Talanta 120 (2014) 408–412

Contents lists available at ScienceDirect

Talanta

journal homepage: www.elsevier.com/locate/talanta

Characterization of an array of Love-wave gas sensors developed using electrospinning technique to deposit nanofibers as sensitive layers

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ARTICLE INFO

Article history:
Received 9 October 2013
Received in revised form
8 December 2013
Accepted 11 December 2013
Available online 21 December 2013

Keywords:
Nanofibers
Love wave
Gas sensor
Chemical warfare agent
Sensor array
Electrospinning

ABSTRACT

The electrospinning technique has allowed that very different materials are deposited as sensitive layers on Love-wave devices forming a low cost and successful sensor array. Their excellent sensitivity, good linearity and short response time are reported in this paper. Several materials have been used to produce the nanofibers: polymers as Polyvinyl alcohol (PVA), Polyvinylpyrrolidone (PVP) and Polystirene (PS); composites with polymers as PVA+$SnCl_4$; combined polymers as PS+Poly(styrene-alt-maleic anhydride) (PS+PSMA) and metal oxides (SnO_2). In order to test the array, well-known chemical warfare agent simulants (CWAs) have been chosen among the volatile organic compounds due to their importance in the security field. Very low concentrations of these compounds have been detected by the array, such as 0.2 ppm of DMMP, a simulant of sarin nerve gas, and 1 ppm of DPGME, a simulant of nitrogen mustard. Additionally, the CWA simulants used in the experiment have been discriminated and classified using pattern recognition techniques, such as principal component analysis and artificial neural networks.

1. Introduction

Arrays of acoustic wave devices (AW) [1–3] have widely been used in sensing applications. Some types of acoustic wave sensors are quartz crystal microbalances [4–6], devices based on Rayleigh waves [7–10] and Love waves [11–13]. The AW devices can be functionalized using a great variety of materials as sensitive layers, such as polymers [4–11], metal oxides [13,14], and nanotubes [15,16], which are deposited on the active area of the AW device in order to adsorb the volatile organic compounds (VOC). The morphology of the sensing layer plays an important role in the molecular adsorption–desorption process, sensor response and therefore in the sensitivity. In recent years, nanostructured materials have been under research in many fields and are also promising to be used as sensing materials due to their large surface to volume ratio which provides high surface area. The nanostructured materials can be used as sensitive layers in AW devices instead of a thin layer, improving the sensitivity and velocity of the response due to their great reaction surface. Therefore, sensitive layers of nanostructured materials can provide higher sensitivity and lower insertion losses than the sensitive thin films coated with methods such as drop, airbrush spray or spinning.

Several reports about nanofibers as sensitive layer of AW sensors have been published [17,18] and there is an increase in the use of the electrospinning technique to deposit them on AW sensors [19–21]. The electrospinning is a simple, efficient and low cost method to deposit nanofibers of very different materials [22–28] since the selectivity and sensitivity of a sensor to a target VOC are very dependent on its chemical and physical properties. Therefore, it is possible to use combinations of a great variety of materials as sensitive layers in order to make efficient arrays of sensors.

The combination of Love-wave devices with different elesctrospun nanofibers can provide substantial advantages to detect VOCs, such as higher sensitivity and selectivity, and lower insertion losses of the devices. However, as far as we know, there are no references of the use of an array of Love-wave devices with electrospun nanofibers to detect VOCs; therefore, it has been made an array consisting of six sensors with different electrospun nanofibers as sensitive layers.

Among the VOCs, chemical warfare agents (CWA) such as nerve, vesicant, incapacitating, lacrimator or emetic agents have been selected, since they are powerful weapons used to kill or incapacitate humans, being a threat to public safety. Due to the above reasons, there is an urgent interest in developing highly

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http://dx.doi.org/10.1016/j.talanta.2013.12.027

sensitive, selective and quick devices to make an early detection system that is capable of detecting low concentrations of CWAs. Therefore, the array of AW sensors, which has been made in this work, has been tested with different concentrations of CWA simulants below the median lethal dose (LD_{50}: dose required to kill half the members of a tested population) of CWA, achieving to improve the performance of this type of sensors. Discrimination and classification of these gases are analyzed and discussed at the end of this paper.

