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# Optical Fiber Sensing Applications: Detection and Identification of Gases and Volatile Organic Compounds

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## 1. Introduction

Optical fiber has produced an authentic revolution in the field of communications. Nowadays, the principal guided data networks of the world use this media. The great success in optical communications raises the question of whether this technology could be also used in other fields, for instance, in the development of sensors. Optical fiber exhibits small dimensions, it is light weight and it is made of an inert and abundant dielectric material, vitreous silica. Even so, regarding to the sensors domain, electronic technology is much more advanced and mature: miniaturization allows mass production and hence, electronic sensors have affordable prices. Photonic devices currently have a justifiable cost in main infrastructures and networks, but they are still less competitive than electronic sensors in applications where there is a wide-spread deployment. Even though the increasing demand for optical devices is reducing its price, some researchers believe that, to be realistic, the success in the communication field does not have to be applicable in sensor technology (Leung, 2001). This idea is far from being pessimistic: applications whose requirements could be satisfied better by the intrinsic features of optical fiber than the ones of electronic devices, have to be identified: for instance, gyroscopes are Optical Fibre Sensors (OFSs) that have been successfully used to measure rotations (Lee, 2003).

Some important physical properties of optical fiber, such as its low size and light weight have been already mentioned, but there are other relevant features:

- The raw material is vitreous silica ( $\text{SiO}_2$ ), which is a dielectric. It means that the fiber is immune from external electromagnetic interference. On the contrary, electronic sensors handle with electric signals and are subject to the resulting noise and cross-talk. A factory with heavy machinery or a high tension installation are places where optical fiber sensors could be a good alternative to electronic devices.
- Optical fiber sensors are passive: there is no need for any biasing electric signal to operate. Passive devices have a great autonomy because they do not have to be electrically fed: only the light source needs that. Moreover, the just mentioned feature is interesting in applications where flammable gases or vapors or even explosives are present. Mines with explosive gases or eco-plants are some examples of environments where electric signals would be dangerous.

- Attenuation suffered by light when propagating through the fiber is very low, down to 0.2 dB/km at 1550 nm. It permits the measuring point and the receiver to be separated by several tens of kilometers without amplification. As a result, it can be possible to work remotely in hazardous environments, such as applications where highly toxic wastes have to be controlled or chemical solvents are shipped.
- The signals from many sensors (up to hundreds) can be guided through the same fiber using Wavelength Division Multiplexing (WDM) techniques (Barbosa et al., 2008), which are explained later in the current chapter. Even sensors measuring different parameters (temperature, humidity, pressure, strain) could be connected to the same optical fiber bus. These features are ideal in multi sensor applications, as for example, the structural control of buildings (Rao et al., 2006).
- A very interesting property that is being studied nowadays is Distributed Sensing: it is based on using the fiber itself as a sensing element, so measurements such as temperature can be determined with a spatial resolution below one meter (Diaz et al., 2008). Distributed sensing is not offered by any electronic sensor but only by optical fiber technology; it is well orientated to structural monitoring applications.

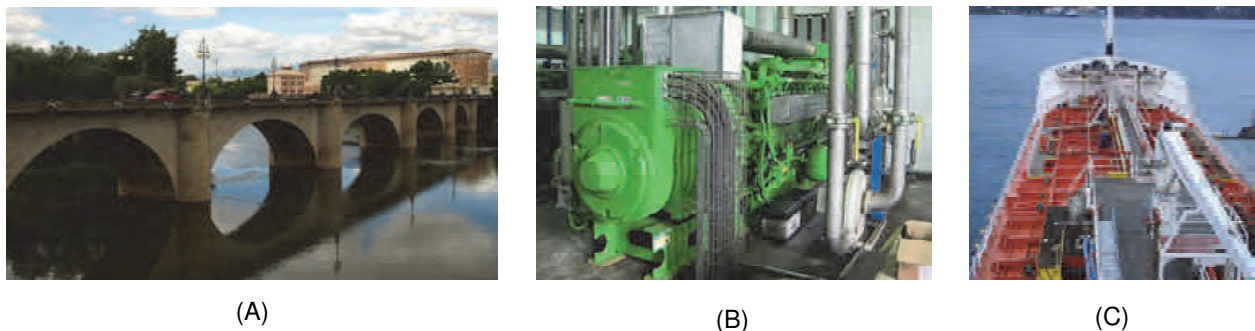


Fig. 1. Some potential market niches where OFSs could be used: (A) structural controlling of bridges; (B) eco plants where methanol is generated; (C) shipping of chemical products.

The most important features of OFSs have been listed, showing some potential applications where their high initial cost would be justified. One field where this technology shows great potential is the detection of Volatile Organic Compounds (VOCs). These substances are present in daily or industrial environments: cleaning products, toxic agents or odors are some examples of VOCs mixtures (Ampuero&Bosset, 2003, Hudon et al., 2000). Although electronic devices already exist for these tasks, they show practical drawbacks such as their large size or high weight (Goschnick et al., 2005).

This chapter is focused on optical fiber sensors used to handle with VOCs because this field can take advantage of optical fiber features. The second section of the chapter shows an overview about OFSs opportunities in VOCs applications; the distinct sensing architectures and construction methods are described in Section 2 as well. The third one covers the factors related to the development of the sensors, whereas some multiplexing networks are described in section 4. Data mining processes typically used to identify VOCs are detailed in section 5 and finally, an all fiber system able to identify beverages is described in section 6.

## 2. Optical fiber sensors to detect and identify VOCs

Applications related with VOCs detection cover several areas such as environmental monitoring, chemical industry, safety at work or food industry, just to mention a few. OFSs offer interesting features that solve some inherent problems of electronic sensors, as it was pointed out at the introduction. In this way, they have been studied and developed since almost three decades ago. This chapter will detail some potential market niches for them.

A volatile organic compound is, by definition, an organic compound that has a vapor pressure between 0.13 kPa and 101.3 kPa: in other words, it evaporates easily. Carbon is usually the base element of these compounds, although there are other ones without it that are considered VOCs as well. These substances are highly inflammable, easy to inhale, and in many cases, toxic (depending on the VOC, concentration and exposure time).

| VOC             | Dangerousness           | Applications                       |
|-----------------|-------------------------|------------------------------------|
| Alcohols        | Inflammables Irritation | Drinks Industry                    |
| Formaldehydes   | Highly Toxic            | Environmental / Safety at work     |
| Methane         | Explosive               | Greenhouse effect / Cattle Health  |
| Ketones         | Industrial Processes    | Inflammable / Irritant             |
| CO <sub>x</sub> | Asphyxia Risk           | Greenhouse Effect / Safety at work |
| O <sub>2</sub>  | Explosion risk          | Air Quality                        |
| H <sub>2</sub>  | Explosion risk          | Storage and Leakage Detection      |

Table 1. Dangerousness and applications of some important VOCs.

There is a large number of potential applications based on identifying the VOCs present in these examples. The production of certain gases, such as methane, during the digestion of cattle (Culshaw et al., 1998) is a good health indicator, and furthermore, this gas affects directly greenhouse effect. In fermentation control applications, these sensors can be useful during the elaboration of alcoholic drinks (Santos et al., 2004), in olive oil production (Stella et al., 2000) or even in maturing stages of sausages (Garcia et al., 2003). Finally, VOCs can be used also to check the condition of foods or drinks as milk, depending on the odors produced by them (Brudzewski et al., 2004). All these tasks can be performed by human operators, but, in cases where a continuous control is mandatory, the health of the workers could get affected, and their smell sense would become damaged.

On the other hand, most chemical solvents are volatile and are widely used to synthesize products present daily: lacquers, paints, dyes, cleaning products, cosmetics, pharmaceuticals, etc... The high toxicity of VOCs and their environmental impact have forced governments to legislate their emissions (in Spain, it is detailed in Real Decreto 117/2003 del 31 de enero). From this reason, it is necessary to control these vapors concentration continuously in the places where they are handled: a long exposure of several VOCs can produce health disorders. This phenomenon is present in many office buildings: it is named the Sick Building Syndrome (SBS) (Elosua et al., 2009).

Thanks to the features that these sensors offer and compared to their electronic counterpart, OFSs have a potential market niche in VOCs detection. Although there is an evident drawback in terms of technological maturity, there are some real applications where they

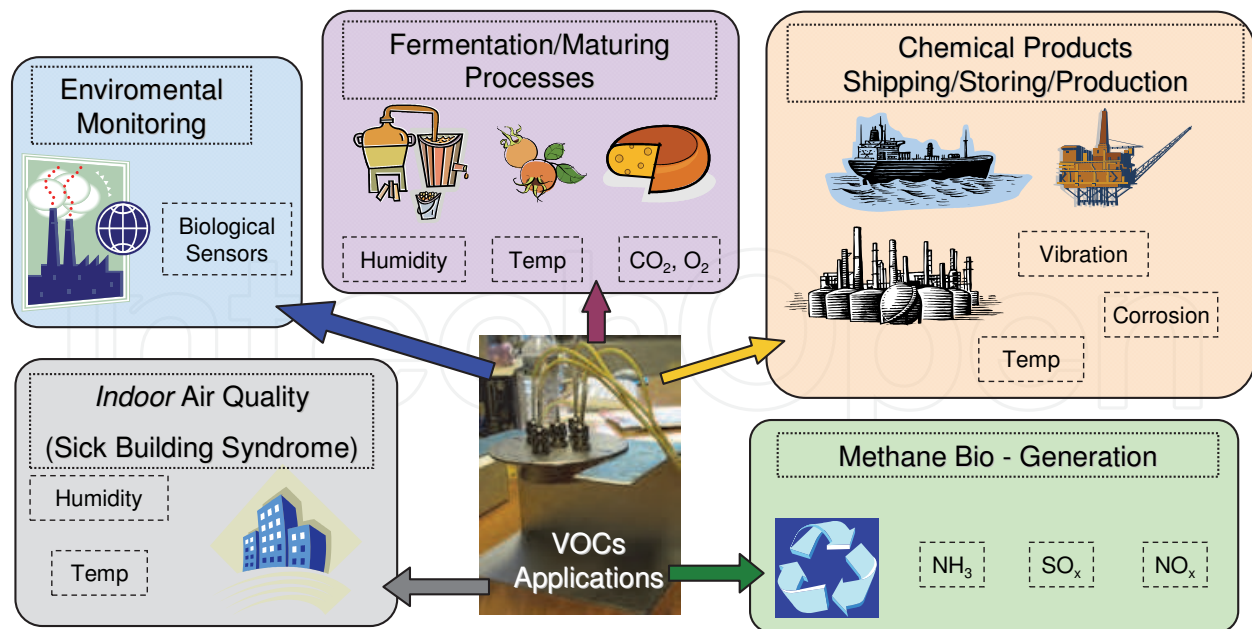


Fig. 2. Some potential market niches related with VOCs for optical fiber sensors. In each one, some other interesting physical or chemical magnitudes are pointed, so all of them could be monitored with optical fiber sensors using the same fiber network.

are successfully been used (Culshaw, 2005). Eventually, requirements of the market will determine whether the use of OFSs is generalized when working with VOCs.

