zehnder interferometer (MZI)) by coupling PCF sections with single-mode fibers (SMFs), which has been successfully demonstrated for the detection of the acetonitrile (CH_3CN), tetrahydrofuran (THF),^[135] acetone,^[136] ammonia,^[137,138] acetylene,^[110] ethanol,^[139] and methanol.^[140] A PCF-based VOC sensor was first proposed by J. Villatoro et al. in 2009 based on an FP interferometer, where a spliced SMF-PCF structure exhibited high sensitivity to acetonitrile (CH_3CN) and tetrahydrofuran (THF).^[135] The collapse area between the SMF and PCF leads to a modes mismatch and excites two core modes, while one open end of the PCF serves as a channel for VOC infiltration. The concentration of VOCs was linked to a wavelength shift in the

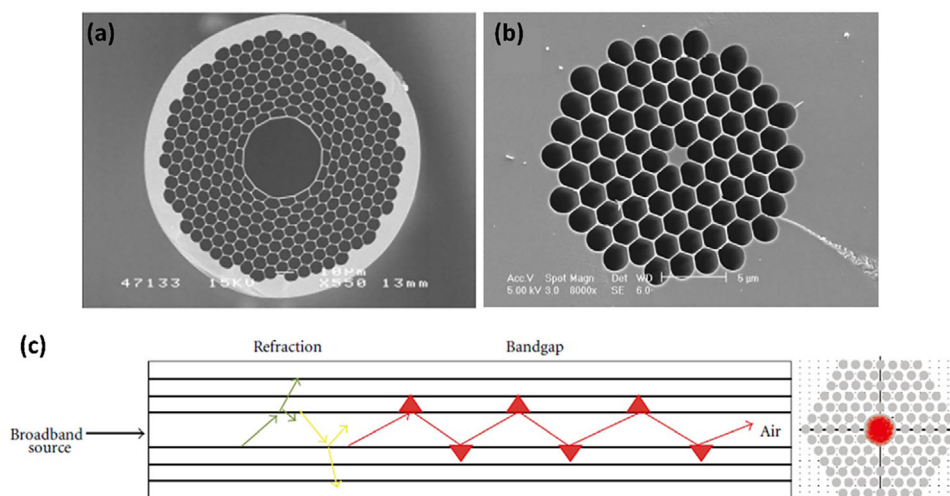


Figure 8. Scanning electron microscope images of the cross-sections of two PCF types: a) a hollow-core and b) a solid-core. a) Reproduced with permission.^[132] Copyright 2005, OPTICA Publishing Group. b) Reproduced with permission.^[133] Copyright 2005, OPTICA Publishing Group. c) Schematic diagram illustrating light propagation in a PCF. Reproduced with permission.^[134] Copyright 2012, Wiley.

reflection spectrum. Kim et al. proposed a similar configuration using a cascaded FP cavity fiber structure (SMF-capillary tube-PCF) for detecting acetone.^[136] A multiplexed sensing platform with six spliced SMF-PCF FP sensor heads was proposed by A. Lopez-Aldaba et al., where the system could simultaneously detect methanol, humidity, and temperature, enhancing detection efficiency.^[140] A fast Fourier transform was applied to isolate the sensors' interference and analyse the data. An alternative approach utilized an FP interferometer consisting of a PCF-fiber Bragg grating (FBG) absorption sensor for acetylene detection, proposed by G. Yan et al.^[110] In this configuration, the FBG with a specific periodicity operated in reflection mode, allowing the intensity to correlate with acetylene concentration. In contrast to reflection-based detection, a transmission-based MZI sensor using high-birefringent PCF as the gas cell was proposed for the detection of ethanol by Niu et al.^[139] The SMF was directly coupled to the high-birefringent PCF with a gap to simplify fabrication and enhance VOC infiltration. The same research group explored ethanol detection in PCFs based on the Rayleigh scattering effect using the same structure.^[141] It should be noted that, like other fiber optic sensors, PCF-based VOC sensors often struggle to achieve high selectivity to specific VOCs in the presence of multiple gases. Several researchers have attempted to address this limitation by applying functionalized coatings to the PCF surface for a more selective VOC detection. For instance, Shrivastav et al. reported an MZI sensor comprising an SMF-PCF-SMF configuration coated with a polyaniline@SnO₂ nanocomposite,^[137] which demonstrates selective detection of ammonia with fast response and recovery times.

In addition to the PCF-based VOC interferometric sensors discussed above, Zheng et al. proposed a PCF-based spectroscopic absorption sensing platform incorporating long-period gratings and a tunable erbium-doped fiber ring laser.^[138] Their design utilized a tunable erbium-doped fiber ring structure as a laser platform, where the infiltration of VOCs influenced the laser intensity. Integrating a tunable filter in such a structure allows for precise laser wavelength tuning, enabling the detection of multiple VOC gases with absorption features in the C-band (1530–1565 nm).

2.6. Cholesteric Liquid Crystals

Cholesteric liquid crystal (CLC),^[142,143] also known as chiral nematic liquid crystal, has molecules ordered in layers with the director axis gradually twisted along the crystal's length (Figure 9). A periodical rotation of the helical pattern, with a complete 360° period called pitch p , is maintained throughout the structure. When the pitch matches the wavelength of light, a Bragg reflection of the circularly polarized light with the peak at $\lambda_0 = n \cdot p$ occurs, where n is the average refractive index of the structure. Usually, the presence of gas molecules diffusing into CLC will cause swelling of the structure, thus elongating the pitch and resulting in a red shift in the spectrum of reflected light (Figure 9d). However, the direction of the shift (red or blue) upon exposure to VOCs can be controlled by the proper choice of liquid crystal and its chiral dopant contents.^[144]

Research on liquid crystals for the detection of gases was initiated in 1965 by Ferguson et al.,^[147,148] and has been further de-

veloped since Dickert et al. in the 1990s demonstrated detectors distinguishing between alcohols and hydrocarbons^[149] and sensing of aromatic and halogenated hydrocarbons with a limit of detection in the ppm range.^[150] Frequent materials chosen for CLC films are cholesteryl chloride, cholesteryl nonanoate, cholesteryl oleyl carbonate, and their mixtures. Many CLC preparation and functionalisation approaches have been proposed, including immobilisation in a polymer film^[151] and cholesterol-based receptor embedding.^[145] Likewise, E7, i.e., a commercially available nematic mixture of several cyanobiphenyls, is often used as an LC to which a chiral dopant is added, creating CLC.^[152,153] E7 doped with TADDOLs, previously reported for oxygen and carbon dioxide sensing,^[154] was implemented for acetone vapour detection.^[155] In this approach, instead of CLC swelling, measurement was conducted due to the chemical reaction between the dopant and the analyte. Other possible dopants include Fe₃O₄ magnetite nanoparticles (carbon monoxide detection),^[156–158] or cholesteric cellulose nanocrystals film doped with copper salt (ammonia).^[159] A promising approach to colourimetric detection is the fabrication of hydrogen-bonded CLC^[160,161] and hydrogen-bridged CLC polymer networks.^[162,163]

Too high concentration of the analyte in the structure can lead to a molecular reorientation of CLC, which becomes disordered and loses its optical properties, i.e., turns entirely transparent. To overcome this, CLC mixing with carbon nanotube was proposed.^[164] Furthermore, thermal stability is crucial for liquid crystals because the mesophase occurs only in a certain temperature range. Below this range, the structure becomes solid, while above, an isotropic phase occurs, where the molecules lose their alignment. In the mesophase, moreover, as the temperature increases, the pitch size increases, which could provide an incorrect sensing response. Thus, gas absorption saturation and temperature dependence strongly limit the sensing range of CLC-based devices. Another significant drawback could be the fluid state of CLC, remaining impractical in many environmental applications. Therefore, many attempts to stabilize CLC, predominantly polymerization techniques, were successfully implemented.

Since polymer-stabilized CLCs remain fluid, limiting performance in an unstable environment, efforts to prepare solid-state CLC without losing its unique properties resulted in an acetone-sensitive soft solid polymer CLC, possessing good mechanical stability and sensing reversibility.^[165] Instead of CLC polymerisation,^[146] a negative strain parallel to the helical axis may also be introduced,^[152] potentially increasing the sensitivity, or CLC may be used to template the polymerization of particles with the ability of colour change through Bragg reflections.^[153] Moreover, embedding CLC in a polymer matrix may enhance the selectivity of the transducer.^[166]

The direction of the photonic bandgap shift could be unstable, alternating from red to blue for increased gas concentration, which may be challenging for sensing applications. According to the spring theory, a larger CLC pitch and lower polymer concentration will increase the device's sensitivity. Li et al. reduced the thickness of the CLC film, achieving a pre-compression effect, mimicking the spring. Thus, the prepared CLC swelled more significantly after exposure to VOC than without pre-compression.^[167]

Sutarlie et al. developed a colourimetric array to measure responses upon the presence of around twelve different chemical

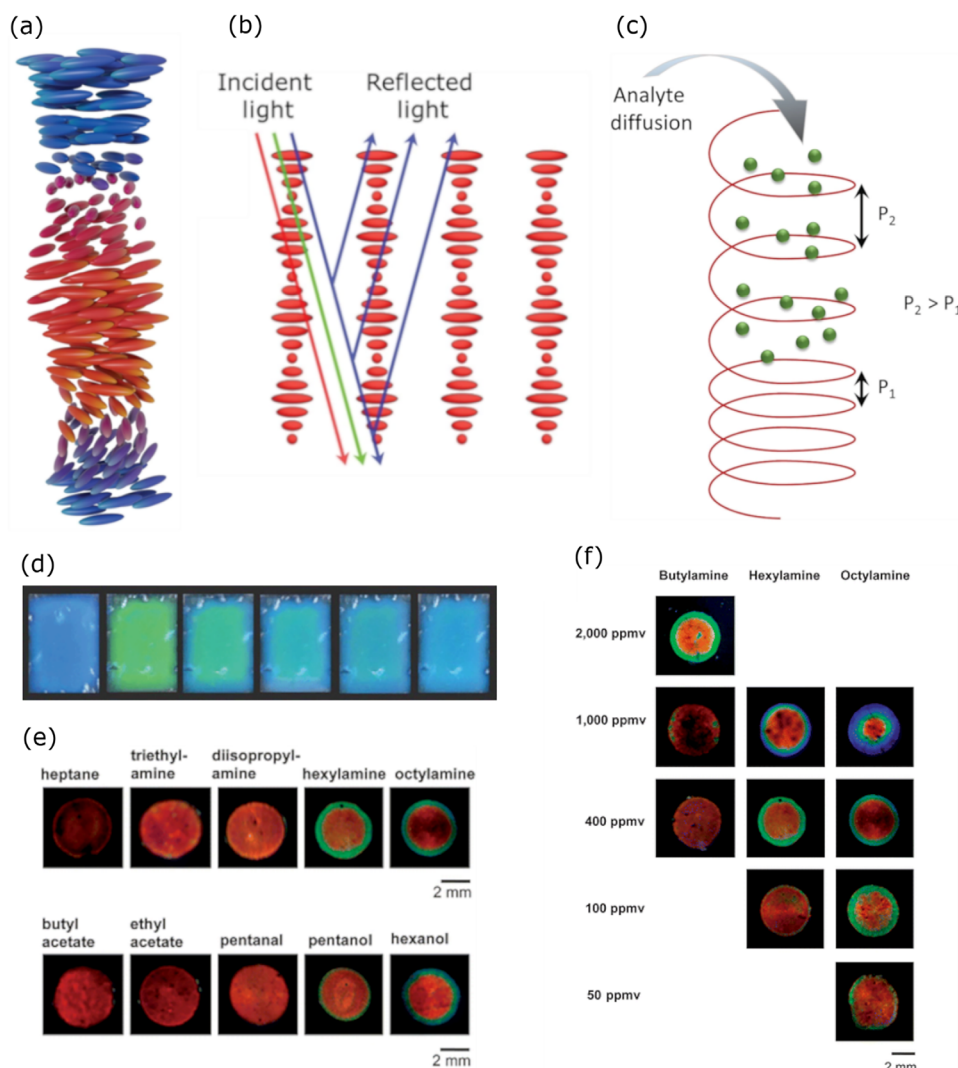


Figure 9. Schematic representation of cholesteric liquid crystal and its behaviour after VOC absorption. a) Rod-shaped molecules in the nematic phase, twisted along the crystal's length. b) Selective reflection of incident light. c) Structure swelling upon interaction with gas molecules, leading to an alteration in the magnitude of the chiral pitch. Reproduced with permission.^[143] Copyright 2020, Wiley. d) Color change of liquid crystal film (1st: initial colouration without analyte, 2nd: exposure to gaseous 1-butylamine, 3rd–6th: gradual recovery after removal of 1-butylamine). Reproduced with permission.^[145] Copyright 2006, Royal Society of Chemistry. e) Response of polymer-stabilized CLC to a variety of organic vapours at 400 ppmv. f) Comparison of the colorimetric responses of polymer-stabilized CLC at 25 °C for butylamine, hexylamine, and octylamine. Reproduced with permission.^[146] Copyright 2010, Royal Society of Chemistry.

vapours (Figure 9e).^[146] Choosing an array format was aimed at avoiding a limited temperature range for measurement, with each component having a different polymer composition. Notably, at room temperature, structures were transparent, and colour changes occurred upon exposure to amine vapours, enabling placing them on goggles or windows.

Tang et al. proposed a side-polished fiber coated with CLC (Figure 10a).^[168] VOC penetration elongated the CLC pitch, decreasing the coating film's refractive index and leading to a blue shift in the fiber resonance spectrum. Yang et al. coated only the ends of the two coupled multimode fibers with CLC, with different pitch lengths each, enabling measurement of the shift in the reflectance spectrum.^[169] Using the duality of the sensor, mea-

surements of concentrations of acetone and ethanol in their mixture were performed.

Optical fibers can enable convenient temperature compensation during VOCs detection utilizing diffraction from CLC. In ref. [171], two types of CLC were used—one, introduced inside the hollow-core fiber, to measure temperature, and second, covered on the fiber's end—to detect VOCs. Zhou et al. show a similar approach, but three CLCs were coated on the tops of two coupled fibers (Figure 10b–d).^[170] On top of the first fiber were coated two misaligned CLCs responsible for acetone and tetrahydrofuran mixture sensing, as they offer different sensitivities to both analytes. The second fiber was used for temperature measurement and compensation.

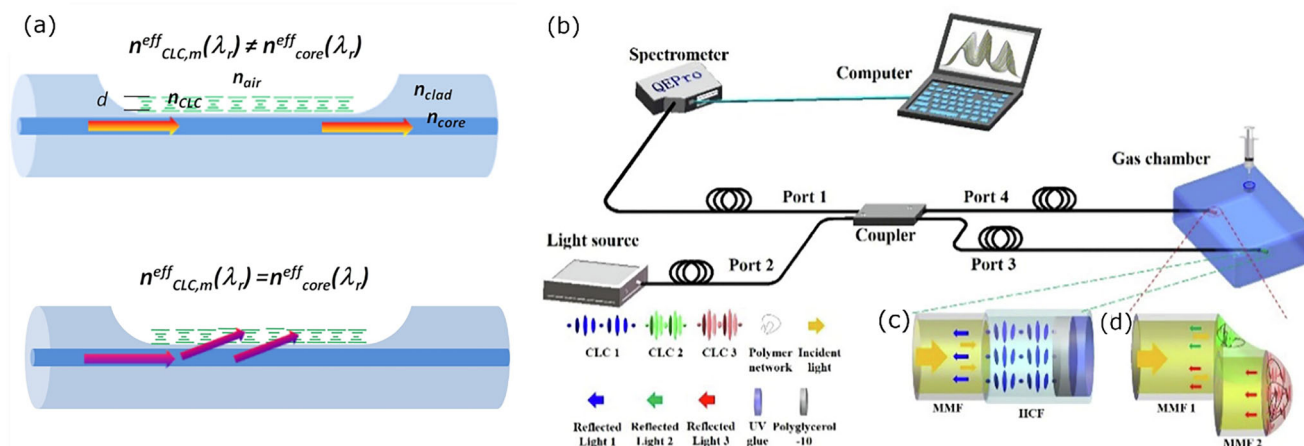


Figure 10. Cholesteric liquid crystals implementation into optical fibers. a) Schematic illustration of the change in the CLC pitch and light coupling between the CLC film and the single photonic fiber. Reproduced with permission.^[168] Copyright 2018, Elsevier. b) Schematic of temperature-compensated optical fiber VOC gas sensing device. c) Schematic diagram of the temperature probe and d) VOC gas probe. Reproduced with permission.^[170] Copyright 2022, Elsevier.

Extensive studies on CLCs were performed by Shibaev et al., including gas sensing applications.^[160,172–176] A broad reflection band was demonstrated for nematic and CLC droplets (Figure 11).^[172,173] Analysis of the distribution intensity across the droplet's fingerprint structure was performed upon exposure to ethanol. Moreover, an attempt to develop an olfaction system for the selective detection of VOCs was presented using six droplets of CLC compositions with variable viscosity.^[174] For low gas concentrations, colour fluctuations and modulation in light reflection were observed, while for high analyte concentrations detec-

tion was based on isotropisation transitions and colour changes of the whole droplet. The authors also presented a sensor array of eight droplets.^[176] In another study, a detection approach utilising atomic force microscopy was demonstrated.^[175] It was found that after exposition to toluene vapour, a glass-forming CLC with molecular structure undergoes significant reconstruction (valleys in the structure were transformed into hills).

A particular type of CLC is a blue phase (BP), appearing between the chiral nematic and isotropic phase, in a very narrow temperature range of circa 1 K (Figure 12). Molecules of BP

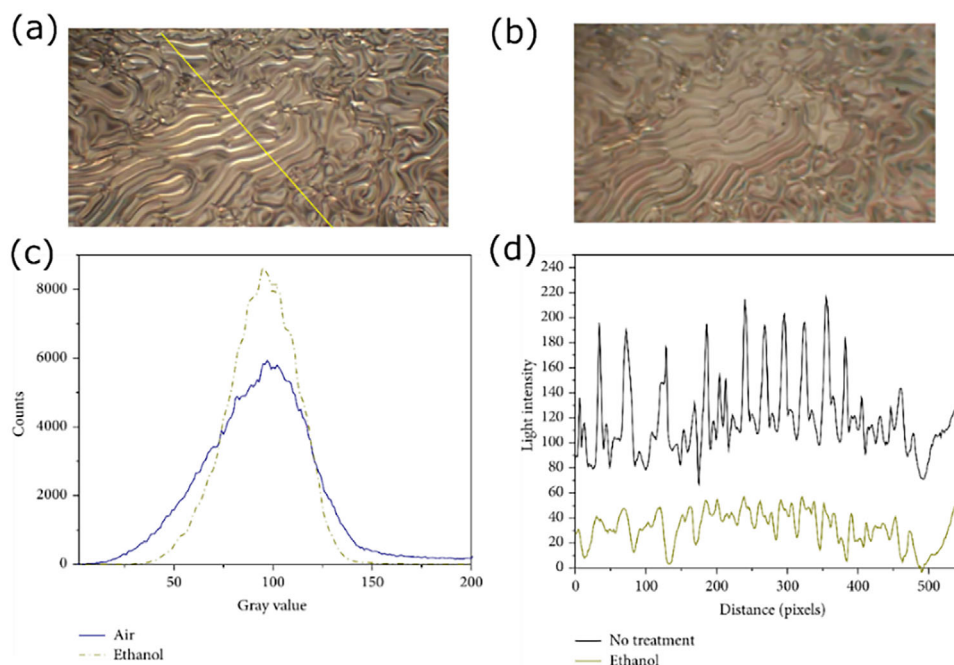


Figure 11. The visual appearance of fingerprint structure before (a) and after (b) the injection of ethanol. c) Brightness analysis of the whole chiral fingerprint structure. d) Analysis of light intensity across the fingerprint structure before. Reproduced with permission.^[172] Copyright 2015, Hindawi Publishing Corporation.

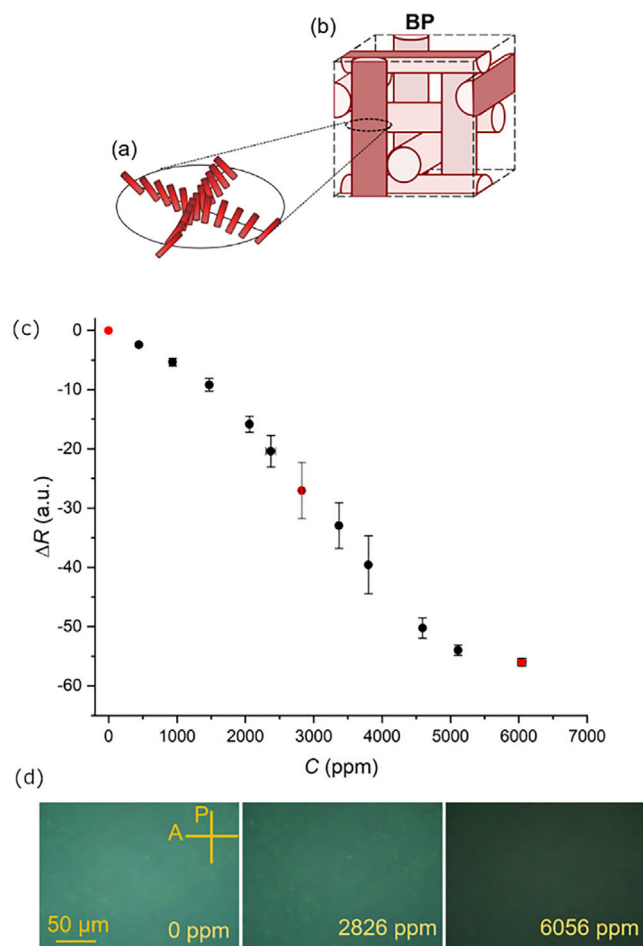


Figure 12. Schematic illustration of the blue phase (BP) structure, showing a) director alignment in the cross section of a double-twist cylinder and b) its configuration. c) Differential optical response (ΔR) of polymer-stabilized BP films as a function of toluene vapour concentration (C) at 25 °C and atmospheric pressure. d) Optical reflection micrographs (crossed polars) of polymer-stabilized BP films at $C = 0$, 2826, and 6056 ppm of toluene vapour (red points in (c)). Reproduced with permission.^[178] Copyright 2020, ACS.

exhibit 3D periodicity, thus forming double-twisted cylinders. The first studies to utilise BPs for gas sensing were proposed by Pantoja et al.^[177] The authors observed phase transitions in BPs after exposure to toluene vapour. For enhanced thermal and mechanical properties during toluene detection, Yang et al. implemented polymer-stabilized BPs.^[178]

2.7. 2D Gratings and Volume Holograms

A diffractive 2D grating is a type of periodic surface relief. The most fundamental and commonly used geometries of such structures are grids (Figure 13a,b,e), i.e., matrices of cuboids, and lines (Figure 13d,f,g) in the form of equidistant grooves. Gratings fabrication methods include various lithographic techniques, namely electron beam lithography, soft lithography, ablation, holography and more.^[179–181] A key advantage from the application point of view is the ability to use once fabricated grat-

ing as a reusable stamp and create unlimited duplicates with the same geometry.

After illuminating with light, 2D gratings produce a characteristic diffraction pattern in the form of discrete bright regions. Upon exposure to the interacting analyte, the structure will undergo swelling, and changes in refractive index may occur. Instead, or together with observing the wavelength shift of incident light, one can measure changes in diffraction efficiency, e.g., a ratio of light deflected in a chosen diffraction order to total incident light.

Early attempts to use 2D diffraction structures to measure diffraction efficiency changes when exposed to gaseous analytes utilized titanium dioxide,^[184] microporous supramolecular coordination compounds,^[185] charge-transfer salts,^[186] and polymer structures.^[187] All of the above were fabricated with PDMS stamps forming a lattice of squares $\approx 5 \mu\text{m}$ wide and not exceeding 200 nm depth. Chen et al., implementing lithography and oxygen plasma treatment, constructed tethered PPMA in the form of cuboidal pillars on a silicon wafer and studied its behaviour under exposure to VOCs with different polarities (Figure 13a–c).^[182] For instance, 200 nm height pillars exposed to toluene swelled reversibly to 313 nm and up to 648 nm upon exposure to chloroform, but with a recovery time of 12 h.

The first implementation of MOF into 2D photonic crystals for VOC sensing was demonstrated by Dalstein et al.^[183] To find a technique enabling structure fabrication below the micrometre range and to overcome issues with light scattering, the authors implemented soft nanoimprinting lithography and utilized crystals with sizes below 50 nm. In both examined scenarios—homogeneous ZIF-8 structures and ZIF-8 deposited on TiO_2 —a PDMS stamp, made through electron beam lithography, was used, as depicted in Figure 13d–j.

Recent studies implement two-photon polymerisation to create periodic photonic structures with good reversibility and the ability to detect vapours.^[188,189] This technique involves the use of a focused femtosecond laser to induce localized polymerization within a photoresist via simultaneous absorption of two photons, typically in the near-infrared range. Because two-photon absorption only occurs at the focal point where photon density is highest, it enables submicron 3D structuring with high spatial resolution and minimal damage to surrounding material. In the work presented by Delaney et al. $2.75 \times 2.75 \mu\text{m}$ grid components, spaced $0.5 \mu\text{m}$ apart, had up to $3.5 \mu\text{m}$ in height.^[188] A matrix with five columns, each comprised of gratings with different heights, enabled redshift observations in the transmission spectrum, more visible for the higher polarity of the analyte. Further implementing two-photon polymerisation, Qian et al. proposed fabricating periodic concentric spiral arrays (Figure 14), working as a transmission phase grating.^[189] One of the reasons for contriving this concept was to increase the polymer-to-void ratio, which was expected to increase colour intensity.

2.7.1. Surface Gratings Fabricated by Holographic Lithography

Holography is a distinctive method of diffraction gratings fabrication, where photosensitive material is illuminated with coherent light from two or more interfering beams.^[190] Reacting on periodic bright and dark regions or a periodically changing

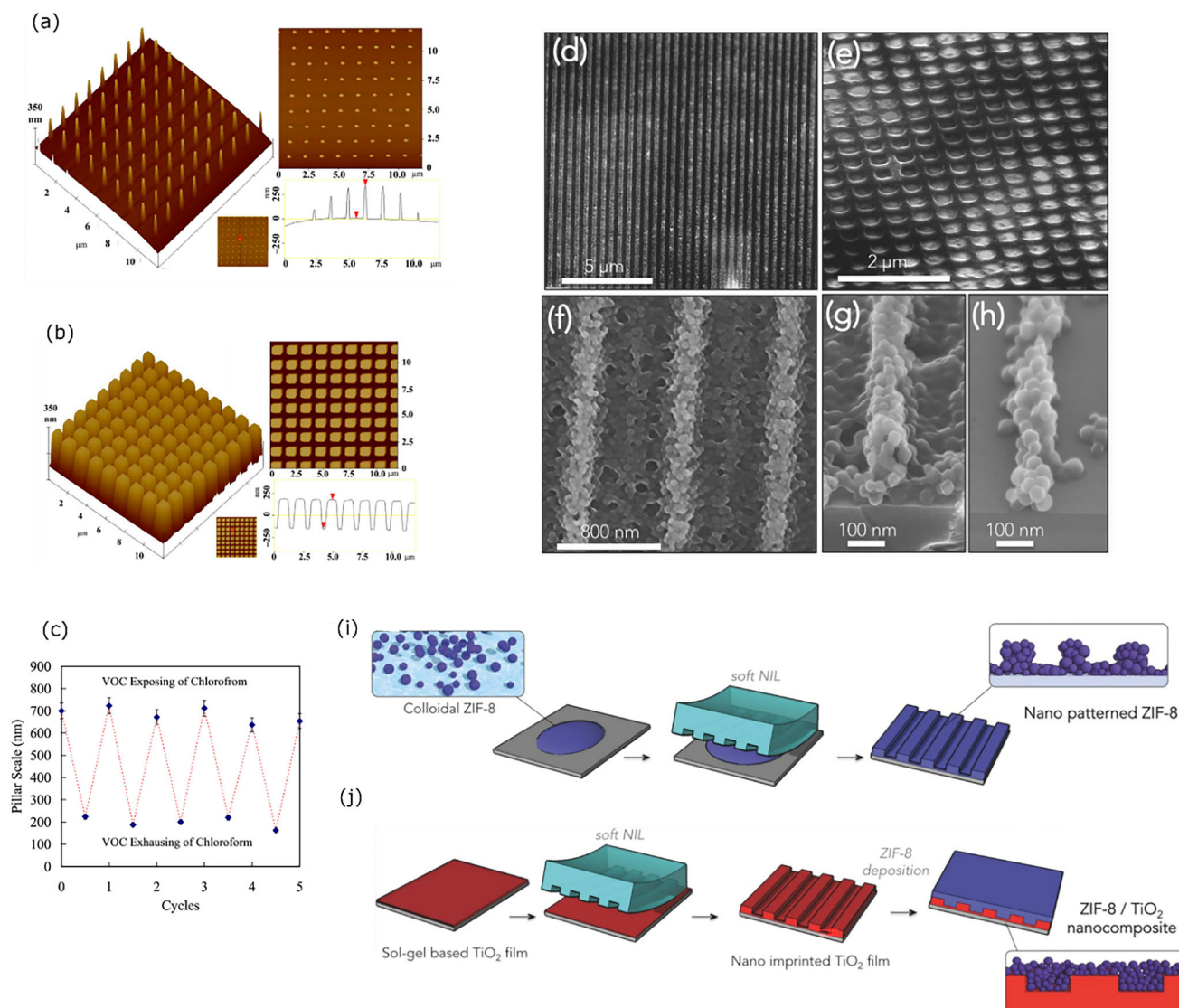


Figure 13. Two-dimensional diffraction gratings. 3D (left), 2D (right), and line cross-section (bottom) analysis AFM topographic images of 200-nm-resolution 2D periodic relief grating of tethered PMMA surface a) in dry state and b) under VOC exposing of chloroform at 20 Torr of partial pressure for 5 h. c) Variation in the scale of the pillar array of 2D periodic relief grating after five cycles of VOC exposing and exhausting of chloroform. Reproduced with permission.^[182] Copyright 2013, AIP Publishing. FEG-SEM micrographs of patterned ZIF-8 colloids showing e) square and d) nanogrooves features. f, g) Different magnifications of d) and h) without a residual layer. Soft-lithographic approaches for the fabrication of i) nanopatterned ZIF-8 and j) ZIF-8/TiO₂ heterostructures. Reproduced with permission.^[183] Copyright 2015, Wiley.

polarisation state, materials undergo permanent refractive index modulation and surface deformation. The surface deformation can usually be further enhanced by thermal treatment.^[191] A significant advantage of the holographic technique is the high precision of creating submicrometric structures with sinusoidal profile (Figure 15). Holograms also offer high flexibility and compact size, and could work both in transmission and reflectance. Moreover, the inscription of complicated structures inside the material enables image display. The response signal can be a modulation in the diffraction efficiency, a shift in the optical spectrum after illumination with white light, and also possible to observe with a naked eye as color or image change. Holographic sensors were demonstrated to detect temperature, humidity, pres-

sure, alcohols in aqua solutions, glucose, ions, and many more analytes.^[192–194] However, the field still seems not exhaustively explored for gas detection, partially due to the susceptibility of the layers in which the structures are typically recorded to water. Three groups of materials for hologram fabrication can be distinguished: photopolymers, silver-halide-based materials, and hybrid organic–inorganic materials.^[195] For gas sensing, the critical role is often the functionalising material, which will interact with the analyte but must not introduce significant light scattering; thus, the functionalising material must be added either in molecular form or in the form of nanoparticles. Due to material limitations, it is often challenging to holographically obtain profound surface relief amplitude, with the current material

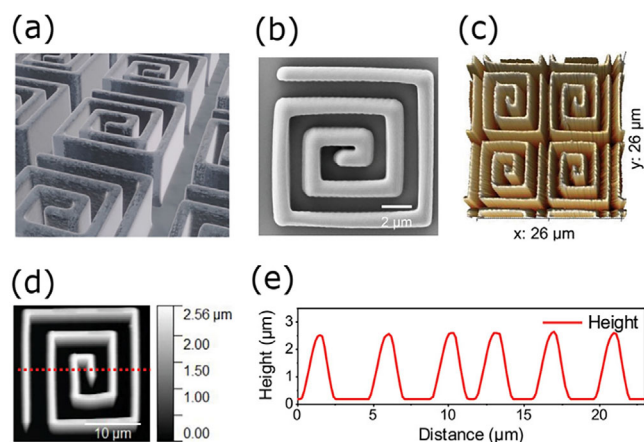


Figure 14. a) Representation of the spiral photonic array. b) Top-down SEM image of a representative hydrogel photonic sensors concentric square spiral. c) AFM image shows the 3D reconstruction of sensor structures, imaged in contact mode. d) AFM image of an extended concentric square spiral fabricated with 27.5 mW laser power; the height analysis across the red trace is shown in (e). Reproduced with permission.^[189] Copyright 2023, Wiley.

limits around 1.6 μm presented in azopolymers.^[196,197] Hence, surface gratings predominantly work in a thin regime with multiple diffraction orders.