2. Material and methods

2.1. Love-wave device

The SAW devices used in this work were of Love-wave type. They were based on a shear horizontal surface acoustic wave (SH-SAW) propagated on the ST-cut quartz substrate, perpendicular to the x crystallographic axis. This SH-SAW, with a wavelength of λ=28 μm, was generated and detected by interdigital transducers (IDTs). The IDTs were made using standard lithographic techniques, depositing an aluminum layer with a thickness of 200 nm by RF sputtering and forming a delay line (DL). A double electrode structure was repeated 75 times to form each IDT. The distance center to center between IDTs was 225 λ and the acoustic aperture was 75 λ.

Finally, the Love-wave was obtained by guiding the SH-SAW in a film of SiO_2 deposited on the piezoelectric substrate by PECVD. The highest sensitivity was found for a thickness of SiO_2 of 3.5 μm, the synchronous frequency being around 163 MHz [12].

The sensors were electrically characterized by means of a vector network analyzer (Agilent 5070B) which measured the frequency response before and after the deposit of the electrospun fibers on the device. Next, each sensor was incorporated, as usually, to an oscillator circuit whose characteristics were measured by a spectrum analyzer (Agilent 9320A).

2.2. Electrospinning process

Electrospinning is a versatile technique for the preparation of continuous nanofibers. Sensitive materials prepared by this method have great potential in the field of the sensors by their large surface to volume ratio, provided by their three-dimensional nanoporous skeleton structure. In the electrospinning process, the solution in the syringe is extruded from the needle tip to the collector, where the device is placed. When high voltage is applied between the needle and the collector, an electrostatic force is induced on the droplets of the solution at the needle tip. The interaction between this electrostatic force and the surface tension causes the droplets to stretch, forming thin jets of polymer solution that dry during flight and are deposited on the collector. If any of the electrospinning parameters, such as the applied voltage, needle-to-collector distance, solution viscosity, or flow rate of solution is changed, the morphology of the fibers obtained on the collector may be affected.

The setup for electrospinning consisted of a 10 ml glass syringe with a metallic needle screwed on the tip. The syringe was filled with polymer solutions, and then placed in the syringe pump (NE-1000), which allowed controlling the flow velocity of the polymer solution. The needle was connected to a high-voltage power supply and was directed to a copper plate which served as the grounded collector, where the Love device was placed. In this way random fibers were produced by the high applied voltage between the needle and the collector (which forms a homogenous electric field), coating with the sensing materials on the device surface.

2.3. Preparation of polymers using electrospinning

The electrospinning technique allows us to obtain nanofibers of composites created by mixing different polymers or additional materials, thereby making it possible to build up an array of sensors with high sensitivity and selectivity. An array of six sensors was developed in this study using the following polymers as sensitive materials:

- *Polyvinyl alcohol (PVA)* is a water soluble polymer produced industrially by hydrolysis of poly(vinyl acetate) and is commercially available in different molecular weights. It has high chemical stability and, due to its excellent physical and mechanical properties, is broadly used in practical applications as fiber and film products, paper coatings, adhesives and contact lens or artificial organs. In our work, PVA samples (Sigma-Aldrich) with two different molecular weights and different composite formations were deposited onto three of our Love-wave sensors.
  The first PVA solution, 10% w/v, was prepared by dissolving PVA (Mw ~94,000 g/mol) powder in deionized water and stirring at 90 °C during 4 h. After cooling to room temperature, it was loaded into the syringe to be deposited as sensitive film onto the surface of the sensor S1 of the array, using the parameters shown in Table 1.
  A second solution was prepared from a PVA / $SnCl_4 \cdot 5H_2O$ composite. A solution of PVA (Mw ~170,000 g/mol) was prepared by dissolving the PVA powder 8% w/v in deionized water and stirring it at 90 °C for 4 h. A solution of 2 g of tin (IV) chloride pentahidrate ($SnCl_4 \cdot 5H_2O$) (Sigma-Aldrich) with 2 g of deionized H_2O was also prepared at room temperature. This solution was slowly added to 20 g of PVA solution and stirred at room temperature for 2 h. The resulting inorganic/organic composite solution was loaded into a syringe to be deposited using the parameters shown in Table 1, and it was deposited onto the surface of the sensors S2 and S3 of the array. Finally, the sensor S2 with the electrospun fibers was introduced into a tube furnace at a temperature of 450 °C for 4 h in ambient atmosphere, obtaining nanowires of SnO_2.
- *Polystyrene (PS)* is a synthetic resin produced by the polymerization of the styrene. It is a waterproof and low thermal conductor. Polystyrene is used extensively in many industries and is the base material for many products. It is one of the most common plastics used in everyday life.
  First, a polymer solution was prepared for electrospinning by dissolving a mixture of PS (Mw ~192,000 g/mol) (Sigma-Aldrich) and the copolymer Poly(styrene-alt-maleic anhydride) (PSMA) (Mw ~350,000 g/mol) (Sigma-Aldrich) at a 2:1 weight ratio in *N*,*N*-dimethylformamide (DMF) solvent. The total