## 2.1 Optical fiber sensors. General concepts

Briefly, a sensor is a device able to transduce a physical or chemical magnitude into a physical measurable signal; however, some authors assume that it covers the whole system, including transduction, transmission and data mining (Lopez-Higuera, 1998). Along the current work, the first definition is considered (referring just to the transducing process). Specifically, the magnitude to be measured is VOC concentration, so it has to be transduced into an optical beam (or beams) travelling through the fiber. The first classification of optical sensors can be made regarding to the parameter affected by transduction: so that, there are amplitude/intensity, phase or wavelength modulated sensors. Sometimes the signal of the sensor is conditioned by applying different modulations looking for making it more robust.

Some parameters, such as response/recovery times, should be always as short as possible, but other ones such as linearity, detection limit or selectivity will depend on the measuring conditions and the final requirements of the application. In the case of VOCs detection, applications can be divided in two main groups:

- Continuous monitoring of VOCs concentration: the concentration has to be known in real time, so the sensors have to be linear and show short response and recovery times. Otherwise, there would be a delay between the real value and the one obtained from the sensor. Moreover, cross correlation must be as low as possible and selectivity as high as possible.
- Individual solvents or VOCs mixtures identification: samples identification is achieved by specific fingerprints. These applications work in a qualitative rather than in a



quantitative way, so, linearity and selectivity are not so necessary. However, it is important that the sensor response was different for each VOC. In fact, mammals smelling sense consists of thousands receptors with overlapped selectivities: their responses conform a fingerprint for each odor (Burl et al., 2001).

## 2.2 Sensing topologies

An overview about the different techniques employed to detect VOCs with OFS will be exposed. In configurations based on spectrometry, each sensing device needs its own light source and detector; in other cases, no fiber is used, so the response of the sensors is first recorded by a CCD camera and then processed. This last solution is optimal for portable systems, but it is not useful in situations where remote sensing is required. In the case of the first configuration, the system is simple but shows a low scalability: the number of active devices grows with the number of sensors. On the contrary, optical fiber allows resources to be shared and also to take advantage of its remote sensing capability (Figure 3).

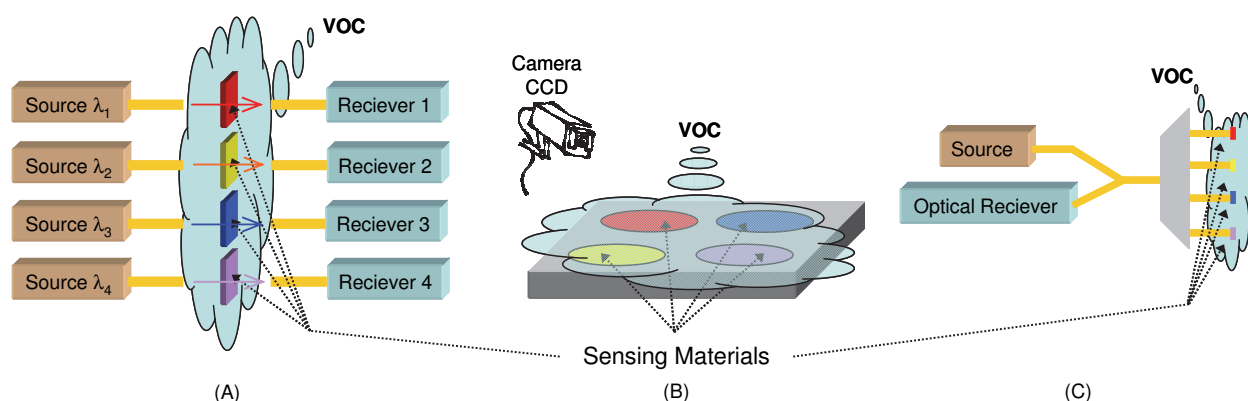


Fig. 3. Different VOCs sensors architectures: (A) one source/receiver per sensor; (B) configuration based on image processing, typically used in portable systems; (C) optical fiber multiplexing network, optimal for remote sensing applications.

There are many criteria to classify all the sensing architectures (Culshaw, 2000, Matias et al., 2006). Among all of them, the one based on where the transduction takes place will be considered in this chapter: in or onto the fiber (intrinsic sensors) or outside it (extrinsic sensors). This classification is followed by several authors (Elosua et al., 2006).

### 2.2.1 Extrinsic sensors

In this type of sensors, fiber acts as a transmission line. The sensing idea lies in guiding out the light from one fiber and then, coupling it back to another one after the signal interacts with the gas to detect. Extrinsic sensors are being used in real applications, multiplexing them in networks that monitor VOCs through wide extensions (Culshaw, 2004).

The way these sensors work is inspired by spectrometric techniques (Garriguesa et al., 2001). Every chemical compound has some specific absorption spectral lines: the material absorbs electromagnetic radiations at certain wavelengths. Therefore, each compound can be identified by the fingerprint formed by its spectral lines. If one of these lines falls into the transmission range of the optical fiber, the gas can be detected launching a light beam centered at this wavelength. If the target gas is present, it will absorb part of the optical

signal, decreasing the power coupled to the receiver; otherwise, the signal will preserve its power level. These sensors are based on an intensity modulation, although frequency modulations have been used to make the measurements more robust (Chambers et al., 2004). The most successful application for this architecture is methane detection.

Another important feature is that a sensing unit can be used to detect different gases, optimizing resources and increasing the versatility of the whole system. It can be achieved by tuning the wavelength of the optical source to match spectral lines from different gases, which is known as Wave Spectral Modulation (WSM). This technique combines both wavelength and time division multiplexing (WDM and TDM): optical signals at different wavelengths are emitted at different time slots. Cross correlation with other gasses can be decreased by using modulations (Whitenett et al., 2004).

Other extrinsic configurations are based on guiding the optical signal from the fiber to a substrate doped with a material sensitive to the VOC to be detected (using the transmitted or reflected signal). Sometimes, if the distance between light source - substrate - receiver is short enough, no optical fiber is needed: with a certain incident angle, the reflected beam is directly coupled to the receiver. Some portable devices to detect formaldehydes have been fabricated following the option that uses no fiber (Kawamura et al., 2006).

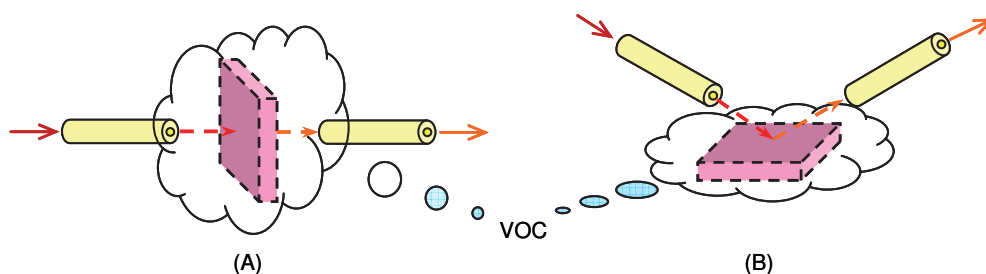


Fig. 4. Examples of extrinsic sensors with a sensing material: (A) the optical signal passing through the substrate with the sensing material is used; (B) the reflected signal is registered.

### 2.2.2 Intrinsic sensors

In this case, transduction takes place on the fiber, either because it is directly affected by VOCs or because a sensing material is fixed onto it. This category can be also organized in several subgroups, depending on the kind of transduction.

#### *Transmission configurations*

Most research efforts during the nineteen nineties were focused on this type of sensors (Khijwania&Gupta, 1999). The optical signal passes through the sensor: it is transmitted across it. The working principle is based on replacing a cladding segment by a material sensitive to the VOC to be detected. In this way, any change suffered by this new cladding will affect the light passing through that segment. For that reason, the sensitivity of the sensor depends on the optical power coupled to the evanescent field and its penetration depth into the modified cladding.

Light propagation through an optical fiber depends on the ratio between the refractive index of the core ( $n_1$ ) and one of the cladding ( $n_2$ ). The modified cladding has a variable refractive index  $n_{2m}$  and the last index to be taken into account is the one of the surrounding

environment  $n_a$ . More rigorously,  $n_{2m}$  has a real part that affects the incident angle (used in Snell law) and an imaginary one that determines the optical absorption of the modified cladding. Depending on the ratio between these refraction indexes, the optical power transmitted through the sensor will vary (Figure 5), so this is the transduction principle.

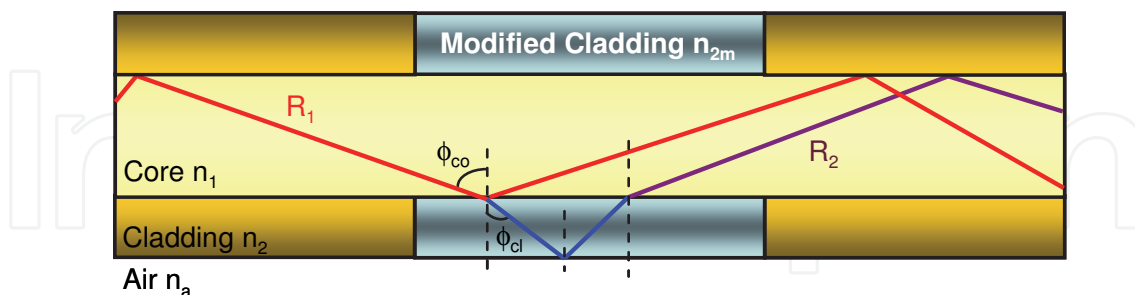


Fig. 5. Ray Theory applied to Evanescent Field Weak Guiding.

The modified cladding fiber can be bended or tapered in order to increase the final sensitivity of the sensor (Scorsone et al., 2004, Tao et al., 2006). Special fibers are also employed to implement sensors: Hollow Core Fibers (Smith et al., 2003) or Photonic Crystal Fibers (Frazao et al., 2008) have been used to develop sensors showing a high sensitivity. Even photonic devices typically used in telecommunication networks, as Bragg Gratings, can be used to implement sensors (Tao et al., 2004). Moreover, other kind of effects such as electromagnetic resonances, can take place when depositing metal oxides nanolayers. In this case, the transduction alters the frequency where the resonance occurs: in this manner, the response of the sensor is modulated in wavelength. Several devices are been developed nowadays, measuring also volatile organic compounds (Zamarreño et al., 2010).