One of the first papers that mentions holography for vapour detection was written by Ye et al.^[200] There, an interferometric approach employs a dynamic way of sensing by recording bright regions with a CCD camera, emerging after exposure to vapours. The authors showed a detection limit as small as 40 ppb to ethyl alcohol. That solution, however, requires bulky instrumentation placed on a stable optic table and, therefore, cannot be implemented in any outside laboratory application.

Hsiao et al. fabricated a reflection holographic grating structure with periodical regions of air voids, enabling VOCs to permeate into them, changing the refractive index of the structure and swelling it.^[201] Li et al. used the grating of hydrogels for carbon dioxide sensing with a detection limit of 2 ppm.^[202] PDMS stamp used for grating fabrication was obtained from copying sinusoidal surface relief grating of azo molecular glass.

Hernik et al. showed PDMS gratings obtained as a duplicate of sinusoidal holographic acrylamide gratings^[198,203] (Figure 15a). Gratings with different surface height amplitudes were compared, proving deeper structures to be more VOC-sensitive. In the newest work, authors present the use of the PDMS hologram replicas to soft-lithographically imprint metal–organic framework ZIF-71 nanoparticles into diffraction gratings^[199] (Figure 15b). Sinusoidal grating with a depth of 530 nm exhibited a limit of detection below 1 ppm to several vapours. The method was implemented further on porous materials from the CAU (Christian-Albrechts-Universität) family, showing improved sensitivity toward acetone vapor (LOD of 200 ppb). The authors' ultimate goal is to develop a compact sensor array consisting of several MOF and zeolite-based gratings with varying depths, calculated with the Raman-Nath theory for thin diffraction gratings to provide optimal sensitivity depending on the material's refractive index.

2.7.2. Volume Holograms

While relief gratings are a type of 2D structure on the material's surface, the second category of holograms is volume gratings, inscribed in a bulk material (Figure 16). They may resemble distributed Bragg reflectors, but due to their achievable complexity can be perceived as 2.5D structures. Compared to surface gratings, swelling in volume holograms is more likely to occur upon external stimuli, which may be utilized in sensing along with refractive index modulation. Furthermore, volume holograms usually work in a regime producing a single order of diffraction,^[204] thus significantly limiting energy losses. The comparison of the holographic volume-based sensor was depicted in reflection (Figure 16a,b) and transmission (Figure 16c,d) working regimes. Simulations of sensitivity upon interaction with the analyte for different geometries in thick and thin regimes were demonstrated by Cody and Naydenova for surface^[34] and volume^[35] gratings.

Leite et al. first proposed a composite of zeolite nanoparticles embedded in an acrylamide-based photopolymer.^[205] After volume grating exposure to toluene vapour, up to 22% change in diffraction efficiency was observed. Later, Yu et al. examined transmission and reflection volume acrylamide holographic gratings doped with nanozeolites, but with a slanted geometry of spatial fringes (recording setups and sensing process, Figure 17a,b).^[206] Subsequently, Mao et al. focused on detecting ethanol with different zeolite types.^[207] Studying reflection and transmission gratings, the authors discovered a blueshift up to 10 nm occurred when a decrease in refractive index overcame the influence of swelling, as the latter typically produces redshift. Therefore, that method could be potentially applied to effectively measure analytes with low refractive indices.

Castagna et al. describe transmission volume holograms doped with the HKUST-1, type of MOF.^[208] The authors examined samples with 5 and 50 μm thicknesses (Figure 17c) and two different mixture formulations. As diffraction efficiencies do not exceed 6%, further improvement to achieve higher diffraction efficiency may be required; however, preliminary experiments indicate the potential for alcohol vapor detection.

Martinez-Hurtado et al. combined laser ablation with the holographic method, enabling the fabrication of the volume hologram directly in PDMS.^[209,210] A pulsed laser was pointed at PDMS with diffused silver particles lying on a mirrored backing (Figure 18). Standing waves occurred after reflection by a mirror placed behind the recording material. The created interference pattern provided energy high enough to ablate the silver particles in a location illuminated with high intensity. The reflection hologram was exposed to a plethora of VOCs, mostly hydrocarbon gases, with the responses seen as modulation in reflected colour intensity upon changes in fringe spacing.

Recently, an experimental and theoretical investigation of the response of undoped and zeolite nanoparticle-doped photopolymer for the detection of VOCs was reported by Graceson et al.^[211] The authors develop a method for the estimation of the relative contribution of the different factors involved in the sensor's response—change in layer thickness and /or refractive index, and change in the refractive index modulation. The benefits of slanted vs unslanted volume holographic gratings are further discussed in.^[212]

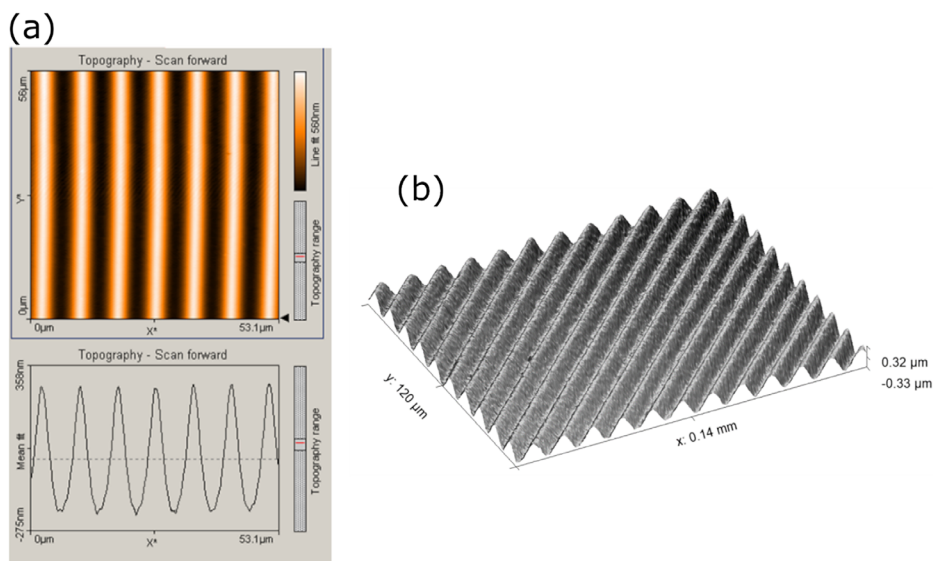


Figure 15. Surface relief gratings. a) AFM image of PDMS copy of sinusoidal holographic surface grating with 6.3 μm fringe spacing and 240 nm depth. Reproduced with permission.^[198] Copyright 2024, American Chemical Society. b) Surface profile of diffraction grating fabricated from ZIF-71 nanoparticles, recorded with a noncontact optical profilometer. Reproduced with permission.^[199] Copyright 2025, American Chemical Society.

3. Sensor Transducers Based on Interference

Interference is observed as a result of the superposition of coherent electromagnetic waves. Due to the phase differences between the two propagating waves, enhancement or weakening of light intensity is present where the waves overlap in space. In most cases, i.e., in the situation when two plane waves are combined, the interference pattern takes the form of bright and dark fringes, with a sinusoidal modulation of the intensity along the normal to the fringes. In the presence of the analyte, the phase difference

between the waves may be altered, thus leading to a shift in the observed fringe pattern. Similarly to diffraction-based sensors, interferometric VOC sensors rely on analyte-induced changes in refractive index or swelling of the sensing layer, which alter the optical path length and result in measurable fringe shifts or intensity modulation.

Generation and implementation of the interference phenomena require an interferometer. The range of possible interferometer configurations is extensive, but two of the most widely used for VOC sensing are the Fabry-Pérot and Mach-Zehnder.^[213] Additionally, Sagnac and Pohl interferometers, as well as reflectometric interference spectroscopy, are also of interest and will be discussed in the sections below. Among the less commonly employed interference techniques for VOC detection Fizeau interferometer^[214] and rugate filter^[215] have also been demonstrated.

The main advantage of the interference techniques is their excellent measurement precision, as demonstrated in applications such as determining the speed of light and detecting gravitational waves. However, the interferometry approach has drawbacks, including the requirement for a coherent light source, complex fringe analysis, exceptional susceptibility to various cross-sensitivities such as mechanical perturbations, and a lack of compactness in double-path interferometers, such as the Mach-Zehnder interferometer. Nevertheless, common-path interferometers such as Fabry-Pérot and Sagnac interferometers can be embedded into the optical fibers, thus facilitating more remote sensing applications.^[216]

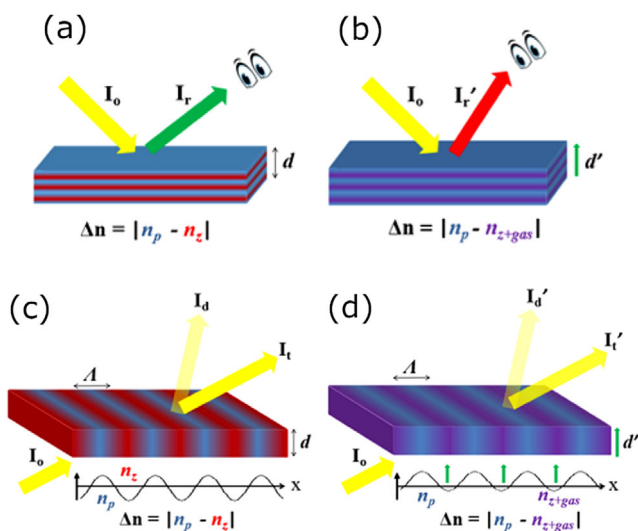


Figure 16. Zeolite-doped reflection mode volume grating with thickness d (a) before and b) after gas exposure. Zeolite-doped transmission mode volume grating with thickness d and fringe spacing Λ (c) before and d) after gas exposure. Reproduced with permission.^[35] Copyright 2018, OP-TICA Publishing Group.

3.1. Fabry-Pérot Interferometer

Fabry-Pérot (FP) interferometers are formed by a cavity separated by two reflective surfaces (Figure 19a). This kind of structure is particularly related to the Bragg mirrors, but instead of

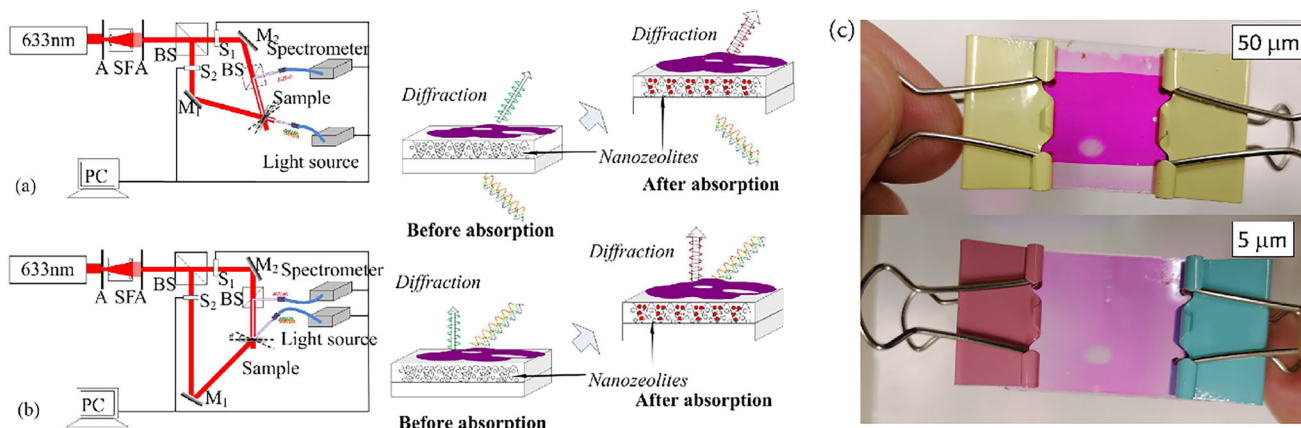


Figure 17. a,b) Experimental setup for holographic recording and schematic diagram for acetone vapor sensing process. a) Transmission recording geometry, b) Reflection recording geometry. Reproduced with permission.^[206] Copyright 2015, OPTICA Publishing Group. c) Thick and thin samples of HKUST-1 doped volume holographic gratings. Reproduced with permission.^[208] Copyright 2022, MDPI.

having multiple alternating layers, it comprises only one resonance cavity; therefore, the reflection spectrum consists of many interference peaks. The positions of those peaks are highly susceptible to the optical path length of the interferometer. Hence, alterations in the cavity length or its refractive index caused by the external stimuli may be utilized for VOCs detection. Sensing material in the FP interferometer may be realized either as the cavity medium or the reflection surface. The majority of the FP interferometers are fabricated on the ends of the optical fibers—either in the extrinsic or intrinsic form, i.e., implementing or not, respectively, the air cavity between the fiber and the reflective material, as shown in Figure 19b. One of the biggest advan-

tages of FP-based sensors is their compactness and ease of integration with other systems, including a gas chromatography column. Key limitations include significant cross-sensitivity to environmental factors—most notably temperature fluctuations and mechanical vibrations—which can easily mask or distort analyte-induced spectral shifts. Furthermore, the interferometric output consists of a series of closely spaced interference fringes, making peak assignment and tracking challenging in dynamic or multi-component environments. These factors often necessitate active stabilization or signal processing strategies to ensure reliable sensing.

While research on the FP systems for sensing dates back to the 1980s,^[220] the first mention of VOC sensing appeared in 2006 when Elosua et al. developed a multimode optical fiber with the cleaved end covered with a nanocavity doped with a vapochromic complex based on pyridine ligands.^[221] Responses to VOCs were compared by measuring the attenuation of the reflected optical power. Further, the authors compared two electrostatic self-assembly fabrication techniques and the sensitivity improvement of repetitive ethanol sensing.^[222]

Among the materials utilized for FP fabrication on the fiber tip are silver and gas-sensitive polymer layer (methanol LOD of ≈ 1 ppm)^[223] carbon nanotube composites,^[224] PDMS,^[225–229] PMMA,^[230,231] graphene,^[231] and zeolite.^[217,232–234] The group working with zeolite fiber-end-coated sensors implemented the spherically shaped fiber end^[217,233,234] (Figure 19c–g). The reason for that modification was to improve sensitivity to VOCs by increasing the adsorption amount in the zeolite film and enhancing the interference performance compared to the flat cavity.

A distinct direction of research is to connect FP interferometers with gas chromatography (GC) detection.^[235–239] Liu et al. proposed an FP cavity fabricated by the deposition of thin metal and polymer layers on the tip of the single-mode fiber, which was then assembled into a hole in the fused-silica capillary wall.^[235] Such a capillary could be further connected to the GC separation column, enabling real-time detection. The idea was further investigated by examining the operation of GC upon exposure to VOC mixtures with the use of two on-column FP sensors placed at each end of the separation column, additionally enabling

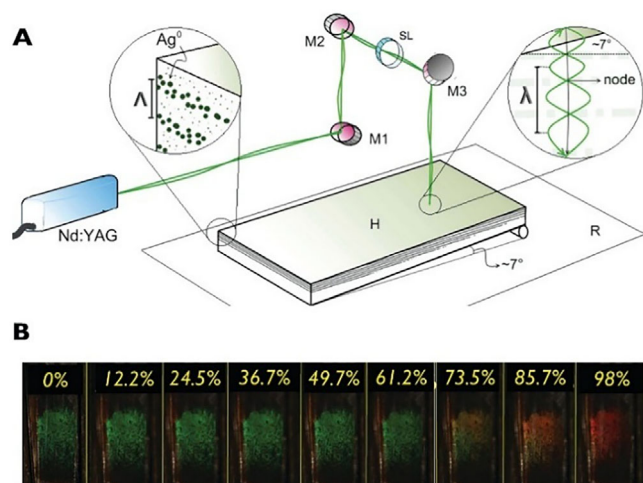


Figure 18. a) Holographic recording setup. A pulsed laser beam deviated with dichroic mirrors (M1, M2, M3) and spreader lens (SL) and reflected from a reflective surface (R) was used to form standing waves with a constructive interference pattern, ablating silver particles in hydrophobic holographic films (H). b) Photographs of a typical holographic film response in a cuvette when exposed to different concentrations of 1-butene at 22 °C. The hologram is illuminated with a white light source, showing a colour transition from green to red. Reproduced with permission.^[209] Copyright 2010, American Chemical Society.

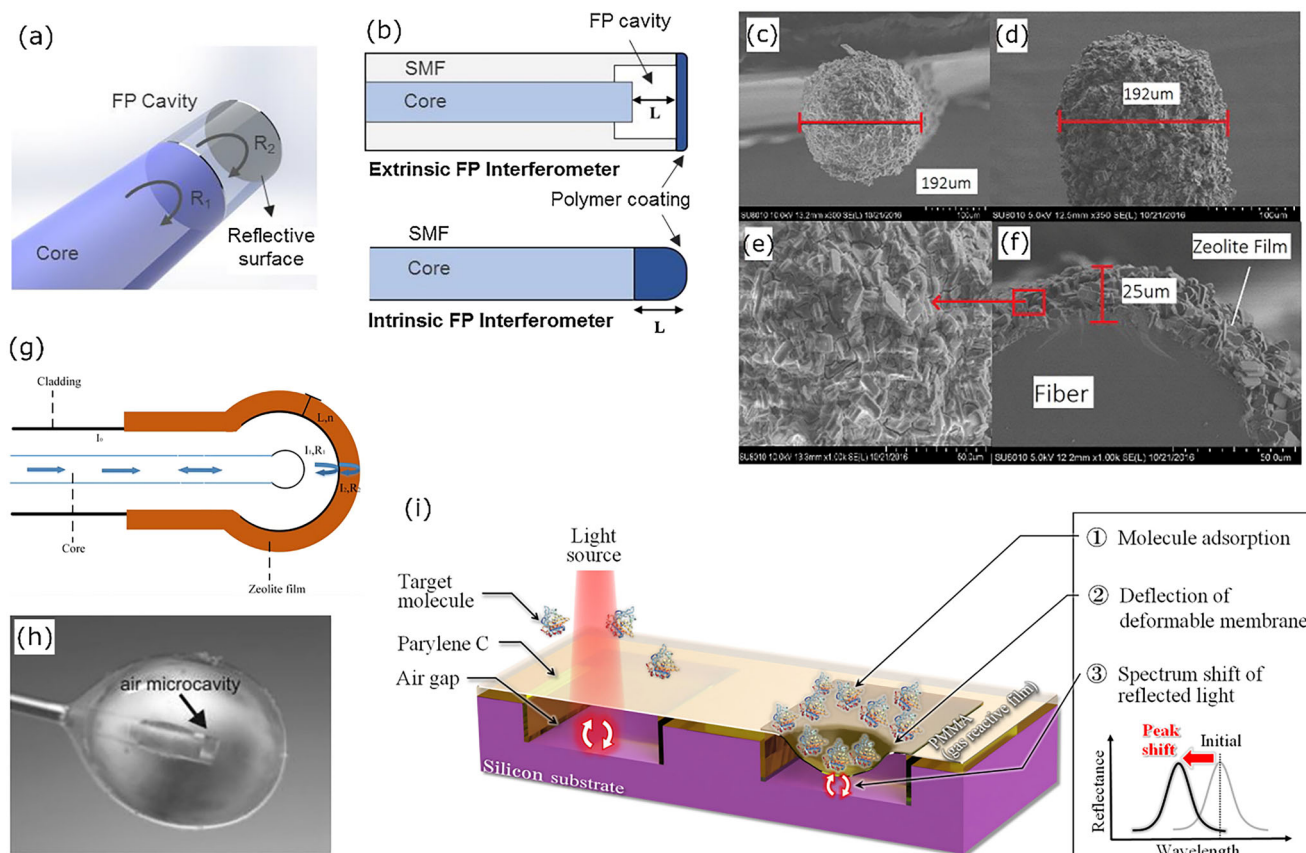


Figure 19. Implementation of Fabry–Pérot interferometer (FPI) for VOC sensing. a) FPI with two reflective surfaces, R1 and R2, and the cavity between them. b) Examples of intrinsic and extrinsic FPIs. Reproduced with permission.^[213] Copyright 2020, Elsevier. Realization of FPI on the fiber-end. SEM images of a zeolite thin film-coated spherical end fiber c) front view, d) side view, e) enlarged view, f) cross-section, g) scheme of the spherical sensing head structure. Reproduced with permission.^[217] Copyright 2017, Elsevier. h) Photograph of an air optical microcavity in PDMS placed at the end of an optical fiber for toluene sensing. Reproduced with permission.^[218] Copyright 2017, Elsevier. i) Schematic diagram and detection principle of microelectromechanical system based on the FPI surface-stress sensor. Reproduced with permission.^[219] Copyright 2020, MDPI.

measurement of time required for the analyte to pass through the column.^[238] Reddy et al. fabricated an FP sensor by spin-coating a polymer layer on a silicon wafer and then interrogated it with a GC column.^[236] In another work, a drop-coating was used to fabricate a sensor array on a chip consisting of four FP sensors for on-column detection.^[237] The device was tested through individual injection with four VOCs and their mixtures.

Melissinaki et al. focused on detecting ethanol^[240] and chlorinated VOCs^[241] using a prism-like microstructure, working as a sensing head, placed on four pillars assembled on the fiber end and separated from it with the FP air cavity. Diffusion into the porous surface of the microstructure resulted in different physisorption processes for each analyte. Kacik et al. also utilized air microcavity at the end of a single-mode fiber but surrounded by a teardrop-shaped layer of toluene-sensitive PDMS^[218] (Figure 19h). With PDMS, too, Kanawade et al. fabricated a negative axicon on the fiber tip to benefit from its good optical quality and a large cavity volume, advantageous for the polymer-swelling-based sensing process.^[229] To improve interference spectrum generation, Pawar et al. synthesized dual-cavity at the end of the single-mode fiber.^[231] Sensing material was placed in the cavities: graphene, PMMA, or their mixture. Upon ex-

posure to VOCs, the refractive index of graphene changes, and PMMA swells, leading to wavelength and intensity shifts in an interference spectrum.

Lopez-Torres et al. theoretically and experimentally compared three microstructured optical fibers with different core cross-sections for FP sensors to evaluate their sensing capabilities.^[242] All of the structures were functionalized with indium tin oxide as a sensing material and exposed to saturated ethanol vapours. Zhao et al. aimed to improve the sensitivity of the PDMS-coated FP interferometer with the use of cascaded hollow-core and large mode area fibers, thus forming two interferometers and achieving a Vernier effect.^[228] The resulting sensitivity to isopropanol was 20 pm ppm⁻¹, while a significant improvement was reported by the same group, i.e., reaching 1.2 nm ppm⁻¹, by utilizing fiber Bragg grating and zeolite as a sensing material on the FP sensor head.^[234]

Takahashi et al. fabricated an ethanol surface stress sensor based on a microelectromechanical system: a deformable membrane and a gas-sensitive PMMA layer on a cavity in the silicon substrate.^[219] In the formed FP interferometer (Figure 19i), after absorption of the gas analyte, the membrane undergoes deformation, resulting in a shift of the peak in the reflection

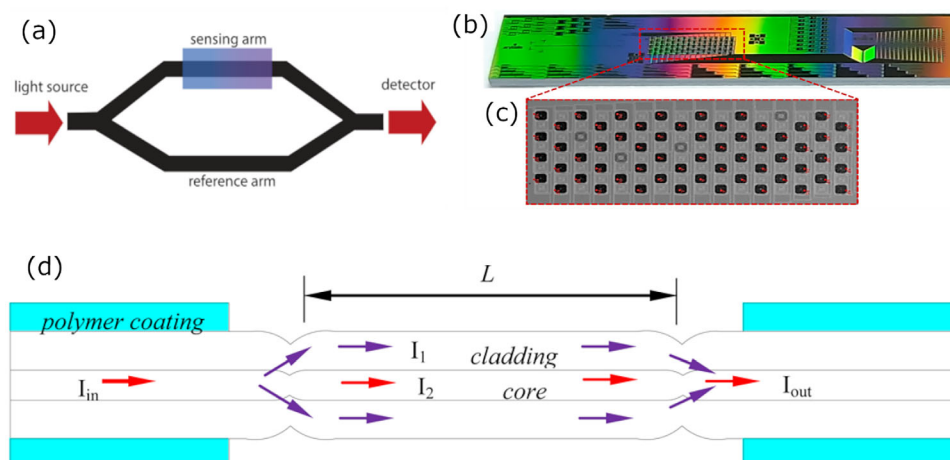


Figure 20. Mach-Zehnder interferometer (MZI). a) Schematic of the classical MZI-based sensor. b) Realisation of a sensor array—picture of a die (22 mm × 4.7 mm). The optical input grating coupler is located on the left, the 64 MZI array in the middle, and the 192 output grating coupler on the right. Test structures are also visible. c) Droplets (in black, with red dot and labelling) of biofunctionalization solutions individually spotted on the sensing arms of the MZI array. Reproduced with permission.^[246] Copyright 2022, Optica Publishing Group. d) Schematic diagram of the MZI where the fiber core and cladding serve as the interferometer arms. Reproduced with permission.^[247] Copyright 2011, MDPI.

spectrum. Ganter et al. developed a nanosheet-based FP interference sensor array, showing a change of colour upon exposure to saturated organic vapours and varying humidity.^[243] The proposed color-change sensing mechanism was based on amine intercalation into the photonic nanosheets and provides distinctive detection due to the different optical shifts and intercalation times for each amine.

Recently, Zhan et al. implemented a colourimetric detection method utilising metal-organic frameworks to form FP films.^[244] Selected MOFs were placed on silicon substrates, and then a 2 nm-thick platinum film was sputtered on top, working as a mirror of the FP cavity. The authors implemented machine learning algorithms to analyze the data from the sensing array and obtained a high accuracy (over 90%) of 6 VOCs classification, maintaining LOD at the 10–50 ppm level. Simultaneously, similar research was presented by Monajezi et al.^[245] MOF films were deposited on a reflective gold layer, then coated with PMMA and a second gold layer, forming an FP cavity. By comparing the responses of the sensor to toluene and ethanol, the authors demonstrated that in both cases, the interference spectrum was shifted towards the longer wavelengths. After exposure to toluene, however, resonance was also broadened, while upon exposure to ethanol, it remained sharp, thus allowing for discrimination between the two VOCs.

