

Detection Optimization Using a Transient Feature from a Metal Oxide Gas Sensor Array

Maryam SIADAT, Etienne LOSSON, Diaa AHMADOU,
Martine LUMBRERAS

LCOMS, University of Lorraine, Metz, France
Tel.: +33 387547626, +33 387315262
E-mail: maryam.siadat@univ-lorraine.fr

Received: 21 December 2013 /Accepted: 12 January 2014 /Published: 2014

Abstract: Metal oxide sensors are largely studied in electronic nose field for gas detection or identification. They are robust, easy to use and have good sensitivity to many gases in the ppm range. However, the drift and low response-time of these chemical sensors constitute an important barrier in their utilization especially when low concentrations of gaseous atmospheres should be detected. To resolve these limitations many possibilities have been investigated to enhance either the sensor properties or the measurement conditions and the data analysis procedures. This paper presents the performance comparison of several features extracted from the sensor response signals. Particularly, a new transient feature corresponding to the inflexion point of the sensor response is processed. This feature is obtained during the first minutes of the gas exposure and shows a good performance, for example to detect a slight concentration variation of a gaseous atmosphere. *Copyright © 2014 IFSA Publishing, S. L.*

Keywords: Metal oxide gas sensors, Transient feature extraction, Performance evaluation, Gas concentration detection.

1. Introduction

Metal oxide gas sensors (MOX) are reliable, very sensitive to many gases, commercially available and now used in different fields of application including continuous gas control, toxic gas detection or gas concentration evaluation. One of the major limitations of this kind of chemical sensors is due to their important response-time, essentially when low concentrations of a gas should be detected. To obtain rapid detection two principal approaches are usually investigated. The first approach is to improve the sensor properties and the second solution is to enhance the response signal processing regarding the

feature extraction and the data analysis procedure [1-3].

Many studies have been reported in the field of feature selection and their processing methods [4-6]. Current features investigated in electronic nose applications can be divided into two main categories: features extracted from the steady-state phase (obtained usually after several minutes of exposure according to the used atmosphere and concentration) and features related to the transient phase of the sensor response (obtained during the first minutes of the gas exposure). The steady-state feature extraction requires more exposure time which causes a notable sensor recovery time and also a rapid ageing or deterioration of the sensors. The transient features

permit a faster measurement which consequently leads a sensor long life with long term stability; but for a robust extraction of this type of features an accurate and high rate acquisition procedure is needed. Moreover, the feature extraction is related to the experimental procedure particularly to the introduction mode of the gaseous atmosphere in the sensor cell: dynamic flow or static measurement [7, 8]. So the criteria of each specific application can make certain features more preferable than others. In electronic nose applications, usually selected features are studied alone as a first stage, and then joined in a set of features to compare their performances to classify the concerned gaseous atmospheres. But taking into account that an important number of features may induce severe computational costs, redundant or non-informative features must be eliminated in the recognition system. So it is often necessary to perform a check of different features to keep the ones which contribute best to the desired classification [9-10].

In this work, we use an array of commercial MOX sensors (TGS and FIS types) for the concentration detection of a unique analyte diluted in a neutral atmosphere in a small concentration range, with a small difference step. This quantitative analysis is requested to regulate, by a feedback, the diffusion of an odorant vapor (pine essential oil) in a closed atmosphere. So, the measurement time and the process time of the recognition features would be particularly important. We propose here the study of several features obtained from both the steady-state sensor response, which are normally more appropriate for a quantitative analysis, and from the transient part of the sensor response. We focus first to adapt our experimental system for an accurate atmosphere generation and the selection of the best measurement conditions. Then we present the analysis of the sensor time responses, and also features extracted from the both transient or stabilized parts of these responses. In this aim, three features among the traditional literature ones and a new transient feature are processed. The performance of each selected feature is tested separately and compared to the others by using classification algorithms. Among them, the maximum of derivative curve of the sensor response signal, which is literature new, shows a good robustness and gives a good alternative for rapid concentration detection. We hope that this study can give new possibility for fast recognition with electronic nose device.

2. Material and Methods

All experiments were performed with a laboratory made set-up equipment, using automation devices and commercial metal oxide gas sensors. As gaseous atmospheres, we have used evaporated pine essential oil diluted in synthetic air at different rates.

