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Array of Love-wave sensors based on quartz/Novolac to detect CWA simulants

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ABSTRACT

An array of Love-wave sensors based on quartz and Novolac has been developed to detect chemical warfare agents (CWAs). These weapons are a risk for human health due to their efficiency and high lethality; therefore an early and clear detection is of enormous importance for the people safety. Love-wave devices realized on quartz as piezoelectric substrate and Novolac as guiding layer have been used to make up an array of six sensors, which have been coated with specific polymers by spin coating. The CWAs are very dangerous and for safety reasons their well known simulants have been used: dimethylmethyl phosphonate (DMMP), dipropyleneglycol methyl ether (DPGME), dimethylmethyl acetamide (DMA), dichloroethane (DCE), dichloromethane (DCM) and dichloropentane (DCP). The array has been exposed to these CWA simulants detecting very low concentrations, such as 25 ppb of DMMP, a simulant of nerve agent sarin. Finally, principal component analysis (PCA) as data pre-processing and discrimination technique, and probabilistic neural networks (PNN) as patterns classification technique have been applied. The performance of the sensor array has shown stability, accuracy, high sensitivity and good selectivity to these simulants.

1. Introduction

Chemical warfare agents as distilled mustard (HD), nitrogen mustard (HN) and phosgene (CG), and nerve agents such as sarin (GB) and soman (GD) are potential threats to public health and these weapons can be acquired or produced for criminal purposes. The attacks in Japan with sarin gas in 1994 in Matsumoto and in 1995 in Tokyo subway are a clear example of the risk of these hazardous agents. CWAs can burn and blister the skin or eyes and enter to the body through the respiratory tract being highly lethal and with detrimental effects on health, for that reason a fast and effective system to detect them is needed. Due to that the CWAs are too dangerous and toxic, and they only can be manipulated in especial laboratories, CWA simulants have been used instead of these ones that closely mimic the chemical structures of real CWA without their associated toxicological properties [1–4]. Methods based on different principles have been developed for detecting CWAs, such as chromatography, mass spectrometry or ion mobility spectroscopy [5,6], but the sensors can be other reliable way of detection.

Arrays of acoustic wave devices are widely used in sensing application, such as medical analysis [7,8], environmental fields [9,10] and food quality [11–13]. Some types of acoustic wave sensors [14–19] are: quartz crystal microbalances, devices based on Rayleigh waves, acoustic plated modes, transverse surface waves and Love waves. Detectors based on surface acoustic wave (SAW) devices can be adequate as a civil defense against CWAs. Love-wave devices are a type of SAW devices in which wave acoustic energy is confined in a guiding layer deposited over the piezoelectric surface [20], being the velocity of propagation of this wave very sensitive to perturbations on the surface, such as mass changes, temperature and pressure. Due to high selectivity, sensitivity, fast response, real time detection, stability and low cost, an array of Love-wave sensors is suitable to detect CWAs.

2. Materials and methods

2.1. SH-SAW devices

Shear horizontally polarized surface acoustic wave (SH-SAW) devices consisting of a piezoelectric quartz delay line (DL) were used in this work in order to make Love-wave sensors (Fig. 1). Quartz has a small piezoelectric coupling coefficient, but it is an ideal crystal for the fabrication of stable oscillators and sensors due to its low temperature coefficient at room temperature. In our case, SH-SAW is propagated on the ST-cut quartz perpendicular to the *x* crystallographic axis. This wave is generated by an interdigital transducer (IDT) when an alternating current is applied and it is

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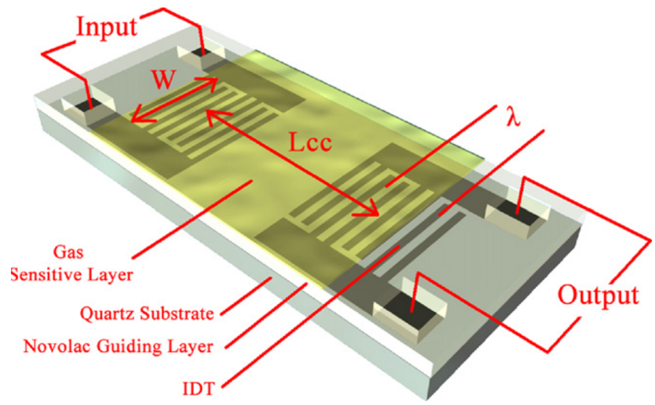

Fig. 1. 3D scheme representing of Love-wave sensor with two RF ports, layer composition, and geometrical parameters.

transmitted through the substrate and converted back to an electric field at the IDT on the other side. They were made by means standard lithographic techniques using aluminium metallization with a thickness of 200 nm deposited by RF sputtering. A structure of four fingers per wavelength ($\lambda = 28\ \mu m$) is repeated 75 times ($N = 75$) to form each IDT. The spacing centre to centre between IDTs, Lcc, is 150 λ and the acoustic aperture, *W*, is 75 λ.