Table 1
Electrospun parameters for preparation of the nanofibers of the polymers used. V is the high voltage applied, v is the flow rate of the syringe pump, d is the needle-to-collector distance, Ø is the external diameter of the needle, t is the time of electrospinning and D is the average of the diameters of the fibers.

Sensor	Electrospun nanofiber	V (kV)	v (μl· min^{-1})	d (cm)	Ø (mm)	t (seg)	D (nm)
S1	PVA[a]	19.5	12	10	0.6	25	~150
S2	PVA[b]+$SnCl_4$ Annealing 4 h 450 °C	19	5	10	0.6	300	~100
S3	PVA[b]+$SnCl_4$	19	5	10	0.6	300	~250
S4	PS+PSMA	18	5	20	0.6	20	~800
S5	PS	18	5	20	0.6	30	~800
S6	PVP	18	5	22	1.1	60	~200

[a] PVA (Mw~94,000 g/mol).
[b] PVA (Mw~170,000 g/mol).

polymer concentration, PS+PSMA, in the solution was 20% w/v and the electrospun nanofibers were then deposited as sensitive layer on the sensor S4. Second, the PS with a concentration of 20% w/v was dissolved in DMF. The resulting electrospun nanofibers were deposited on the sensor S5. The parameters of the electrospinning for PS and PS+PSMA fibers can be seen in Table 1.

- *Poly-vinyl-pyrrolidone (PVP)* is also a water soluble non-conducting polymer which has excellent wetting properties and readily forms films. This makes it suitable to use as a coating or an additive to coatings in a wide variety of fields such as, medicine, pharmacy, cosmetics and industrial production. It is also used as sensing material to detect VOCs in gas sensor. In this work, the PVP fibers were fabricated by electrospinning and deposited as sensitive film on the surface of sensor S6 of the sensor array.
  Pure PVP (Mw ~360,000 g/mol) powder (Sigma-Aldrich) was dissolved in deionized water to make a PVP solution with a concentration of 20% w/v and was then stirred at room temperature for 3 h. This solution was loaded into the syringe and the resulting nanofibers were deposited on the surface of S6. The parameters of the electrospinning for PVP fibers can be seen in Table 1.

2.4. Samples and experimental setup

The CWA simulants used in our experiment were dimethylmethyl phosphonate (DMMP), dipropyleneglycol methyl ether (DPGME), dimethylmethyl acetamide (DMA) and dichloroethane (DCE). The concentration of the simulant was calculated using Antoine's Equation. The volatiles were extracted and diluted with synthetic air which was controlled by a mass flow controller in order to provide the desired concentration. The volume of the liquid samples was 10 ml. They were kept at a constant temperature of 10 °C in a thermal bath for 30 min (headspace time) before being carried to the chamber. Air flow in the chamber was 200 ml min^{-1} and the exposition time was 30 min. The experimental control and data acquisition in real time were implemented with a PC using home-made software.

2.5. Statistical treatment

Principal component analysis (PCA) and a probabilistic neural network (PNN) were used for data analysis. PCA is a statistical method for reducing the number of dimensions of numerical dataset without heavy loss of information. Once the analysis is finished, all data can be plotted in two or three axes. The neural networks were trained with the three principal components (PC1, PC2 and PC3) which resulted from PCA, and its performance was evaluated with leave-one-out cross validation.

3. Results and discussion

3.1. Electrical characterization of the Love-wave device

Nanostructured materials, nanofibers in this work, may provide better sensitivity with lower amount of mass than continuous thin films, made by methods such as drop, airbrush or spinning, due to the high ratio between mass and surface [17], because they have a specific surface area between one and two orders of magnitude larger than the specific surface area of continuous flat films. Therefore, the frequency responses of the Love-wave devices were studied before and after the deposit of electrospun nanofibers and small insertion losses, as low as 2.6 dB, were measured due to the small amount of material deposited. Fig. 1a shows the attenuation of the Love-wave device before and after depositing PVP nanofibers. Furthermore Fig. 1b shows a small frequency shift in the phase response of 25 kHz. The low insertion losses produced by the nanofibers are an advantage of using Love-wave devices, because the lower the insertion losses, the lower the noise in the measurement.