2.1. Experimental System

Eight metal oxide sensors (SP-AQ1, SP-MW0, TGS 882, TGS 880, TGS 816, TGS 2620, TGS 2602, TGS 2600) are placed in a stainless steel circular cell with a reduced volume to optimize the gas exchange time. They have been chosen for their sensitivities to solvent vapours, odorous gases or volatile organic components. Fig. 1 presents the experimental set-up with the sensor cell and the gas lines maintained at a constant temperature (to avoid condensation) using a heater ribbon and a controlled temperature box (experimental chamber) using a PID regulator. The oil diffusion unit is based on a constant inflow of synthetic air (line 2) bubbling into a bottle containing the liquid essential oil (EO). The outlet flow carrying the evaporated EO substances is combined with a synthetic air flow (line 1 for a dry atmosphere; lines 1 and 3 for a humid atmosphere) to generate a desired concentration of EO mixture [11]. The line 3 is utilized to add humid air with different relative humidity rates [12]. For all the measurements, the total gas flow rate through the sensor cell is maintained constant (100 ml/min) to eliminate the influence of this parameter on the sensor responses. So, the concentration of the EO mixture is defined in terms of percentage of the bubbling air rate (inflow rate) over the total gas flow rate. By varying the inflow and the carrier air flow rates, different evaporated EO concentrations are created (for example to generate a concentration of 2 %, 2 ml/min EO inflow rate is combined with 98 ml/min of carrier gas). In this paper, we present our results obtained with dry atmospheres. Gas lines are controlled by Mass Flow Controllers (MFC, Brooks) using an automation device (PLC, Adamtech) equipped with several boards such as a controller, an analogical output and an analogical input board. The sensor responses are recorded using a PC acquisition board (National Instruments). The voltage variations are measured on the sensor load resistor with 1 second time resolution and they constitute the sensor response signals.

Measurements are controlled via a dedicated software tool developed in our laboratory in Visual C++. This tool permits to have automatic, repetitive and accurate measurements. Several working parameters are configurable as the sampling rate, the gas exposure or purge times, gas mixtures creation (composition or concentration) and humidity rates. The main window of the controlling software is shown in Fig. 2. On the left side we can observe the measurement evolution as the sensor response signals (voltage), the recorded data and the acquisition delays. The right side of the screen is dedicated to define the measurement parameters as the gas mixture generation, the preparation of the desired measurement cycles, the sensor exposure time or the sampling frequency. Each gas exposure is preceded and followed by a pure air exposure at the same flow rate than the gas exposure flow rate (100 ml/min).