3.2. Mach-Zehnder Interferometer

Mach-Zehnder (MZ) interferometer comprises two wave-propagating arms with a common light source, generating a coherent and collimated beam, guaranteeing a constant in time phase difference between the waves. Then, two waves merge again at the device output, producing an interference pattern. When the medium of one arm is influenced by external stimuli (i.e., extending its optical path), the phase of the light coming through it will also alter, thus shifting the interference spectrum.

By demodulating the phase difference, one can measure the concentration of a gas analyte around the sensing arm. A simple scheme of the MZ interferometer for sensing purposes is presented in **Figure 20**.

Fabrication methods of MZ sensors may vary, as possible common implementations of the setup include open space, fiber-based, and chip-based interferometers. Therefore, MZ-based sensors offer high flexibility and versatility. Importantly, a well-designed MZ interferometer can minimize thermal and spectral noises. However, they still remain a complicated sensing platform, generating a complex optical spectrum that has to be analysed. Drawbacks of MZ interferometry come from its mathematical working principle,^[246] i.e., sensitivity varies as a sine function, even vanishing for certain phase delay differences between the two interferometer arms. Moreover, the direction of the differences in the two arms is ambiguous, also due to the sine shape of the intensity variations. To address those issues, a coherent detection scheme can be utilized.

Siarkowski et al. fabricated an MZ interferometer consisting of sensing and reference arms,^[248] in accordance with the representation depicted in Figure 20. Both arms were coated with plasma polymerized thin film, adsorbing polar and nonpolar organic compounds. A similar approach was chosen by Nitiss et al. but utilized an all-organic waveguide with PMMA-based sensing material as a cladding.^[249] In a recent (2023) paper, Zhao et al. reached pyridine sensing with a limit of detection of 476 ppb by using a waveguide with a dipolar polycarbonate cladding.^[250]

Another approach to realising the MZ interferometer was proposed by Ghosh et al., which developed the composite plasmonic waveguide, exhibiting surface plasmon polaritons excitation, coated with porous ZnO for ethanol detection.^[251] Implementing a similar plasmonic-based approach, Zhou et al. proposed a sensor with an improved sensitivity.^[252] MZ interferometers integrated on-chip for ethanol^[253] and acetone^[254] detection were presented. Ethanol sensing was performed with MOF coated SiO₂ waveguide chip. An evanescent wave

propagating through the MOF was enhanced by refractive index change caused by VOC adsorption and was then affecting the interference spectrum. Detection of acetone with concentrations below 1 ppm was possible with polyethyleneimine/graphene oxide gas-sensitive film.

B. Li et al. developed a zeolite-coated MZ interferometer for sensing changes in environmental refractive index caused by acetone vapour.^[247] In the proposed interferometer, one beam propagated through the fiber core and the second through the cladding (see Figure 20b,c). Part of the light was coupled back from the cladding to the core, thus interfering with the first beam. The detection method was based on refractive index modulation, changing the effective propagation constant of the cladding modes, thus enabling sensing by monitoring the phase shift of the interference fringe. Choosing a similar approach, Wu et al. composed an in-line MZ interferometer in a MOF-coated fiber by fabricating two long-period gratings in it.^[128] Half of the light from the first grating was coupled into the cladding, thus exciting a co-propagating mode, sensitive to changes in the refractive index of the MOF cladding after VOCs adsorption. In the second grating, the cladding mode was coupled back to the core, combining two modes and producing interference fringes in the optical spectra propagating out of the fiber.

Y. Li et al. performed a detection of VOCs with a liquid crystal-based fiber.^[255] The MZ configuration was obtained with the interference between two waves with opposite polarisation states propagating through the anisotropic crystal medium. Response to VOCs was observed as a blue shift of the attenuation peak wavelength when the vapor was adsorbed into the liquid crystal.

Laplatine et al. demonstrated a sensor array comprised of 64 MZ interferometers integrated on a silicon nitride substrate (Figure 20d).^[246] On each element of the array, a bioreceptor, i.e., a short peptide, was grafted to obtain high selectivity to a wide range of VOCs. The created photonic integrated circuit comprised a single grating coupler in the input and an array of them at the output. In between, biofunctionalized MZ interferometers were placed, and the light in the system was propagating through the multimode interference couplers, equally distributing light into the sensing array, splitting it between the sensing and reference arms, and finally recombining them. Limits of detection in the ppm range were reported to several VOCs.

3.3. Sagnac Interferometer

In the Sagnac interferometer, both light waves travel through the same path in the loop, but in different directions. Therefore, any distortion in that path will affect both the interferometer arms, which is not directly applicable to most sensing scenarios. As such, Sagnac interferometers are most known for their highly precise rotation measurements and in gyroscopes, due to the movement of the device influencing the phases of the propagating beams differently.^[256] That gives the Sagnac interferometer excellent immunity to temperature effects, acoustic noise, and other environmental stimuli, but it also elicits a need for modification for gas sensing applications.

Ning et al. implemented two interferometers: Fabry–Pérot and Sagnac, to detect two VOC mixtures using PDMS as a sensing material.^[226] The authors differentiate between ethanol and 2-

propanol with the use of an inverse matrix algorithm. In both cases, the sensing response was PDMS swelling; in the Fabry–Pérot interferometer, it induced modulation of the cavity length at the end of the fiber. For the Sagnac interferometer fabrication, a polarization-maintaining fiber was used. The scheme of a proposed sensor is depicted in Figure 21. Here, tension force from swelling causes strain on the fiber, modulating its length, thus changing the path difference of the two polarization modes, which in turn shifts the wavelength in the interference spectrum. Response of the Sagnac interferometer was nonlinear, stronger than the Fabry–Pérot interferometer to 2-propanol, but weaker to ethanol.

Li et al. developed an ethanol sensor based on a liquid crystal-embedded fiber Sagnac interferometer.^[257] Nematic liquid crystal, into which analyte molecules were permeating, was placed in the ceramic tube between two single-mode fibers, creating a loop. Upon ethanol adsorption, birefringence increased, thus shifting the wavelength of the interference peak.

Elandaloussi et al. modified a Sagnac interferometer to measure the optical path length of cells, which can be implemented into absorption-based sensing.^[258] In the proposed interferometer, beams are slightly separated, so the gas cell was placed in the path of one of them. The optical path length difference between the beams was close to zero for an empty gas cell and changed after the gas was introduced into it.

3.4. Reflectometric Interference Spectroscopy

Reflectometric interference spectroscopy (RIS) is based on the interference of light reflecting from multiple layer interfaces. An example of such a structure is shown in Figure 22. Upon analyte interaction with the material, the optical path length changes, leading to a shift in the interference pattern. This technique resembles Bragg-based methods; however, RIS is less commonly utilized for VOC sensing. The main difference is that RIS measures shifts in interference fringes reflected from a thin multilayer system, while Bragg-based methods measure shifts in the peak reflected wavelength from the structure with periodically modulated refractive index. While the interrogation of the Bragg-based sensors can be carried out by an incoherent light beam, a coherent light source is critical for RIS to produce clear and stable interference patterns.

In one of the early attempts, Reich et al. proposed a compact VOC sensor system based on RIS and utilizing four independent wavelengths.^[260] The authors compared its performance with a system utilizing a white light source. Low limits of detection, in the range of several dozens of ppm for the compact sensor and below 10 ppm for a sensor with white light, were obtained for several VOCs. Sensing performance was based on changes in the thicknesses of polymer layers.

Kim et al., by comparing reflectance spectra before, during and after exposure of porous silicon multilayer to VOCs, observed a reversible red shift of the reflectance peak.^[259] The fabricated 3 μm thick multilayer (Figure 22) was characterized by a random distribution of pores of an average diameter of 20 nm each. Wang et al. proposed a silica-nanochannel membrane to detect VOCs at ppb level.^[261] They compared reflectometric spectra obtained from one, two, and three silica layers, showing that a more

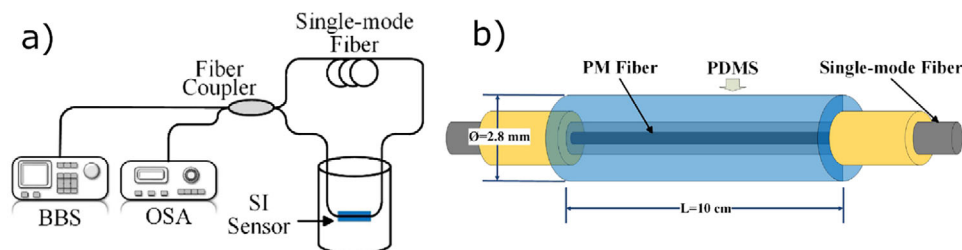


Figure 21. a) System setup for the Sagnac interferometer-based VOCs detection. b) Construction of the PDMS-coated Sagnac interferometer sensor. Reproduced with permission.^[226] Copyright 2016, Optica Publishing Group.

complex interference spectrum with multiple peaks is generated when the number of adjacent interfaces is increased. To find the trade-off between simplicity and the largest wavelength shift, the authors investigated a 3-layered silica membrane. From the measured VOCs, the lowest limit of detection obtained was for acetone (55 ppb), thanks to high refractive index variations after VOC adsorption.

Sebok et al. measured n-hexane adsorption on ZnO₂ nanohybrid film of 500 nm thickness containing 20 bilayers.^[262] Based on quartz crystal microbalance and contact angle measurements, the authors selected gold nanoparticles as the most promising film-modifying agent for further RIS experiments and adsorption studies. That functionalization was aimed at hydrophobicity and selectivity enhancement and resulted in two to three times stronger adsorption of n-hexanol compared to other VOCs, such as methanol, ethanol, and toluene, while before functionalization there was no significant distinction between those four examined vapours.

3.5. Pohl Interferometer

VOC sensing based on the Pohl interferometer was proposed by Martínez-Hipatl et al.^[263–265] The interferometer design is simple—consists of a VOC-sensitive film placed on a glass substrate, light source, and detector measuring the optical signal reflected from the structure (**Figure 23a**). In such a system, three

reflected beams are present (**Figure 23b**), producing an interference pattern (**Figure 23c**).

The authors utilized a spin-coated PDMS layer as the sensing material. In the initial work, the photodetector was covered with a mask to capture a small part of the fringes, so the changes in the intensity of light were measured when the fringes were moving.^[264] Such configuration depends on manually finding the optimal point on the fringe pattern. As an improvement, the authors proposed capturing the pattern with the CCD camera and acquiring the data with a computer program.^[265]

4. Sensors Utilizing Propagation of Evanescent Wave

Optical fibers have played a critical role in modern telecommunications since the development of low-attenuation optical fibers for long-distance light transmission in the 1970s.^[266] In parallel with the progress in optical fibers and fiber-based components for the telecoms industry, significant effort has also been invested in exploiting optical fibers' potential for sensing in various application areas, including sensing of VOCs. However, while optical fiber's immunity to environmental influences, such as temperature and humidity, is a significant strength for optical communications, for sensing applications, this strength becomes a disadvantage since sensing requires a strong interaction of guided light with the local environment or analyte.

Most optical fibers have only a slight refractive index difference (RI) between the core and the cladding and satisfy the weakly

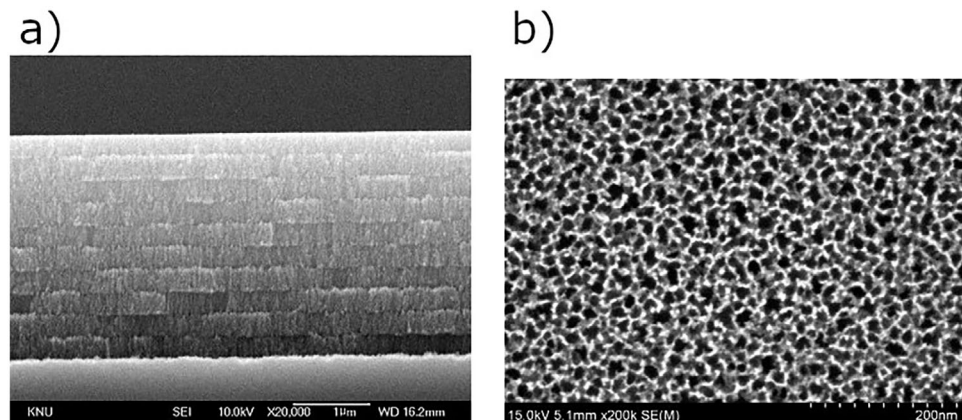


Figure 22. a) Cross-sectional image of the porous silicon multilayer on the silicon substrate for the reflectometric interference spectroscopy. b) Surface image of the porous silicon multilayer produced by electrochemical etching. Reproduced with permission.^[259] Copyright 2011, Elsevier.

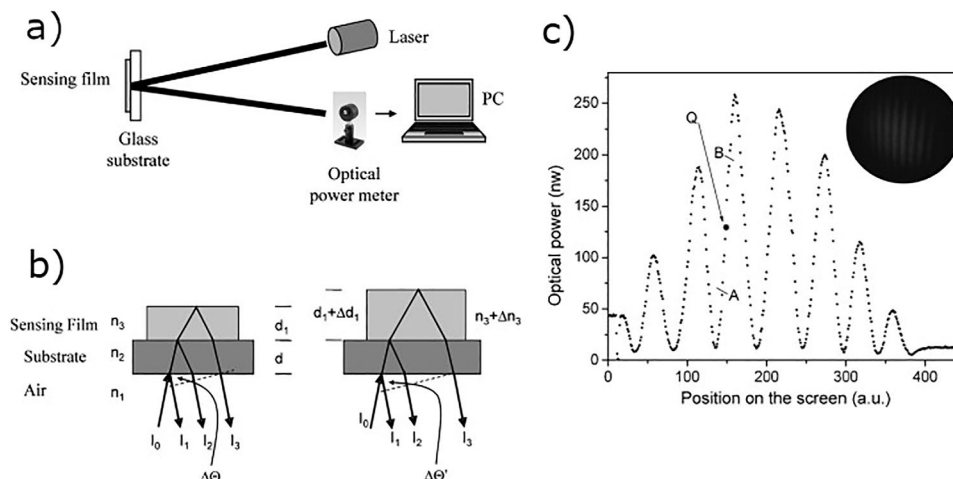


Figure 23. Pohl interferometer for VOC sensing: a) arrangement, b) operation principle, and c) optical power profile of the interference pattern for a laser with a Gaussian beam; the inset shows the fringe pattern obtained on a screen. Reproduced with permission.^[264] Copyright 2010, Elsevier.

guiding condition.^[267] Due to the small RI difference between the core and the cladding, the weakly guided fundamental modes have a mode field diameter which exceeds that of the core and thus extends into the cladding with the result that a substantial fraction of the light (up to 20%) propagates within the cladding as the evanescent field. The penetration depth of the evanescent field (d_p) in the cladding is given by:^[268]

$$d_p = \frac{\lambda}{2\pi \sqrt{n_1^2 \sin^2 \theta_1 - n_2^2}} \quad (3)$$

where λ is the wavelength of the light, θ_1 is the incident angle at the interface of the fiber core/cladding and n_1 and n_2 are the RIs of the fiber core and cladding, respectively.

However, this evanescent field is not accessible for sensing purposes due to the large diameter of the cladding relative to the mode field diameter.^[266] As a result, a wide variety of approaches to fiber modification have been developed that allow the fiber sensor head to interact effectively with the local environment. Many optical fiber sensors expose a large proportion of the evanescent field to the environment under test through fiber tapering, removing the fiber cladding by etching, and utilizing U-bends or D-shaped fiber structures. Such sensors are called “evanescent wave (EW) fiber sensors.”^[269] EW sensors operate based on the interaction of the evanescent field of light traversing an optical fiber with the surrounding environment or analyte. The measurements obtained by these sensors are not affected by the bulk surrounding RI because the penetration depth of the evanescent field ranges from ten to several hundred nanometers.^[270] Numerous types of EW fiber optic sensors have been developed to date, with many of them using different kinds of sensitive coatings that enhance the sensitivity and selectivity and lower the limit of detection (LOD) of fiber-optic sensors.

Generally, a typical optical fiber EW sensing system can be divided into four parts, as illustrated in **Figure 24a**, including the Optical source, Sensor head, Detector, and the optical path, usually realized by Optical fibers.^[271] Depending on the specific application, the sensor head could be a bare or functionalized fiber

structure with an additional coating layer of sensitive material on the fiber surface. The sensing is realized by allowing the transmitted or reflected light in the sensor head to interact with the surrounding medium. Due to the change in environmental parameters, molecular binding, or chemical reactions within the coating materials, the local RI close to the fiber surface is changed. In turn, the change in the parameters of interest can be determined by a suitable calibration of these variations in the RI.

According to the type of optical fiber structure in the sensor head, EW sensors can be categorized into unclad, D-shaped, U-bent, and tapered sensors.

4.1. Unclad Fiber with a Straight Geometry

If a section of the cladding is removed from the fiber (**Figure 24b**), the approximation of weakly-guided light used for the description of transmission in a standard singlemode fiber (SMF) is no longer applicable since, for the standard SMF, the RI difference between the core and cladding is less than 1%, whereas, for an unclad fiber, the difference in RI is dictated by the external environment and can be much more significant. This leads to the variation of the output intensity as a function of the optical properties of the medium surrounding the unclad section. Once the fiber cladding is removed, its refractive index n_2 in Equation (1) will be replaced by the refractive index value of air or absorptive media surrounding the core.

The skip length model, depicted in **Figure 24c**, assumes that the evanescent wave is absorbed at each point of total internal reflection along the entire length of the exposed fiber core. The skip length, L_1 , defined as the distance along the fiber axis between successive internal reflections of a single light ray, can be shown to be:

$$L_1 = D \sqrt{\frac{n_1^2}{\sin^2(\theta_L)} - 1} \quad (4)$$

where D is the fiber’s core diameter in the sensing region, n_1 is the core refractive index, and θ_L is the light launch angle into

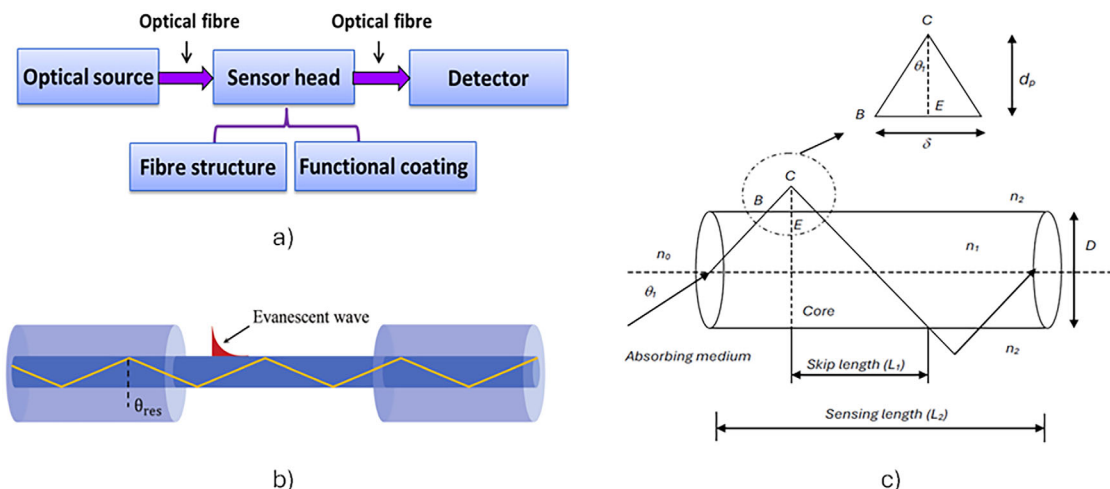


Figure 24. a) Schematic diagram of an EW fiber sensor system; b) Schematic diagram of an EW sensor based on an unclad sensing fiber. Reproduced with permission.^[272] Copyright 2023, Wiley; c) Reflected light track in the fiber sensing area (skip length model).

the fiber core. At each point of internal reflection, the distance in the direction of propagation travelled by the electromagnetic wave while it has penetrated the surrounding air or absorbing medium is defined as the displacement, δ . The displacement can be considered the effective absorbing path of the electromagnetic wave by the medium with each internal reflection down the fiber:

$$\delta = \frac{2d_p L_1}{D} \quad (5)$$

If the length of the fiber core is exposed to the surrounding medium, the fiber interaction length is denoted L_2 , and the total number of internal reflections, N , in the L_2 can be calculated as:

$$N = \frac{L_2}{L_1} \quad (6)$$

and the total length of the absorption path, L_{abs} , will be:

$$L_{\text{abs}} = N\delta = \frac{L_2 \delta}{L_1} = \frac{2d_p (L_2)}{D} \quad (7)$$

Equation (5) shows that the expected absorption is proportional to the exposed core's length, and the absorption measurements' sensitivity is also inversely related to fiber diameter. Therefore, accurately controlling the etching depth in the fiber etching process is the key to making high-sensitivity EW sensors.

Fiber cladding can be removed by mechanical or chemical etching.^[274–276] For example, in ref. [274], a mixture of ammonium fluoride and hydrofluoric acid was utilized for fabricating the sensing zone of the multimode silica fiber (MMF), and in ref. [275], pure acetone was employed as the chemical solvent to remove a portion of the fluoropolymer cladding from a polymethylmethacrylate (PMMA) fiber. In the same study,^[275] the mechanical etching procedure used sandpaper and abrasive aluminium oxide polishing film of various sizes to etch the fiber's cladding. Mechanical etching delivers a smooth fiber surface and efficiently avoids fiber breakage.

Various materials, such as metal oxide semiconductors (MOS), polymers, and polymer–MOS hybrid nanomaterials, have been explored extensively for VOC detection. MOS has unique features like a large surface-to-volume ratio, good crystallinity, and high chemical and thermal stability. Zinc oxide (ZnO) and tin oxide (SnO₂) are n-type wide bandgap semiconductors ($E_g = 3.37$ eV and 3.6 eV) that have been widely used as sensing layers for the detection of VOCs, respectively. Moreover, these metal oxides have appropriate optical conditions for evanescent sensing, such as a higher refractive index ($n > 1.9$) and good optical transparency. In addition, few reports have suggested that doping the metal oxides with metal nanoparticles, such as Al, Cu, Zn, and Pt enhances the sensing performance of VOCs.^[277]

Renganathan et al. reported a sensor for detecting methanol, ethanol, and ammonia vapours.^[278] They studied a modified clad using Ce, Li, and Al-doped ZnO as sensing layers separately, with the highest sensitivity of 58, 54, and 56 counts/100 ppm for ammonia, methanol, and ethanol, respectively. Prasanth et al.^[277] demonstrated sensing of acetone, acetophenone, ethanol, and isopropanol (IPA) with sputter-coated thin films of ZnO, AZO, and SnO₂ as the sensing layer uniformly deposited over an unclad core of the optical fiber. SnO₂-coated sensor probe shows a maximum wavelength shift of 2.4% toward 250 ppm IPA. The detection limit of the SnO₂-coated sensor probe was 300 ppb. Bagchi et al.^[279] investigated a cladding-stripped multimode optical fiber sensor probe coated with polypyrrole-polyethylene oxide (PPO) nanofibrous mat for detecting ammonia, ethanol, methanol, and triethylamine vapours with detection limits of 0.1 ppm, 2 ppm, 2 ppm, and 1 ppm, respectively.

4.2. D-Shaped Fiber

In an EW sensor based on a D-shaped fiber, a D-shaped sensing region is made by removing part of the cladding and even the fiber core on one side of the optical fiber, as shown in Figure 25a,b.

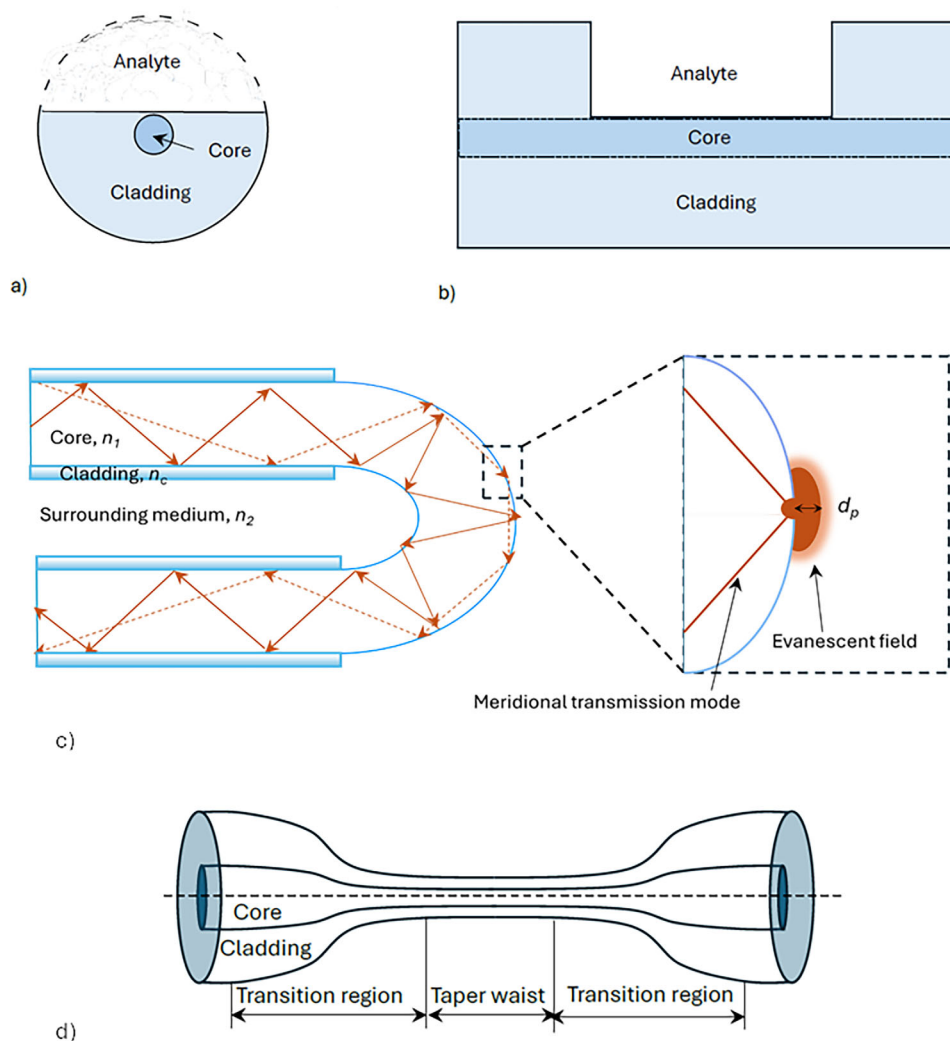


Figure 25. Conceptual illustration of a D-shaped optical fiber sensor: a) cross section view and b) detailed schematic of a D-shaped optical fiber; c) Geometry of the U-shaped sensing region and the representation of a meridional transmission mode (left); illustration of the penetration depth (right); d) Schematic of a tapered microfiber with a uniform waist region in the center connected to the original fiber ends via two transition regions.

The sensitivity of D-shaped EW sensors depends on three main factors: the light power coupled from the surface of the cross-section, the attenuation of the EWs, and the decrease in the number of propagating modes originating at the surface of the cross-section within the fiber. For a given refractive index of the surrounding air or analyte, the attenuation of the EWs is determined by their penetration depth and the number of total reflections on the surface of the sensing region.^[269,280] The D-shaped fiber EW sensors can achieve very high sensitivity, but their precise polishing in practice proved complex. The polishing depth needs to be optimized to achieve maximum light coupling in the core region with the surrounding environment, resulting in a high sensitivity. Additionally, the polishing of fiber may make it fragile, limiting the longevity of the sensor.

In 2015, Yeom et al. reported a D-polished fiber sensor for VOC detection using a wavelength interrogation technique^[281] with enhanced sensitivity, selectivity, and dynamic range. The sensing head consisted of a standard SMF functionalized with a thin

layer of the sensing membrane that required five types of solvatochromic dyes for its synthesis. The sensor was investigated for five kinds of VOCs: dimethylamine, benzene, ethanol, acetic acid, and toluene. The results showed linear response characteristics with high resolution in the range from 1 to 10 ppb. Khan et al.^[282] developed a gas sensor array also using four different types of solvatochromic dyes and two different types of polymers, to make the five different types of sensing membranes. These were deposited on the five side-polished optical fibers by a spin coater to make the five different sensing elements of the array, and then principal component analysis (PCA) was applied to discriminate between the different VOCs. Gordon et al.^[283] studied a single-mode D-fiber with a 2.5 μm polydimethylsiloxane layer applied after removing a section of the fiber's cladding. The sensor was naturally reversible and responded exponentially to gas and liquid concentrations of dichloromethane and acetone. In another study, Lowder et al.^[101] also utilized a polydimethylsiloxane layer for sensing dichloromethane and acetone, but in their proposed

structure, it was coated on top of a surface-relief fiber Bragg grating to enhance the sensitivity.