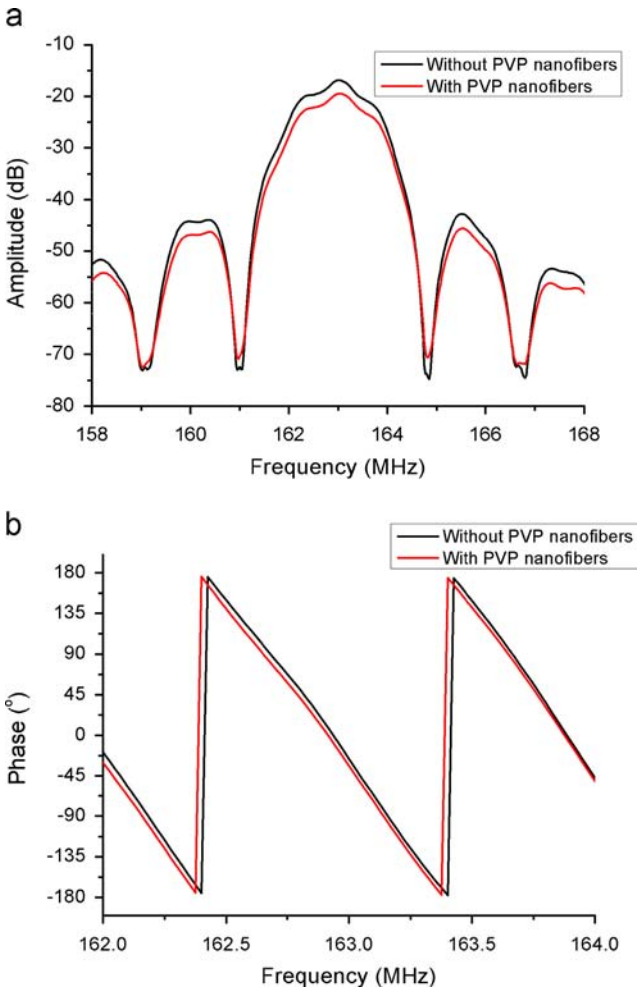

Fig. 1. Frequency response of the sensor S6 before and after the electrospinning of the PVP nanofibers (a) Amplitude and (b) Phase.

3.2. SEM characterization of the electrospun nanofibers

Nanofibers were coated with aluminum using a vacuum thermal evaporation deposit technique for characterization by scanning electron microscopy (SEM) (Quanta 3D FEG - FEI Company). The films, formed uniformly by the nanofibers deposited randomly were first observed under a magnification of 650 × and all of them showed an appearance similar to a tissue, as it can be seen in Fig. 2, which shows the PVA electrospun nanofibers deposited on S1. However, as it is shown in the left upper panel of the same figure, at a magnification of 20,000 ×, the SEM showed the diameters of the fibers deposited on the Love-wave sensors that, in this case, are about 150 nm. For this study, the parameters of the electrospinning process mentioned in Section 2.2 were adjusted in order to obtain the smallest possible diameter of the fibers (Table 1). By electrospinning process, the diameter of the fibers always was of nanometers, but in a different range for each material. Thus, by means of SEM, the diameter of the fibers was measured and an average for 10 fibers was calculated

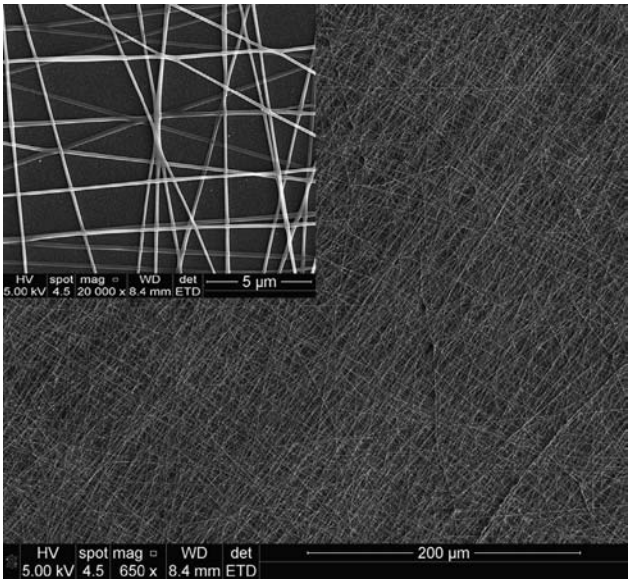

Fig. 2. SEM image of the PVA fibers deposited on the sensor S1. In the overall image the magnification is 650× and in the left upper panel the magnification is 20,000×.