#### *Reflection configuration*

In this category, sensors consist of an optical fiber pigtail cleaved ended, on whose extreme is fixed the sensing material. Thus, light travelling through the core reaches the end of the pigtail, interacts with the deposition and a part of the incident signal is reflected depending on the transduction. The sensor head looks like a chemical electrode; therefore in applications where the concentration of some chemical species has to be measured, it is called optrode (Wolfbeis et al., 1998).

The working principle is, a priori, simple: if any of the optical properties of the sensing material changes in presence of any VOC, it will cause a variation in the reflected signal (Giordano et al., 2005). In a quantitative way, the response of reflection sensors can be explained by interferometry (Lee et al., 1992). After the sensing layer is fixed, the interface between the fiber and it, together with the interface between the layer and the surrounding media, conform an interferometric cavity. If the refractive index of the sensing layer, its thickness or its optical absorbance changes in presence of VOCs, so will do the reflected power in the fiber - sensing layer interface. This idea is summarized in Figure 6 (Consales et al., 2009):

#### *Other configurations*

It is also possible to combine reflection and transmission topologies: it is known as the *hybrid configurations* (Mitsubayashi et al., 2003). Another possibility is based on fixing onto the fiber a luminescent material whose emission varies in presence of the target VOC. The most



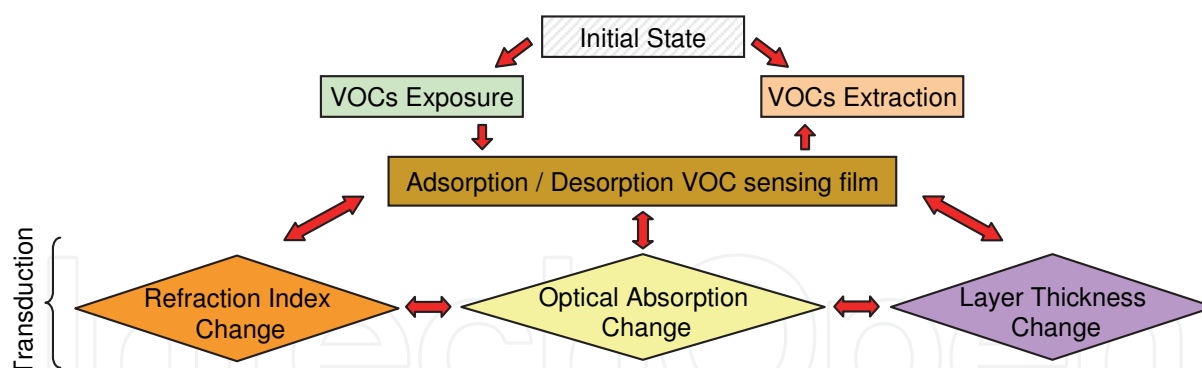


Fig. 6. Scheme with the parameters present in the response of a reflection sensor.

relevant application so far based on luminescent sensors is the detection and measurement of oxygen (O'Neal et al., 2004).

### 2.2.3 Choosing the sensing architecture

All the configurations described so far are summarized in Figure 7; as it can be inferred, choosing one of them is challenging. The best way to select one is thinking about the requirements of the final application and check which one better meets them.

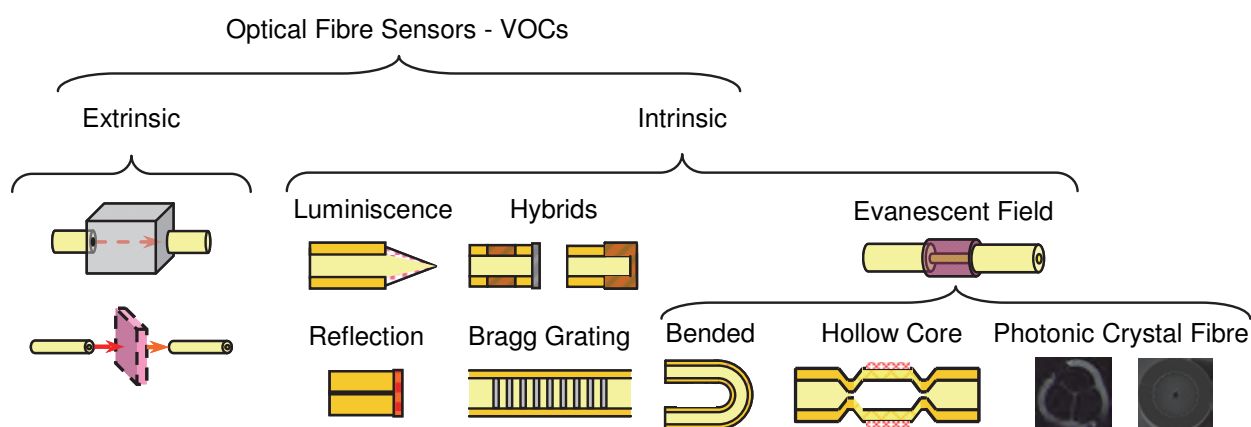


Fig. 7. Classification of sensors described in this section.

## 3. Construction techniques

The second basic step when preparing OFS is the deposition of the sensing material along or onto a cleaved ended pigtail. This stage is necessary only in intrinsic sensors because with extrinsic sensors, the transduction takes place outside de fiber. The deposition has to be repetitive and as homogeneous as possible. The VOC to be detected has to get diffused into the sensing layer, so its morphology is a critical factor. The construction techniques have been adapted for OFSs, so the final thickness is in the nanometric scale, which ensures a fast response. The most relevant two procedures will be described along this third section.

### 3.1 Dip coating technique

The simplest method consists of dissolving the compound of interest in a solvent that does not alter its sensing properties and then, dipping the fiber in this mixture. This technique is

called *dip coating* (Bariain et al., 2000). The morphology and thickness of the final deposition are affected mainly by the times that the fiber is immersed into the solution of the sensing material and the velocity at which it is dipped (Arregui et al., 2002). An alternative option consists of leaving the fiber dipped in the solution for a certain time (Scorsone, et al., 2004).

This procedure is employed when preparing sensors in transmission configuration. The fiber is dipped perpendicularly into the mixture, so a thin layer is fixed along it. In the case of reflection sensors, the layers deposited onto the end of the pigtail do not conform a regular shape but one similar to a match head (Figure 8). Although simple, when preparing reflection sensors, this method is limited by its poor reproducibility.

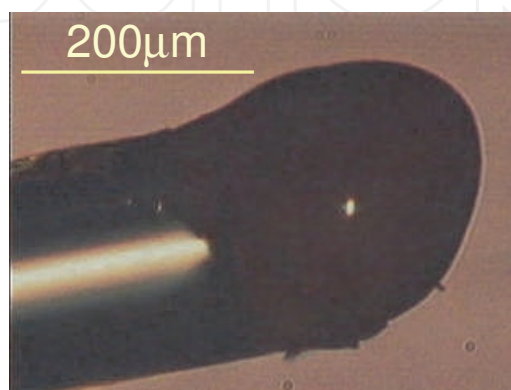


Fig. 8. Image obtained from an optical microscope of a reflection sensor prepared with Dip Coating technique.

### 3.2 Layer by layer method

The small dimensions of optical fiber can take advantage of deposition techniques that do not depend on the size and morphology of the substrate. One of them is the Layer by Layer method (LbL): it belongs to a series of procedures whose aim consists of creating matrixes or supports on different substrates at a molecular scale. To achieve this, the electrostatic attraction of molecules with opposite electric charge can be used, so that they get assembled one with each other. Long polymer chains are used in this way: they exhibit ionic groups when they are dissolved. Polymers that exhibit this property are called polyelectrolytes. Multilayer structures based on this idea have been studied since the 90s (Decher, 1997). One important feature is that the LbL is carried out at room temperature; moreover, it allows the thickness of the layers deposited to be controlled (Choi&Rubner, 2005).

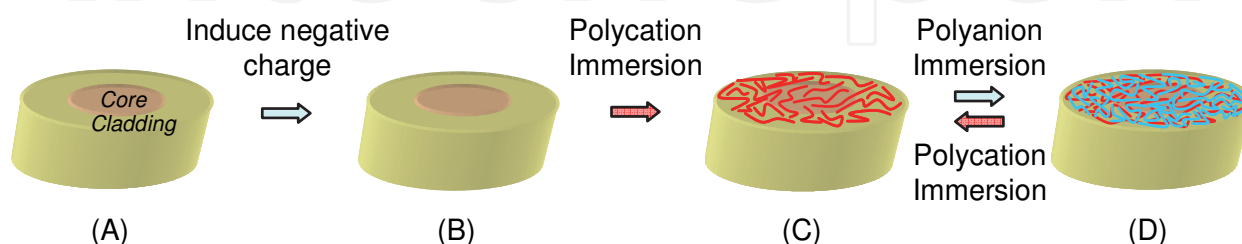


Fig. 9. Main steps of LbL method.

The LbL does not depend on the geometry of the substrate, so it can be used to develop either transmission or reflection sensors. In case of the last ones, the construction process can

be registered thanks to the interferometric nature of that configuration. Every time a monolayer gets fixed, the thickness increases, so that the reflected power changes, recording a response similar to the one of an interferometer as the layers are deposited. The thickness of each individual layer is at nanometric scale, which allows the growth of the nanocavity to be studied by the Ray Theory (Arregui et al., 1999). Employing the experimental set up shown in Figure 10, the construction process can be controlled: plotting the reflected power in function of the immersions, an interferometric sinusoidal response is obtained.