4.3. U-Bent Fiber

The evanescent field of an unclad fiber can be further enhanced by bending it into a U-shaped structure, as shown in Figure 25c. The main parameters influencing the sensitivity of the U-bend EW sensors are the bend radius, core diameter, sensing region length, and fiber numerical aperture, where optimum values were reported for each parameter to reach maximum sensitivity.^[284] Meanwhile, each fiber material, glass or plastic, has advantages in different applications. The main concern for U-shaped EW sensors is the integrity of the fiber, which ensures sustainable operation. Therefore, a mechanically strong fiber is more desirable. Plastic fibers have inherent advantages over glass fibers, such as lower cost, better mechanical stability and strength, lower possible bend radius, and greater flexibility and robustness. In contrast, silica fibers are brittle, which results in lower mechanical strength and affects long-term performance.^[285]

In 2017, Memon et al.^[286] reported a highly sensitive U-bend evanescent wave plastic optical fiber sensor for ethanol concentration measurement in water. The sensor was fabricated using a low-cost, large core (1000 μm diameter) plastic optical fiber and investigated in the broadband visible light spectrum with significant response in the range of 0.0126%/v to 0.0633%/v ethanol concentration, which corresponds to the detection of a change in refractive index as low as 10^{-7} .

It should be noted that despite the high sensitivity of the U-bend structures to the surrounding refractive index, detecting VOCs in the gas phase in the air through EW sensing remains a significant challenge. Gas is a low-density matrix (approximately three orders of magnitude lower in density than liquid or solid matrices), resulting in a small number of molecules at the fiber surface at any given time and consequently causing a low efficiency for these sensors operating in the gas phase.^[287] Nanoporous materials, such as porous silicon (pSi), are highly useful in adsorbing and condensing VOCs due to capillary action in the nanopores of the pSi.^[288] In ref. [287], an EW U-bend sensor was reported utilising a nano-porous cladding capable of concentrating volatile molecules near the fiber's surface, thus increasing the number of molecules interacting with the evanescent field. The sensor exhibited high sensitivity to isopropyl alcohol, ethanol, and acetone. Gao et al.^[289] reported a U-bent fiber sensor coated with a graphene oxide (GO) coating for aqueous ethanol detection. Li et al.^[290] reported a U-bent multimode fiber sensor covered by a molybdenum disulfide (MoS_2) film, grown on the fiber directly by thermally decomposing the precursor of $(\text{NH}_4)_2\text{MoS}_4$. It showed good sensitivity, linearity, repeatability, and stability in detecting aqueous ethanol.

Another approach to increasing the sensitivity and selectivity of EW sensors to various VOCs is utilising the localized surface plasmon resonance (LSPR) phenomenon. To achieve this, in ref. [291], an unclad U-bent portion of a multimode fiber was coated with a colloidal layer of noble metal nanoparticles (Ag and Au) with an average diameter of 20 nm. The electric field of the plasmons interacts with the VOC vapours, causing

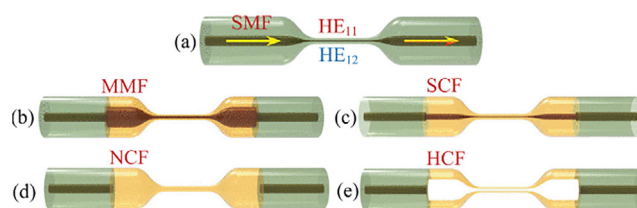


Figure 26. A schematic diagram illustrating different tapered fiber structures a) SMF, b) SMS, and heterostructures c) SSCS, d) SNCS, and e) SHCS.^[37] Reproduced with permission.^[299] Copyright 2021, IEEE.

a change in resonance conditions of the localized plasmons, which eventually modulates the output light signal of the fiber. The Ag-nanoparticles-coated sensor demonstrated sensitivities of 0.01453, 0.02072, 0.03722, and 0.02567 mV ppm^{-1} to methanol, acetone, ethanol, and propanol, respectively. In contrast, for the Au-nanoparticles-coated sensor, the corresponding sensitivities were 0.03156, 0.07150, 0.04319, and 0.08366 mV ppm^{-1} .

4.4. Tapered Fibers

In the case of tapered optical microfibers, the total fiber diameter is reduced to a few micrometers. The evanescent field extends into the surrounding medium, effectively propagating outside the fiber. This means that the evanescent field of optical microfiber is accessible and can be utilized for sensing. The EW penetration depth of a tapered fiber can be expressed using Equation (1).

A typical tapered optical microfiber has a shape and structure, as shown in Figure 26d. It comprises a uniform-diameter waist region at the centre and two tapering transition regions. The fiber ends retain the original untapered dimension and thus can be easily connected to other standard optical fibers.

In principle, the optical properties of tapered microfibers depend on the geometry of both the transition and the waist region. The decrease in the waist diameter results in multiple modes coupling, enhancing the sensor's sensitivity. Tapering can be performed on cladded and uncladded fibers.^[292] Tapering is typically performed by pulling the fiber during heating so that the diameter of the tapered region is smaller than the core diameter.^[293]

Gravina et al. introduced a plastic tapered EW sensor for sensing methylene blue in water.^[294] The very low refractive indices of the core-cladding perfluorinated polymers ($n = 1.35\text{--}1.34$) permitted a strong enhancement of the evanescent wave power fraction in aqueous environments, leading to good sensitivity. Jarzebinska et al.^[295] proposed an ammonia gas sensor based on a single-mode tapered optical fiber with nano-assembled coating. The film coating was composed of alternate layers of tetrakis-(4-sulfophenyl) porphyrine (TSPP) and poly(allylamine hydrochloride) (PAH), which were deposited using the electrostatic self-assembly process. The fiber with the smallest (9 μm) diameter was able to provide the best sensitivity ($0.440 \pm 0.002 \text{ mV/ppm}$) and detection limit ($2 \pm 0.3 \text{ ppm}$). Ibrahim et al.^[296] reported another ammonia sensor with polyaniline-coated multi-mode fiber, whose sensitivity was enhanced by 61% when the waist diameter decreased from 40 μm to 20 μm . Aziz et al.^[297] demonstrated a tapered multimode fiber probe coated with a silver nanoparticle-incorporated reduced graphene oxide (Ag/rGO) nanocomposite

for sensing ethanol in water. Huang et al.^[298] demonstrated an optical microfiber probe functionalized with ZIF-8/GO multilayers concentration-specific responses to ethanol and methanol vapours with a dynamic range of up to 6850.1 ppm and a low detection limit (5.26 ppm).

Multiple studies focused on enhancing the evanescent field-surrounding medium interactions by tapering various fiber heterostructures instead of simple single- or multimode fibers,^[271,299] as illustrated in Figure 26. A simple example of a fiber heterostructure is a single mode–multimode–single mode (SMS) structure consisting of a short section of multimode fiber (MMF) fusion spliced between two SMS fibers. Other examples include single mode–no-core–single mode (SNCS), single mode–small core–single mode (SSCS), single mode–hollow core–singlemode (SHCS).

Tapering the above fiber structures allows access to the evanescent field of multiple propagating modes within the taper waist section, making them suitable for VOC sensing, usually in combination with appropriate functional layers. For example, in refs. [300,301], Liu et al. fabricated a tapered SSCS fiber structure with an ultrahigh RI sensitivity of 19 212.5 nm RIU⁻¹. The tapered SSCS fiber was then functionalized with sol–gel for ammonia sensing^[302] and simultaneous measurement of methanol and ethanol concentrations.^[303]

If two SMS fiber structures physically in contact with each other are fused and tapered to a small diameter, a strong evanescent field coupling between them will occur, making such an optical fiber coupler structure suitable for sensing.^[299] The functionalisation of tapered SMF couplers enables the sensors to detect ammonia^[304] and ethanol.^[305]

4.5. Integrated Waveguides

Planar dielectric waveguides (WGs) also have several attractive features for evanescent-wave sensing and demonstrate comparable performance in situations where evanescent-wave sensing using optical fibers can be challenging. Planar optical waveguides offer several distinct advantages over fibers in sensing applications, although fiber sensors are more versatile. The benefits of planar waveguide sensors include their mechanical robustness, the potential for integration with other optoelectronic components for compactness, and the relative ease of applying specific coatings on the waveguide surface for evanescent sensing.^[306]

Different WG geometries, such as rib, ridge, and slot WGs, have different properties, including the characteristics of the evanescent field. Rib WGs typically comprise a thin strip (rib) of high-refractive-index material on a lower-refractive-index substrate. The evanescent field in rib WGs is generally confined within the high-index region of the WG. The field extends into the cladding but is typically more localized compared to other types of WGs. A ridge WG has a raised section (ridge) on the WG core, which helps confining and guiding light. Like rib WGs, the evanescent field in ridge WGs is primarily localized within the high-index region.^[307] Whereas slot WGs consist of a low-index region (slot) between two high-index regions, these WGs are designed to have a substantial part of the optical field concentrated in the low-index slot region, making them advantageous for sensing.^[308] While rib and ridge WGs primarily con-

fine the evanescent field within the high-index regions, slot WGs intentionally utilise a low-index slot to enhance the interaction between light and the surrounding medium. The choice of WG depends on the application's specific requirements, such as the desired level of confinement, interaction strength, and the nature of the surrounding medium.

Another important consideration in the design of the EW sensors is the choice of the material platform. Silicon-on-insulator (SOI), silicon nitride (Si₃N₄), and polymer materials are the main available technologies, each offering unique advantages and tailored properties to meet specific sensing requirements.^[307]

Jin et al.^[309] demonstrated evanescent-field detection of ethanol at the C–H absorption band using a single-mode chalcogenide waveguide. This work demonstrates the principle of selective evanescent-field detection in the gas phase; however, the microfabrication process is complex, and single-mode operation limits the spectral range of the measurement. Later, the same group demonstrated chip-scale chemical sensors using optical waveguides consisting of amorphous silicon (a-Si) and aluminum nitride (AlN). Label-free chemical identification and real-time monitoring of heptane and methanol were demonstrated experimentally by scanning the mode spectrum.^[310] Gutierrez-Arroyo et al.^[311] designed and developed a selenide-integrated platform working in the mid-infrared, where Ge–Sb–Se multilayered structures were deposited by RF magnetron sputtering. Limits of detection of 14.2 and 1.6 ppm for methane and nitrous oxide, respectively, were experimentally demonstrated. Kumari et al.^[312] reported a silicon-on-nitride (SON) slot waveguide for methane detection in the atmosphere with a limit of detection down to 5 ppm.

In summary, EW VOC sensors offer the advantages of relatively low cost, compact size, high sensitivity, simple design, and remote and continuous monitoring capabilities. Various fiber and waveguide geometries and functional coatings have been developed to enhance the sensors' sensitivity and selectivity toward a wide range of VOCs, allowing them to achieve detection limits in the ppb order or better. Nevertheless, several main challenges still exist in the field that limit the practical applications of the EW sensors in VOC detection. Among such challenges is lowering the costs for sensor read-out systems, since currently they are relatively expensive, comprising an optical source and a photodetector. Another challenge is tackling sensor contamination issues, limiting their lifespan. Furthermore, since in practice, any VOC sensor is exposed to a mixture of gases and other environmental factors (e.g., temperature and relative humidity fluctuations), addressing the cross-sensitivity issues becomes a key factor, resulting in the need for both a high degree of selectivity of the individual sensors and high multiplexing capability to provide reliable information.

5. Optical Transducers Utilizing Absorption of Light

5.1. Confinement of Luminescent Guest by Nanoporous Host

The concept of “LG@MOF” concerns the confinement of a luminescent guest (fluorescent dye or fluorophore) incarcerated within a porous host, comprising a vast range of metal-organic

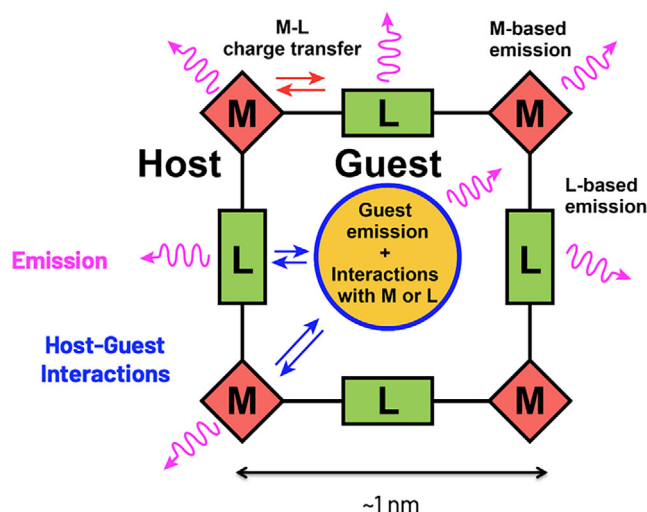


Figure 27. Luminescent guest confined in the MOF host, where metal clusters (M: red squares) are linked by organic linkers (L: green rectangles) with an incorporated guest (orange circle). Plausible emission pathways include metal-based emission, linker-based emission, ligand–metal charge transfer (LMCT), and varied energy transfer routes between the host and the nanoconfined luminescent guest.

framework (MOF) structures.^[313] The varied structural and physical properties of MOFs coupled with their rich chemical affinities (in terms of polarizability, hydrophobicity, or hydrophilicity) offer a platform for constructing photoluminescent sensors with high sensitivity and high selectivity.^[314] There are multiple energy and charge transfer pathways that might be accessible in the LG@MOF composite system, as depicted in **Figure 27**.

Of note, the resultant LG@MOF composite system exhibits strong host-guest interactions (**Figure 27**), which give rise to tunable luminescence and sensing behavior. LG@MOFs are excitable by absorption of ultraviolet (UV) lights, and upon exposure to VOCs, toxic solvents, and gases (VTGs) the distinctive changes observed in spectra (in emission intensity and/or frequency shift) can be used to track the presence of different analytes and their concentrations. The porosity as well as the crystallinity of the MOF host allow nanoscale partitioning of the luminescent dye guest molecules, thereby solving the aggregation-caused quenching (ACQ) problem. This is significant because commercially available fluorophores are susceptible to ACQ and thus become non-emissive in the solid state. But nanoconfinement by LG@MOF yields composites that remain highly emissive in the solid state, suitable for optoelectronic device applications. For example, the confinement of fluorescein dye in ZIF-8 (host) yields the Fluo@ZIF-8 composite (incorporating 0.02 to 10 wt.% guest) that exhibits a high luminescent quantum yield (QY) of $\approx 98\%$ in the solid state (see **Figure 28**).^[315] Likewise, confinement of rhodamine B (RhB) in ZIF-8 produces RhB@ZIF-8, where the QY was found to be extremely high at $\approx 99\%$ in solid state (vs RhB solution $\approx 60\%$) when the lowest guest concentration of ≈ 0.5 wt.% was used.^[316] Remarkably, the photostability of LG@MOF systems is notably improved thanks to the protection provided by the MOF host by shielding the encapsulated guest fluorophores from rapid photochemical deterioration under exposure to UV and/or humidity.^[317]

The research on LG@MOF based optical devices is very much in its infancy, with only pilot studies being reported, demonstrating the efficacy of luminescent MOFs in proof-of-concept devices for the detection of VOCs, anions, and cations, pH sensing, and solid-state lighting. Encouragingly, it is generally straightforward to synthesize the LG@MOF systems, enabled by one-pot reaction performed under ambient conditions. For example, rapid synthesis of ZnQ@OX-1 [ZnQ = zinc(II) bis(8-hydroxyquinoline)] by employing the high concentration reaction (HCR) strategy leads to the formation of a gel material.^[318] Subsequently, crystalline nanosheets exhibiting tunable fluorescence (both emission intensity and wavelength) are obtained, which are shown to be responsive to VOC sensing of alcohols (methanol, ethanol, isopropanol), *N,N*-dimethyl formamide (DMF), dimethyl acetamide (DMA), dioxane, and acetonitrile (see **Figure 29**). Furthermore, it was demonstrated that a paper sensor device containing ZnQ@OX-1 reveals fast reversible colour change when exposed to acetone vapor, where a red shift of 30 nm was observed on acetone adsorption followed by a blue shift on desorption. The mechanism and detection threshold of the acetone sensor was systematically characterized by in situ studies via high-resolution spectroscopic techniques based on synchrotron-radiation FTIR radiation, inelastic neutron scattering, and nearfield infrared nanospectroscopy.^[319] This is a “turn-off” type sensing mechanism in which the emission intensity declines upon VOC uptake. Importantly, only confined ZnQ guest within the OX-1 host can display intense luminescence and acetone vapour sensing response due to host-guest interactions conferred by LG@MOF. In contrast, unconfined ZnQ adhered on the surface of OX-1 surface showed only a weak luminescence and no VOC sensing response.

Understanding the precise configuration and number of guest molecules confined within the LG@MOF system has implications for the mechanistic design of improved photoluminescent sensors and photonic devices. By way of an example, for an anthracene (ANT) encapsulated system employing a ZIF-8 host, termed ANT@ZIF-8,^[320] it was elucidated that four ANT molecules (as monomers) could be accommodated in the sodalite cage of ZIF-8. Upon UV irradiation for 30 min of a photopatterned device, a detailed mechanism was proposed whereby half of the ANT monomers would photodimerize to yield an intense switching of emission colour from 550 nm (yellow) to 415 nm (violet). This phenomenon is reversible, employing an extended thermal treatment (120 °C for 2 days), dissociating the dimeric guests via an excimer-to-monomer photoswitching process that may be facilitated by the ZIF-8 host. For yet another large-pore ZIF host, termed ZIF-71 [Zn(dclm)₂; dclm = 4,5-dichloroimidazole] featuring the RHO topology, the guest aggregation effect pertaining to rhodamine B (RhB) was studied to reveal guest configuration and host-guest interactions for fluorescence VOC sensing.^[321] RhB@ZIF-71 can afford a maximum of two RhB molecules per ZIF-71 cage (pore diameter ≈ 2 nm), i.e., as a RhB dimer, because of the π -conjugated planar structure of RhB. With varying guest concentration introduced during synthesis step, it was proposed that the H-aggregates, J-aggregates, or monomers could form within the RhB@ZIF-71 composite. Under pressure, it exhibits a mechanochromic sensing response, the H-aggregate transforms into J-aggregate through shear deformation of the

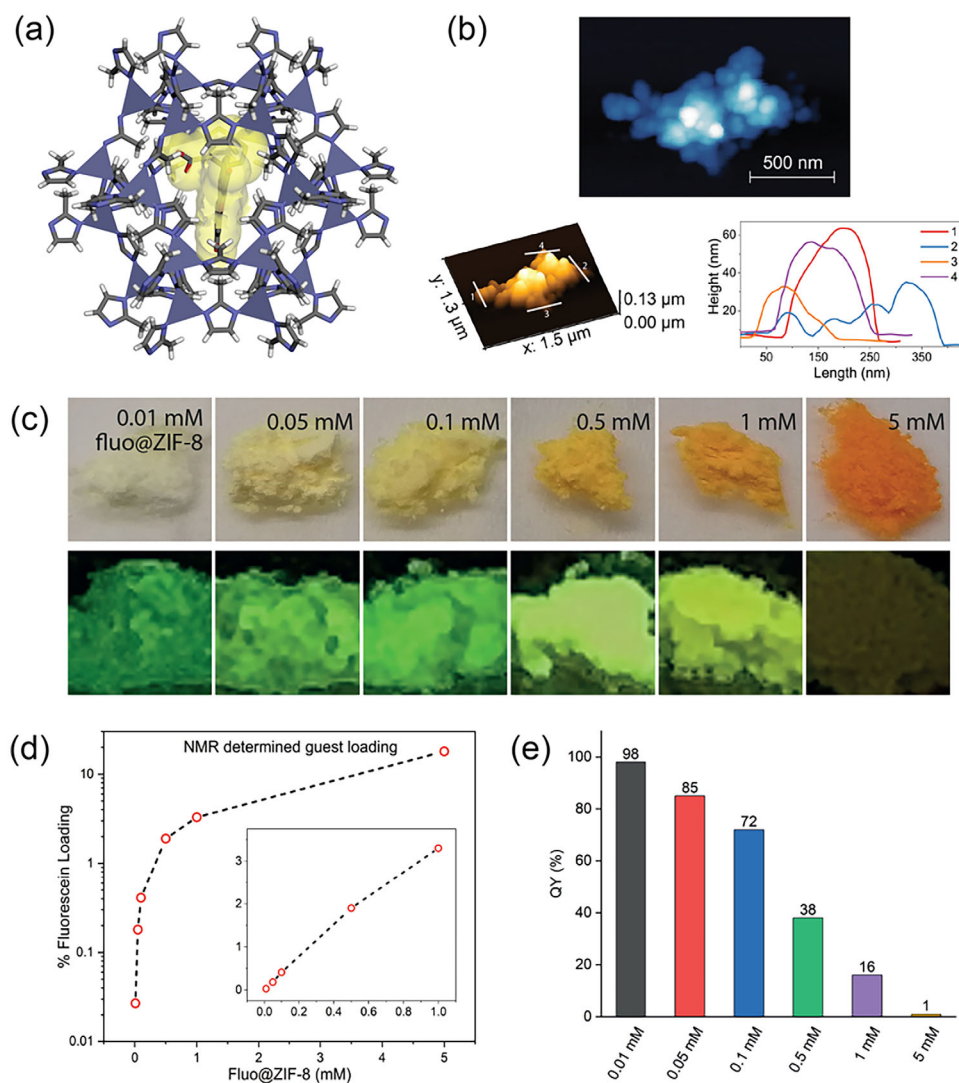


Figure 28. a) Schematic representation of Fluo@ZIF-8. Purple tetrahedra are Zn_4N_4 , and color codes of atoms are red: oxygen, gray: carbon, blue: nitrogen, and white: hydrogen. b) Nanocrystals of Fluo@ZIF-8 characterized by atomic force microscopy. c) Samples with different fluorescein guest loading observed under the ambient light (top row) and fluorescence emission when excited by a 365 nm UV lamp (bottom). d) Fluorescein guest concentration characterized by ^1H nuclear magnetic resonance. e) Luminescent quantum yield declining with increasing guest loading concentration in MOF host corresponding to (d). Reproduced with permission.^[315] Copyright 2021, ACS.

ZIF-71 cages, giving a red-shifted emission as a function of the applied stress. Likewise, the solvatochromic sensing response of RhB@ZIF-71 can be attributed to the stronger caging effect of the J-aggregates in ZIF-71 host (Figure 30), providing VOC selectivity for the polar protic, polar aprotic, and non-polar solvents that was previously intractable employing the pure RhB alone.

Interestingly, LG@MOF systems enable the design of self-calibrating ratiometric optical sensors. Ratiometric sensors possess dual-emissive properties, from which the differential emission intensity of the two distinctive peaks can be leveraged to pinpoint specific analytes or to correct for unwanted external interferences. For example, the Rh6G@MOF system^[322] exhibits two distinct emission peaks, the first peak (363 nm) originates from its tpt (2,4,6-tri(4-pyridyl)-1,3,5-triazine) linker, and the sec-

ond (580 nm) comes from the rhodamine 6G guest. It can be seen in Figure 31 that when the Rh6G@MOF was subjected to a nitroaromatic explosive termed TNP (2,4,6-trinitrophenol) from 0 to 200 ppm concentration, the luminescence due to the linker at 363 nm was strongly quenched, whereas the emission intensity recorded at 580 nm from the encapsulated Rh6G guest was negligibly affected. Subsequently, by establishing the intensity ratios of these two peaks the ppm concentration of TNP can be determined. Because of the specific interaction between the MOF host and the acidic TNP molecule, this ratiometric material exhibits unique selectivity towards sensing of TNP, evaluated against a range of nitroaromatic and non-nitroaromatic analytes. The concept of ratiometric fluorescent sensors is promising, for which more exploration is warranted to understand the design of specific LG@MOF for targeting selected analytes, and to uncover the

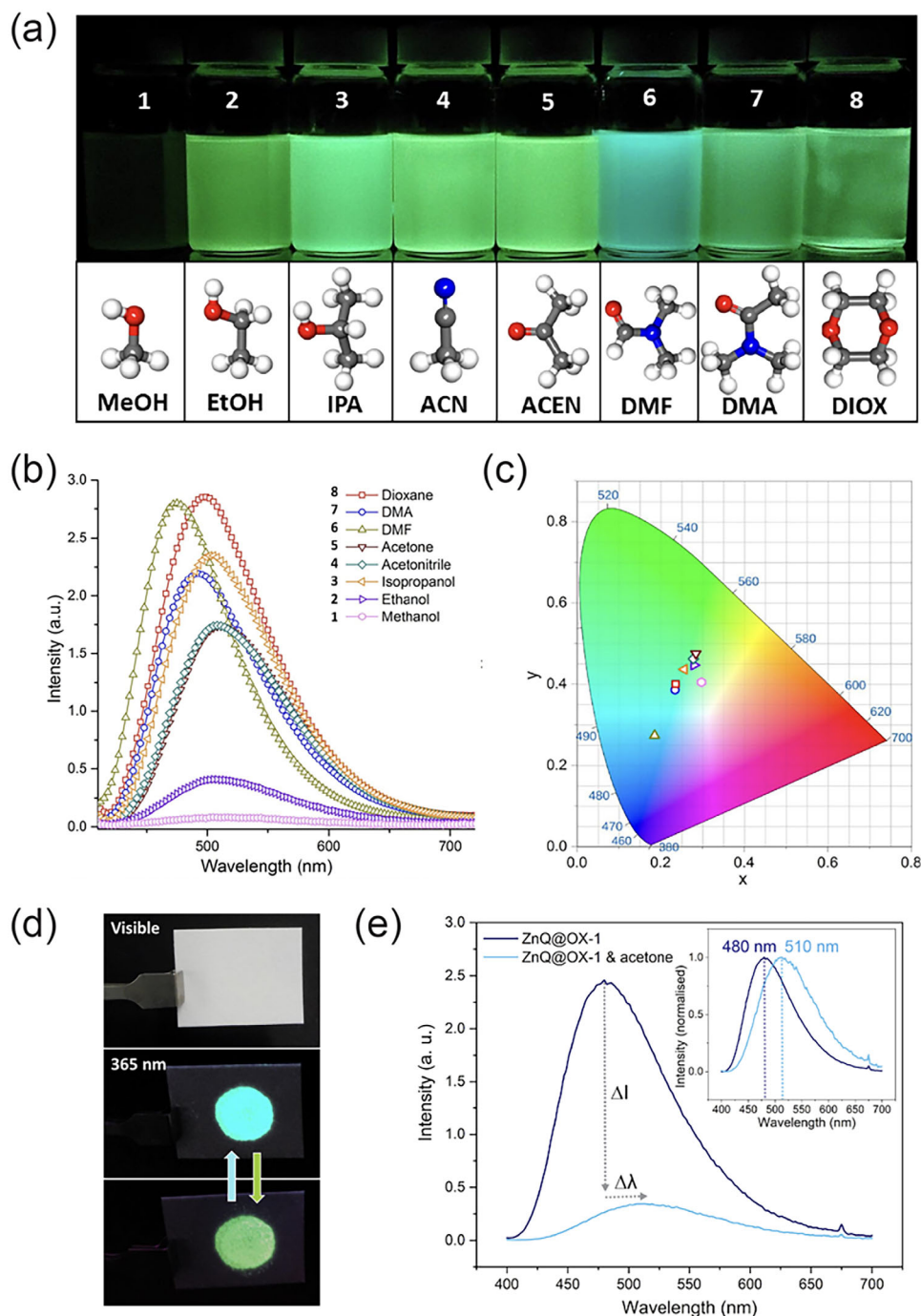


Figure 29. a) Photochemical sensing behavior of ZnQ@OX-1 nanosheets in VOCs, where methanol quenches while other solvents result in changes of emission intensity and wavelength. b) The corresponding emission spectra and c) CIE chromaticity subject to different solvents. d) Acetone vapor sensing using a paper device coated with ZnQ@OX-1, giving a spontaneous red shift in emission wavelength, which is reversible upon acetone desorption. The excitation source is a 365 nm UV lamp. Reproduced with permission.^[318] Copyright 2017, Wiley. e) Emission spectrum showing quenching effect in conjunction with a 30-nm frequency shift when exposed to acetone vapor. Reproduced with permission.^[319] Copyright 2023, Wiley.

underlying host–guest mechanisms. Particularly, fluorescent turn-on and ratiometric optical chemical sensors are still uncommon in the literature. For instance, a lanthanide $\text{Eu}^{3+}/\text{Cu}^{2+}$ @UiO-66-(COOH)₂ was demonstrated for selective sensing of hydrogen sulfide (H_2S) compared with various analyte

solutions (5 mM concentration) after 30 s exposure. The mechanism was attributed to the antenna effect that controls energy transfer between the MOF linker and the $\text{Eu}^{3+}/\text{Cu}^{2+}$ cations in the absence or presence of sulfide.^[323] A turn-on type BTX sensor for the optical detection of benzene, toluene, and xylene was

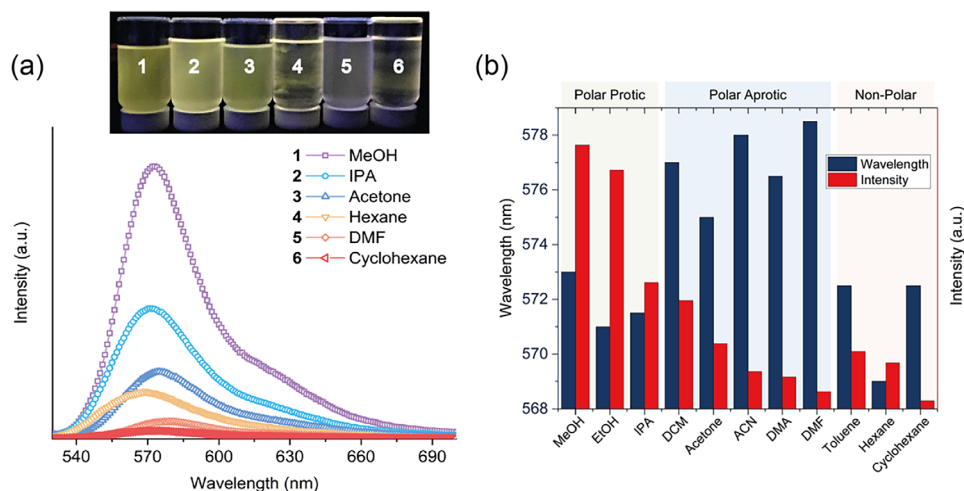


Figure 30. a) Fluorochromic sensing of VOCs using RhB@ZIF-71 nanocrystals suspended in a range of solvents. b) Differential changes observed in emission wavelength (left) and emission intensity (right) of RhB@ZIF-71 subject to the polar protic, polar aprotic, and non-polar analytes. Reproduced with permission.^[321] Copyright 2020, ACS.