2.2. Guiding layer

Novolac photoresist [21] has been used as guiding layer because this material has low propagation velocity for SH-SAW and is very resistant to chemical agents. The devices were put into acetone and isopropanol for cleaning them before the deposition of guiding layer. Then, different solutions of Novolac photoresist (AZ® nLOF™ 2070) prepared whit the PGMEA solvent (AZ® EBR™ Solvent) were deposited on the device by spin coating (Laurell WS-650SZ) at 4200 rpm for 30 s, in such a way that a high uniformity and low surface roughness were achieved.

2.3. Sensitive layer

An array was made up of six Love-wave devices coated onto guiding layer with different rubbery and amorphous polymers [22–26] as sensitive layers. CWAs, which are volatile organic compounds (VOCs), are adsorbed on polymers, each one with specific sorption properties for the different CWAs, This allows us to classify the different CWA simulants. Each polymer was dissolved in the suitable solvent (Table 1) and then each device was coated with this solution by spin coating at 4200 rpm for 30 s.

2.4. Samples

The CWA simulants used in our experiment were: dimethylmethyl phosphonate (DMMP) as sarin (GB), dipropyleneglycol methyl ether (DPGME) as nitrogen mustard (HN), dimethylmethyl acetamide (DMA) as distilled mustard (HD), dichloroethane (DCE) as soman (GD) or distilled mustard (HD), dichloromethane (DCM) as phosgene (CG) and dichloropentane (DCP) as distilled mustard (HD).

In order to get different concentrations of CWA simulants, a 10 ml volume of the liquid sample was kept at constant temperature (10 °C) in a thermal bath. The vapour pressure of the simulants with regard to the temperature was calculated using Antoine's Equation [27], which allowed us to know the concentration of each VOC in the headspace for a wide temperature range.

After 30 min of headspace time, a first flow of synthetic air, used as carrier gas, was controlled by one mass flow controller (MFC) of range 0–10 ml/min or 10–200 ml/min, which was chosen depending on the flow required for the desired concentration. The VOC concentration was diluted with a second flow of air controlled by other MFC of range 10–200 ml/min in order to achieve a total flow of 200 ml/min transporting the simulant concentration to the chamber where the sensor array was placed.

2.5. Instrumentation and data acquisition for characterization

The acoustic devices were electrically characterized by means of an automatic network analyzer (ANA) Agilent 5070B with the purpose of studying the frequency response. A profilometer (Veeco Dektak with stylus of 2.5 μm of diameter) was used to measure the guiding layer profile and to check its roughness.

The integration of the device in an oscillator circuit leads the oscillation or feedback with a specific frequency, f_0, which can be used as output signal. This frequency is mainly defined by the wave velocity in the device.

The array was made up of six sensors and a reference, for this reason a microwave switch system (Keithley S46) was used to alternate the different signals, making possible their measurement in a unique channel of the frequency counter (Agilent 53131A). Both, microwave switch system and frequency counter, were controlled by a GPIB protocol. The temperature of the sensors was kept at 25 °C, similar at room temperature, using a PID system, in such a way that the temperature was measured with a platinum resistance sensor (Pt100) and controlled by a Peltier device. The sensor temperature, the power sources that fed electrovalves, the flow controllers and the Peltier device were communicated with a computer by a Zee Bee protocol. The experiment control and data acquisition in real time were implemented with a PC by means of software made at home. The experimental setup is shown in Fig. 2.

2.6. Pattern recognitions methods

Principal component analysis (PCA) is a statistical method for reducing the number of dimensions of numerical dataset without much loss of information. Mathematically, PCA projects the data onto a new coordinate base formed by orthogonal directions with data of great variance. The principal components are ordered, thus the first greatest variance is the first coordinate (called first principal component, PC1), the second greatest variance on the second coordinate, PC2, and so on. PC1, PC2 and PC3 allow the visualization of dataset main information in 2-D and 3-D representation. In our

Table 1
Sensor array composition and the corresponding solvent.