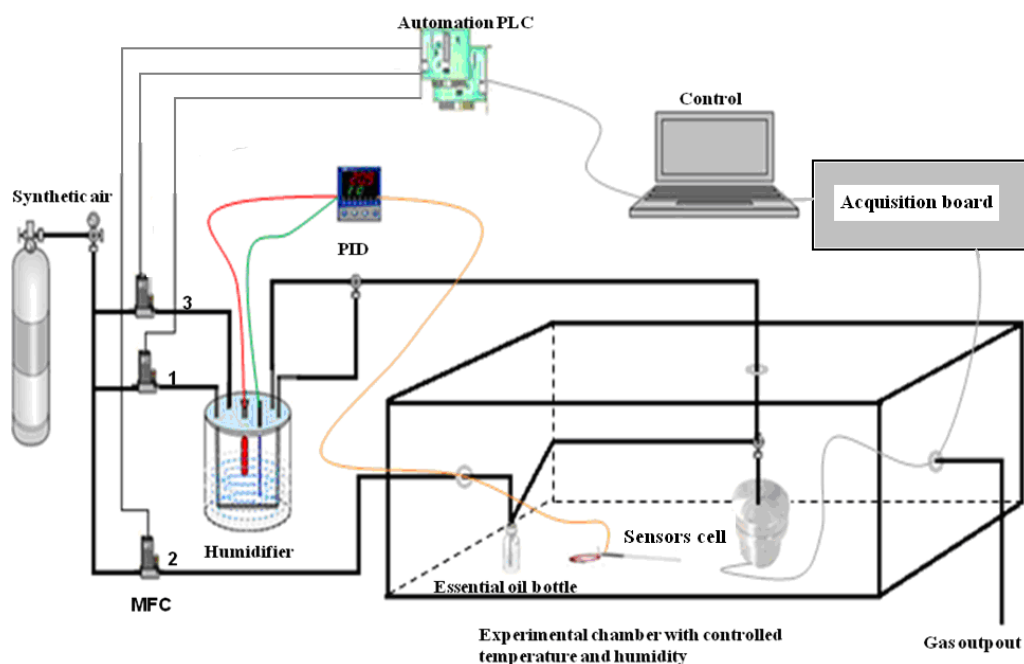


Fig. 1. Experimental set-up.

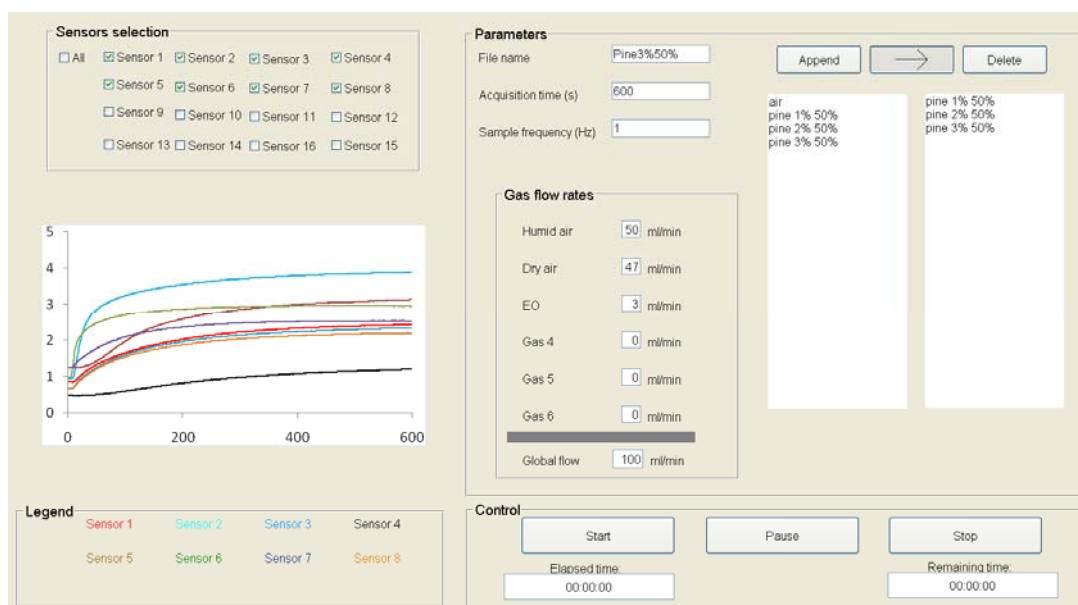


Fig. 2. Screen capture of the main window of the measurement software tool.

2.2. Measurement Protocol

Pine oil is used with different dilutions in synthetic air (1, 2, 3, 4, 5, 6, 7 %). The choice of these dilutions (concentrations) was established according to their odorous feeling (from slight to unpleasant smelling) by a human panel. All the sensors present a measurable effect for this concentration range. The gas exposure time is fixed to 10 minutes to obtain the steady-state response of all the sensors particularly when the lowest EO dilution is employed. The purge time is fixed at 20 minutes to permit a good recovery of all the

sensors when the highest EO dilution is used. Each measurement day starts with the cleaning phase of the sensors during approximately 1h to reach the stabilization. Fig. 3 presents the recorded signals obtained for repetitive cycles of a constant EO concentration, and shows the good regeneration of all the sensors whatever the concentration value.

Characterization measurements have been done randomly for all the chosen EO concentrations. Each measurement was at least repeated 10 times to control the stability of the sensor responses and to constitute a consistent data basis for the training of the recognition system.