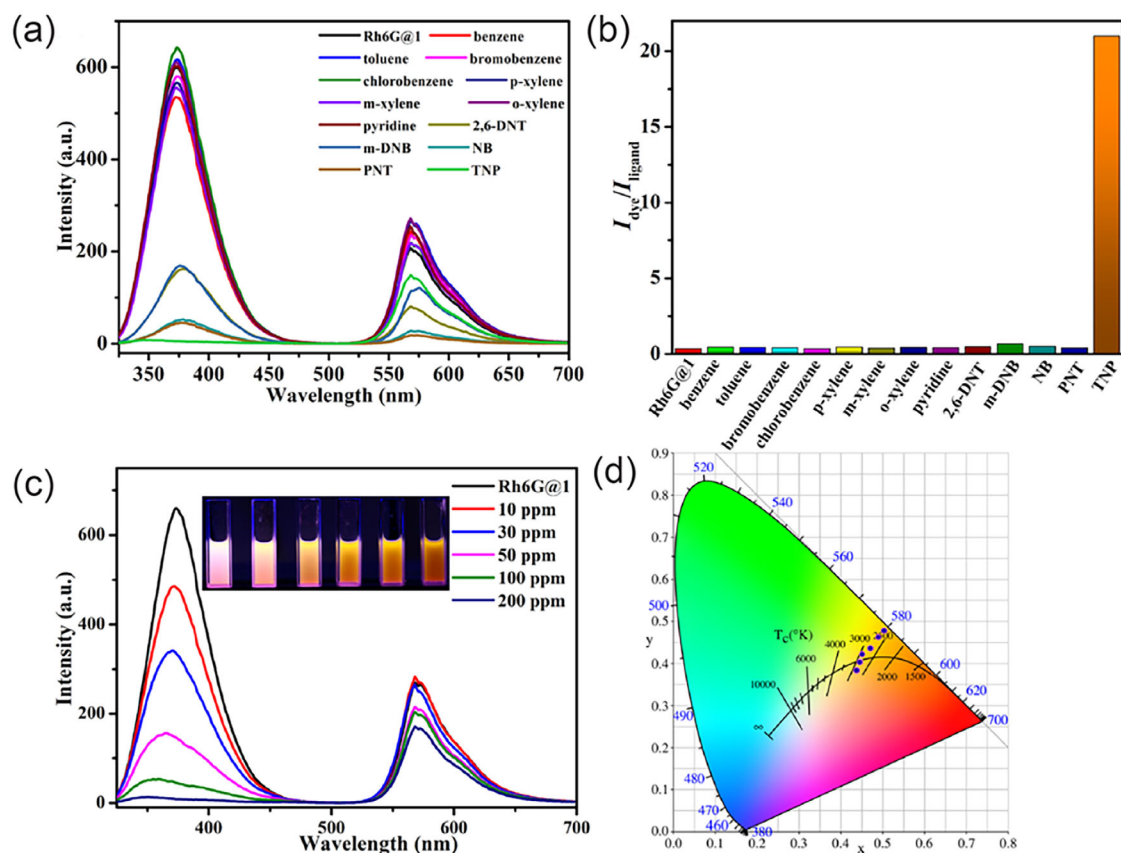


Figure 31. a) Ratiometric optical sensing of Rh6G@MOF when exposed to a variety of analytes each at a concentration of 200 ppm. b) Relative peak intensity (580 nm:363 nm) showing the high selectivity for TNP analyte. c) Ratiometric response of Rh6G@MOF tested in different concentrations of TNP, and d) the corresponding CIE chromaticity chart for the different concentrations of TNP analyte. Reproduced with permission.^[322] Copyright 2017, ACS.

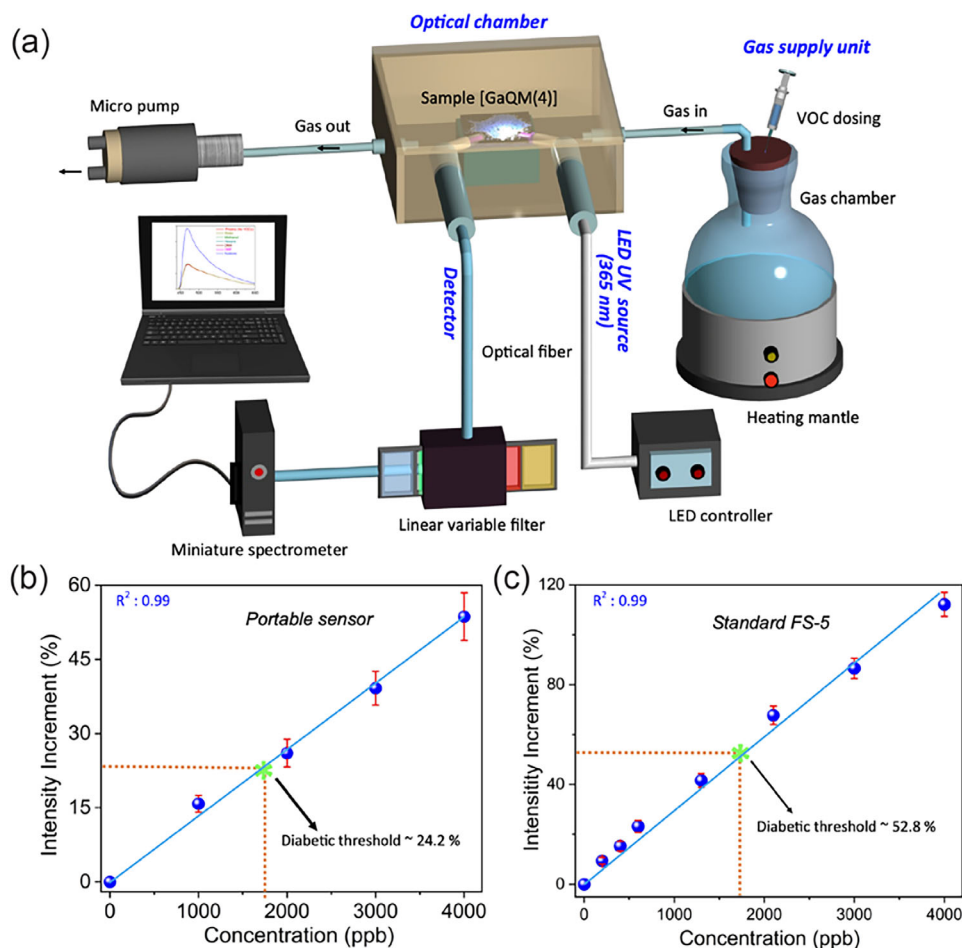


Figure 32. a) Portable optical sensor for acetone vapor with sub ppm-level sensitivity, employing a 365-nm UV excitation optical fiber. b) Calibration curve obtained from the GaQ@MAF-5 powder sample within the diabetic detection range for a breathalyzer application, compared with c) the output determined from a standard (not portable) benchtop spectrofluorometer. Reproduced with permission.^[325] Copyright 2023, Wiley.

recently realized employing a perylene@MIL-68(In) system, where the mechanism was attributed to self-trapped exciton states of the dimeric guest excimers nanoconfined in the one-dimensional channel of the MOF host.^[324] Upon exposure to BTX, the luminescent quantum yield of perylene@MIL-68(In) increased from its initial QY value of 14% (pristine powder), to $\approx 59\%$, $\approx 66\%$, and $\approx 75\%$, respectively, when sensing benzene, toluene, and xylene analytes.

Another recent exemplar concerns the GaQ@MAF-5 [GaQ = gallium(III) tris(8-hydroxyquinolato) metal complex] based acetone vapour sensors with a turn-on fluorescence response for sub-ppm level photochemical sensing.^[325] The reported minimum detection limit is 200 ppb, which places it well within the range for application in noninvasive diabetes diagnosis of exhaled human breath. Combined with electrospinning, it was found that PVDF fibers integrating the GaQ@MAF-5 crystals exhibit enhanced QY and emission intensity upon acetone vapour exposure at ≈ 2 – 3 ppm concentration level, and the device was reusable and tested above 60 cycles. Of note is the chemical selectivity of GaQ@MAF-5 toward acetone vapor in a complex mixture containing water vapor, alcohol vapor, and common gases such as nitrogen, oxygen, and carbon dioxide prevalent in ambient condi-

tions. A proof-of-concept portable sensor device was constructed to demonstrate the feasibility for practical application, as depicted in Figure 32.

5.2. Solid-State Photochromism via Photoswitchable Guest

The colour or chromaticity of a photochromic material (termed photochrome) can be modulated through light stimulation, this phenomenon involves the photoisomerization process, which is reversible on light removal.^[326] Common photochromes include spiropyran, diarylethenes, fulgides, viologens, azo and imide molecules, and donor-acceptor Stenhouse adducts.^[327] Such organic photochromic molecules have limited long-term stability, thereby hampering their practical use in aggressive settings such as VOC sensing.^[328] To this end, the encapsulation of photochromes within porous solids (e.g., MOFs, COFs, MOP cages) provides an effective pathway to improve the photofatigue resistance and photoswitching efficiency (reducing switching time) of the resultant composite systems in the solid state. The photostability and mechanical durability of the photochromic films may

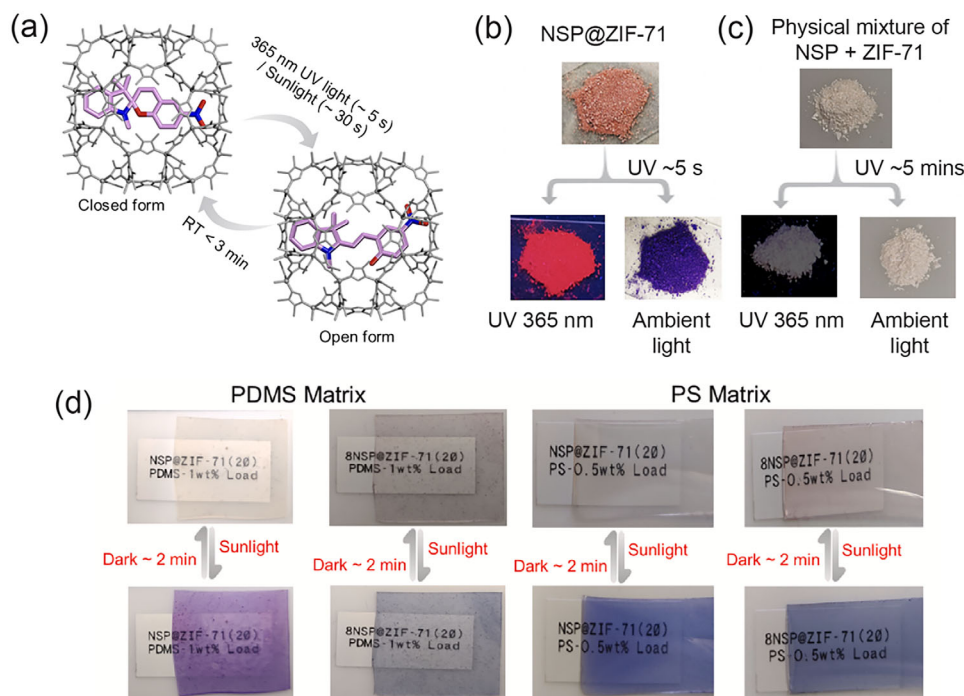


Figure 33. a) Schematic of the photoswitching mechanism of NSP@ZIF-71 through the open form (highly polar merocyanine) $\rightleftharpoons$ closed ring (non-polar) forms of NSP, under 365-nm UV irradiation. b) Photochromic switching in the solid state for NSP@ZIF-71 composite system. c) Controlled sample showing no switching for a physically combined mixture of NSP + ZIF-71. d) Thin-film membranes of NSP@ZIF-71/PDMS and NSP@ZIF-71/PS subject to 365-nm UV lamp or natural sunlight. Reproduced with permission.^[333] Copyright 2023, Elsevier.

be further enhanced by incorporation of the caged photochromes into a suitable polymer matrix.

Solid-state photoswitching materials can be constructed via an intrinsic approach, in which photochromic molecules function either as the organic bridging ligands or as a side group appended to the MOF linker. For example, a naphthalene diimide (NDI)-based ligand was used to yield a 2D MOF structure through solvothermal synthesis. A thin film of this NDI-based MOF deposited on a fluorine-doped tin oxide (FTO)-coated glass substrate was demonstrated to exhibit photochromic and electrochromic properties. It is redox active, reversibly switching between a colourless and yellow state.^[329] Likewise, 3-D MOF structures comprising NDI linkers have been reported, for which these photochromic MOFs will switch their colour upon sunlight irradiation.^[330] The second approach is by guest encapsulation or nanoconfinement of photochromes (e.g., azobenzenes, spiropyrans, diarylethenes) within a porous MOF host, which is akin to the LG@MOF strategy described in Section 5.1. A vast range of photochrome@MOF compounds that are photoswitchable can be achieved; the resultant composite materials display a solution-like photochromic response from nanoscale partitioning of photochromic molecules, which are spatially isolated by the crystalline MOF pores.^[331] Exposed to VOC analytes, spiropyran@MOFs display solvent-specific color switching.^[332]

Crucially, nanoscale pores in the MOF host offer a tunable chemical environment to facilitate reversible structural switching of an encapsulated photochromic guest, upon UV light irradiation. For instance, this effect has been clearly demonstrated in Figure 33a,b for a spiropyran composite, termed NSP@ZIF-

71,^[333] showing rapid transformation between the closed and open forms of NSP guest within the RHO cage; this was not evidenced for a physical mixture (combined powders) comprising simply of NSP and ZIF-71 (Figure 33c). Moreover, composite films prepared in different polymer matrices demonstrate differential colors and switching characteristics. Design guidelines for selecting the optimal nanoporous MOF host for optimal solid-state photochromism include: (i) pore size of the host compatible with the size of the photoswitchable guest; (ii) surface hydrophobicity of the MOF cages; and (iii) electron deficiency of the host surface. Recent studies employing a series of MOF hosts suggest that it is preferred to adopt a MOF host with an intermediate pore size relative to the guest molecule, combined with an intermediate hydrophobicity and non-electron-deficient host surface.^[333]

5.3. Aggregation-Induced Emission (AIE) for Multistimuli Optical Sensors

A major limitation of conventional luminescent materials is that they quench in the solid state, i.e., becoming less emissive by non-radiative decay, because of ACQ (Figure 34a).^[334] As opposed to the quenching phenomenon, materials exhibiting aggregation-induced emission (AIE) will emit more intensely under a highly concentrated state. Such AIE materials are termed AIE luminogens or AIEgens, encompassing tetraphenylethylene (TPE) and triphenylamine (TPA) molecules depicted in Figure 34b, and their associated derivatives. In the aggregated state, the enhanced radiative decay of AIEgens can be attributed

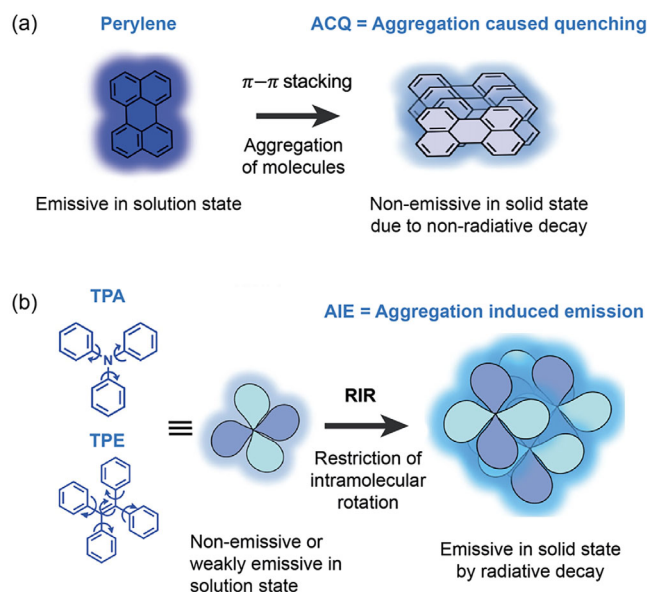


Figure 34. Molecular mechanisms to explain the phenomena associated with a) aggregation-caused quenching (ACQ), and b) aggregation-induced emission (AIE). Reproduced with permission.^[338] Copyright 2023, Wiley.

to restricted intramolecular motion of the rotational phenyl rings of the adjacent molecules.^[335] This unique phenomenon enables the turn-on type AIE sensors, whose emission intensity will increase when subjected to a physical or chemical stimulus. When combined with electrospinning, the fabrication of AIE-based polymer fibers^[336] and porous membranes with a high surface area extends the practical applicability of AIE materials to VOC fluorescent sensing,^[337] solid-state white light emission, mechanoluminescence, and smart textiles for chemosensors.^[338]

Integration with MOFs, either as a ligand in the extended hybrid framework (AIE-MOF) or as an encapsulated guest in porous host (AIE@MOF), could yield novel composites with unconventional optical properties for turn-on type sensors. Indeed, this is a relatively new but promising topic of research in the MOF field, thus it deserves more attention in light of the exciting recent discoveries and opportunities elucidated below. Hitherto, AIEgens have been employed as an organic linker in the synthesis of several AIE-based MOF structures.^[339–341] Nevertheless, the resultant materials typically have a low fluorescence QY and do not exhibit the turn-on sensing behavior. For example, the coordination of tetrakis(4-carboxyphenyl)ethylene (TCPE, an AIEgen) with Zn^{2+} or Cd^{2+} ions yielded AIE-immobilized Zn- and Cd-MOF structures.^[342] These AIE-MOFs are fluorescent and show some selectivity for optical sensing of solvent analytes, such as ethylenediamine, cyclohexanone, and acetaldehyde. The ability to distinguish different guest solvent molecules as evidenced through a red or blue shift in emission spectra can be explained by host-guest interactions that impede intramolecular rotations of the AIE linkers (Figure 34b). The reported QY values of the foregoing AIE-MOF compounds, however, are relatively low at only 1–2% ascribed to the lack of strong molecular restriction imposed by the MOF framework. Therefore, it is interesting to note that the immobilization of TCPE-based AIE linkers in another Zn-based MOF^[343] and Zr-based MOF^[344] has been reported to

double the QY of the AIE-MOF compounds, compared with that of the isolated ligands (typical QY $\approx$ 20–30%). While this major improvement may be attributed to the reduction in non-radiative decay of the immobilized AIEgens within the MOF structure, the precise mechanism remains to be established, and it is likely to be framework dependent.

Yet another challenge of AIE-MOF lies in the complex synthesis steps to afford grafting of AIEgens onto common organic ligands, which later serve as a building block for constructing AIE-responsive MOFs. Promising results could be achieved such as that illustrated in **Figure 35**, if the aforementioned obstacle can be overcome. In this instance, by grafting TPE onto NUS-13 MOF, this approach enables chemical sensing with selectivity for a series of VOC solutions, encompassing mesitylene, xylene, toluene, benzene, hexane, and nitrobenzene.^[339] Notably, by subjecting the resultant NUS-13-TPE compound to a rising concentration of benzene vapour at room temperature, a turn-on fluorescence response was recorded from 0 to 600 ppm, revealing a linear correlation of emission intensity with VOC concentration (Figure 35c,d). In terms of mechanism, it is anticipated that the synergistic effect conferred by grafting TPE molecular rotors on ligand strut positioned in the rigid MOF channels allow high sensitivity detection of VOC at ppm levels. Again, the turn-on response of NUS-13-TPE is attributed to the hindrance of the motion of TPE linkers upon guest adsorption, thereby boosting its radiative decay.

Turning to the AIE@MOF strategy, this pathway involves the nanoconfinement of AIEgens within the pores of a MOF host for engineering an LG@MOF system with turn-on luminescence. A recent example concerns the encapsulation of TPE (as AIE guest) molecules in the pores of ZIF-71 with a RHO topology, whereby the close proximity of nanoconfined TPE bestows stimuli-responsive fluorescence absent in the isolated TPE molecules.^[345] The choice of ZIF-71 as a host was based on the fact that it affords encapsulation of a TPE monomer, which is comparable in its dimension to the pore size of ZIF-71 (16.5 Å). When subject to pressure, the externally applied stress deforms and collapses the RHO cages of ZIF-71, allowing an enhanced $\pi-\pi$ interaction between adjacent TPE dimers. Such a mechanism generates a turn-on type red-shifted fluorescence as a function of pressure, observed in the lower stress regime of 10s to 100s MPa, see **Figure 36a,b**. This is a significant finding, because pristine (unconfined) TPE molecules could only demonstrate a weak turn-off type sensing even under a substantially high pressure of 1.5 GPa.^[345,346] However, the caging effect from ZIF-71 hinders the unrestricted intramolecular rotation of TPE molecules, thereby resulting in mechanofluorochromism at a relatively lower stress state. This pressure-responsive behavior may be coupled with the VOC chemical detection character of TPE described above, for devising a multistimuli chemo-mechano sensor.

Towards practical applications, processing of AIE-MOF or AIE@MOF in combination with technical-grade polymers such as polyurethane (PU) or polyvinyl difluoride (PVDF) yields a resilient composite membrane (Figure 36c) for fabricating robust sensors that will withstand real-world conditions.^[345] Here, the challenge lies in retaining the high sensitivity and selectivity of the powder sensor materials after introduction of a polymer matrix, while at the same time enhancing the mechanical

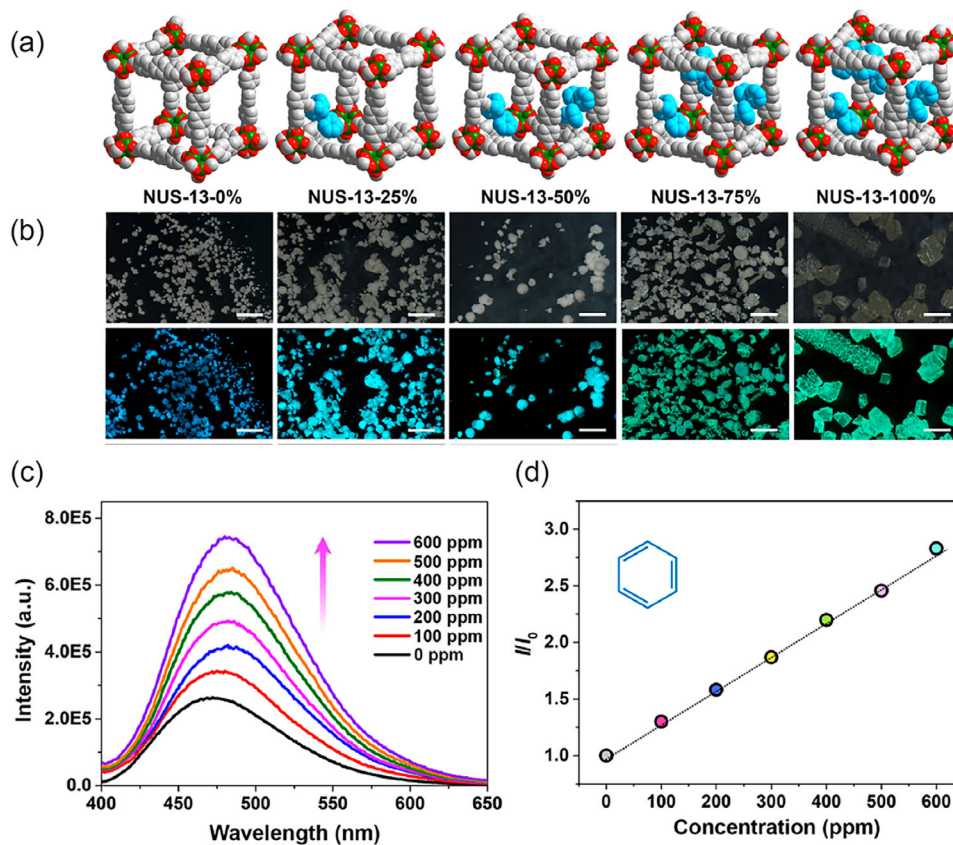


Figure 35. a) AIE-MOFs based on NUS-13 (host) incorporating different amounts of TPE (AIE guest) grafted on the 4,4'-terphenyldicarboxylate linkers acting as the molecular struts. b) Optical (top) and fluorescent (bottom) microscopy images of the NUS-13-TPE crystals; scale bars = 500 μm. c) Emission spectra of NUS-13-TPE ($\lambda_{\text{ex}} = 360$ nm) subjected to different ppm-level concentrations of benzene vapour at room temperature, and d) relative emission intensity (I/I_0) as a function of benzene concentration. Reproduced with permission.^[339] Copyright 2020, ACS.

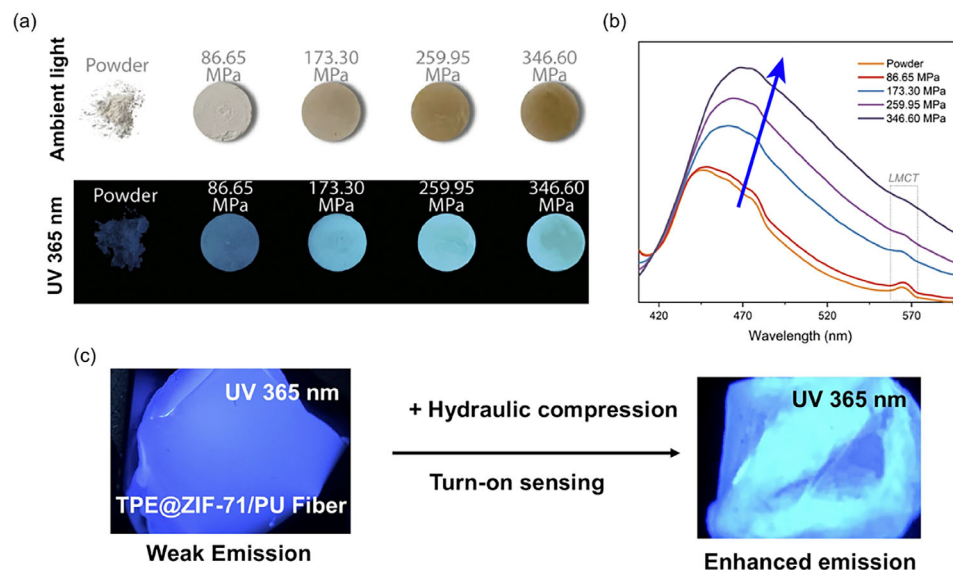


Figure 36. a) Pressed pellets of TPE@ZIF-71 viewed under the ambient light (top row) and when excited under a 365-nm UV light (bottom). b) Higher emission intensity obtained with increasing pressure as indicative of the turn-on type mechanofluorochromic response. c) Electrospun membrane of TPE@ZIF-71/polyurethane (PU) fibers inspected under UV excitation before and after being compressed under a hydraulic press. Reproduced with permission.^[345] Copyright 2022, Elsevier.

resilience for device handling and durability for long-term performance. In addition to nanocomposite membranes, electrospinning of fluorescent fibers and thin films augmented with conventional AIEgens and donor–acceptor type systems offer high surface area per unit volume that may be exploited for multistimuli sensing, photothermal, and photodynamic applications in the solid state.^[338] An interesting exemplar involves the study of luminescent textiles incorporating TPE for acetone vapour detection, where a turn-off response was reported through halving of its emission intensity upon acetone exposure. This phenomenon was ascribed to acetone boosting the rotational motion of TPE molecules, thus increasing the non-radiative decay.^[347] A detailed study to investigate the effect of confined TPE in MOFs for textiles, either as AIE-MOF or AIE@MOF, is warranted to understand the pathway to accomplish turn-on response, important for reaching a higher signal-to-noise ratio (SNR). Particularly, this is relevant to the future development of high-sensitivity explosive vapour sensors incorporating AIE molecules. Another example demonstrates the feasibility of employing porous films of varying morphologies, fabricated via electrospinning, drop-casting, and spin coating for optical sensing of DNT (2,4-dinitrotoluene) vapours; albeit giving a turn-off type response whereby 90% of emission intensity was quenched on DNT exposure.^[348] Tuning of AIE emission for a specific target VOC at a defined detection threshold will be relevant for future research.

6. Optical Transducers Utilizing Optically Anisotropic Materials and Structures

A promising approach to VOC detection is based on transducers that facilitate the detection of the change of state of polarization of a probe beam propagating through optically anisotropic material or structure that can change their properties in the presence of the target analyte (VOCs).^[143] One of the early examples demonstrating the potential of this approach was reported in 1999.^[349] The authors utilized a nematic liquid crystal between two polarisers to detect meta-xylene and para-xylene. The measurements revealed that the birefringence and the phase transition of a liquid crystal can be exploited in VOC sensing.

The following section reviews more recent successful examples of this approach utilizing polarized light optical microscopy (POM).