Sensor number	Polymer	Solvent	Formula	Supply
0	Reference	–	–	–
1	PECH (polyepichlorohydrin)	Acetone	$[CH(CH_2Cl)CH_2O]_n$	Aldrich
2	PEI (polyethylenimine)	Ethanol	$(C_2H_5N)_n$	Fluka
3	PDMS (polydimethylsiloxane)	Hexane	$(C_2H_6OSi)_n$	Sigma
4	PCPMS (polycyanopropylmethyl siloxane)	Dichloromethane	$(C_4H_{13}ClO_2Si_2)_n$	ABCR
5	CW (carbowax)	Ethanol	$H(OCH_2CH_2)_nOH$	Sigma
6	PMFTPMS (trifluoropropylmethyl siloxane-dimethylsiloxane)	Dichloromethane	$CH_9SiO(SiOC_2H_6)_m\text{-}(SiOC_4H_9F_3)_nSiCH_3$	ABCR

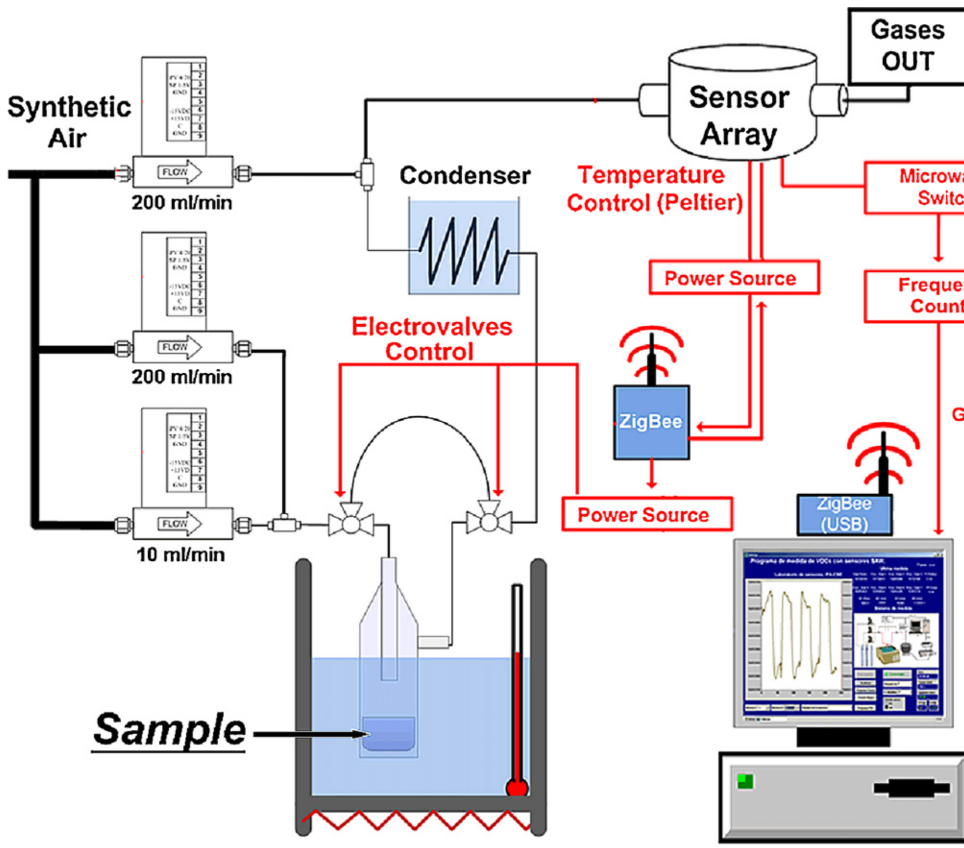

Fig. 2. Scheme of the instrumentation and experimental set up used for the data acquisition in real time.

case, 2-D plot was enough to perceive a good separation among the tested simulants.

A probabilistic neural network (PNN) was applied to the PC1, PC2 and PC3 in order to recognize the type of VOCs patterns under study. Neural networks are mathematical models that process information by means of an adaptive system that changes its structure based on external or internal information that flows through the network during the learning phase. Thus, a PNN creates a function for capturing and representing complex input/output relationships. A PNN was trained, and the performance was evaluated with leave-one-out validation method. This method consists of training N distinct nets (in this case, N is the number of the measurements) by using the remaining vector, excluded from the training set. This procedure is repeated N times until all the vectors are validated.