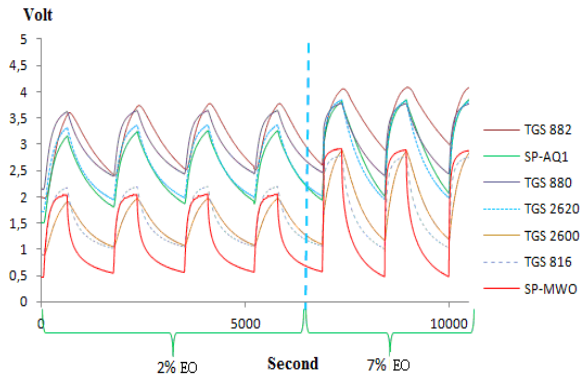


Fig. 3. Sensor reproducibility during cycle repetition at two pine EO concentrations.

3 Signal Processing

3.1. Sensor Response Characterization

Sensor responses were first analyzed to determine several characteristic parameters as sensor detection thresholds, response-times, sensitivities and stabilities. In each sensor response, three parts can be distinguished. Part 1, with a constant value (the initial value), corresponds to the response-time of the whole set-up device. Part 2 presents an increasing response (transient phase), whereas part 3 is devoted to the stabilized final phase.

As shown in Fig. 4, sensors react differently to a same atmosphere. When considering the response-times, we can note that FIS sensors (SP-MW0 and SP-AQ1) are the faster ones. The TGS sensor responses are more or less rapid. Concerning the sensitivity, the FIS SP-AQ1 presents the highest response magnitude, the Figaro TGS 882 gives also a good sensitivity but its response-time is longer than the other sensors. One of them (TGS 2602) was damaged very soon; this sensor was removed from the array. This diversity in the response signal shapes shows the importance of exploring different features in order to select the best ones which best optimize the concentration detection.

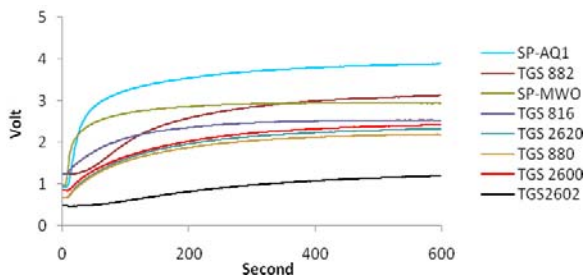


Fig. 4. Response signals of all the sensors at a fixed atmosphere (5 % pine EO).

We summarize in Table 1 the response-time ranges of each part of the sensor response considering all the studied concentrations. In all the three parts of the response, the two FIS sensors give

the best response-times. The Figaro sensors present longer response-times (approximately 2 times in comparison with the FIS sensors). These differences in the response-times of the sensors could be taken into account for a rapid gas concentration determination. So, our interest was focused on the selection of features characterizing the response-time of the sensors.

Table 1. Sensor response time comparison when exposed to all the studied concentrations.

Sensors	Sensor response times for all the EO concentration range: 1 to 7 % pine oil in [s]		
	Part 1 (initial level)	Part 2 (transient)	Part 3 (stabilized)
TGS882	<30	30 to 400	>400
TGS2620	<24	24 to 400	>400
TGS816	<23	23 to 300	>400
TGS2600	<19	19 to 450	>450
SP-AQ1	<18	18 to 180	>180
SP-MW0	<15	15 to 200	>200
TGS880	<16	16 to 380	>420

Our application concerns the gas concentration detection, so we have also analyzed how the response of each sensor varies along with the EO concentration. Fig. 5 presents the TGS 880 and SP-MW0 responses to the total concentration panel. We can observe notable variations during the transient phase as well as the stabilized phase. The other sensors of the array show also a notable variation along with the concentration.