Table 2
CWA simulants tested, concentrations measured and LD50 for each CWA.

Simulant	CWA	Formula	Concentrations (ppm)	LD50 (ppm· min)
DMMP	Sarin	$C_3H_9O_3P$	0.2, 0.4, 0.6, 0.8, 1	18
DPGME	Nitrogen mustard	$C_7H_{16}O_3$	1, 2.5, 7.5, 10	180
DMA	Distilled mustard	C_4H_9NO	50, 100, 150, 200	140
DCE	1,2-Dichloroethane	$C_2H_4Cl_2$	100, 200, 300, 400, 500	140

(Table 1). It is well known that the difference of the diameters for each material is a consequence of the properties of the polymer solution, such as surface tension or solution viscosity, and the parameters of the electrospinning, such as the applied voltage or flow rate solution. However, both the diameters and the material properties can be changed with a treatment after nanofiber deposit. Therefore, the characterization by means of SEM showed that the annealed nanofibers of PVA+$SnCl_4$ had lower diameter due to the carbonization of the polymer (Table 1).

3.3. Gas characterization with CWA simulants

Different behavior for each sensitive layer with regard to adsorption properties for VOCs was expected due to the difference in morphology and chemical composition among the different layers of nanofibers. To verify that the array of Love-wave devices with nanofibers was suitable to detect VOCs, different concentrations of the CWA simulants were measured and the frequency shift of each sensor was recorded in real time, obtaining different responses from each sensor. Table 2 shows the different CWAs simulants tested, their concentrations measured and their LD_{50}. The sensor array was repeatedly exposed to each one of the different concentrations of simulant to ensure the repeatability of the measurements, and the exposures always produced significant and similar frequency shifts. The array was then purged with clean synthetic air, which shifted the frequency back to the initial value. Fig. 3 shows the response for the case of DMMP detected by the sensor S4 coated with PS+PSMA.

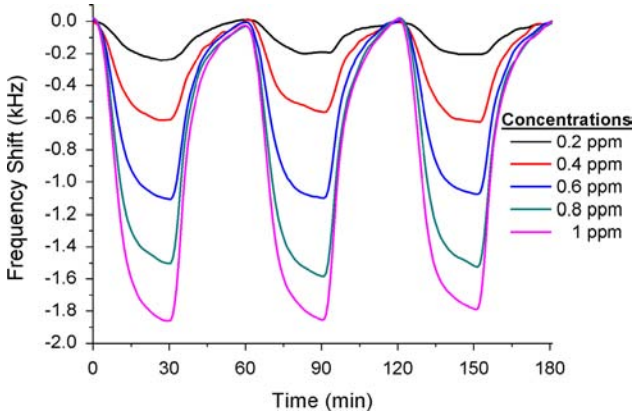

Fig. 3. Real time response of Sensor S4 to different concentrations of DMMP.

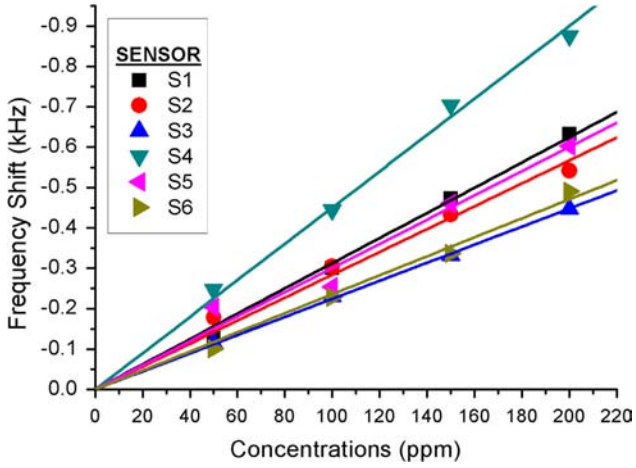

Fig. 4. Linear relation between frequency shift and the concentration of DMA for each sensor of the array.