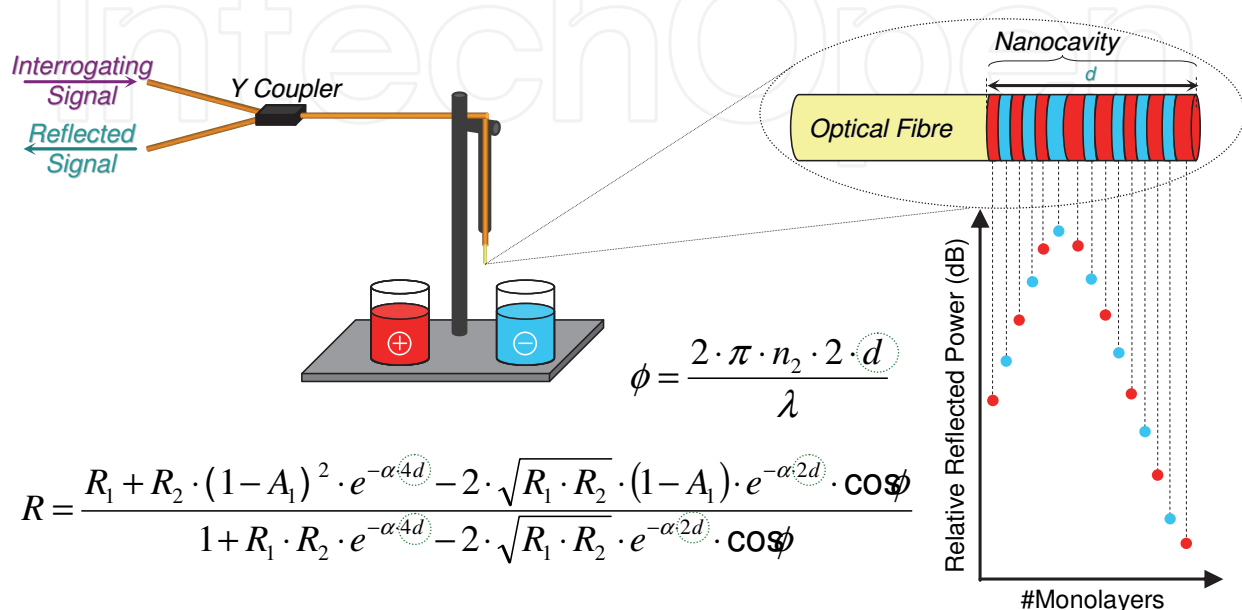


Fig. 10. The nanostructure is supposed to be homogeneous, its optical absorption  $\alpha$ , scattering losses  $A_1$  and refraction index  $n_2$  are constant; therefore,  $R_1$  and  $R_2$  too (reflectivities of the fiber/nanocavity and nanocavity/media interfaces respectively). In this manner, the reflected signal depends on the thickness  $d$  of the structure.

#### 4. Experimental set ups and multiplexing networks

This section will be about the photonic equipment used to study the sensors individually and multiplexed in optical networks. It is assumed that all the devices are intensity modulated: with other modulations, the optical source and detector would show different devices, but the topology would be similar. The multiplexing networks described are supposed to work with reflection sensors, although they can be generalized to any type.

##### 4.1 Individual sensor experimental set ups

Extrinsic sensors are easy to interconnect: the light path is “interrupted” by the cell where the gas to detect interacts with the ray and is coupled again to the fiber. Therefore, the cell is connected between the light source and the receiver. For transmission extrinsic sensors, it is just the same situation: the OFS is connected between the emitter and the receiver (Figure 11). The signal level is much higher than noise, so that it is not necessary to use high sensitivity detectors or high power optical sources.

Regarding to reflection sensors, the interrogating signal must reach the sensor head from the optical source; thereafter, the response signal has to be guided back to the receiver. The

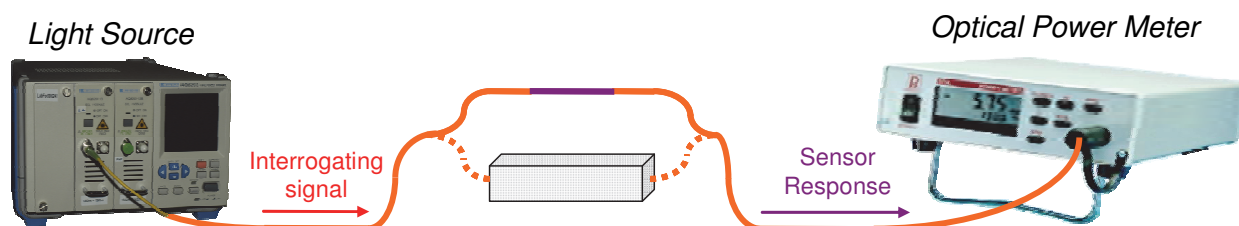


Fig. 11. Experimental set up for an extrinsic sensor (dotted line) and an intrinsic transmission one (solid line).

photonic device most used to achieve it is the optical coupler: typically, it has 4 ports, but for sensing applications, one of them is removed, being called “Y couplers” (Figure 12). These devices are widely used, so it is easy to find one for almost any kind of fiber. On the other hand, there is a device with three ports that can be used to guide back the sensor response introducing only insertion losses: it is the optical circulator. It is able to guide the signal from its port 1 to its port 2 and from that port to its port 3. In this manner, the sensing signal is coupled entirely to the receiver.

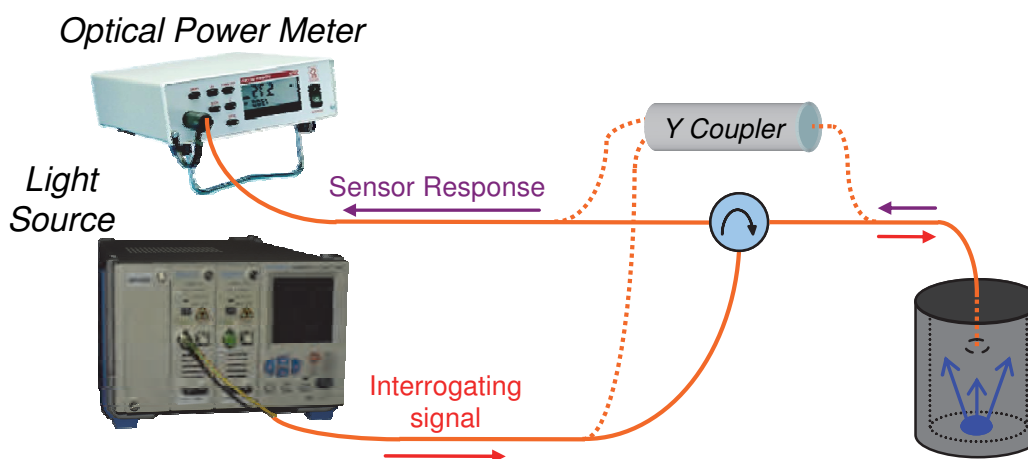


Fig. 12. Reflection experimental set up with an optical coupler (dotted line) and with a circulator (solid line). A thermal jacket is used to ensure isothermal measurements.

#### 4.2 Multiplexing networks

One of the most relevant features of optical fiber is the multiplexing ability. Actually, if several sensors have to be used at the same time, it is critical to share resources such as the optical source, the receiver and the light path. It is important to keep in mind that it has to be done with devices used in telecommunication networks: therefore, the sensors have to be implemented with standard monomode fiber. The configurations to be described are based on the optical wavelength division multiplexer: it is able to multiplex in one direction (join several channels into a single fiber) and demultiplex in the other one (separate several channels into several fibers) (Orazi et al., 1996). Thereby, the interrogating signal can be guided to each sensor through it; meanwhile the responses can be directed back to the receiver. Sensors connected to the multiplexor in this manner follow the *star topology*.

The active components of the system are grouped in a modular way, making the system more robust in case of failures. As an example, the optical source and the receiver can be

place at the same unit, so that they can be repaired or replaced easily. This unit is called *Optical Header*: this a concept widely used in remote sensing applications (Vallejo, 2009).

#### 4.2.1 Wavelength division combined with time division multiplexing

Intuitively, each sensor should be interrogated with a wavelength that matches each channel of the multiplexer. Using one laser per sensor would increase significantly the cost, and thereby, the scalability of the system would be poor. Following the idea of illuminating the sensors with signals at different wavelengths, a tunable laser can be employed. In order to interrogate the sensors, the laser is tuned at the wavelength of one of the multiplexer optical channels, then the signal from the sensor is registered, and finally, the laser is tuned at another channel wavelength. There is no moment when signals at two different wavelengths travel together through the fiber: therefore, an optical power meter can be used instead of an Optical Spectrum Analyzer (Figure 13).

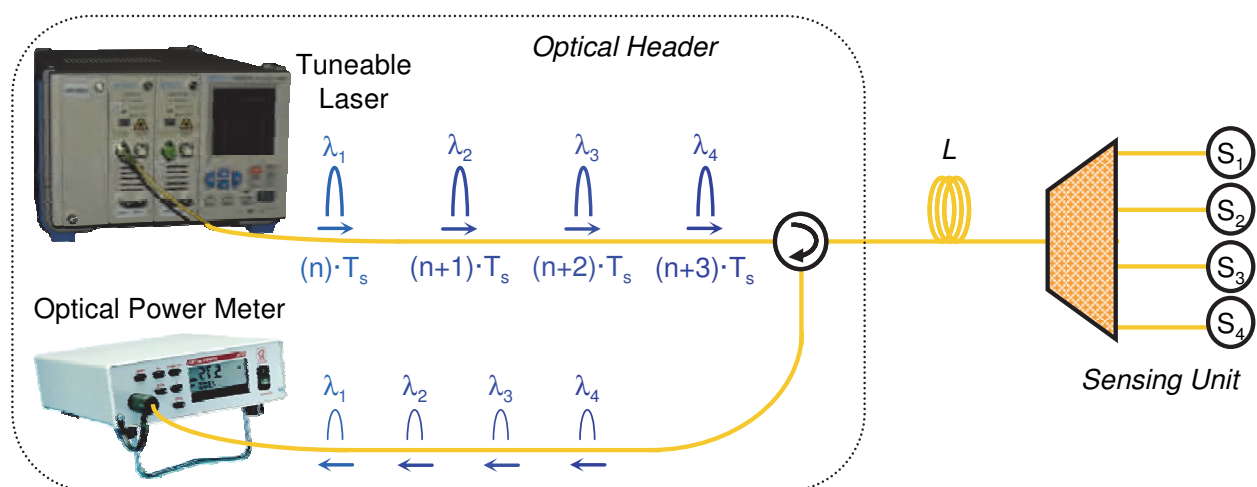


Fig. 13. Experimental set up that combines wavelength and time division multiplexing. Every  $\lambda$  is emitted at multiples of sampling period  $T_s$ , as it is shown in the figure for the  $n^{\text{th}}$  sampling cycle.

The experimental set up combines Wavelength Division Multiplexing (WDM) in the sensing unit with the Time Division Multiplexing (TDM) of the tunable laser. This configuration is limited by the sampling rate: more specifically, by the tuning speed of the laser. A low sampling rate would produce an information loss. It would not be a problem in sensors whose recovery or response times were slow or when they could be characterized only with the power level; nevertheless, it is a great drawback in real time applications.

#### 4.2.2 Multiplexing based on a white light source

The WDM technique allows all the sensors to be interrogated and sampled at the same time; moreover, all the signals share the optical path. It is better to use only one optical source than one per sensor to keep the scalability of the system. Taking advantage of the filtering capability of the multiplexer, a white light source can be used to illuminate the sensors: the multiplexer will filter the broadband signal towards the sensors and it will guide back the narrow band responses of the sensors centered at each optical channel. The number of sensors could be increased using narrower channels and/or wider spectral sources.