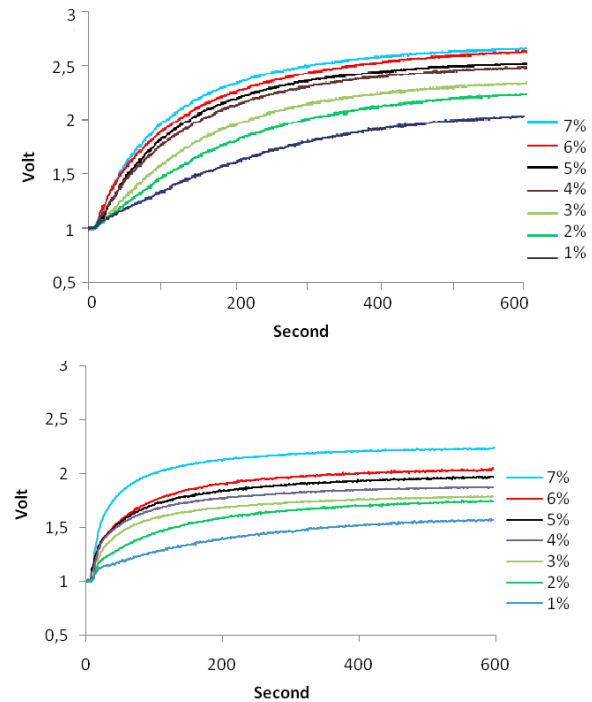


Fig. 5. TGS 880 and SP-MW0 responses for all the studied pine oil concentrations.

All these observations encourage us to select different features from the transient and/or the stabilized part, and then to study the performance of each of them for our application. So, we have decided to extract characteristic parameters either directly from the recorded time-dependent signals (i.e. voltage taken at the load resistor connected in series with each gas sensor) and from the derivative curves which highlight the transient phase [13].

3.2. Derivative Curve Determination

To permit the extraction of characteristic features from the derivative curve (especially its maximum value), we have to use low-pass filtering process on the time-dependent response in order to obtain first a smoother form of the derivative signal. Fig. 6 shows a time-dependent signal and its derivative curve without any filtering (raw signal).

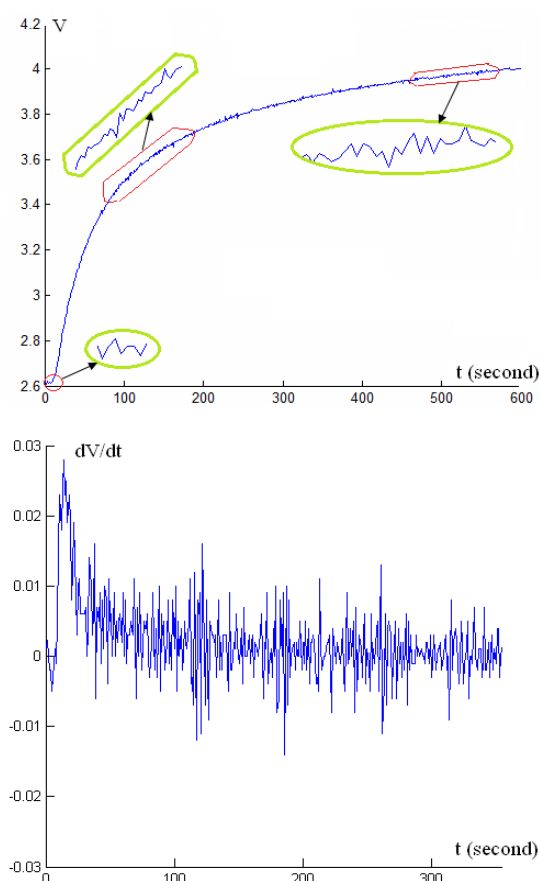


Fig. 6. Example of raw time-dependent signal and its derivative curve.

For each gas sensor, a same selected filter must be applied whatever the gas concentrations (from 1 % to 7 % in this study). In this way, several types of digital filters are tested and optimized, as simple or moving average, Butterworth and Chebyshev algorithms. In our case, second order Butterworth type filtering with

adequate cut-off frequency gives the best results. Fig. 7 shows the results obtained from three different normalized cut-off frequency (ω_n) for the TGS 2620 sensor signal. We can note that if $\omega_n = 0.08$, the magnitude of the peak is too much reduced. For $\omega_n = 0.38$, noise is still apparent and the peak height value remains difficult to obtain. So, for this sensor a normalized cut-off frequency of 0.2 has been chosen as the optimal value. This filtering study is done for each sensor, and according to their dynamic response an optimal frequency (varying from 0.08 to 0.2) is determined.