3. Results and discussion

3.1. Guiding layer deposition

In the development of preparation of these sensors various aspects of the guiding layer must be taken into account, because in this one most of the energy of the wave is transported, and therefore some features of the device depend largely on this film. The operating synchronous frequency, f_0, of the device is directly related to Love-wave velocity, v_L, through: $f_0 = v_L/\lambda$. A Love-wave is obtained when the velocity in the guiding layer material, v_{GL}, is lower than in the substrate, v_S. The Love wave velocity is $v_{GL} < v_L < v_S$ depending on the thickness of the guiding layer. Then the synchronous frequency, f_0, depends on the thickness of this layer (Fig. 3). The velocity of the SH-SAW propagated in quartz ST-cut perpendicular to x crystallographic axis is $v_S = 5060$ m/s, being 180.71 MHz the synchronous frequency in SH-SAW, and the velocity in Novolac is $v_{GL} = 1100$ m/s [28]. The Love wave velocity for thin guiding layers is similar to the SH-SAW velocity in the substrate, but if the thickness increases its velocity is closer to the SH-SAW velocity in the guiding layer material. Several Novolac solutions were prepared as guiding layers with different thicknesses. A profilometer was used to measure the thickness and check the roughness. The transmission spectrum response of each device was characterized by means of an ANA in order to choose the optimum guiding layer thickness. The data were compared with the results of the article of Rasmus-

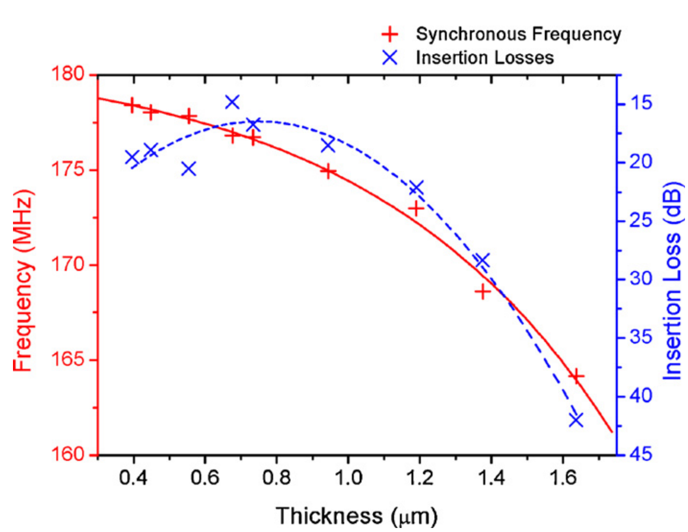

Fig. 3. Effect of the deposition of the guiding layer on the synchronous frequency and insertion losses in air as a function of the thickness.

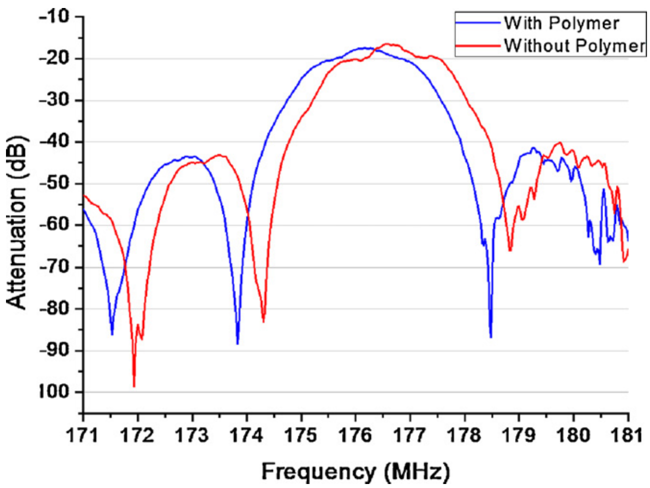

Fig. 4. Amplitude of Transmission S21 for a Love-wave device before and after deposition of PEI as sensitive layer.

son and Gizeli [21] and similar results were obtained. Polymers, such as Novolac, are interesting from the point of view of the sensitivity because they have low velocity for SH-SAW. However they have high acoustic damping, being the main problem when polymers are used as guiding layer. Because of this reason our election of the thickness was based on the lowest insertion losses and an optimum value of 0.8 μm approximately was chosen. The insertion losses for different thickness are shown in Fig. 3.

3.2. Device characterization

The devices were characterized in RF, before and after being coated with the polymer and the response frequency was recorded, allowing to measure the insertion losses and the phase shift. As example, attenuation and frequency shift after the deposition of the polymer PEI are shown in Fig. 4.