Since all the signals share the optical path, they have to be separated at the receiver. The most economical solution consists of using a demultiplexer in the optical header whose channels match the ones from the multiplexer in the sensing unit. This configuration sets out two problems: first, as many receivers as the number of sensors are needed, and second, the demultiplexer has to match its optical channels with the multiplexer ones. One alternative to overcome these problems consists of coupling the multiplexed signals into an OSA. The responses from the sensors can be obtained sampling the spectra registered by the OSA at the wavelengths where the channels of the multiplexer are centered (Figure 14). The drawback of using an OSA is its high cost compared to optical power meters.

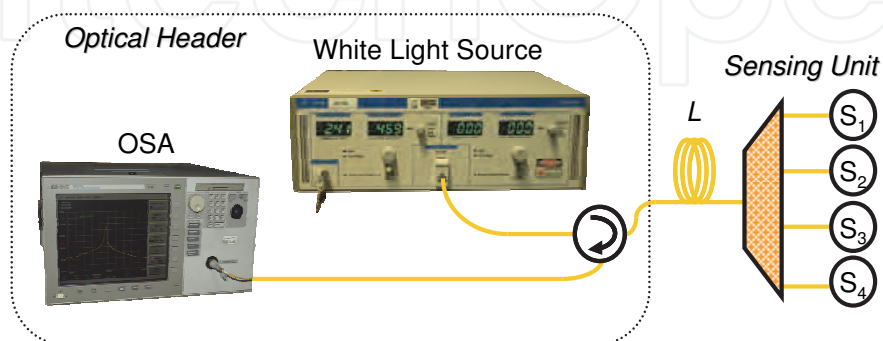


Fig. 14. Experimental set ups used for sensors multiplexing with a white light source.

#### 4.2.3 Multiplexing network architectures

The number of sensors in the array based on the star topology is limited by the number of its optical channels of the multiplexer. The configuration drawn in Figure 14 can handle several sensors but just one sensing unit. There are some applications where it might be necessary to work with several sensing units at the same time: as an example, sensors arrays might be placed in the fermentation tanks of a cellar. In this situation, the optical signal has to be distributed among all the arrays, which can be done only using optical couplers. The sensing units can be multiplexed with the different topologies (star, tree, single bus, ladder): the optimum one depends on the characteristics of the topology (number of arrays, types of sensors and multiplexing technique) and on the requirements of the final application (physical distribution of the points where the arrays are to be placed).

Let us suppose that the arrays are connected in a single bus, which is drawn in Figure 15. The optical header contains the optical source, the receiver and the circulator, as before: now, the broadband signal has to be distributed to all the sensing units by the couplers. Thereby, each one of them couples part of the signal to the array and the rest forward to the bus in order to interrogate the other units. The optimum ratio of the optical couplers (50:50, 90:10, 95:5, 99:1) depends mainly on the number of sensing units (Abad Valtierra, 2002).

### 5. Signal processing and data mining techniques

The response of any sensor when expose to the VOCs sample or mixture is sampled in almost every case: in this way, it can be stored and processed. Although this section will handle with intensity modulated sensors, it can be applied to any other modulation. If the concentration of a certain VOC has to be detected, a parameter that shows a linear relationship with it is typically used (intensity, wavelength shift, just to mention some).

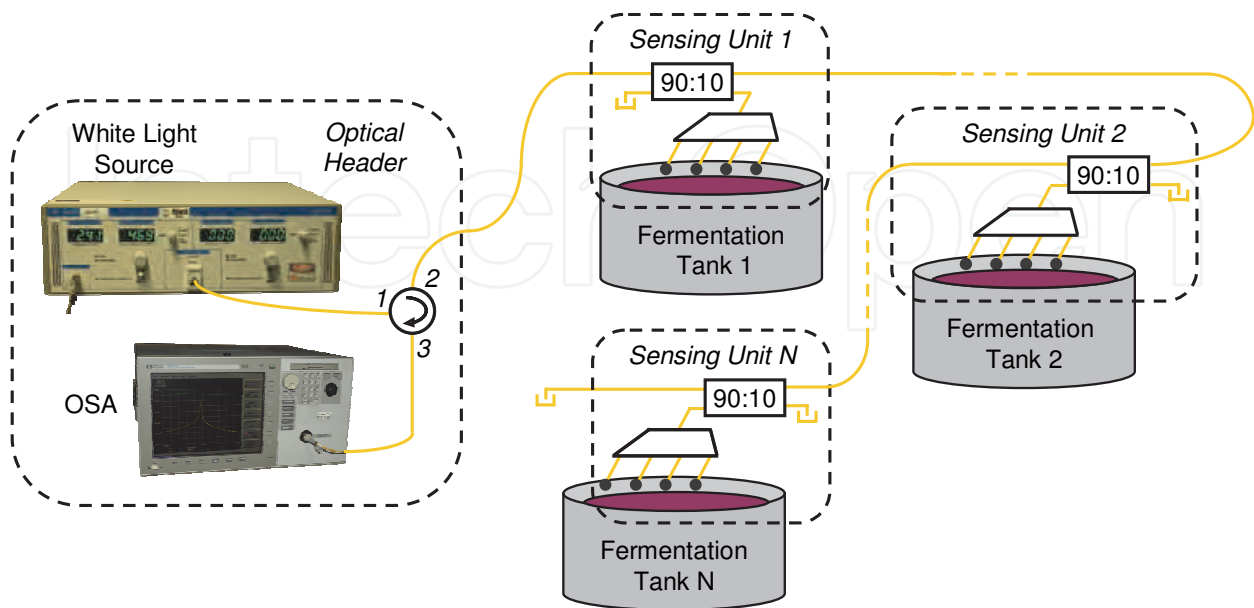


Fig. 15. Example of a network in which the arrays are connected in a single bus topology and the sensors of each unit follow a star configuration.

Nevertheless, if the sample is to be identified and not quantified, it is not necessary a linear response: either linear or not linear data mining techniques can be used to extract features from the sensor signal and so, to identify the sample.

### 5.1 Spectral analysis and temporal response

The signal detected from the sensor can be registered in terms of optical power along the time (with an optical power meter) or converted into the frequency domain (by an OSA). The last option is employed with broadband signals or when several sensors are wavelength multiplexed. The shape of this spectrum can be used to identify different VOCs or mixtures, and its amplitude can determine the concentration of a certain volatile compound. Figure 16 illustrates three examples of signals converted to the frequency domain of some sensors exposed to organic vapors. In these cases, the spectra are compared before and after the exposition to the organic vapors. This kind of measurements is known as *Absorbance Spectra*, and it is expressed in dB.

Another manner to obtain information from a spectrum consists of monitoring the amplitude at a certain wavelength: moreover, it can be achieved by using an optical source centered at that wavelength combined with a power meter as receiver. The optical power variation or response/recovery times of these signals are used to characterize the response of the sensors. As an example, in Figure 17 are plotted the signal fluctuations of a sensor in presence of different concentrations of organic vapors individually: there is a linear relationship between the power variation (in dB) and each vapor concentration.

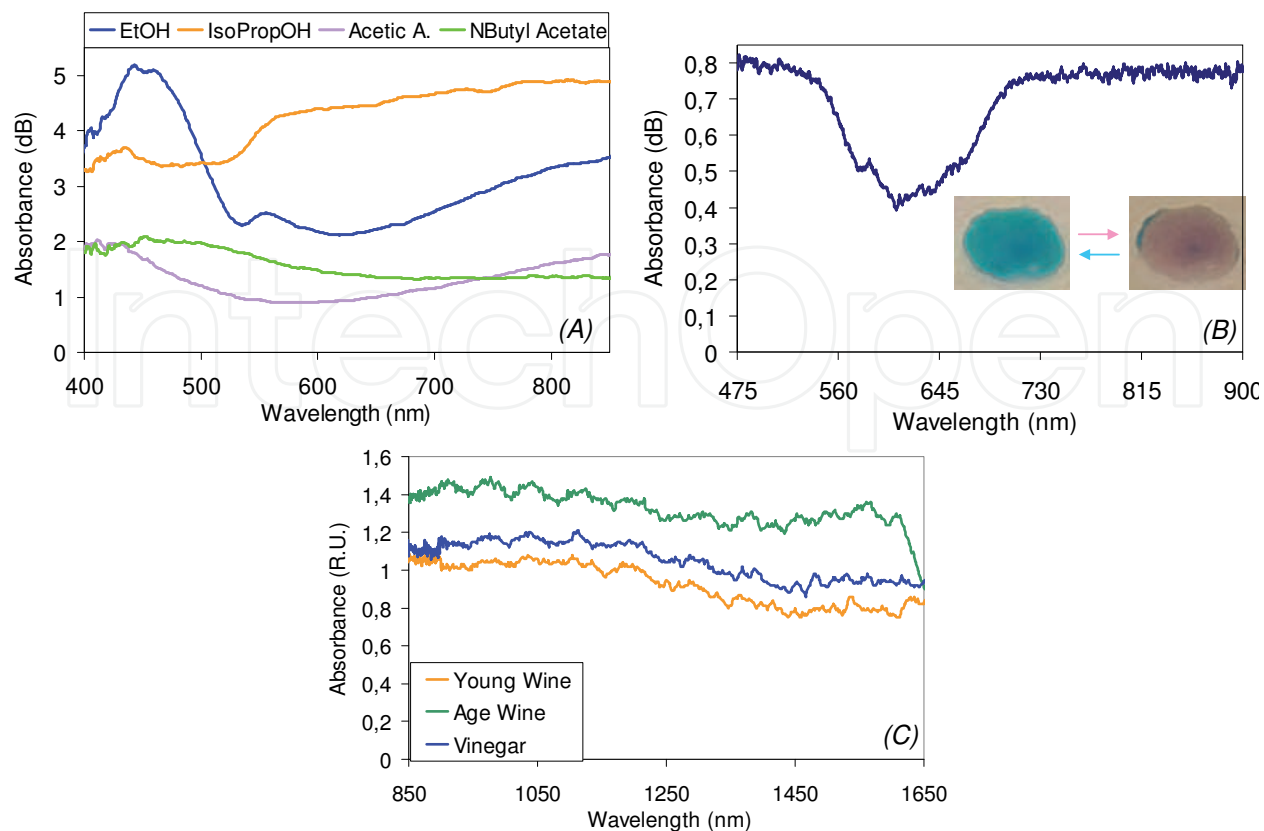


Fig. 16. Different spectra from distinct sensors: (A) signals registered for a sensor when exposed to different VOCs in the VIS; (B) color change from a sensing material shown in its absorbance spectrum; (C) absorbance spectrum in the NIR for different wine related drinks.