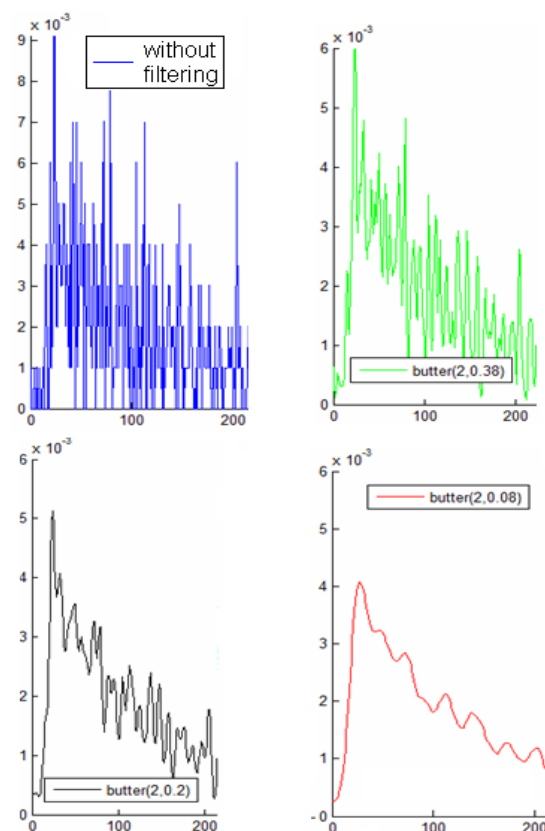


Fig. 7. Example of a derivative curve without filtering and after Butterworth filter application using different cut-off frequencies for TGS 2620 sensor.

3.3. Feature Extraction

In electronic nose applications usually a set of selected features are studied in term of their performance to classify the used substances. Of course, to obtain an accurate classification, the feature selection and data processing are very important.

For this work, we have first selected two main traditional features as steady-state characteristic parameters:

- Amplitude response ($V_S - V_0$), where V_S is the final value and V_0 the initial value of the time-dependent response. V_S and V_0 are calculated by averaging respectively 10 data recorded at the end of

the 5 minutes of gas exposure and 5 data recorded during the first seconds of the gas exposure.

- Surface under the response signal (St-5 min), obtained during the first 5 minutes of gas exposition. This feature considers the amplitude and also the rapidity of the response.

We note here that the total surface (obtained during 10 minutes of EO exposition) and the St-5min give a very correlated information, so we have decided to work with the feature extracted on 5 minutes of exposure.

As transient features, we have also chosen two characteristic features, a traditional one and a new one, both extracted in the first minutes of the gas exposure:

- Initial slope of the signal response (Slope) corresponding to the gradient of the transient phase. This dynamic slope is generally used when a rapid detection is needed. This feature is calculated using linear regression on 25 recorded data after the transient phase is detected.

- Maximum value of the response derivative curve (called Peak) is also determined. This peak is directly related to the inflexion point of the sensor response. This maximum appears for all the sensors in the 100 first seconds of the gas exposition. For each sensor, the Peak values show an appreciable variation along with all the oil concentrations.

4. Data Analysis and Discussion

The four selected features are tested for their capacities to differentiate the oil concentrations. Fig. 8 shows the evolution of the St-5 min and the Peak features along with the EO concentrations for three sensors. To take into account all the information contained from the 4 features and for 7 sensors, the values of each extracted feature are grouped to create a data basis and then treated by classification methods.

The capacity of each feature to classify the pine EO concentrations was studied using first the Principal Component Analysis (PCA). This non-supervised statistical method permits to reduce the dimensionality of a multidimensional data basis. The orthogonal projection of all the observations on a plane defined by the two first components (CP1 and CP2) allows to evaluate the classification power of the chosen variables. Fig. 9 presents the PCA diagrams obtained using each of the four selected features taken separately. For each analysis, the data basis is formed with 7 explicative variables (one feature per sensor) and 126 observations (approximately 15 measurements per concentration). In the case of the Slope and the St-5 min features, the 4, 5 and 6 % concentration groups are overlapped and not differentiable. With the V_S-V_0 feature, the 2, 3, 4, 5 and 6 % concentration groups seem to be very close and difficult to be distinguished. By using the Peak feature, we obtain a better classification, only the groups 4 and 5 % are close.