3.3. Simulant sample measurements

The sensor array was exposed to series of repetitive measurements of the CWA simulants with different concentrations (Table 2), being the frequency of each device monitored in real time. In general the response of the sensors was fast (from the first measurement the frequency value was shifted), reversible and reproducible with a good linear correlation between concentration and frequency shift. In Fig. 5a is possible to see three different DMMP exposures for each concentration. For 1 ppm the response time and the recovery time to reach an approximately 80% of their frequency final values were about 6 min. The linear correlation between the frequency shift and the DMMP concentration was $R^2 = 0.99927$ (Fig. 5b).

A radial plot of the slopes shows a specific finger print for each simulant, which could be used for a visual differentiation among

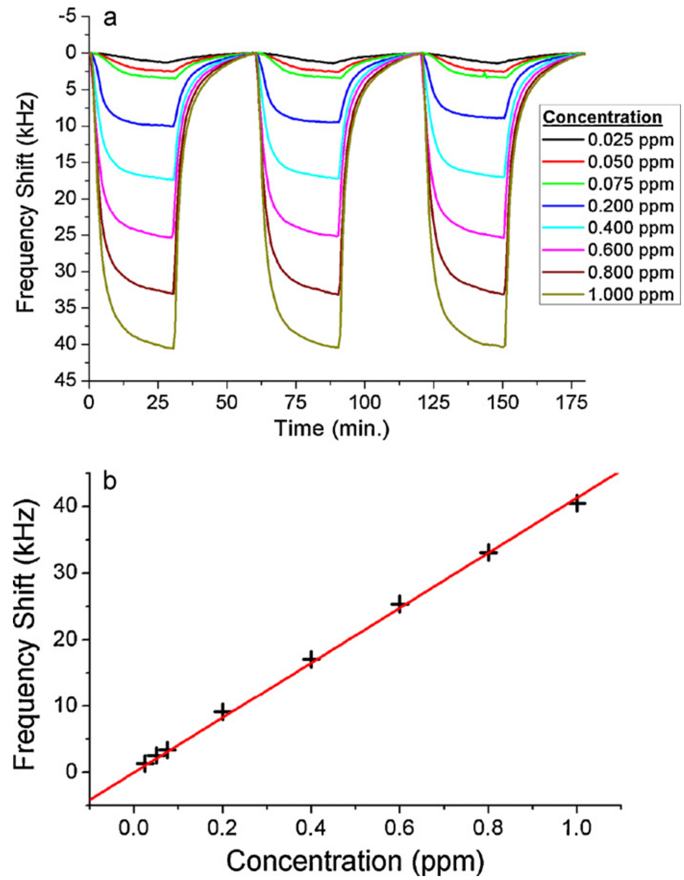

Fig. 5. (a) Real time response of the PECH coated sensor for different concentrations of DMMP. (b) The linear relation between frequency shift and the concentration for the PECH coated sensor and different concentrations of DMMP.

them (Fig. 6). Since the data present very different magnitude orders, the logarithm of the slopes was represented.

3.4. Methods for discrimination and classification

In the PCA the input data were the normalized responses with respect to the concentration, choosing as responses the maximum frequency shifts during exposure time (Fig. 7). The ellipses include the measurements of each simulant, therefore separated ellipses indicate that the CWA simulants can be clearly discriminated. The low scattering of datapoints is attributed to the variance of the frequency shift for the same concentration measurements, which was greater for low concentrations, as it was expected. Good separation is observed among the tested simulants.

The PCA has been used as pre-processing technique, eliminating irrelevant and redundant data and taking into account the essential information. A PNN was applied to PC1, PC2 and PC3 in order to classify the CWA simulants. A 100% classification was achieved.

Table 2
Chemical warfare simulants an the concentrations measurements.

Simulant	Abbreviation	Concentration (ppm)	Formula
Dimethyl methylphosphonate	DMMP	0.025[a], 0.05, 0.075, 0.1, 0.2, 0.4, 0.6, 0.8, 1	$C_3H_9O_3P$
Dipropylene glycol monomethyl ether	DPGME	0.25[a], 0.5, 0.75, 1, 2.5, 5, 7.5	$C_7H_{16}O_3$
Dimethylacetamide	DMA	25[a], 50, 100, 150, 200, 250	C_4H_9NO
1,2-Dichloroethane	DCE	75[a], 100, 125, 150, 200, 250	$C_2H_4Cl_2$
Dichloromethane	DCM	125[a], 150, 175, 200, 225, 250,	CH_2Cl_2
1,5-Dichloropentane	DCP	5[a], 10, 15, 20, 25	$C_5H_{10}Cl_2$

[a] Limit of detection for each simulant.