6.1. Transducers Utilizing Optically Anisotropic Materials

The potential of Liquid Crystals (LC)-based transducers for VOC detection has been extensively investigated. These materials are characterized by significant orientational order and, in some cases, positional order, which are considered fundamental requirements for self-organization and formation of hierarchical structures. It has been previously demonstrated that LC respond readily to external stimuli, such as electrical and magnetic fields, light, mechanical shear, pressure, temperature, and chemical analytes with a change in their orientation and hierarchical structure.^[143,350–352] Their sensitivity to chemical analytes has been explored in the development of LC-based gas sensing technologies, by tailoring the LC-sensing materials to respond to

targeted VOCs. It has been observed that certain VOCs can diffuse or dissolve into liquid crystal materials and can change the LC order parameter, director orientation, and/or phase properties. This leads to changes in their optical properties, which can be observed macroscopically.

A major advantage of the LC-based sensing materials is that they operate at ambient temperature^[350] and have been proven to exhibit high selectivity at low concentrations of the analytes.^[350] LC-based sensing materials have the potential to be used in portable,^[353] low-cost gas sensing devices for in-situ, real-time detection of VOCs since this approach does not require the use of expensive and complex instrumentation.

There is a substantial research effort in understanding and exploring the applicability of different LC-based systems (nematic, smectic, chiral, columnar phases) in gas sensing.^[143]

6.1.1. Detection Based on VOCs Interaction with LC Supported on a Chemically Functionalized Surface

In surface-based LC devices, thin LC film is supported on a functionalized surface, which assists the LC orientation and hierarchical order. The principal mechanism responsible for the change in the LC molecular order is the competition between the gas analyte and LC molecules for the same binding sites on the substrate, as presented by the example in **Figure 37**. The LC reorientation is typically monitored by measuring the intensity of polarized light transmitted through the LC system. The binding and adsorption of specific gas analytes to the surface of a substrate can be controlled by the chemical functionalization^[354–379] of the substrate with specific chemical groups or by the surface topography.^[349,353,380–385] A comprehensive review of studies exploring either of these two mechanisms is included in.^[143]

6.1.2. Detection Based on VOCs Interaction with LC in Solid Surfaces with Pre-Defined Topography

A promising solution to the development of low-cost, durable, and allowing easy handling VOC sensors is proposed by a technology involving fiber production with liquid crystal contained within the fiber core. This approach is attractive, because the LC-functionalized fibers can be produced at low cost, following a simple one-step standard fiber production procedure that yields flexible non-woven responsive fiber mats. Different techniques have been utilized for fiber production—electrospinning, the core-sheath geometry ensured using a coaxial spinneret,^[386–390] in-situ phase separation,^[391] and airbrushing.^[392] A variety of LCs have been incorporated in the fibers, including non-chiral nematics,^[390,391] shortpitch cholesterics,^[387–389,393] and smectics.^[394] A convenient aspect is that the cylindrical polymer sheath, which is stretched during spinning, acts like an alignment layer on the LC,^[388,394] thus ensuring a ground state director orientation primarily along the fibers. Provided that an isotropic polymer is used to fabricate the fiber, the typically very thin sheath does not obscure the LC characteristics. Macroscopically, the LC determines the optical properties of the system and the fiber mat as responsive to VOC as the liquid crystal itself.^[386]

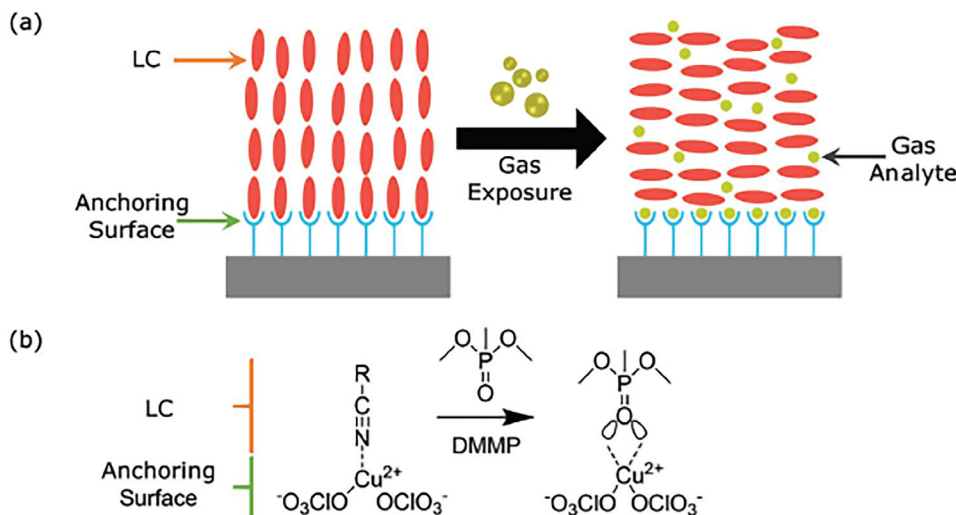


Figure 37. Orientational transition upon gas exposure for a liquid crystal supported on a chemically functionalized surface. a) Schematic illustration of the change in molecular ordering of a homeotropically aligned liquid crystal to a planar reorientation, upon gas exposure, caused by the competition between gas and liquid crystal molecules for the binding sites on the aligning substrate. b) Molecular interactions between copper (II) perchlorate (chemically modified substrate) and the nitrile group of 5CB and the imminent disruption by the phosphoryl group of the chemical analyte DMMP (dimethyl methyl phosphonate). Reproduced with permission.^[143] Copyright 2020, Wiley.

Following this approach, non-electronic gas sensors from electrospun mats of liquid crystal core fibers for detecting volatile organic compounds at room temperature are developed by Reyes et al.^[353] The authors study the optical response of coaxially electrospun fibers, with a poly(vinylpyrrolidone) (PVP) sheath surrounding a core of nematic-forming LC 4-cyano-4'-pentylbiphenyl (5CB), under exposure to toluene vapour. Both aligned and random fiber mats are investigated using POM and scanning electron microscopy (SEM), for their optical properties and fiber morphologies, respectively. The response is a function of fiber orientation, relative humidity of the spinning atmosphere, and LC-to-polymer solution flow rate ratio during spinning. It is demonstrated that the fiber mats retain their response for long periods (at least 1 month after fabrication). The response can be detected either by the naked eye due to a change in the microscopic appearance of the mat or with the use of crossed polarisers.

6.1.3. Detection of VOCs Based on Interaction with Doped Liquid Crystals

One approach providing an increased sensitivity of the LC-based VOC sensors is the use of doped LC. Most systems reported in the literature are nematic LC doped with molecules that can either enhance or induce an orientation in the LC,^[378,395–397] or, more interesting from the VOC detection point of view, interact with the gas analyte.^[398,399] An example of the case when the dopant interacts with butylamine vapour is demonstrated in.^[398] The LC, 5CB was doped with a range of concentrations of lauric aldehyde (from 0.01 to 4 wt%) and used to fill copper grids placed on a glass slide, thus forming two LC interfaces – LC/glass and LC/air. When exposed to butylamine vapour, a bright-to-dark optical response was observed due to an orientational transition of the LC,

triggered by a reaction between lauric aldehyde and butylamine (Figure 38). The proposed gas sensor appears to be reusable, and its reversibility is attributed to the hydrolysis of the imine in humid/wet environments.

6.1.4. Detection of VOCs Based on Interaction with LC Droplets

When compared with the solid surface-based sensing systems described in sections 6.1.1–6.1.3, interface-anchored or (bio)polymer-in-capsulated LC droplets exhibit a larger surface area, which can be advantageous in chemical sensors designed for higher sensitivity. When LCs assume a spherical geometry, the molecular mechanism of gas sensing (recognition of the target occurs through a competition between the analyte and either the surface of (droplet) deposition or any

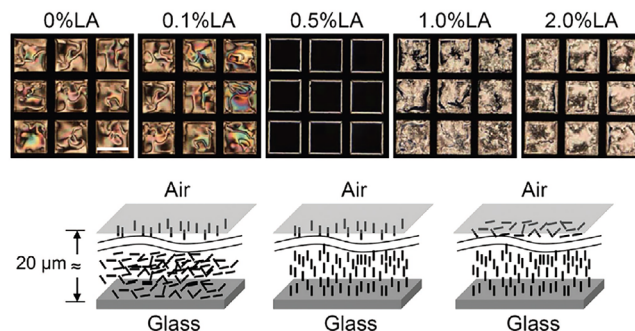


Figure 38. Representation of the director configuration of 5CB doped with lauric aldehyde (in a range of different concentrations), and confined in copper grids, at the LC/glass and LC/air interfaces. Corresponding polarized optical microscopy images were captured 2 h after sample preparation. Scale bar, 250 μm . Reproduced with permission.^[398] Copyright 2012, Elsevier.

components of the droplet itself.^[143] Both nematic^[400–403] and chiral LC droplets^[146,150,151,161,404] based sensors have been reported in the literature.

An example of the use of chiral liquid crystal droplets for VOC detection is reported in ref. [405]. Chiral liquid crystal formulations are confined as droplets by an interface formed by ionic liquids and further encapsulated within a gelatin matrix. It is demonstrated that the polydisperse self-assembled droplets have different topological architectures, which depend on their relative chirality parameter. The response of the samples to the presence of VOC analytes, such as heptane, toluene, chloroform, acetone, ethanol, diethyl ether was observed in real time through POM. It is reported that the occurring textural transformations are unique for each VOC tested. The CLC-based materials have a semi-selective response, a dynamic alternation between the chiral nematic and the isotropic phases when exposed to the analyte within seconds, as shown for two different liquid crystal mixtures in (Figure 39). It is proposed that the complexity and richness emerging from the observed morphological changes could be coupled with image recognition tools, for automated report and identification of gaseous analytes suitable as semi-selective sensors in arrays of gas sensors used in electronic nose devices.

In further development of this approach, a class of cooperative hybrid gels incorporating stable droplets of LCs self-assembled in the presence of room temperature ionic liquids with surfactant-like properties and biopolymers is reported in ref. [400]. The self-assembly properties result from the control of noncovalent intermolecular interactions and create structures within structures, where each component has a cooperative effect in structure and function. The authors provide proof-of-concept data (Figure 40) to show that hybrid gels can further enhance the performance LCs in gas sensing.

The approach to VOC detection described above has been used in conjunction with deep convolutional neural networks (CNN) as pattern recognition systems to analyse optical textures in LC droplets and their dynamics when exposed to a set of different VOCs. LC droplets' responses to VOCs were video recorded under POM. CNNs were then used to extract features from the data and, in separate tasks, to recognise and quantify the vapours to which the films were exposed. It is demonstrated that a single droplet can recognise 11 VOCs with small structural and functional differences (F1-score above 93%). The optical texture variation pattern of a droplet also reflects VOC concentration changes, as suggested by applying a regression model to acetone at 0.9–4.0% (v/v) (mean absolute errors below 0.25% (v/v)). The CNN-based methodology is thus a promising approach for VOC sensing using responses from individual LC-droplets.^[406]

6.2. Transducers Utilizing Optically Anisotropic Structures

6.2.1. Holographic Metasurfaces

A sensor platform utilizing the sensitivity and fast response of LC to VOC in combination with a holographic metasurface has been reported by Kim et al.^[407] The metasurface is designed to reproduce different holographic images according to the polarization of incident light and maximizes the polarization conversion efficiency by simultaneously using the geometric and propaga-

tion phase of each nanostructure. A nematic LC (5CB) is used to demonstrate the operation of the proposed device when exposed to isopropyl alcohol (IPA) (Figure 41). To provide high-quality holographic visual information, asymmetric coupled metasurface is designed, which produces a high diffraction efficiency (>50%) with very little cross-talk and/or afterimages between the two states, which diffract light to produce “alarm” and “safety” signs. The added advantage of the holographic metasurface sensor over other metasurface sensors or, indeed, the typical LC sensors is that it can produce an informative image; for example, rather than simply changing colour, it can produce a warning message. To demonstrate the compatibility of the proposed sensor with wearable devices, the metasurface is fabricated on a flexible substrate using a one-step nanocasting process and then incorporated in safety goggles.

6.2.2. Birefringent Porous Glass

Pinet et al.^[408] report on a simple design involving a birefringent porous glass oriented between two crossed polarizers. Their broad-spectrum chemical sensor for detecting volatile organic compounds such as acetonitrile vapours can detect concentrations as low as 50 ppm. The sensor measures either the change in the intensity of polarized light transmitted through the anisotropic porous material at a defined wavelength or a change in colour detectable by eye. The optical effects resulting from exposure to the VOCs are reversible.

The authors report a significant response to concentrations of acetonitrile as low as 50 ppm. Different VOCs have been examined, for example, toluene and diethyl ether. It is proposed that the nonspecificity of the sensor is a consequence of physicochemical interactions related to adsorption and solvent capillary condensation properties. It is however proposed that selectivity, can be obtained by modifying chemically the surface energy and by controlling the pore size distribution.

7. Detection of VOCs by Optical Resonance Phenomena

When an electromagnetic wave of suitable wavelength irradiates a nanostructure containing free electrons (predominantly metals), the wave induces a periodic oscillation of the free charges in the nanostructure.^[409] These oscillations cause a variation in the local electric field, known as a plasmonic wave. This effect has been used for biological, chemical, and vapour-sensing applications.^[410–414] Sensing applications exploit two features of plasmonic waves: plasmonic peak shift, which is determined by variations in the dielectric environment, and the surface-enhancement of various spectroscopic techniques (Figure 42).

Several factors influence plasmon excitation, including the incident angle and wavelength of the light, the material and geometry of the plasmonic structure, and the refractive index of the environment.^[409,415] Adsorption of molecules on the plasmonic substrate or within an adsorbing layer alters the refractive index of the environment,^[410,416] subsequently modifying other resonance parameters. Therefore, the adsorption of analyte molecules can be detected by measuring a full spectrum to determine the wavelength at which resonance occurs. Alternatively, for

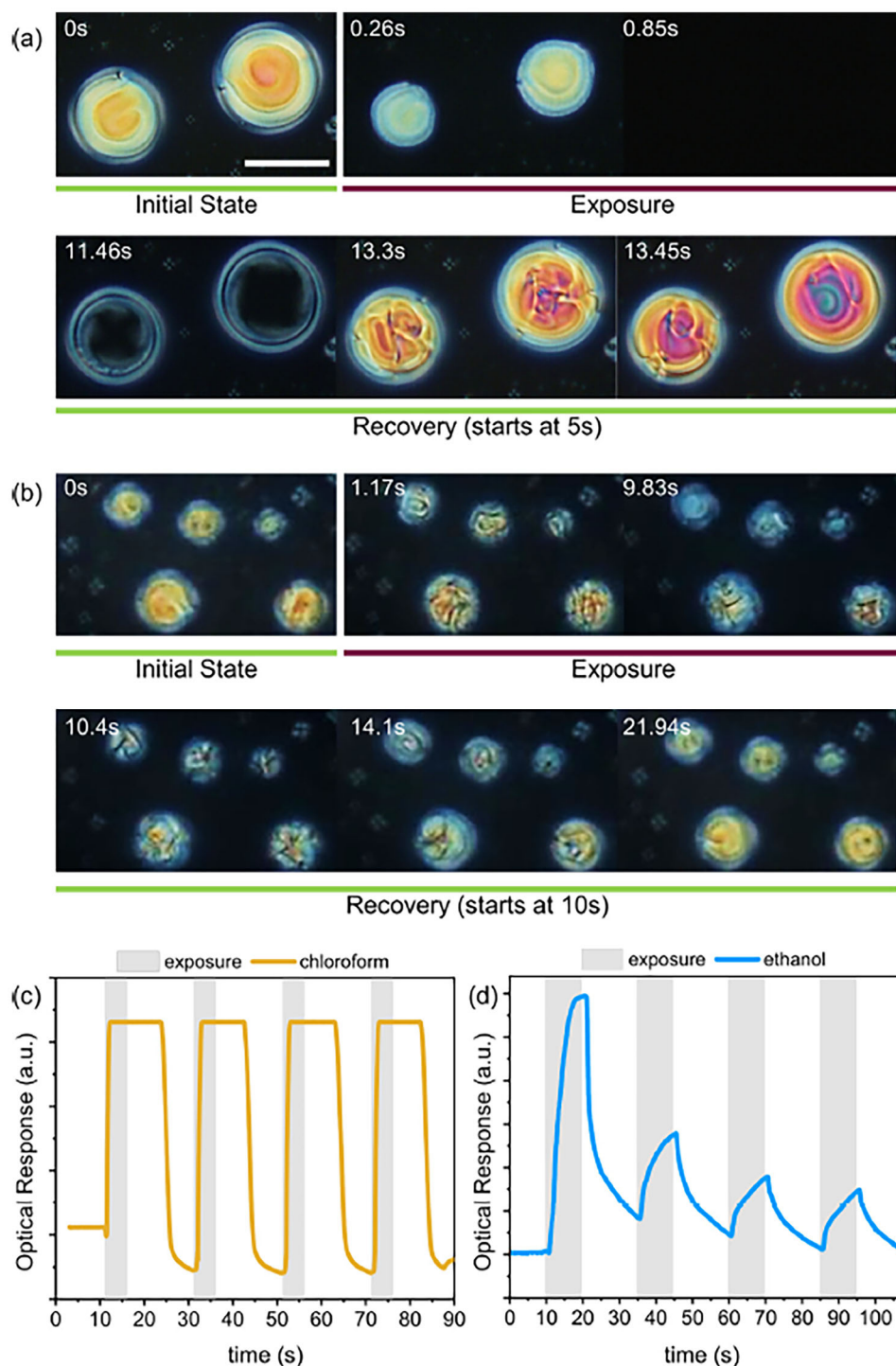


Figure 39. Textural changes of the hybrid gels during exposure to vapors of a) chloroform and b) ethanol and subsequent recovery, as observed through the POM, and c,d) corresponding signals extracted from video analysis. POM photos were taken with crossed polarizers at room temperature: a) M1 (3.5% w/w CB15–96.5% w/w 5CB) hybrid gel and b) M2 (5% w/w CB15–95% w/w 5CB) hybrid gel. The white line corresponds to 50 μm . Reproduced with permission.^[405] Copyright 2023, AIP Publishing.

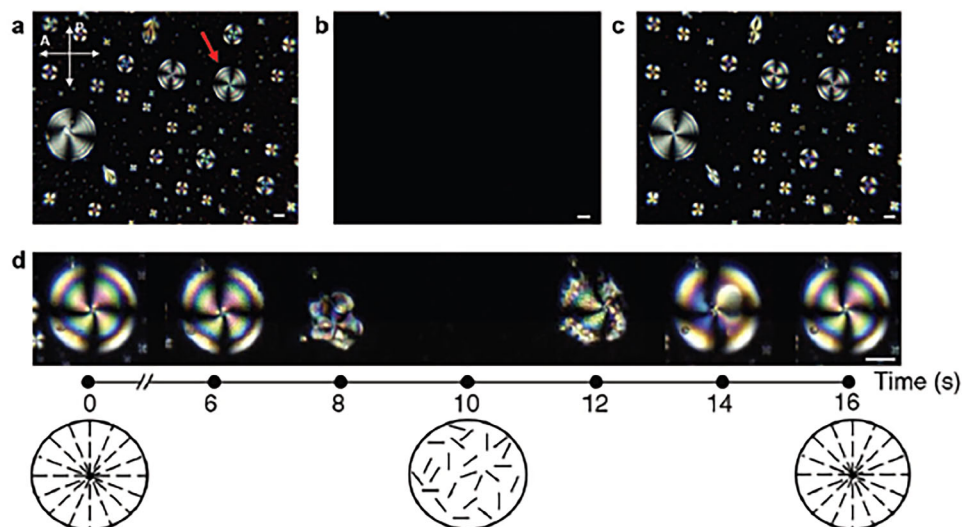


Figure 40. Optical response of hybrid gel films when exposed to air saturated in chloroform. Hybrid gel films composed of gelatin, [BMIM][DCA], 4-cyano-4'-pentylbiphenyl (5CB), and water were observed by POM, with crossed polarizers. a–c) Before, during, and after exposure to chloroform VOCs for 2 s. The red arrow indicates the droplet pictured in detail in (d). d) Detailed time analysis of the 15 μm diameter droplet highlighted in (a) (exposure begun at $t = 0$ s). The scale bars in the microscopy images correspond to 5 μm . Reproduced with permission.^[400] Copyright 2017, Wiley.

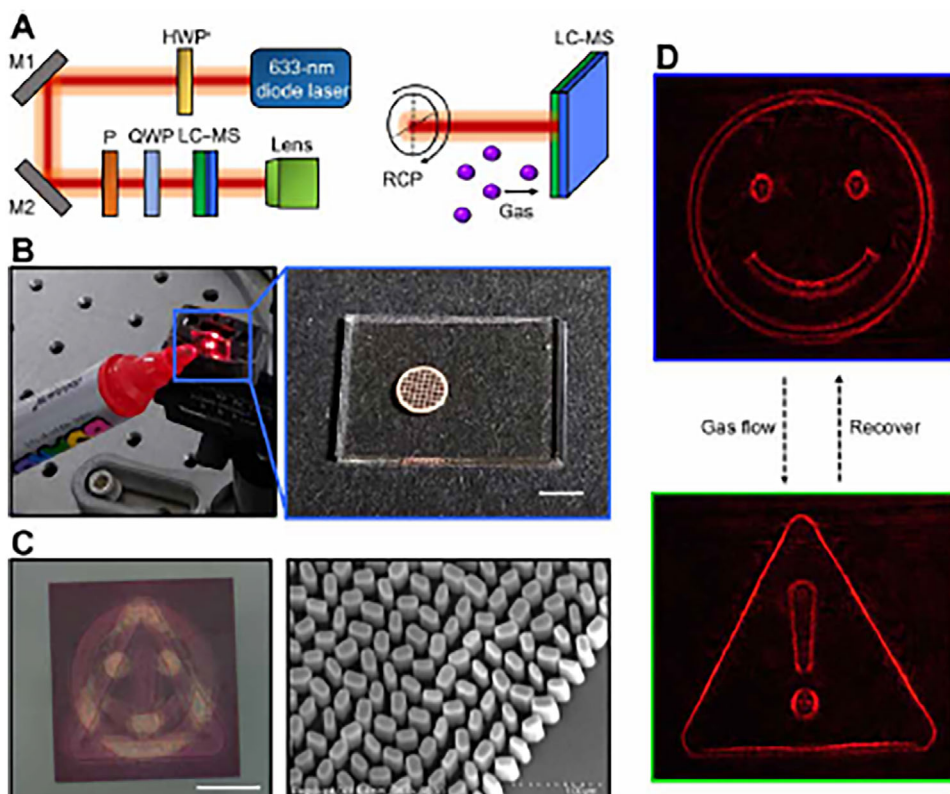


Figure 41. Demonstration of an LC-MS gas sensor. A) Optical setup for an LC-MS gas sensor (HWP, half-wave plate; M1, mirror 1; M2, mirror 2; P, polarizer; QWP, quarter-wave plate). In the absence of IPA gas, the RCP light illuminated on the LC-MS sensor passes the LC layer without any polarization conversion and is transmitted into the metasurface. In contrast, the LC layer converts the incoming RCP into LCP light upon exposure to IPA gas. B) Photographs of an LC-MS gas sensor with a board marker as a source of volatile gases, including IPA. Scale bar, 3 mm. Photo credit: Inki Kim, POSTECH. C) Optical and SEM images of the integrated dielectric metasurface. Scale bar, 100 μm . D) Resulting holographic image alarms. Upon the exposure of gases from the board marker, the LC-MS sensor rapidly exhibits the alarm sign within a few seconds and recovers the initial safety sign once the gases are removed. Reproduced with permission.^[407] Copyright 2021, AAAS.

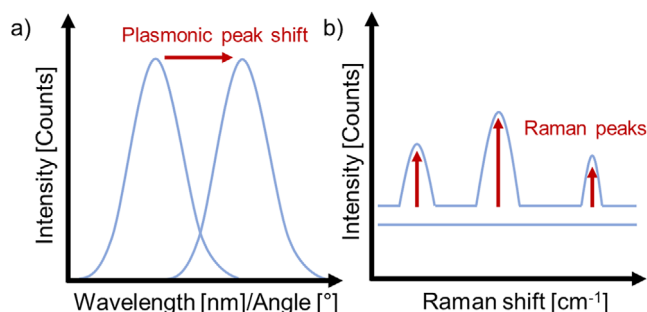


Figure 42. Schematic illustration of plasmonic sensing methods. a) Plasmonic peak shift. b) Surface-enhanced spectroscopic techniques as represented by Raman scattering.

planar films, a fixed wavelength may be used, and the incident angle at which resonance occurs is determined (Figure 42a).^[417,418]

Spectroscopic techniques such as fluorescence, infrared, luminescence, and Raman have been reported to be more sensitive within these enhanced electric fields and are therefore generally referred to as surface-enhanced techniques.^[419,420] Surface-enhanced Raman scattering (SERS) is particularly intriguing in vapour sensing applications as it generates a characteristic fingerprint of the analyte, facilitating selectivity (Figure 42b). Therefore, SERS is by far the most prevalent surface-enhanced method reported for VOC sensing.

7.1. Surface Plasmon Resonance in Films

A coupling strategy is essential for inducing a plasmon in a planar structure because the frequency and wave vectors (thus momentum) of the surface plasmon and the incident beam must match. Common coupling strategies include the application of an attenuated total reflection (ATR) geometry or a grating coupler.^[414,415,418,421] In the ATR strategy, p-polarized light undergoes total internal reflection inside a prism, generating an evanescent wave parallel to the surface. This wave can excite a plasmon in the planar layer. Only p-polarized light can excite surface plasmons, as s-polarized light does not have an electric field component parallel to the surface.^[418,422] The Kretschmann geometry is the most prevalent ATR geometry in sensing (Figure 43).^[414,415,418,421,423,424] In this configuration, the thin metal film can be deposited directly onto a prism,^[423,425] or, in a more practical and economical implementation, on a glass slide that is combined with a separate prism.^[426–431] Physical vapour deposition techniques, such as sputtering or thermal evaporation, are commonly employed to deposit these thin metal films, with the latter preferred due to lower surface roughness, minimizing dampening of surface plasmons and, consequently, peak width.^[432]

An ATR setup can operate in two modes: the constant angle mode, where the variation in refractive index manifests as a wavelength shift of the plasmonic peak, or constant wavelength mode, where a single wavelength is monitored and alterations in the dielectric environment are discerned by illuminating the structure at different angles.^[415,417,422,431]

7.1.1. Planar Films

Bare plasmonic substrates exhibit limited interaction with analytes, so a coating is often applied to enhance sensitivity and selectivity. A thin film of an organic liquid can selectively dissolve compounds from the gas phase. Both polyethylene glycol and silicon oil in trichloroethane have been used as a liquid adsorbing layer in the Kretschmann geometry to detect various VOCs.^[433,434] A polymeric coating can also serve this purpose, where selectivity depends on the chosen polymer. This approach has been demonstrated with a silicon-glycol copolymer and polyimide films.^[429,435]

Attention has recently shifted toward porous materials because of their high surface area, which facilitates adsorption and results in a concentrating effect that enhances sensitivity. Moreover, partially selective adsorption is possible by the presence of functional groups in pores, enhancing selectivity.^[436–438] Metal oxide thin films such as ZnO and TiO₂ have been used to detect various alcohols and hexane using the Kretschmann geometry with a gold film.^[427,428,430] MOFs are attracting attention for sensing applications because of their high surface area and tunable pore interior.^[437,438] For example, utilizing the Kretschmann geometry with a silver film coated with ZIF-8 resulted in a methanol sensor with sub-ppm LOD (Figure 43c,d).^[426] One advantage of surface plasmons over localized plasmons (see below) is their far-reaching evanescent fields (100–200 nm vs 10–30 nm),^[421,439–444] resulting in a larger interaction volume in which the plasmon is sensitive.^[445]

7.1.2. Optical Fibers

Light traveling through an optical fiber undergoes continuous total internal reflection because of the higher refractive index of the core relative to the cladding. Removing the cladding in a specific region and coating the fiber with a metal film of an appropriate thickness allows the light in the fiber to generate a surface plasmon. In this case, the optical fiber serves the same purpose as a prism in the Kretschmann configuration. The occurrence of a plasmonic peak is observed as a wavelength shift of the resonance peak.^[446–448]

The synergistic properties of graphene and carbon nanotubes were explored to fabricate a plasmonic sensor using an optical fiber. A graphene-carbon nanotube-polymethylmethacrylate hybrid nanocomposite overcoat layer on an optical fiber exhibited enhanced selectivity for methane over other tested analytes.^[449] Alternatively, a graphene monolayer coating was applied over a periodically internally perturbed optical fiber (Figure 44a). This approach increased the intensity of the evanescent field, enhancing the methane sensitivity. The graphene layer amplified the evanescent wave rather than functioning as an adsorbing layer.^[450] The MOFs ZIF-8 and ZIF-93 have been used as adsorbing layers on fiber optic plasmonic sensors (Figure 44b). These MOFs showed high sensitivity, with ppm-range LODs for alcohols.^[451] Furthermore, the difference in hydrophilicity between the two frameworks was used to obtain vapour composition data.^[451] In these works, the metal layer (Ag or Au) was deposited by thermal evaporation or sputtering. We envision an improvement of sensor performance if a less directional metal

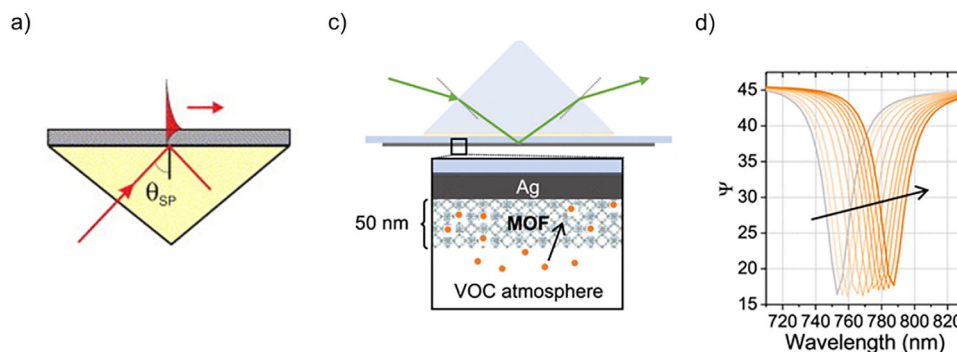


Figure 43. Surface plasmon resonance in planar films. a) Schematic representation of plasmonic coupling using the Kretschmann geometry. Reproduced with permission.^[424] Copyright 2005, Elsevier. VOC sensing in the Kretschmann geometry by using a metal-organic framework thin film adsorbing layer. a) Sensor structure, and b) Observed plasmonic peak shift upon methanol dosing. Reproduced with permission.^[426] Copyright 2022, ACS.

deposition method, such as chemical vapour deposition or atomic layer deposition, would be implemented.^[452–455]

7.1.3. Grating Couplers

Another coupling strategy is to use a grating coupler, a periodic nanostructure designed to couple light into the surface plasmon (Figure 45a). The explanation and derivation of grating couplers exceed the scope of this review. For more in-depth insights, we refer to the book by Raether et al.^[418] A significant upside of this approach is that no prism is needed for coupling, although more expensive lithographic techniques are often required to fabricate the gratings.