There are good linear correlations between the frequency shifts of the sensor and concentrations of the CWA. Fig. 4 shows the frequency shift for the case of DMA detected by each sensor of the array. The slopes of these lines correspond to the sensitivity of the different sensors with respect to the concentration for each CWA simulant. Fig. 5 shows a complete visual comparison among the sensitivities of each sensor with the different VOCs considered in this study. It can be clearly seen that, as expected, these sensitivities were very different from each other. This was because each sensitive layer had different interaction with each CWA simulant, which is essential to classify and discriminate the different CWA.

3.4. Methods for discrimination and classification

This different response of the array to each chemical warfare agent simulant allows its discrimination and classification. In order to prove this, the maximum frequency shift during the exposure time was taken and PCA was applied to these data to obtain the principal components. The most important components, PC1 and PC2, are represented in Fig. 6. The results of the plot were used to study the classification. The ellipses include the measurements belonging to each simulant and, therefore, separated ellipses indicate that the CWA simulants have been correctly discriminated. The scattered points are the result of the variance in the frequency shift for the measurements of the same concentration, which, as was expected, was greater for low concentrations. Fig. 6 shows a clear separation among simulants, proving that the chosen sensitive layers detect differently the simulant considered

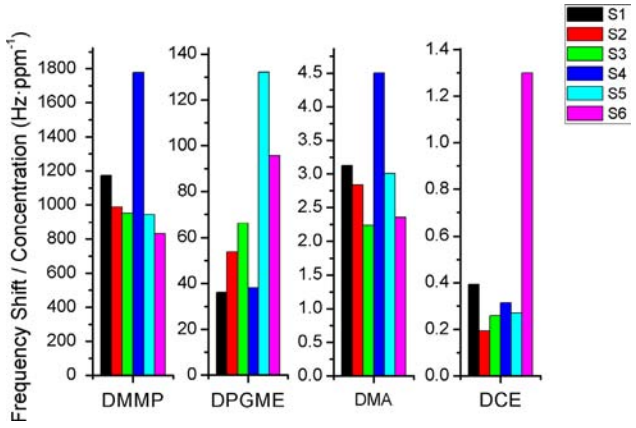

Fig. 5. Sensitivity values (Hz ppm^{-1}) of each sensor to every CWA simulant.

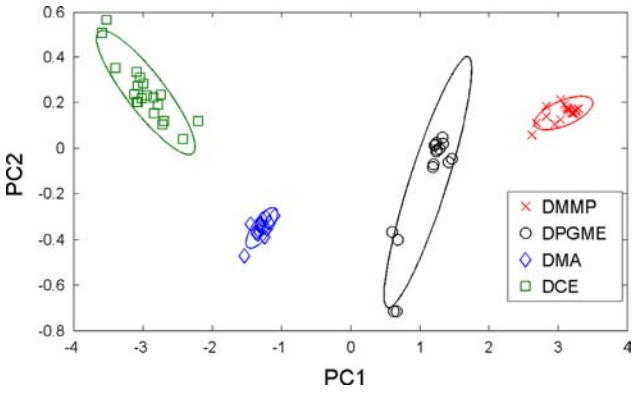

Fig. 6. Principal components analysis applied to data to discriminate CWA simulants.

and therefore the array is able to discriminate different VOCs. In order to validate the correct classification of the CWAs, the above information of the PC1, PC2 and PC3 was used to train the PNN in order to recognize the CWA simulants, then with the leave-one-out method, the classification was carried out and a 100% classification success rate was achieved.

4. Conclusions

Electrospinning technique is an efficient method to deposit a very large range of materials in the form of nanofibers. The nanofibers are suitable to use as sensitive layer on Love-wave devices in order to detect VOCs. Efficient detection by means of an array of Love-wave sensors with electrospun nanofibers of several materials as sensitive layers was demonstrated by measuring different concentrations of the four CWA simulants considered. The result was a significant change in frequency for each concentration and the responses were the same for different exposures to the same CWA simulant concentrations. The experiment also demonstrated that the responses for different concentrations of each CWA simulant were linear in the range of these measurements.

The results have confirmed that the Love wave sensor array was suitable to achieve high sensitivity and selectivity. The different signals produced by the array for each CWA were a consequence of the different morphology and chemical composition of the different nanofibers deposited on each Love-wave device of the array. For this reason, the CWA simulants were discriminated and correctly classified through PCA and PNN respectively.

Acknowledgment

This work has been supported by the Spanish Science and Innovation Ministry under the project TEC2010-21357-C05-04.

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