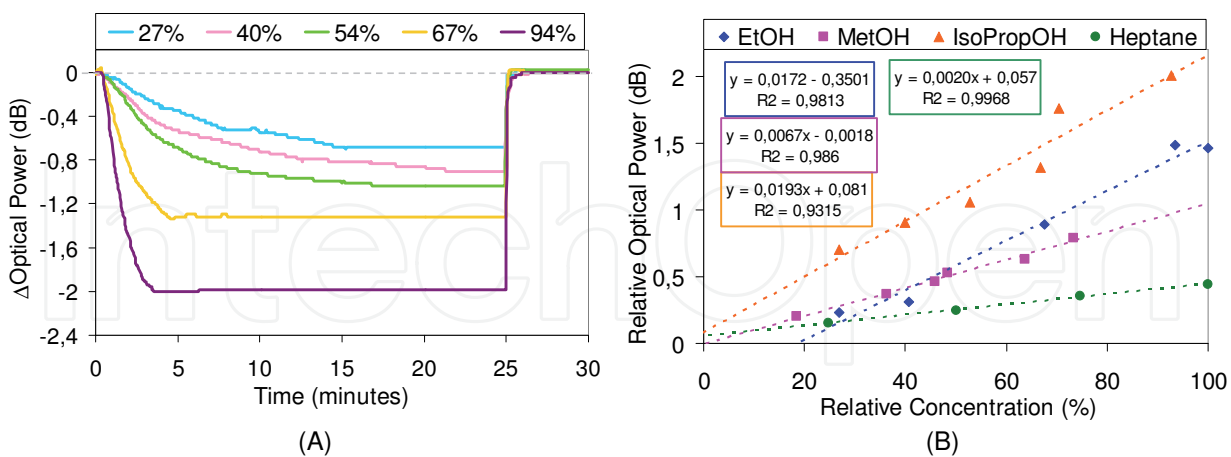


Fig. 17. (A) Signals from the sensor in presence of different isopropanol concentrations and (B) linear approximations for that sensor for distinct vapors.

## 5.2 Principal component analysis

There is abundant information available in the spectral or in the temporal response of the sensor: it can produce a high computational cost. It exits a linear procedure to compress

information that reduces the number of parameters to use, which is the Principal Component Analysis (PCA) (Penza&Cassano, 2003). This transformation is used either with discrete temporal (Guadarrama et al., 2004) and spectral signals (Perez-Hernandez et al., 2009). PCA is based on the idea of removing redundant information: it can be achieved assigning the whole covariance or the cross correlation of the input space to a few components. Thereby, each transformed principal component stores an amount of the initial information. The principal components chosen contain thus a percentage of the total covariance, but not 100%: this difference is the resulting error of the dimensional reduction. Another important factor is how the information is distributed along the components, or better said, the weight that they have: typically, the principal components are ordered from the highest to the lowest weight. Once the PCA components are placed in the transformed space, they can be clustered to identify groups that show similar characteristics. Therefore, PCA is used in applications related with classification of VOC samples (Boholt et al., 2005).

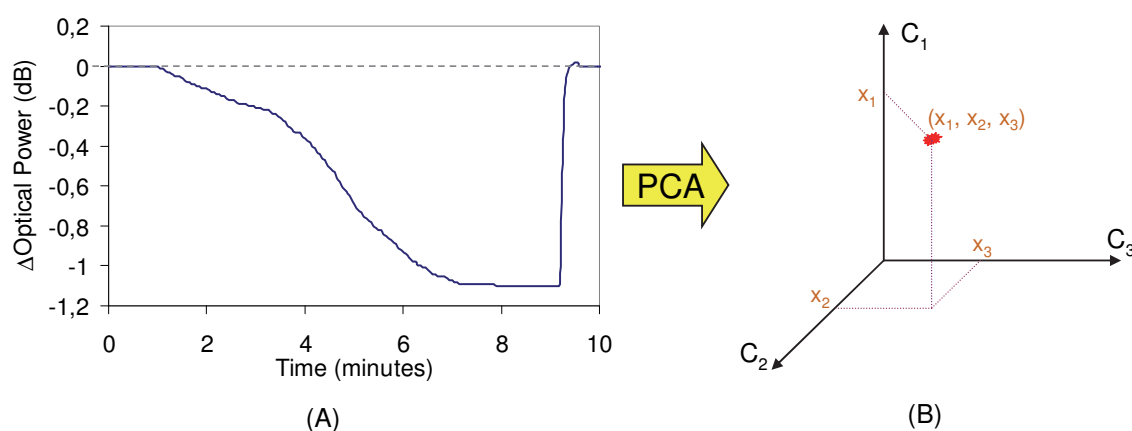


Fig. 18. Conversion of a temporal response (A) into a three dimension space (B) by PCA.

### 5.3 Artificial neural networks

Whereas PCA is a linear transformation, Artificial Neural Networks (ANN) are non linear algorithms that process information in a manner inspired by brain behavior (Shaham et al., 2005). In its most general form, an ANN is a machine designed to model the way in which the brain performs a particular task or function. Neural networks use a massive interconnection of simple computing cells referred to as neurons. An ANN is similar to the brain with regard to acquiring knowledge from its environment through a learning process, and using interneuron connection strengths (synaptic weights) to store this knowledge. There are several types of ANN, and even nowadays, new models are being optimized. Among all of them, one of the most used so far is the Feed Forward Network (FFN) to process the input information. The network has also to be trained: a popular learning algorithm employed to train the ANN is Back Propagation.

The basic unit of an ANN is the neuron: it is an information-processing unit that is fundamental for neural network operation. In Figure 19, it is shown the model of a neuron which can be used in a large family of neural networks. The basic elements are: a set of synapses, each of which is characterized by a weight of its own; an adder for summing the input signals weighted by the respective synaptic strengths and an Activation Function (non linear but differentiable) for limiting the output amplitude of a neuron.

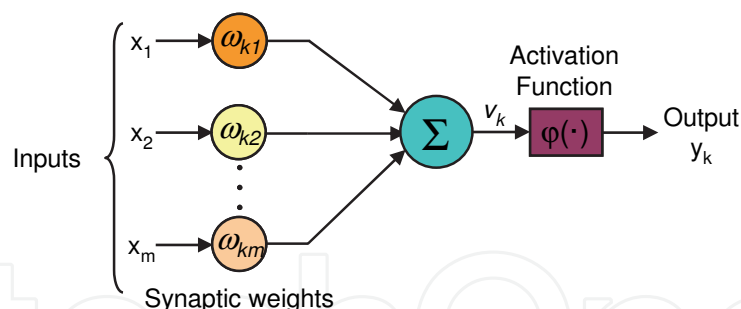


Fig. 19. Nonlinear model of a neuron labeled.

The training stage is performed in an iterative way. Firstly, the synaptic weights of the ANN are randomly initialized; then, the input data feeds the network and the resulting output is compared with the desired one. This error (back propagated) is used to correct the synaptic weights, repeating the process with another sample of the training subset. There are advanced ANNs with no training stage, but this FFN and the Back Propagation Algorithm offer a general idea about how neural networks compute data. Thanks to their learning ability, ANNs have been widely used with VOCs identification tasks (Luo et al., 2004)

## 6. An application related to VOCs identification: Wine fermentation process

After showing the most important factors related to OFSs that handle with VOCs, a specific application will be described taking into account all the topics exposed so far. Artificial systems able to identify odors can take advantage of the optical fiber features. In these applications, several sensors are grouped in arrays: this amount of information can be transmitted through multiplexing configurations. The data processing has to be optimized because there is much redundant information available. In this section, a system able to distinguish between grape juice, wine and vinegar will be described. Although this task might seem trivial, it is remarkable that the system would allow the fermentation process to be monitored on line and in real time, which is hard to do with traditional methods.

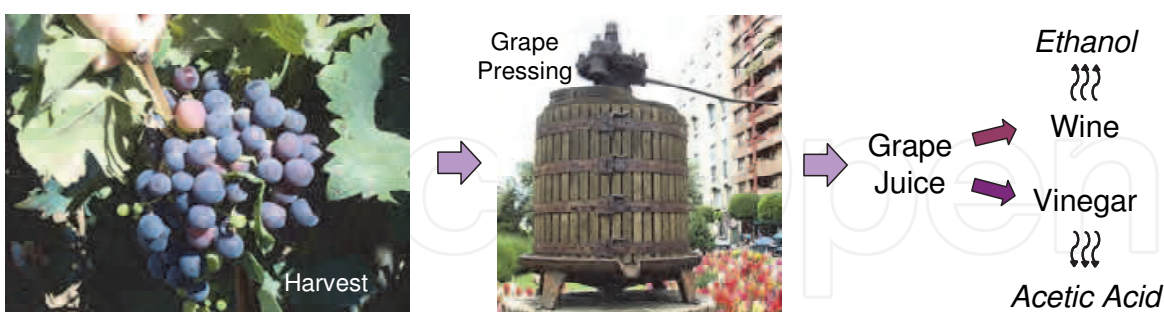


Fig. 20. Some VOCs present along wine fermentation. They characterize each stage: thereby they can be used to identify each one of them.

### 6.1 Preparation and multiplexation of the sensors

Although reflection sensors show a low signal level, the high mechanical robustness makes them a good choice for this type of application. It is compulsory to use singlemode fibers if the sensors have to be multiplexed, so it could lower the sensitivity of the devices: the core of this fiber is much smaller than the multimode ones. This inconvenience can be overcome



by the LbL method: the morphology of the depositions can be altered, so that the roughness is increased, improving the interaction between the VOCs and the sensing materials. Organometallic compounds that suffer changes on its optical properties in presence of organic vapors were chosen as sensing materials. These products belong to a family of products this chemical structure but each one show a distinct molecular marker: it ensures a different selectivity for different VOCs (Luquin et al., 2005). Furthermore, these variations are reversible, which guarantees real time monitoring. Up to 4 different sensing materials are used to develop 4 devices: Sensor A, Sensor B, Sensor C and Sensor D (Figure 21).