A 100 nm Ag layer on a sinusoidal modulated structure was used without further modification for the detection of condensing vapours (Figure 45b).^[456] A modified grating coupler was prepared by depositing a bimetallic layer (7 nm Au, 37 nm Ag) on top of a sinusoidal phenyl-bridged polysilsesquioxane layer. Another layer of this hybrid sol-gel material was then coated over the bimetallic layer. The hybrid layer adsorbs organic compounds as a result of its porosity and functionality. This structure was used for xylene sensing (at 30 ppm) by ellipsometry.^[457]

7.2. Localized Surface Plasmon Resonance

Many of the geometric limitations associated with surface plasmons are circumvented by using nanoparticles, while the free

electrons showcase similar plasmonic behaviour to metal films. When light at the resonant wavelength is irradiated, a periodic oscillation of the free charges in the particle occurs because the particle experiences an oscillating electric field, prompting the free charges within the particle to oscillate. (Figure 46). The electric field around the particle is enhanced upon excitation (near-field enhancement), which is strongest at the surface but diminishes rapidly with distance.^[440,444,458,459] The wavelength at which the LSPR can be excited again depends on the dielectric environment. Like the surface plasmons in planar structures, this refractive index sensitivity allows sensor fabrication.^[460]

Solvothermal synthesis, such as the well-known Turkevich method, is the most common and simple way to fabricate LSPR structures.^[461–463] In such synthesis methods, a metal precursor (e.g., HAuCl_4 , AgNO_3) is combined with a reductant (e.g., sodium citrate, formaldehyde, KBH_4 , ascorbic acid),^[461,463–466] to form small metal clusters surrounded by an electronic double layer in which further metal ions are reduced to grow existing particles.^[462] Protective agents are added to avoid agglomeration, stabilizing through either electrostatic stabilization (e.g., citrate), strong binding to the surface (e.g., thiols), or steric hindrance (e.g., PVP).^[467–469] These methods are widely adopted because of their simplicity and reproducibility, although they offer limited flexibility regarding the size or shape of the nanoparticles. Seed-mediated growth and other advanced solvothermal methods have been developed for size control.^[470–472] The shape of the synthesized particles can be altered either through kinetic control or selective surface passivation (Figure 47b).^[473–475]

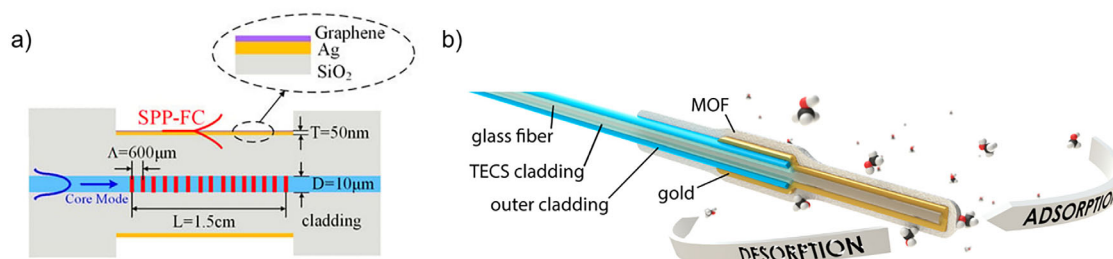


Figure 44. Fiber-optic-based plasmonic VOC sensing. a) Evanescent wave of Ag enhanced by a graphene layer (Surface plasmon polariton-fiber cladding hybrid mode, SPP-FC). Reproduced with permission.^[450] Copyright 2016, MDPI. b) MOF coated on an Au layer. Reproduced with permission.^[451] Copyright 2017, ACS.

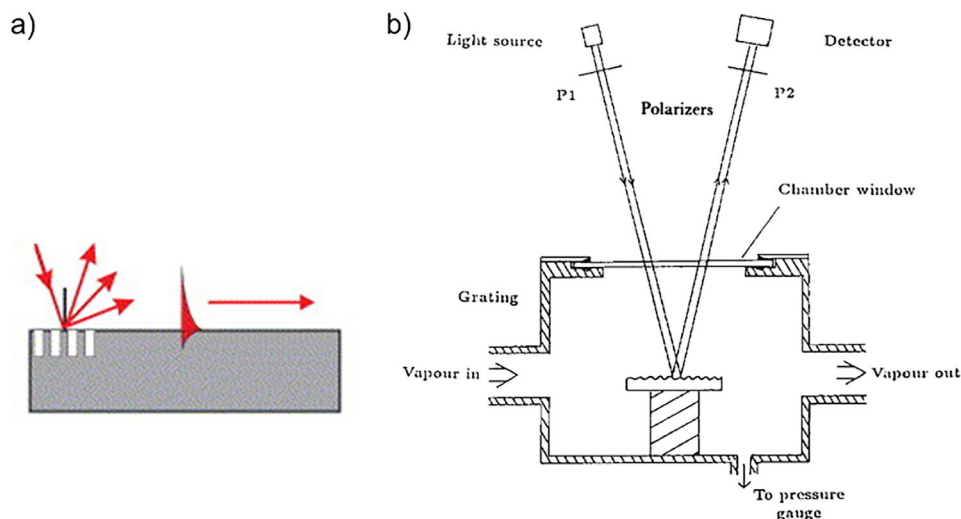


Figure 45. Surface plasmon resonance using grating couplers. a) Schematic representation of grating coupling strategy. Reproduced with permission.^[424] Copyright 2005, Elsevier. b) Grating coupling of plasmon in Ag film for VOC detection. Reproduced with permission.^[456] Copyright 1992, Elsevier.

Alternatively, lithographic techniques can be employed, in which a pattern is drawn by irradiating (e.g., e-beam, UV, X-ray) a resist material. The unwanted regions are removed in the development and etching steps.^[476] While these methods can achieve precise size, shape, and spatial control, their high cost and low throughput limit their practical usability.^[415] These constraints are partially relieved by soft lithographic techniques, which avoid the need for precise irradiation by relying on techniques such as self-assembly, moulding, or stamping.^[477]

The size and shape of the metal nanoparticles significantly influence the extent and spatial distribution of the enhanced fields (Figure 47). The enhancement typically diminishes more rapidly for smaller particles.^[442,443] For non-spherical particles, the field is not uniformly enhanced. Instead, areas around sharp edges are exceptionally amplified, referred to as hotspots (Figure 47a).^[478,479]

When particles are deposited on a substrate, as is often the case for sensors, the substrate alters the plasmonic characteristics.^[480–482] The resonance conditions of isolated spherical plasmonic nanoparticles are independent of the incident angle since the dielectric medium uniformly surrounds the particle.^[483] However, an angle dependence arises when the nanoparticle is deposited on a surface.^[484] Upon excitation, enhancement occurs perpendicular to the incident beam

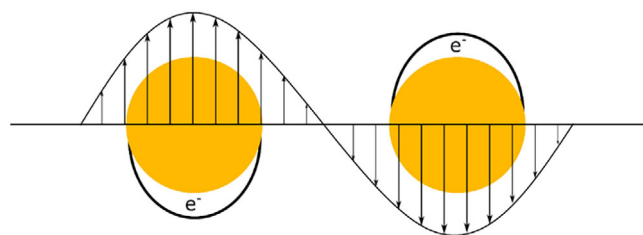


Figure 46. Plasmonic excitation in nanoparticles by electromagnetic irradiation.

(Figure 48).^[443,485,486] The particle is most sensitive to the area where the enhancement occurs.^[487] So, changing the angle will alter the extent to which the particle experiences the effect of the underlying substrate.^[486]

LSPR excitations are especially sensitive to interactions with other plasmonic particles.^[415] The plasmon coupling between two nanostructures can be rationalized by the dipole interaction of the polarized particles in a hybridization model (Figure 49a,b).^[488–491] An increasing red-shift of the resonance peak occurs with decreasing interparticle distance, and a hotspot is formed between the particles (Figure 49c).^[415,442,489,491] Plasmonic coupling is a short-range effect, with a decreasing range for smaller particles.^[442,492,493] Although an enhanced refractive index sensitivity can be obtained,^[492] accurately controlling the spacings is challenging, requiring high-tech lithography techniques.^[442,489] Alternatively, a functionalized coating can be applied around the nanoparticles, which dictates the interparticle spacing.^[492,494–496] However, the most sensitive region, the hotspot, is occupied by a non-sensitive material, thus rendering it inaccessible to the analyte.

Numerous plasmonic particles are present on the surface of an LSPR-based sensor device. When these particles are spaced sufficiently apart, they behave as isolated entities. However, close packing leads to plasmonic coupling. These extended plasmonic structures can be constructed in two ways: the lithographic approach^[497] and the self-assembly of solution-synthesized particles.^[498]

7.2.1. Plasmonic Peak Shift

Using the plasmonic peak shift originating from a change in environmental refractive index is one approach to creating LSPR sensors. For instance, bare silver nanoprisms made through soft lithography were used for ethanol sensing (Figure 50).^[499] However, an adsorbing layer is often advantageous for enhancing

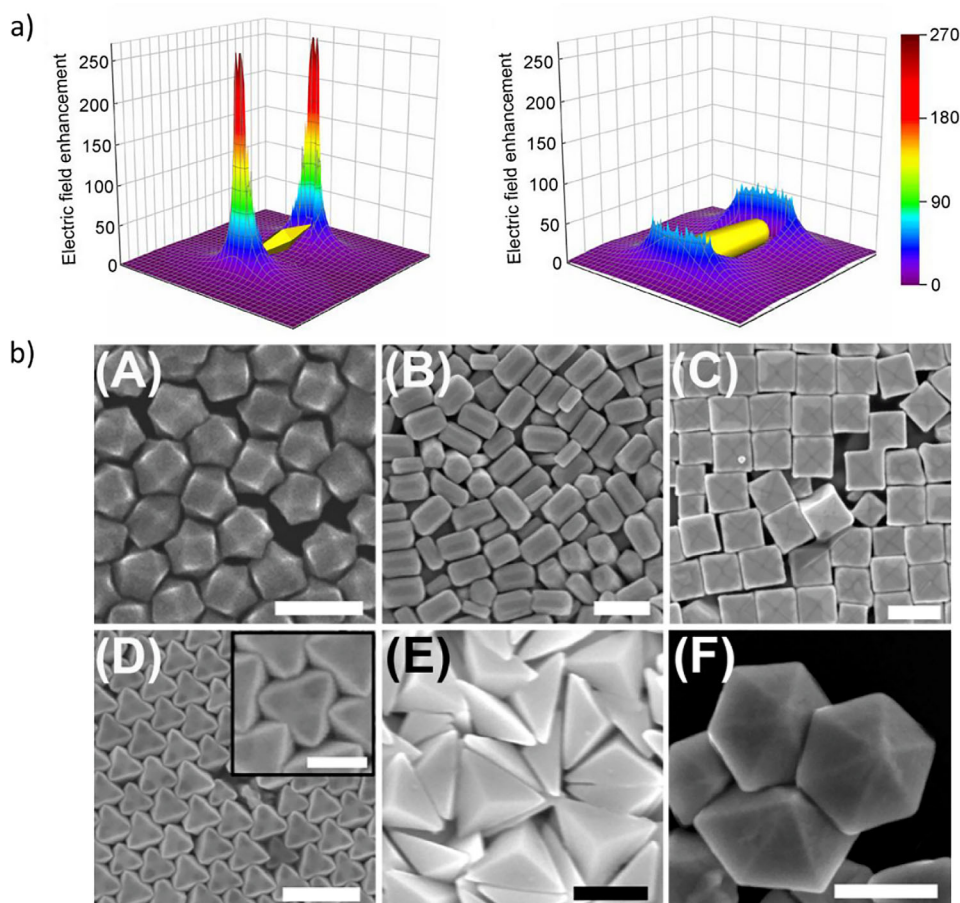


Figure 47. Non-spherical plasmonic nanoparticles. a) Near-field enhancement comparison between gold nanobipyramids and nanorods. Reproduced with permission.^[478] and b) Examples of different possible shapes of Au nanoparticles (A) trisoctahedra, B) tetrahexahedra, C) concave cubes, D) octahedra, E) bipyramids, and F) hexagonal bipyramids). Reproduced with permission.^[473] Copyright 2013, ACS.

sensor performance. In the simplest form, the citrate linker from a Turkevich-style solution-based Au nanoparticle synthesis remains present during sensing. The citrate can significantly influence VOC adsorption, favouring more polar molecules. A citrate-capped immobilized Au nanoparticle layer has been used as a detector in gas chromatography.^[500] In this case, selectivity emerges from the chromatographic separation, so there are fewer requirements on the adsorbing layer.

Immobilizing molecules alters the surface termination of nanoparticles and, therefore, their adsorption. Sputtered Au nanoparticles coated with various thiolated molecules have been used to detect terpene vapours,^[501] while n-octanethiolate-functionalized Au nanoparticles have been spray-coated on

a surface to allow for the differentiation and sensing of toluene and hexane.^[502] The sensitivity of an assembly of tetraoctylammonium-coated Au nanoparticles to saturated toluene and ethanol vapour has also been demonstrated.^[503] Dalfovo et al. showed that their Au nanoparticle assembly sensing mechanism is governed by both the changing inter-particle distance (i.e., degree of plasmonic coupling) and the change of the environmental refractive index.^[503] Borah et al. compared methanol sensing performance between a normal incidence UVVIS transmission measurement and an ellipsometry measurement in the Kretschmann geometry of an Au nanoparticle assembly (Figure 51a,b), favouring the latter. In this work, the nanoparticles were capped with hydrophobic oleylamine, influencing methanol adsorption.^[504] Immobilizing SAMs (4-pyridine thiol; benzenethiol; 3-mercaptopropionic acid) on silver nanoparticles has been illustrated to improve selectivity and LOD.^[505] Decanethiol, naphthalene diol, and 2-mercaptopbenzothiazole have been immobilized on Ag nanoparticles, Au nanoparticles, and Au nanoshells, respectively. The resonances of each plasmonic structure are non-overlapping; therefore, the different chemical selectivities can be read simultaneously. By forming an array of plasmonic nanoparticles with different adsorption preferences, the selectivity of the sensor

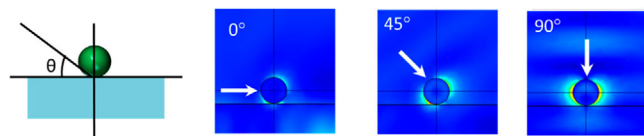


Figure 48. Simulation of the enhanced electric field around a Cu_2O spherical particle irradiated at different angles. Reproduced with permission.^[486] Copyright 2019, MDPI.

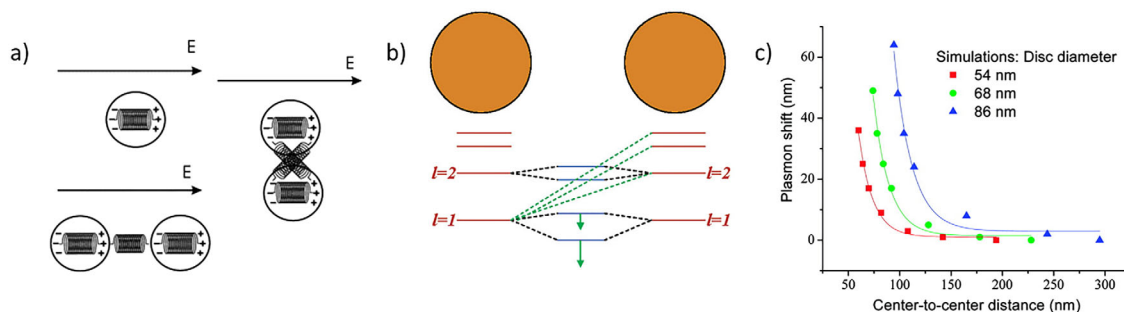


Figure 49. Coupling of closely spaced plasmonic nanoparticles. a) A dipole-dipole interaction model illustrating the interaction between two particles depending on their orientation relative to the exciting field. Reproduced with permission.^[489] Copyright 2003, Elsevier. b) A hybridization model showing the energy levels of the bonding and anti-bonding states. This approach is more accurate when small particle spacings are considered. Reproduced with permission.^[491] Copyright 2004, ACS. c) Effect of particle size and distance on plasmonic coupling. Reproduced with permission.^[442] Copyright 2007, ACS.

setup can be significantly improved (Figure 51c).^[506] In addition, spin-casted Au nanoparticles functionalized with 1-mercapto-(triethylene glycol) methyl ether have been used for individual vapour and mixture sensing (ethyl acetate and benzene) by leveraging chemometrics for signal processing.^[507]

A simple yet effective coating for sensors is a polymeric layer. PMMA has been used for vapour sensing because it swells upon organic compound exposure. High sensitivity and selectivity for chloroform have been reported for sensors with a spin-coated PMMA layer over silver triangular nanoprisms.^[508] Another study explored different sensing behaviours of sensors prepared with PS or PSS overcoats on evaporated gold islands. The organic vapours initiated polymer swelling, with hydrophobic vapours, such as chloroform, swelling PS, and hydrophilic vapours, such as water, swelling PSS. Consequently, the distinct swelling behaviour resulted in opposite selectivity for the two polymeric coatings.^[509] In another example, a plasmonic nanohole array was applied to an analyte-sensitive substrate (UV-curable adhesive, Norland 61). When the structure was exposed to ethanol vapour, it was detected with high sensitivity (LOD 10 ppm).^[510]

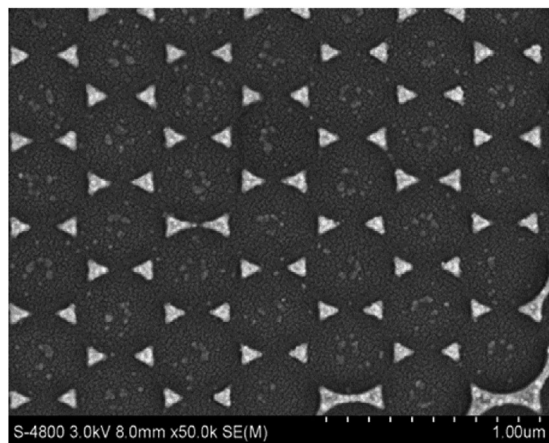


Figure 50. Ethanol vapour sensing with bare Ag plasmonic nanoparticles, morphology of the sensing surface by SEM. Reproduced with permission.^[499] Copyright 2011, MDPI.

Another category of adsorbing layers is porous metal oxides. Gaspera and coworkers spin-coated NiO and TiO₂ sol-gel films over a sub-monolayer coating of Au nanoparticles and showed the potential of this structure for ethanol sensing (only tested at 180 ppm).^[511] The same group made a different VOC sensor capable of detecting 180 ppm of ethanol by spin-coating a mixture of anatase TiO₂ and Au colloids.^[512] TiO₂ fractals with sizes significantly exceeding the near-field enhanced region deposited on Au nanodiscs have been demonstrated as a method to improve ethanol sensing performance (Figure 52).^[513] In another example, Au dots were patterned by e-beam lithography, which were subsequently coated with porous silica. The selectivity and sensitivity of this architecture were screened with eighteen different VOCs, with toluene having the lowest LOD (0.4 ppm).^[514] The authors showed that the selectivity originates from the adsorption preferences of the porous silica rather than differences in the refractive index of the analytes.^[514] In contrast to the previous examples, Güntner and Schenk reported a chemical reaction-mediated LSPR sensor with an extremely low LOD for formaldehyde (5 ppb).^[515] In this sensor, a Ag/AgO_x core-shell structure on TiO₂ was fabricated, in which the AgO_x is reduced by formaldehyde to metallic Ag. This reduction strongly influences the resonance conditions of the architecture and, therefore, the optical characteristics. Selectivity is obtained since only compounds capable of AgO_x reduction are detected. Furthermore, the authors integrated this technology in a wearable sensor with a smart-phone read-out.^[515]

7.2.2. Surface-Enhanced Spectroscopic Techniques

A second approach to creating a sensor using plasmonic nanostructures is to enhance the signal from other spectroscopic techniques, such as Raman, fluorescence, infrared, and luminescence.^[419,420] Plasmonic nanostructures are exceptionally sensitive within hotspots.^[419,420] However, fabrication reproducibility remains a challenge because the signal strongly depends on the intensity of the hotspot. Changes in the geometry of the plasmonic structure lead to variations in enhancement and, consequently, signal intensity.^[516] Surface-enhanced spectroscopic techniques offer a means to increase the selectivity of a sensor device relative to plasmon peak shift. For example, using

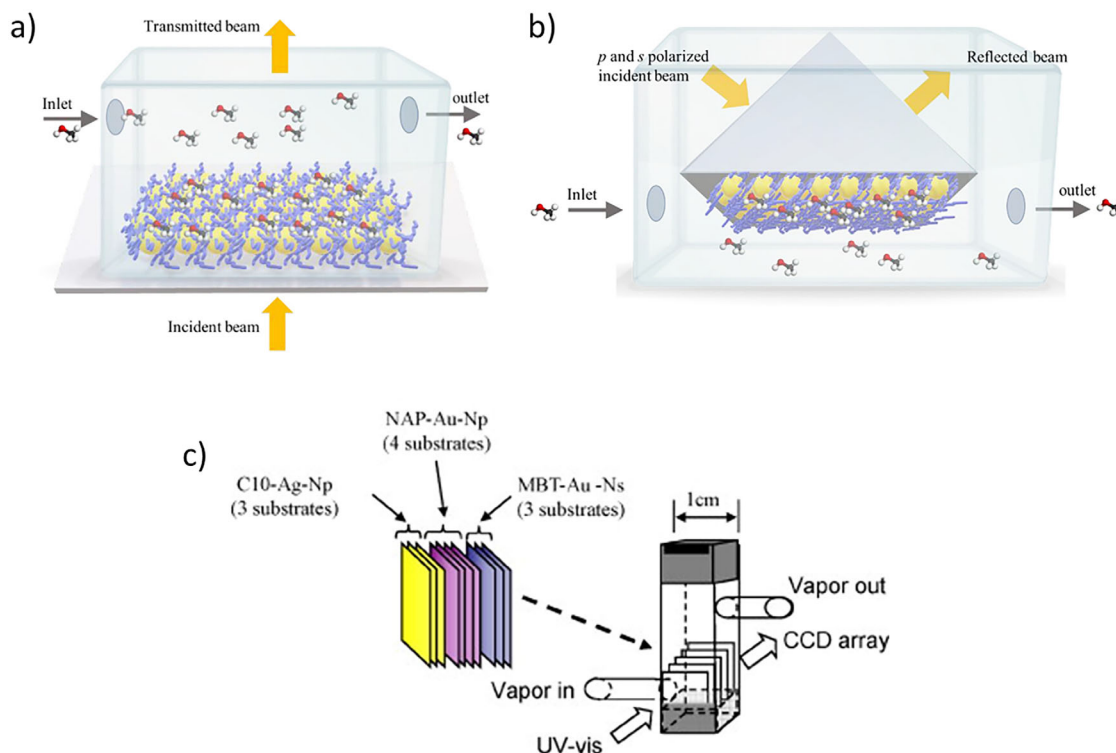


Figure 51. Functionalized plasmonic nanoparticles for vapour sensing. (a-b) Comparison of sensing geometries for methanol sensing with a Au nanoparticle self-assembly. a) Transmission UVIS spectroscopy, and b) Ellipsometric read-out in the Kretschmann geometry. Reproduced with permission.^[504] Copyright 2022, ACS. c) Stacking substrates with different plasmonic nanoparticles for the simultaneous read-out of multiple surface chemistries. (Decane thiol, C10; naphthalene thiol, NAP; 2-mercaptobenzothiazole, MBT; nanoparticle, Np; nanoshell, Ns) Reproduced with permission.^[506] Copyright 2010, Elsevier.

SERS, a characteristic fingerprint of the analyte can be obtained, even at minute concentrations.^[517,518] Raman spectroscopy is a branch of vibrational spectroscopy in which chemical information about molecules is obtained from the inelastic scattering of photons by measuring the shift in energy between ingoing and outgoing photons.^[415,519]

Due to the high sensitivity and selectivity of SERS, as-synthesized particles can meet the requirements for sensor integration. An assembly of citrate-coated Au nanoparticles was used for sub-ppm detection of a chemical warfare agent (Figure 53a).^[520] Similar particles have been assembled into multihole capillaries for the fast detection of saturated pyridine

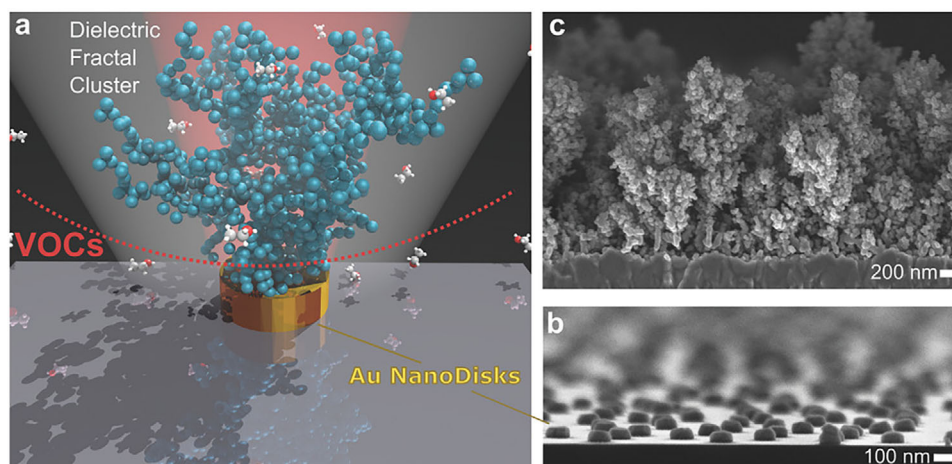


Figure 52. VOC sensing via plasmonic peak shift with dielectric fractal LSPR sensor. a) Schematic. b) Cross-section of Au nanodisc metasurface, and c) cross-section of TiO_2 dielectric fractals. Reproduced with permission.^[513] Copyright 2018, Wiley.