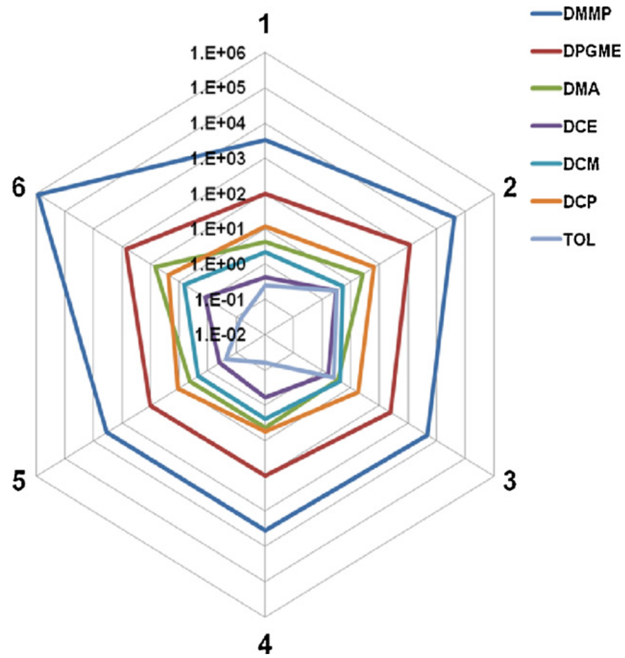

Fig. 6. Sensitivity values (Hz/ppm) in a radial plot. A specific fingerprint for each simulant is formed.

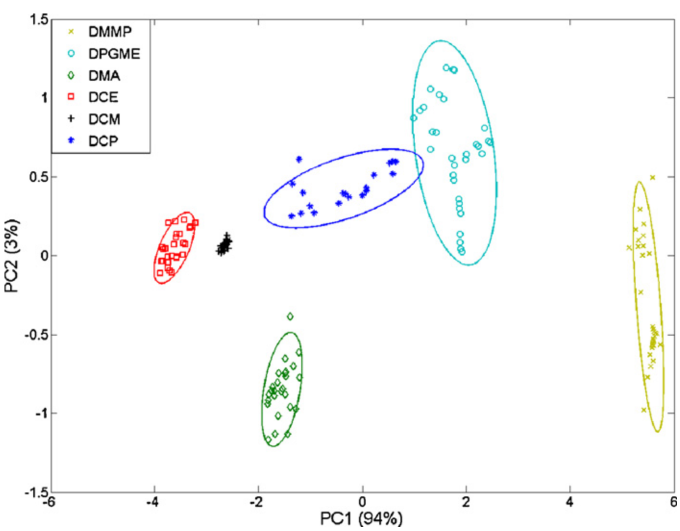

Fig. 7. Principal components analysis applied to data for discrimination of CWA simulants.

4. Conclusions

Our goal has been to develop an array of Love wave sensors based on quartz as piezoelectric substrate and Novolac as guiding layer to detect low concentrations of CWA simulants.

In the development of the device a drawback is the damping of the wave due to of the material used as guiding layer. With regard to this fact, a first study of the Novolac guiding layer thickness has been realized showing that for thicker layer of 0.8 μm the Love wave damping increase suddenly, for this reason the thickness has been chosen with respect to minimize the insertion losses instead of the improve the sensitivity. In any case these sensors have shown very good characteristics with respect to stability, linearity, reversibility, response, repeatability and sensitivity.

This array has detected very low concentrations with very good sensitivity, for instance, it has been possible to measure concentrations as low as 25 ppb of DMMP.

In addition good discrimination and classification of tested CWA simulants have been achieved through PCA and PNN.

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Isabel Gràcia joined the National Microelectronics Centre (NCM) in 1986 working on photolithography. In 1993, she received her PhD in physics from the Autonomous University of Barcelona, Spain, working on chemical sensors, which is also her current research field.

Carles Cané received his BSc degree in telecommunication engineering in 1986 and PhD degree in 1989 from the Universitat Politècnica de Catalunya in Barcelona, Spain. Since 1990, he has been permanent researcher at the National Microelectronics Centre (NCM) in Barcelona. He is currently working on sensors and microsystems and their compatibility with standard CMOS technologies.

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