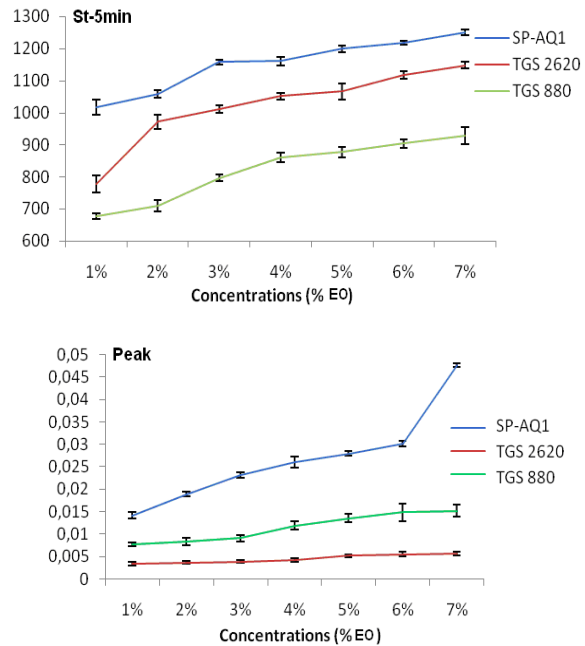


Fig. 8. Evolution of two selected features (St-5 min and Peak) along with the pine oil concentrations.

Regarding these results, we can conclude that the Peak feature is more accurate than the classical features for our application. In addition the Peak feature is less dependent on the sensor drift which disappears due to the signal derivation and it is obtained faster than the other ones.

To confirm this finding, the discrimination power of each feature is tested using the supervised Linear Discriminant Analysis. The concentration identification is validated by the cross validation technique: a success rate of 95.2 % is obtained with the Slope, 99.2 % with the V_S-V_0 , 99.1 % with St-5 min and 100 % when using the Peak feature.

5. Conclusions

We have studied an array of MOX sensors for a rapid analysis of an odorant vapour. Some sensors, particularly FIS sensors have shown good performance in term of stability and short response-time. Four steady-state and transient features have been processed and compared separately in terms of their capacity for gas concentration detection. A new feature extraction method is proposed, which represents the maximum value of the derivative sensor response curve. This feature (called Peak) is calculated after an adapted filtering of the sensor response signal and it is obtained in the first 100 seconds of the gas exposure. We have used each feature separately in a recognition system using PCA and LDA techniques. With the two methods, the Peak feature proposed in this paper gives the best results for the concentration detection of the pine oil among the seven used concentrations.