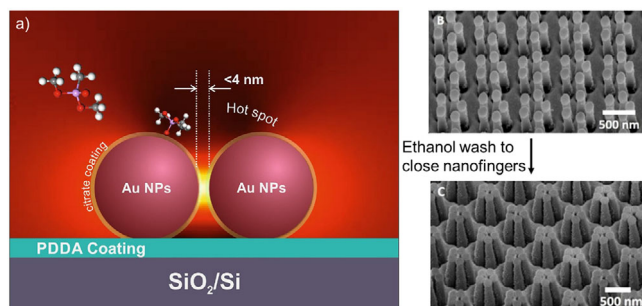


Figure 53. SERS detection with minimally functionalized plasmonic nanostructures. a) Use of hotspot creation through plasmonic coupling to enhance SERS sensitivity. (Positively-charged poly(diallyl dimethylammonium), PDDA) Reproduced with permission.^[520] Copyright 2018, Elsevier. b,c) Formation of plasmonic nanogaps by tilting metal-capped posts to facilitate SERS detection. Reproduced with permission.^[523] Copyright 2017, Wiley.

vapour (within 1 s) and the sub-ppb detection of 4-nitrophenol.^[521] Circular nanostructures have been patterned using deep UV photolithography, in which hotspots were formed by controlled spacing. These layered Ag (30 nm)/Au (15 nm) structures were used for acetone sensing.^[522,523] Plasmonic nanogaps can be created by tilting metal-capped posts towards each other (Figure 53b-c). A leaning nano-pillar substrate with Ag-coated tips identified the ratio of ethanol and acetone in mixtures without the need for an adsorbing layer.^[524] A gold-capped version was used for the SERS identification of the VOC signature of *E. coli* inoculated in biofluids (blood or urine).^[523] This structure had a LOD of 0.001 ppb for indole and benzothiazole, the former being the main VOC produced by *E. coli*.^[523] A grid of Au nanoparticle assemblies located the sources of VOCs through machine learning-enhanced analysis by leveraging spatial variations in chemisorption. This approach succeeded with complex mixtures, demonstrated by the localization of *E. Coli* biofilms through their VOC signature.^[525]

Functionalizing the surface, for example, with thiolated molecules, is one of the most simple adsorption layers. An electrochemically roughened Ag layer was demonstrated as a SERS platform. The sensitivity was enhanced via thiol coating and cooling to increase VOC adsorption.^[526] Similarly, a sensor was fabricated by oblique-angle thermal evaporation, resulting in a Ag nanorod array, which was functionalized with propane thiol (Figure 54). When the sensor was cooled to -80°C , an LOD of 1 ppb for benzene was achieved.^[527] A SERS structure was created by coating a sputtered Au layer with reduced graphene oxide and then depositing Au nanoparticles solvothermally. This configuration enabled particle-film coupling with the reduced graphene oxide acting as both an enhancer and spacer. This setup was combined with chemometric analysis for breath analysis to detect gastric cancer.^[528] A Ag film was e-beam evaporated on top of a polystyrene self-assembly to form a film over a nanosphere structure capable of SERS. The metal layer was functionalized with decanethiol, enabling the detection of a chemical warfare agent.^[529] Three distinct plasmonic nanoparticles (Au@Ag@Au cubes with $\text{Ti}_3\text{C}_2\text{T}_x$, Au@Ag@Au with $\text{Ti}_3\text{C}_2\text{T}_x$ cubes with linker, and Au@Ag cubes) were combined in a microfluidic setup. As a result of their different surface function-

alization, they exhibited sensitivity (ppb-level LOD) to different groups of VOCs. Combining the SERS signal of these structures enabled the quantification of VOCs in mixtures.^[530] Methyl salicylate could be detected with an LOD of 0.608 ppb by a multi-layer of Au nanoparticles coated with 4-mercaptophenylboronic acid.^[531] Patients could be screened for COVID-19 by detecting a decrease in ketones and alcohols and an increase in aldehydes in their breath. This screening was accomplished via a SERS sensor composed of Ag nanocubes functionalized with three different thiols (4-mercaptopyridine, 4-mercaptopbenzoic acid, 4-aminothiophenol) where the signal output was analysed with chemometrics to allow diagnosis.^[532]

Polymer coatings have been used as cheap and effective adsorption layers in SERS sensors. Drop-cast Ag nanoparticles were coated with a Tenax-TA polymer for VOC detection of live cotton plants and dried teas. Through chemometrics, this sensor could differentiate between different tea types and healthy and infested cotton plants.^[533] Another sensor was developed by assembling polystyrene nanospheres and forming pits on these polymeric particles. Polyvinylpyrrolidone-coated Au/Ag bimetallic nanocubes were then deposited in the pits and further coated with ultra-flexible TiC_2T_x MXene, as a universal adsorption layer. The LODs of benzaldehyde, 2,4-dinitrotoluene, and indole were 10, 10, and 50 ppm, respectively. Furthermore, individual components could be identified and quantified in a ternary mixture by classical least-square analysis.^[534]

Porous metal oxides can be leveraged as adsorption layers to enhance SERS performance. For example, Ag nanoparticles were embedded in porous sol-gel SiO_2 . The SiO_2 network acts both as an adsorbing layer, and a cavity that enhances the Raman signal. This configuration detected various VOCs (LOD benzene 56 ppm) and enabled the determination of component ratios in mixtures.^[535] In a similar example, an Au nanoarray was coated with porous zinc oxide to detect benzene-type molecules sensitively (LOD toluene 10 ppb).^[536]

Integrating SERS substrates with MOFs has been a prolific area of research. For example, ZIF-8 was coated over soft lithographic Au nanoparticles for toluene detection (LOD 100 ppm)^[537] and a Ag film over silica nanospheres coated with ZIF-8 for benzene detection (LOD 540 ppm).^[538] Au@ZIF-8 core-shell nanoparticles have been employed to detect saturated VOCs (Figure 55a).^[539] This MOF was also coated over a self-assembly of Ag nanocubes, using hotspot generation from their sharp features and plasmonic coupling for the sensitive SERS detection of diverse VOCs.^[540] ZIF-8 has been coated around microfluidic-integrated AuNanoparticle@AgNanocubes functionalized with cysteamine as a gas-capturing agent for the detection of various aldehydes (LOD benzaldehyde 10 ppb), even in mixtures.^[541] The same MOF was coated around Au@Ag core-shell nanorods for the selective detection of two chemical warfare agents (0.2 and 76 ppbv, respectively).^[542] A multilayer of Ag/ZIF-8 core-shell particles has been used for long-distance (2–10 m) VOC sensing (ppb-level LODs), resulting in multiplex detection via chemometrics.^[543] Breath analysis via chemometrics for the diagnosis of gastric cancer has been achieved with Ag@ZIF-67 core-shell particles packed in a glass capillary. The selectivity of the sensor was tuned towards ketones and aldehydes by functionalizing the Ag@ZIF-67 particles with 4-aminothiophenol, allowing a Schiff-base reaction.^[544] In a related study, Ag nanocubes coated

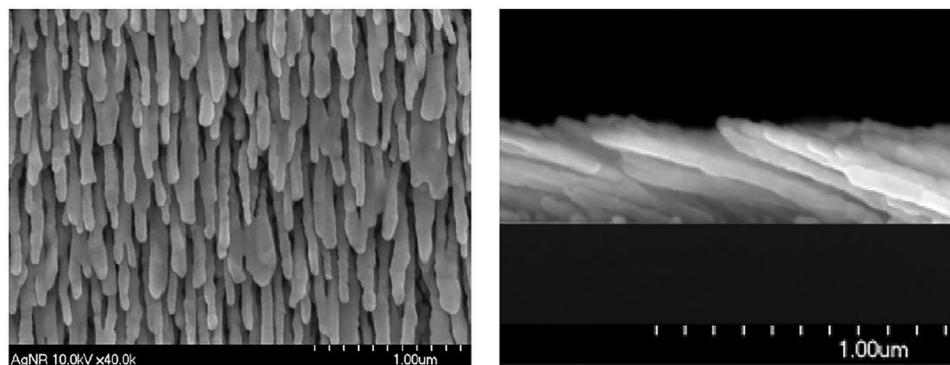


Figure 54. SEM images of thermally evaporated Ag nanorods used for benzene detection. Reproduced with permission.^[527] Copyright 2018, Wiley.

with ZIF-67 and functionalized with 4-aminothiophenol were embedded in a $g\text{-C}_3\text{N}_4$ solid phase extraction membrane. This configuration allowed aldehyde-specific detection of benzaldehyde, which was envisioned for early lung cancer screening.^[545] Another study used gold nanoparticles assembled into superparticles, forming hotspots through coupling. These superstructures were functionalized with aminothiophenol for selectivity towards aldehydes and were further coated with ZIF-8 for selective adsorption (Figure 55b). This setup enabled discrimination between different aldehydes for early-stage lung cancer screening (on simulated breath) when enhanced with chemometrics.^[546] Fu et al. reported MIL-100(Fe) as a Raman substrate because of a charge transfer mechanism, $\pi\text{-}\pi$ stacking for aromatic molecules, and reactive open metal sites. The bare MIL-100(Fe) has a LOD for toluene of 2.5 ppm, but when this material is further en-

hanced with plasmonic hotspots from Au nanoparticles, a LOD of 0.48 ppb for toluene was obtained.^[547] The preferential adsorption of the MOF could be tuned by partially substituting the Fe with Zr; when sensors with different interaction preferences are available, a sensor array can be constructed, further enhancing selectivity.^[547]

Similar to MOFs, COFs (covalent organic frameworks) have recently emerged as promising materials in sensor design. COFs are characterized by their construction from solely organic linkers. For instance, a COF constructed from 1,3,5-tris(aminophenyl) benzene and 1,3,5-benzenetricarbaldehyde was coated around thiol-polyethylene glycol-amine-coated nanoparticles. These Au@COF particles were assembled in a microchannel, resulting in hotspot formation. This sensor configuration was used to sense VOCs with ppb-level LODs.^[548] By using

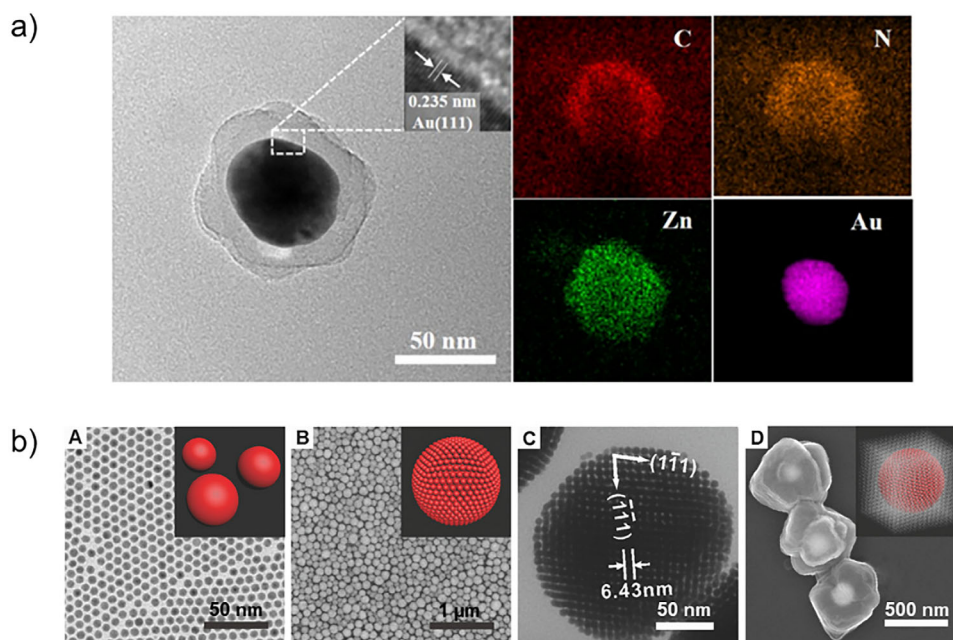


Figure 55. MOF coating around plasmonic particles and superstructures for SERS. a) HRTEM image and EDX mapping illustrating the morphology and elemental composition of an Au@ZIF-8 particle. Reproduced with permission.^[539] Copyright 2021, ACS. b) Gold superparticles formation and MOF coating for SERS detection of lung cancer biomarkers (A) TEM image of gold nanoparticles (B) SEM image of assembled gold superparticle (C) TEM image of gold superparticle, and (D) SEM image of gold superparticle@ZIF-8. Reproduced with permission.^[546] Copyright 2018, Wiley.

chemometrics, individual components could even be identified from within mixtures.^[548]

Colombelli et al. introduced surface-enhanced UV-VIS optical absorption for VOC sensing.^[549] Their method leverages terbium(III) bis(phthalocyaninato) (TbPc₂) complexes, which exhibit altered optical properties upon interaction with electron-donating or accepting VOCs. In their study, TbPc₂ complexes were sublimated to form a thin film over an assembly of Au nanoparticles. The plasmon excitations increased the optical cross-section of the TbPc₂ complexes, thereby enhancing the sensitivity.^[549] While many spectroscopic techniques can be surface-enhanced by plasmonics, SERS is currently dominant in the field.

7.3. Whispering Gallery Mode Resonators

Whispering gallery mode (WGM) resonator-based sensors are promising tools for detecting VOCs due to their compact size, high sensitivity, and high-quality (Q) factors.^[550,551] Similar to the WGMs phenomenon initially observed for acoustic waves,^[552] a micron-sized dielectric cavity with circular symmetry can support optical WGMs and enable light confinement by total internal reflections at the boundary between the cavity and its surroundings. Typically, light is coupled into the WGM cavity utilising the evanescent field of a planar waveguide, a prism, or tapered fiber. Various circular, e.g., spherical, cylindrical, or ring geometries of the WGM resonators allow the light to be trapped inside the cavity. Hence, it propagates along the internal boundary with minimal losses, leading to a long photon lifetime inside the cavity while allowing for multiple cycles of interaction with the surrounding microresonator medium. It should be noted that only specific wavelengths that meet the resonance condition would be coupled into the microcavity, while non-resonant light couples back to the tapered fiber or prism. The following equation governs the resonance condition for the WGM cavity:^[553,554]

$$m\lambda = 2\pi Rn_{\text{eff}} \quad (8)$$

where m is an integer number, λ is the resonant wavelength, R is the radius of the microcavity, and n_{eff} is the effective refractive index (RI) of the resonator. In practice, using an optical spectrum analyser, the resonant wavelengths for a particular WGM resonator can be observed as periodic resonant dips in the transmission spectrum of the coupling fiber taper. Even minute changes in the resonator's radius and/ or effective RI result in spectral shifts of the resonant wavelengths as:^[554]

$$\frac{\Delta\lambda}{\lambda} = \frac{\Delta R}{R} + \frac{\Delta n_{\text{eff}}}{n_{\text{eff}}} \quad (9)$$

Therefore, measurements of the spectral shift of the WGM resonances allow for the detection of environmental perturbations, such as changes in the effective RI of the WGM resonator or its radius, which can be quantitatively correlated to VOC concentrations. When a WGM resonator is exposed to the atmosphere containing VOCs, the VOC molecules are absorbed on the surface of the resonator, changing the effective RI governed by the distribu-

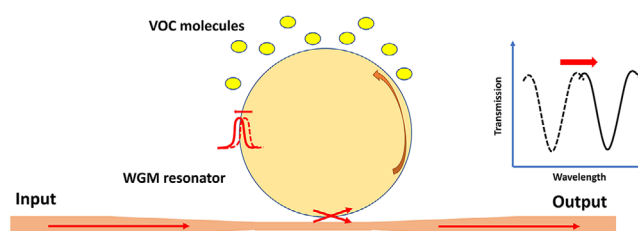


Figure 56. Schematic diagram of a WGM-based VOC sensor.

tion of the electrical field, which can be expressed as follows:^[555]

$$n_{\text{eff}} = \frac{\int n(r) E(r)^2 dr}{\int E(r)^2 dr} \quad (10)$$

where $n(r)$ represents the RI along the radial direction and $E(r)^2$ is the corresponding electrical field intensity. The VOCs with higher RIs lead to redistribution of the electrical field from the interior of the resonator to the interface between the resonator and surroundings, increasing the effective RI according to Equation (8). Additionally, resonators coated with specific materials may swell upon VOC adsorption, changing the resonator radius and causing a resonant wavelength shift. These changes collectively result in measurable shifts in the resonant wavelengths, enabling precise quantification of VOC concentrations. The schematic diagram of a WGM-based VOC sensor is illustrated in **Figure 56**.

WGM-based VOC sensors can be mainly categorized as passive (operating in transmission or reflection modes), as well as active WGM-based laser sensors. Various WGM sensing structures have been reported utilising new materials for surface functionalisation and new sensor structure designs supported by advanced technologies, increasing the readout speed and selectivity. For WGM-based VOC transmission/reflection sensors, resonators fabricated from fused silica demonstrated limited sensitivity for VOC detection due to their lower RI and solid structure. One approach to enhance sensor sensitivity is to use specially treated resonators. For instance, a sodium borosilicate glass resonator subjected to a heating and leaching process has been successfully utilized for ethanol detection (**Figure 57a**).^[556] Alternatively, various coating materials have emerged as a straightforward and effective way to overcome the limitations of silica resonators by increasing the surface interaction area with VOC molecules and improving the sensitivity when the resonator is exposed to VOCs. Application of thin functional layers by dip coating^[557] or inkjet printing^[558] enables the deposition of a thin layer on the surface or inner surface of the various WGM resonators, such as microspheres,^[559] microdisks,^[560] microbottles,^[561] and microcapillaries.^[562] Porous materials, such as sol-gels designed to absorb specific VOCs, have been reported. For example, a microspherical WGM resonator with a sol-gel coating achieved selective and highly sensitive detection of ammonia vapour.^[559] In addition, polymers have been investigated as other candidate materials for WGM-based VOC sensors due to their wide variety and transparency.^[563] Various polymers (e.g., methyl phenol polysiloxane, polyethylene glycol (PEG),^[562] SU-8,^[560] polyvinyl alcohol (PVA),^[564] and polydimethylsiloxane (PDMS))^[565] absorb VOC molecules, allowing for the size expansion and

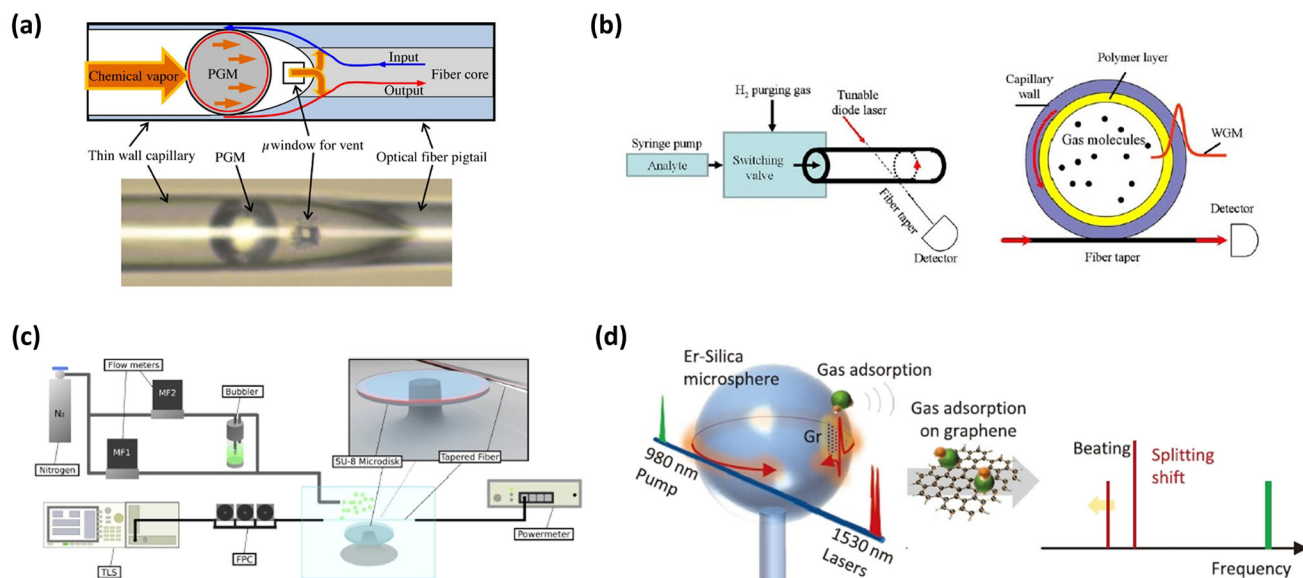


Figure 57. Schematic diagrams of various WGM-based VOC sensors. a) Porous microsphere reflection sensor. Reproduced with permission.^[556] Copyright 2015, Elsevier. b) PEG-coated microfluidic sensor. Reproduced with permission.^[562] Copyright 2008, Optica Publishing Group. c) SU-8 microdisk sensor. Reproduced with permission.^[560] Copyright 2021, Optica Publishing Group. d) Graphene-coated microsphere laser sensor. Reproduced with permission.^[570] Copyright 2022, Wiley.

corresponding RI change of the resonator, thus leading to the resonant wavelength shift that can be linked to the concentration of a specific VOC. Microcapillary resonators, also known as optofluidic ring resonators (OFRRs), open up a novel strategy for VOC sensing since they can significantly reduce the volume of samples under test. Capillaries with inner coatings of methyl phenol polysiloxane and PEG have been investigated for rapid and low-volume detection of ethanol and hexane (Figure 57b).^[562] The same method was then utilized for 2,4-dinitrotoluene detection using PEG as a coating material.^[566] Using inkjet printing as a fabrication method, a SU-8 microdisk WGM VOC sensor has also been reported for multiple VOC detection. It should be noted, however, that the sensor suffered from cross-sensitivity to humidity (Figure 57c).^[560] Polydimethylsiloxane (PDMS) has also attracted considerable attention as a functional material for VOC detection due to its hydrophobicity.^[567] The hydrophobicity effect of PDMS enables the resistance to the polar molecules, such as H_2O , while absorbing weakly polar molecules (e.g., methanol and ethanol) and non-polar molecules (e.g., ethylene), ensuring its interaction with the WGM resonator and improving sensing efficiency.^[557] In addition, the $-\text{OSi}(\text{CH}_3)_2-$ group in PDMS interacts effectively with acetone, making it suitable for acetone detection.^[557] Coating with hybrid materials offers another promising method for enhancing WGM-based VOC sensing. Metal-organic framework (MOF) integrating the metal with polymer material improves the surface-to-volume ratios and introduces various active sites for molecule attachment, realizing selective and sensitive VOC catalytic analysis.^[568] For instance, a WGM microbottle coated with MOF-Fe-PD doped PDMS polymer has been demonstrated for highly sensitive and selective toluene detection.^[569]

To enhance the efficiency of VOC detection, transmission-type WGM VOC sensors have been integrated with advanced algo-

rithms and data analysis techniques, enabling multiple parameters detection and mixtures ratio analysis. The characteristic matrix method has been used for the calculation of ammonia concentration and relative humidity based on two WGM microsphere resonators for multi-parameter sensing.^[571]

WGM lasers are another frequently used scheme for WGM-based VOC sensing. A WGM microlaser comprises a laser pump source, gain media, and resonator cavity for the stimulated laser emission. The laser pump source provides the energy required for the population inversion in the active particles of the gain medium, while the resonator serves as the cavity for laser amplification and stimulated emission.^[540] Similar to the WGM transmission and reflection-mode VOC sensors, the operation of the WGM laser-based VOC sensors relies on the radius changes caused by resonator size expansion and the effective RI changes resulting from absorbing VOC molecules. All these changes result in a shift in the laser peak wavelength, which can be linked to the concentration of the VOC. Fluorescent dyes (e.g., Rhodamine B (RhB)),^[572] Poly(9,9-dioctylfluorene-alt-benzothiadiazole) (F8BT)^[573] and rare-earth elements (e.g., erbium^[570]) have been utilized as gain media for WGM laser-based VOC sensors. Additionally, two-dimensional (2D) materials offer unique transduction properties when coated on the resonator's surface, enhancing photon–electron interactions, leading to a laser mode change when absorbing molecules, and making it suitable for VOC sensing. For instance, a graphene-functionalized erbium-doped WGM laser has been reported for the detection of multiple VOCs, where the attachment of VOC molecules to the surface of the microsphere resonator alters the graphene's Fermi level, resulting in the splitting of the WGM laser modes, enabling precise measurement of the concentrations of mixed VOCs (Figure 57d).^[570] In addition to the previously discussed WGM-laser-based VOC sensors using a

gain medium, the Brillouin laser sensing scheme incorporating graphene has been proposed, where the laser modes and frequency are tuned by the graphene relating to the number of molecules absorbed on its surface, enabling quantitative sensing of VOC.^[574] On the other hand, the Brillouin optomechanical microresonators sensing scheme involving Brillouin optomechanical mode that results from surface elastic modulation in graphene caused by molecular adsorption demonstrates high sensitivity for ammonia gas detection.^[575]

Despite the numerous advantages and applications of WGM-based VOC sensors, the signal stability of the WGM-based transmission sensing scheme is still an issue, requiring a rigid packaging approach to maintain reliable performance. Additionally, the high cost of optical laser sources and high-resolution photodetectors, particularly high-power or pulsed lasers used in WGM laser-based VOC sensing, renders these systems expensive, limiting their further applications.

8. Conclusions

Along with electrical and gravimetric methods, optical detection methods underpin a vast and important subgroup of VOC detection techniques. In this review, we classified optical sensors based on the optical phenomena utilized in the sensing transducers and discussed the functionalized materials that are used to enhance their response. The key advantages of most optical sensing approaches include fast response time, operation at room temperature, compact and affordable electronic elements, ease of operation, possible integration with optical fibers and thus implementation in remote sensing, immunity to electromagnetic fields, further possibilities for sensitivity improvements, and sensor array development due to the versatility of functionalising materials.

An optical sensor is based on three key components: a light source, a transducer, and a detector of the optical signal, depending on the modality of operation. Because most of the optical sensors can operate with incoherent/low-coherence light sources, they are well suitable for the implementation of low-cost sensors. Some sensors, for example, the interferometric sensors, require a coherent light source, which affects their applicability in cost-effective devices. In all cases, for prototype development, light sources with stable output characteristics (output power and spectral characteristics) will be required.

Predominantly, optical transducers react to VOC through refractive index alterations or dimensional changes of the sensing material. These changes, depending on the detection method, affect the photonic bandgap (in diffraction-based sensing), the interference pattern created by two or more light beams, the propagation of evanescent wave, or resonance phenomena. Other important sensing approaches include the detection of the change of polarisation state of a probe beam propagating through optically anisotropic materials (mostly liquid crystals) or the use of spectroscopic techniques, such as surface-enhanced Raman scattering. The latter provides a unique opportunity for characteristic fingerprint production, directly facilitating the selectivity of gas sensing.

The optical methods offer visual and/or electronic detection of the signal. In the case of colourimetric sensors, such as those based on DBRs, volume holographic gratings, opal-like crystals, or luminescent/photochromic materials, the human eye can play

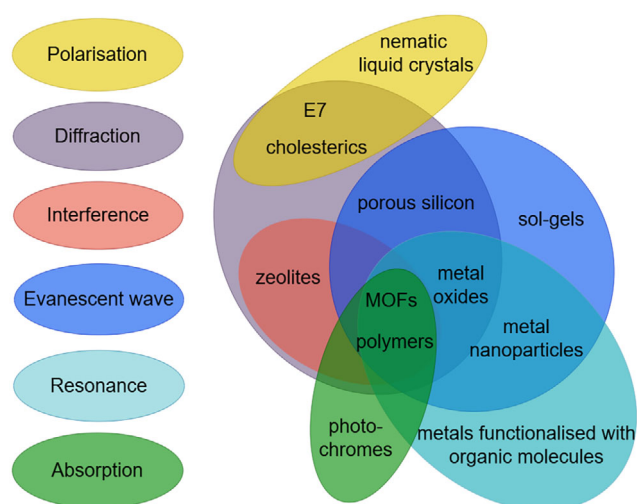


Figure 58. Functionalized materials are used in different types of optical VOC sensors.

the role of the photodetector by detecting a colour change when the structure is exposed to dangerous VOC levels in hazardous environments. Because of the strong dependence of the observed colour on the incident beam angle and the limited wavelength resolution of the human eye, visual detection is prone to misinterpretation, especially when small changes in colour are to be detected. Thus, for a precise quantitative measurement of the analyte concentration, an electronic read-out is preferable. To achieve high selectivity to a target analyte, however, the development of a sensing array would be needed. This imposes several challenges, such as processing of a large number of output signals/images in real time and the use of several functionalized materials, each responding in a unique manner to a mixture of analytes.

Materials that provide reaction-mediated sensing response usually offer higher sensitivities but lack reversibility. Adsorption-based sensing materials, on the other hand, may be used multiple times. An overview of materials implemented in the discussed sections is presented in **Figure 58**. Polymers are often utilized as a flexible, low-cost, and versatile medium across most of the optical techniques, but, in terms of their sensitivity, further improvements are needed. In recent years, a relatively new class of highly porous materials called metal-organic frameworks (MOFs), is reported to offer significantly improved sensing capabilities.

Despite the broad research efforts, a relatively small number of optical sensor transducers have been reported to be further investigated for device implementation. Key studies vital for the real-life applications focus on ageing, sensor lifetime, sensitivity to environmental factors such as humidity and thermal fluctuations in the environment, reversibility, and hysteresis of the material after VOC adsorption and desorption. The development of prototypes emphasises challenges associated with a light source and detection system selection, both requiring compactness, affordability, and reliability. Important impediments are the availability of low-cost stable light sources with sufficient power, beam shaping, and management, and the achievement of high signal-to-noise detection, all impacting the limit of detection. Moreover, in

array format, the device must be programmed to read and process the data, composed of a complex set of simultaneous responses provided by several transducers or, in spectroscopic techniques, to match the single readout with the fingerprints collected in the database. Often, machine learning or chemometrics implementation is required to reliably differentiate between the different target analytes. As such, after enhancing the sensitivity of the single transducer and obtaining sub-ppm and lower limit of detections for a set of analytes, real-life environmental tests are required, involving exposure to a complex gaseous mixture made of different VOCs at varying temperature and humidity conditions. Moreover, currently, the field seems to lack uniform protocols for estimation of the limit of detection, a challenge significantly impeding the accurate comparison of the different techniques. Providing a way of detection limit calculation when reporting a sensitivity outcome, for example in ranges where applicable, following a linear regression-based approach ($LOD = 3.3 \times \text{standard deviation of the response, divided by a slope of the calibration curve}$),^[576] may foster a more uniform comparison of the different research achievements reported in the literature.

Nevertheless, sensor array approaches demonstrated in most of the discussed subcategories promise rapid development of optical sensors. A significant effort is dedicated to the integration of optical sensors in devices, including wearable sensor devices, colourimetric sensors on goggles or windows, or optical transducers connected to gas chromatography. Achieving the goal of highly sensitive, selective, stable, and reversible optical sensor devices with a high signal-to-noise ratio is a complex task and requires the concerted effort of interdisciplinary teams. Collaboration with different stakeholders, for example, relevant industrial partners, environmental agencies, and the health care sector, will be needed to introduce to the market optical sensor devices for air quality monitoring and point-of-care breath analysers.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

Conflict of Interest

The authors declare no conflict of interest.

Keywords

optical sensors for VOC detection, optical materials for VOC detection, VOC biomarkers, VOC sensor arrays, VOC sensors

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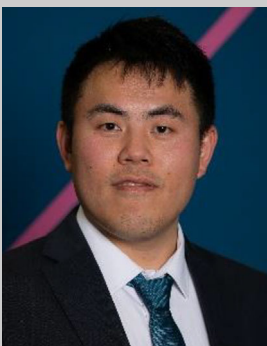
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