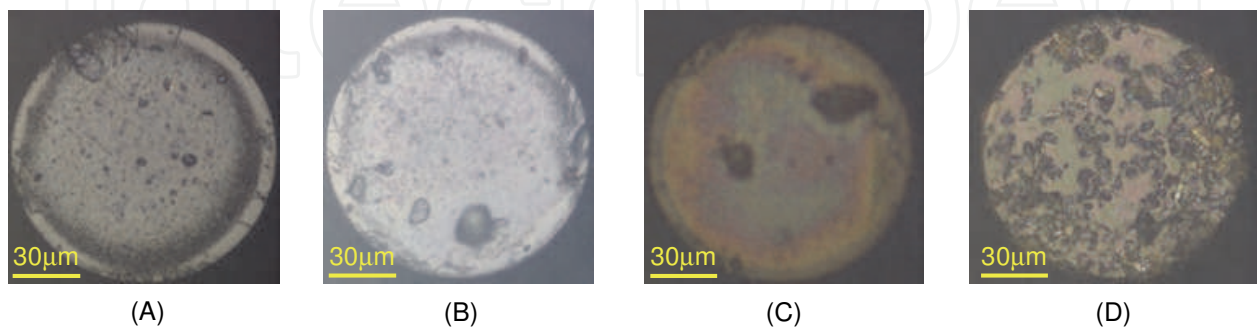


Fig. 21. Plan images from the sensors prepared following LbL to identify the drinks.

The sensors developed are exposed to 1 mL samples of grape juice, wine and vinegar individually. The initial experimental set up uses a laser at 1550 nm, an optical circulator and a power meter. The responses for each drink are plotted in Figure 22.

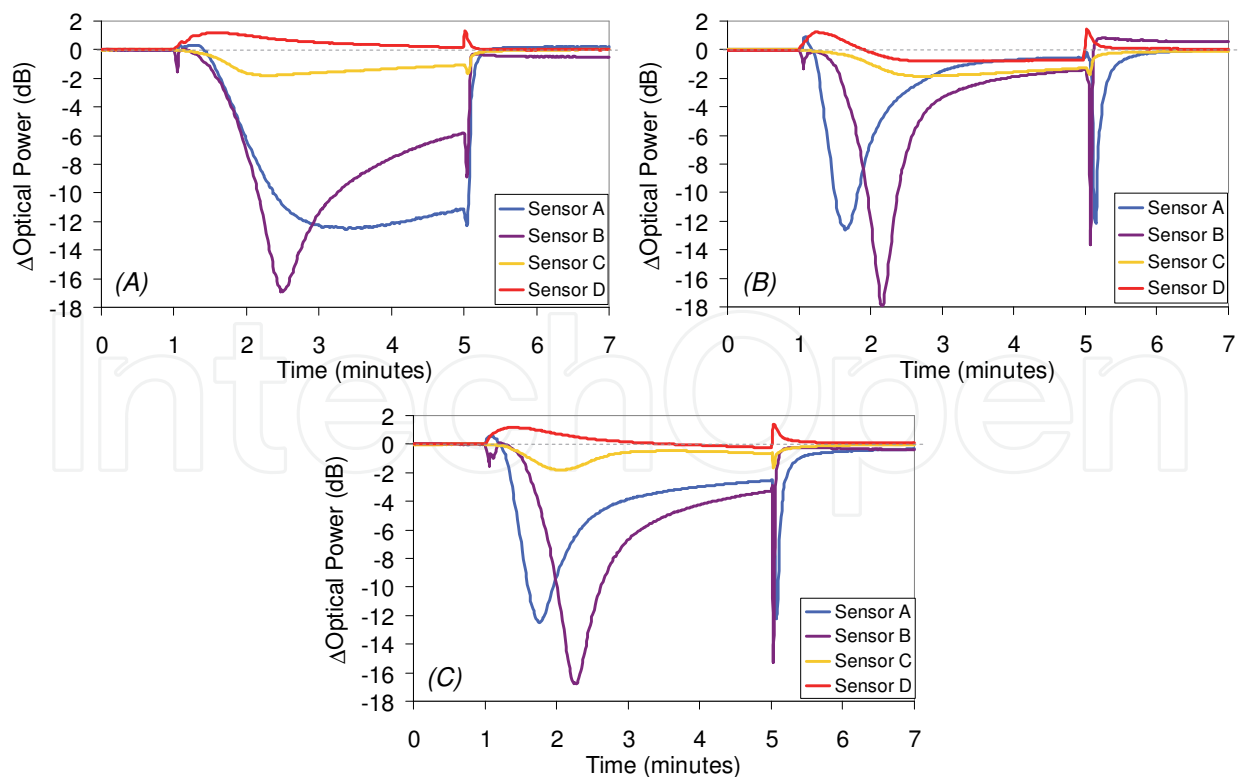


Fig. 22. Temporal responses in terms of optical power from the sensors for each beverage: (A) grape juice, (B) wine and (C) vinegar.

The four devices are connected following a star configuration with an optical multiplexer. As was described in section 4, the array is linked to an optical header through a circulator; inside the header are located a white light source and an OSA (as in Figure 14).

### 6.2 Processing the array information

Looking the responses from the sensors shown in Figure 22, parameters such as the recovery time or the signal variation could be used to identify the different beverages. Nevertheless, as intensity modulated devices, power fluctuations due to the optical source or to the aging of the sensors, might alter these factors. In order to check these inconveniences, a total of 100 samples per drink along 1 month were recorder from the sensor array (Figure 23). It can be observed that for all the sensors there are severe optical power fluctuations that make impossible the identification of the drinks.

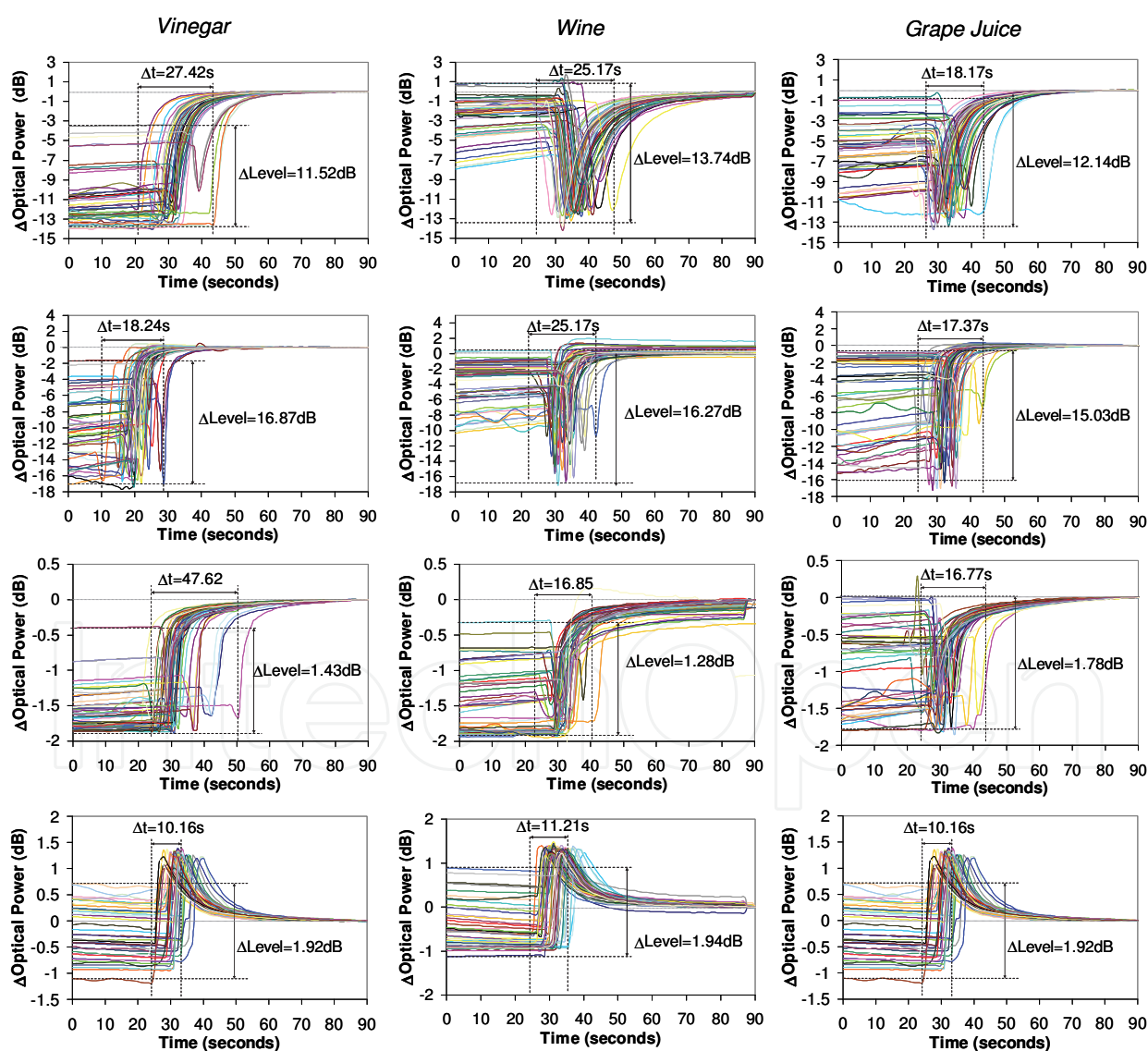


Fig. 23. Recovery responses from 50 samples per each drink and sensor. Each row corresponds to a different sensor: from up to down, Sensor A, Sensor B, Sensor C and D.

It is evident that a more robust data mining is needed to overcome the fluctuations observed in Figure 23. Processing the transitories by an ANN or by PCA is still low efficient, in terms of computational cost. Actually, the transitory is a quick change in the signal level, so that the most relevant information can be obtained by applying the Fast Fourier Transform. This transformation is also a linear operator: once computed, the spectrum of the signal can be shortened eliminating redundant coefficients. The most relevant information is in the low frequencies: therefore, the sequences can be truncated after the second lobule. In any case, the reduced spectrum sequences are still too heavy to be computed: PCA can be applied to compress this information into a three dimension space. The combination of the sensors responses makes the system more robust, but as there might be several fluctuations in the signals, these two linear transformations might not be able to handle with them. The non linear nature of ANN is a good option to deal with this kind of signals. In this way, the whole data set transformed into the frequency domain and then compressed by PCA, can be used to train and test an ANN. Moreover, the network can be designed to have 3 outputs, one per drink to identify, which will behave as binary indicators.

To sum up the complete data mining computation, the recovery response of the sensors is transformed and processed along the data mining process. Initially, the sensor array returns 320 samples from the signals of the 4 sensors, and eventually, they become a 3x1 binary vector that indicates the beverage the array has been exposed to. The process is summarized in Figure 25 for a wine vapors test.