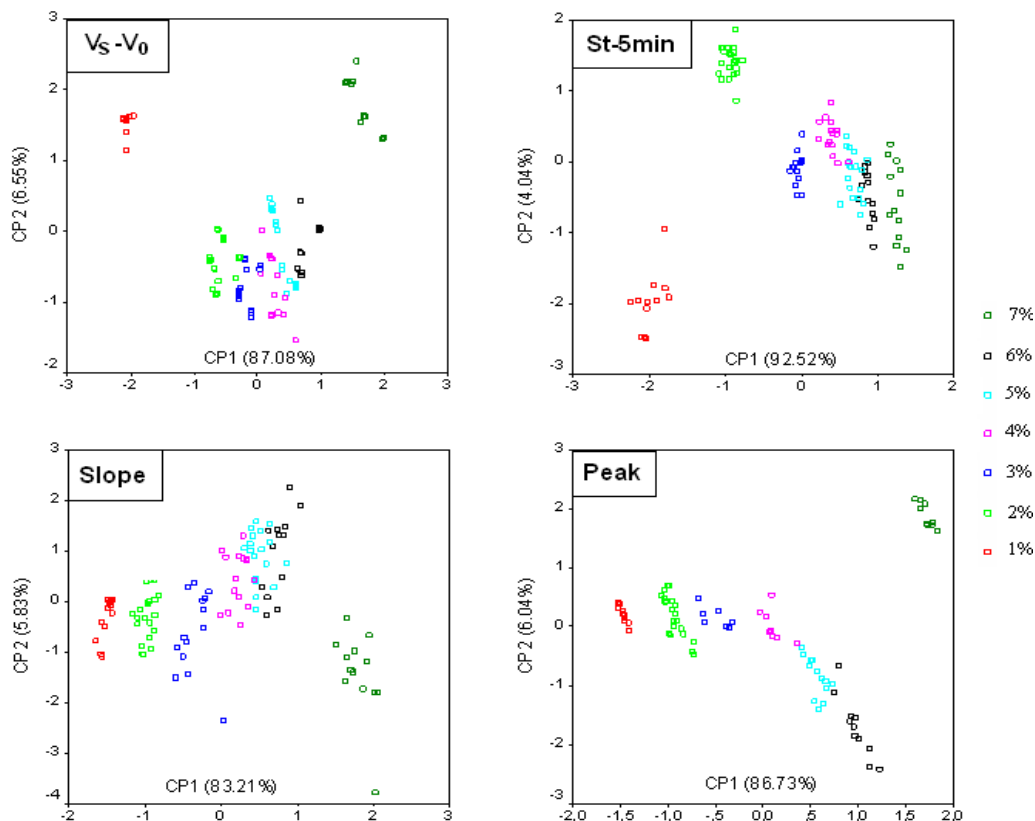


Fig. 9. PCA diagrams obtained with each feature taken separately: V_S-V_0 , St-5min, Slope and Peak.

References

- [1]. J. W. Gardner, E. L. Hines, H. C. Tang, Detection of vapours and odours from a multisensory array using pattern-recognition techniques, Part 2, Artificial neural networks, *Sensor and Actuators B*, Vol. 9, 1992, pp. 9-15.
- [2]. C. Distante, M. Leo, P. Siciliano, K. C. Persaud, On the study of feature extraction methods for an electronic nose, *Sensor and Actuators B*, Vol. 87, 2002, pp. 274-288.
- [3]. S. Roussel, G. Forsberg, P. Grenier, V. Bellon-Maurel, Optimisation of electronic nose measurements, Part II: Influence of experimental parameters, *Journal of Food Engineering*, Vol. 39, 1999, pp. 9-15.
- [4]. T. Eklöv, P. Martensson, I. Lundström, Enhanced selectivity of MOSFET gas sensors by systematical analysis of transient parameters, *Analytical Chimica Acta*, Vol. 353, 1997, pp. 291-300.
- [5]. A. Szczurek, M. Maciejewska, L. Ochrowicz, Sensor array data profiling for gas identification, *Talanta*, Vol. 78, 2009, pp. 840-845.
- [6]. K. Brudewski, J. Ulaczyk, An effective method for analysis of dynamic electronic nose responses, *Sensor and Actuators B.*, Vol. 140, 2009, pp. 43-50.
- [7]. T. C. Pearce, S. S. Schiffman, H. Troy Nagle, J. W. Gardener, Handbook of machine olfaction: Electronic Nose technology, *Wiley-VCH Weinheim*, 2003.
- [8]. N. Yamazoe, K. Shimano, New perspectives of gas sensor technology, *Sensors and Actuators B*, Vol. 138, 2009, pp. 100-107.
- [9]. N. Paulson, E. Larsson, F. Winquist, Extraction and selection of parameters for evaluation of breath alcohol measurement with an electronic nose, *Sensors and Actuators B*, Vol. 84, 2000, pp. 187-197.
- [10]. X. Vilanova, E. Llobet, R. Alcubilla, J. E. Suegras, X. Correig, Analysis of the conductance transient in thick-film tin oxide gas sensors, *Sensor and Actuators B*, Vol. 31, 1996, pp. 175-180.
- [11]. H. Sambémana, M. Siadat, M. Lumbreras, Gas sensing evaluation for the quantification of natural oil diffusion, *Chemical Engineering Transactions*, Vol. 23, 2010, pp. 177-182.
- [12]. C. Delpha, M. Lumbreras, M. Siadat, Discrimination and Identification of a Refrigerant Gas in a Humidity Controlled Atmosphere Containing or not Carbon Dioxide: Application to the Electronic Nose, *Sensors and Actuators B*, 98, 1, 2004, pp. 46-53.
- [13]. S. Zhang, C. Xie, D. Zeng, Q. Zhang, H. Li, Z. Bi, A feature extraction method and a sampling system for fast recognition of flammable liquids with a portable E-nose, *Sensor and Actuators B*, Vol. 124, 2007, pp. 437-443.