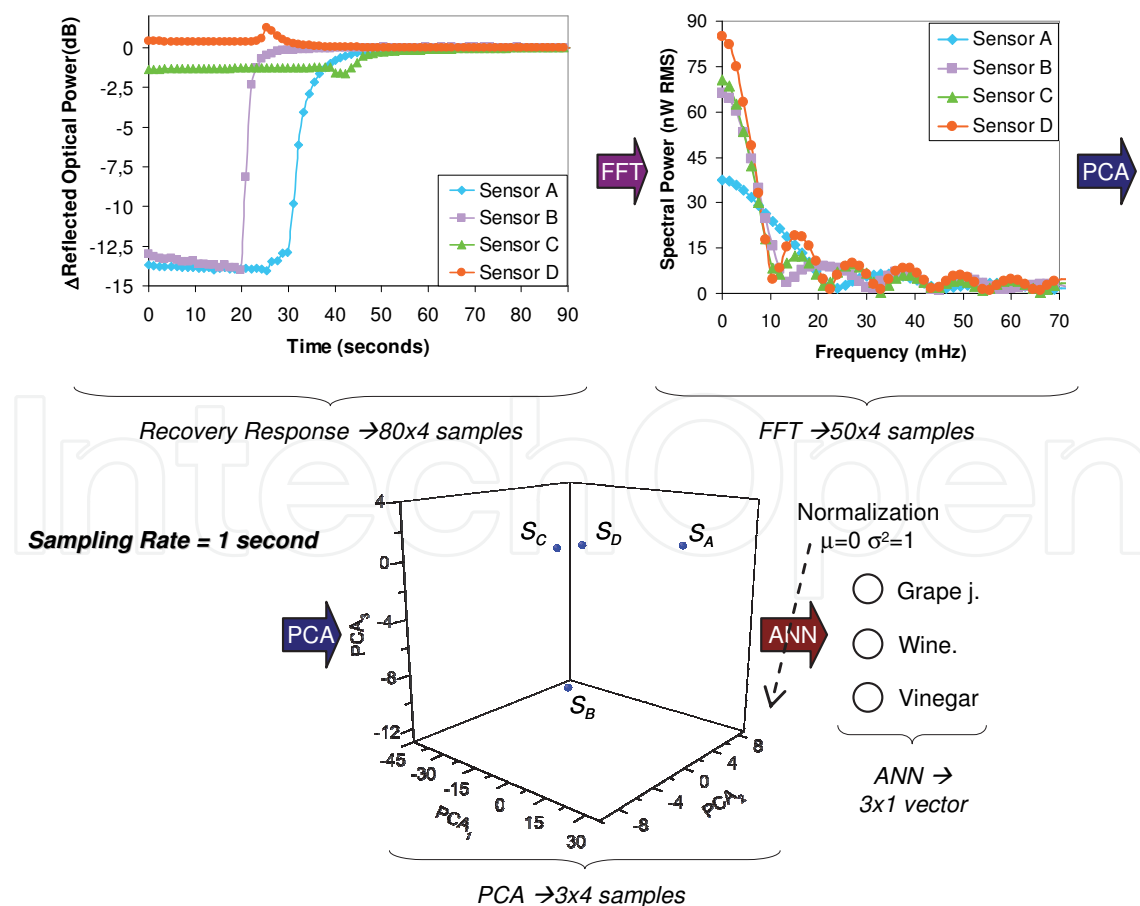


Fig. 24. Scheme that resumes the data mining process of the sensor array.

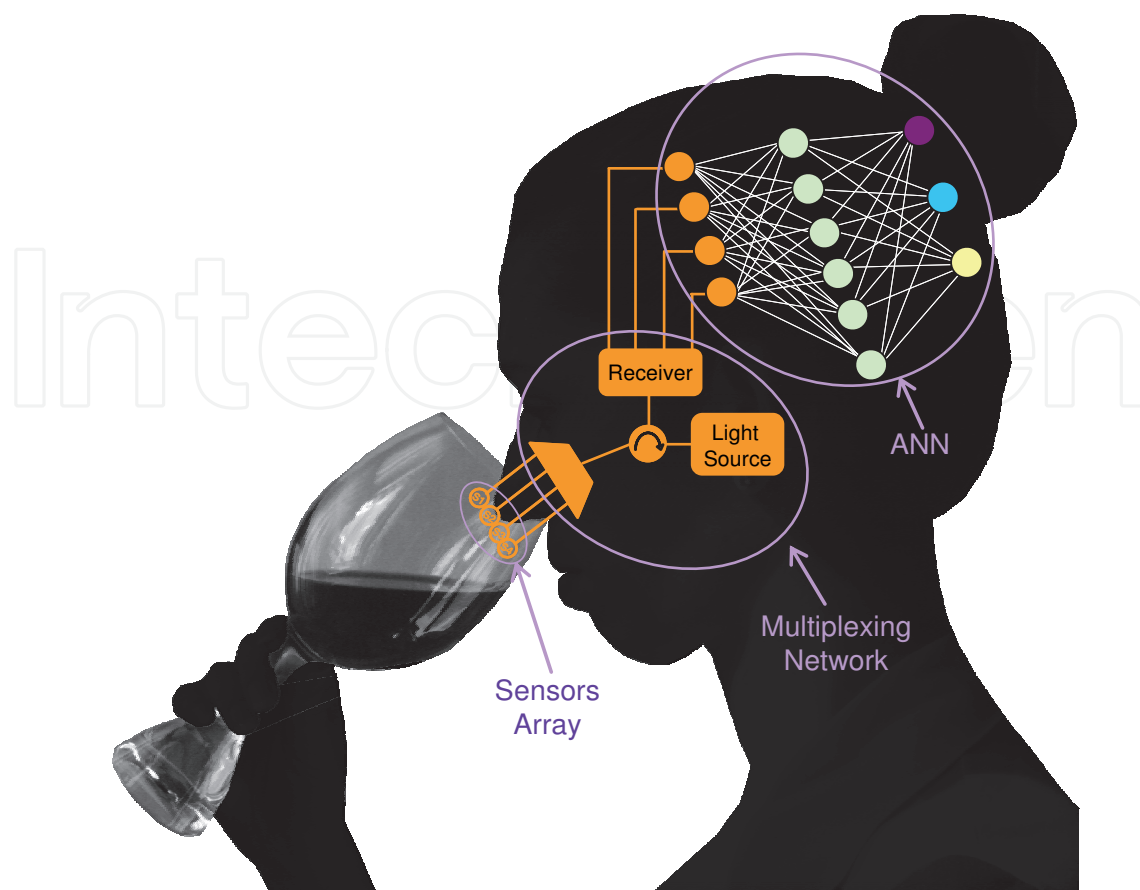


Fig. 25. Analogy between the sense of smell and the optoelectronic nose, together with the blocks involved in the odor identification.

### 6.3 Features of the whole system

The system described in this section is able to identify the most important stages during the wine fermentation process. It is just one of the potential applications where this technology can be used. In this specific case, the device allows the state of the beverage fermentation to be followed in situ with a maximum delay of 4 minutes. In contrast, traditional analyses require the samples to be taken from the fermentation tanks and brought them to a laboratory, where the alcoholic grade and volatile components are analyzed separately. Nowadays this second option is still preferred, mainly in small installations, and it is also more precise from an analytical point of view. The photonic equipment would be lower cost by multiplexing several optoelectronic noses in one single network. Then, other important magnitudes, such as temperature, humidity, CO<sub>2</sub> or O<sub>2</sub> could be controlled too, taking advantage of the transparency of this type of networks. The VOCs detection based on OFSs can be improved and extended in the future to other applications in the food industry: in fact, some researchers are already focused on it (Garcia, et al., 2003).

The final system can be divided into three blocks: the sensors array, data transportation and data mining (Brezmes et al., 2000). Making an analogy with mammalian sense of smell, the first one would correspond to the olfactory cells placed in the pituitary; the second one acts as the nervous system that transfers the information from these receptors and the third is the brain, where the processing occurs. It is illustrated in Figure 25.

The block configuration makes it scalable, modular and versatile. Thereby, should one constituent be damaged, only the affected part need to be repaired or replaced, while the others can keep on working properly. Even in the case of the array, if a sensor fails, it could be replaced without compromising the behavior the other devices. Regarding to the versatility, other types of sensing configurations can be integrated in the same network, whether they are discrete or distributed and it even could transmit others types of data.

## 7. Conclusions

There are several solutions in the VOCs detection field based on electronic sensors: some of them are even commercially available. Even so, there are certain environments where the optical fiber intrinsic features offer important advantages. In the VOCs market niche, optical fiber sensors show a great potential. Electromagnetical immunity, no biasing signals (and no explosion risk) or remote sensing are just some the most relevant features. There is not an optimal configuration to handle with VOCs; actually, there are several different sensing architectures depending on the requirements of the final application. Extrinsic sensors are optimal to detect gases individually with a relatively low interference from other volatile compounds. On the contrary, intrinsic sensors need a sensing material that provides them selectivity to certain VOCs. There also other factors to be taken into account such as the kind of modulation or the type of fiber: all these design parameters have to be chosen depending on the measuring conditions and requirements.

Remote sensing and multiplexing are very useful in VOCs applications. The block design that distinguishes between optical header and sensing unit allows measurements to be done in dangerous places; moreover, the scalability also permits several sensing units to be connected to the same network. Transparency is another important factor: sensors measuring different VOCs or any other parameters such as temperature and humidity can share the same light path, optical source and receiver. Among all the potential market niches, wine fermentation monitoring can take advantages of all the properties exposed in this chapter. The samples have to be analyzed qualitatively, so intrinsic sensors are the best option to handle with complex mixtures of VOCs. Up to 20 sensors could be multiplexed in a single array with the current technology, and of course, several arrays could be connected also to the same bus. Furthermore, thanks to the optical fiber low attenuation, the network might be implemented in medium size cellar, for instance.

Nowadays, it is evident that photonic technology is essential in communications networks. There also some sensing applications that have shown a great utility and are used in many environments. The success of optical fiber sensors to detect and identify VOCs will be determined by the requirements of the market. The best strategies have to find the environments and applications where these sensors offer features that other technologies do not, making them an attractive and affordable option.

## 8. Acknowledgments

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This book presents a comprehensive account of recent advances and researches in fiber optic sensor technology. It consists of 21 chapters encompassing the recent progress in the subject, basic principles of various sensor types, their applications in structural health monitoring and the measurement of various physical, chemical and biological parameters. It also highlights the development of fiber optic sensors, their applications by providing various new methods for sensing and systems, and describing recent developments in fiber Bragg grating, tapered optical fiber, polymer optical fiber, long period fiber grating, reflectometry and interferometry based sensors. Edited by three scientists with a wide knowledge of the field and the community, the book brings together leading academics and practitioners in a comprehensive and incisive treatment of the subject. This is an essential reference for researchers working and teaching in optical fiber sensor technology, and for industrial users who need to be aware of current developments and new areas in optical fiber sensor devices.

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