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Article

Response Optimization of a Chemical Gas Sensor Array using Temperature Modulation

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Abstract: This paper consists in the design and implementation of a simple conditioning circuit to optimize the electronic nose performance, where a temperature modulation method was applied to the heating resistor, in order to study the sensor's response and determine whether they are able to make the discrimination when are exposed to different Volatile Organic Compounds (VOC's). This study was based on determining the efficiency of the gas sensors to be used in order to perform an Electronic Nose, improving the sensitivity, selectivity and repeatability of the measuring system and selecting the type of modulation (e.g. Pulse Width Modulation) for the analytes detection (i.e, *Moscatel wine* samples (2% of Alcohol) and *Ethyl-Alcohol* (70%)). The results demonstrated that using temperature modulation technique to the heater of sensors, it is possible to achieve a good discrimination of VOC's in fast and easy form, through a chemical sensors array. A discrimination model based on Principal Component Analysis (PCA) was implemented to each sensor, and data responses obtained gave a variance of 94.5% and 100% accuracy.

Keywords: temperature modulation; gas sensors; volatile organic compounds; electronic nose; conditioning circuit

1. Introduction

Different studies related to the electronic noses have had a rise in many fields. These have been used to monitor odors in the food industry in order to determine the quality of certain products, like for detecting gases in places where it can endanger human life since they can be harmful health. Other applications related to the diseases detection and as abroad aspect of current research make contributions to mining and the whole field concerning to hydrocarbons [1].

Currently, all these applications have been made following the same pattern (i.e. using sensors that are selective and repetitive) which will be effective when they detecting a sample of a volatile compound [2]. In previous studies conducted by different researchers, for example, the research group of Polese has made a study entitled "Self-Adaptive Thermal Modulation of Gas Sensors" [3]. Other studies in different institutions have done some projects which used temperature modulation through metal-oxide sensors [4-8]. On the other hand, some works have been focused to research about frequency spectrum analysis, in order to improve the gas sensor performance, discriminating odors of similar response under noisy condition and modulation techniques using processing methods such as: Cluster analysis, principal components and neural networks, where a signal from a device for data acquisition was acquired and afterwards the gas sensor response was analyzed [9-10]. The work done by Anindita and Kanak about "A Temperature Modulation Circuit for Metal Oxide Semiconductor Gas Sensor" was taken as reference in this research in order to try to apply equivalent procedure but obtaining different outcomes [11].

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1.1. Gas sensor

1.1.1. Tin oxide sensor

Tin oxide gas sensor containing oxygen in clean air which is adsorbed on the surface of the sensing material on the crystal structure [12]. When the sensing material is heated to high temperatures (around 400 ºC) and in the presence of reducing gases, free electrons flow easily through the conduction band of tin oxide particles. Furthermore, the pure oxygen air is absorbed by the particles of tin oxide surface and traps free electrons due to high electron affinity, forming a potential barrier in the conduction band. This potential barrier restricts the flow of electrons causing an increase in electrical resistance [13]. When the sensor is exposed to an atmosphere, for example, with reducing gases, CO, CO_2, etc., tin oxide surface absorbs gas molecules, producing an oxidation reaction between the gas and the absorbed oxygen, which decreases the potential barrier and thus reduces the electrical resistance. The relationship between the sensor resistance and gas concentration deoxidized can be expressed by the following experimental equation and is valid for a certain range of concentration of a gas:

$$\boldsymbol{R} = \boldsymbol{A} * [\boldsymbol{C}]^{-\propto} \tag{1}$$

where R is the electrical resistance, A and α are constants and [C] the gas concentration [14, 15]. Tin dioxide sensors are characterized by having a good selectivity, repeatability and accuracy. Also, they can detect up to 20 gases at low concentrations, but the principal disadvantage of them is that they are very sensitive to moisture and this could generate drifts in the response. Another possible failure is that they might present contamination of active layer when is applied any volatile compound in high concentration, which avoid that they can be recovered due to saturations. Energy consumption of the gas sensors is one of the most important factors to be analyzed, in order to make an implementation of this device through powered systems modulated sources and to reduce the consumption.

Typically, gas sensor consumption is around 15 mW when a continuous signal is applied to the heater, but if is applying a modulated signal to heater wire the consumption decreases, being favorable for systems whereas batteries are used (e.g, FIS Inc and Figaro Engineering Inc) [16, 17]. Figure 1 shows the establishment time of the step-type signal applied to the sensor heater.

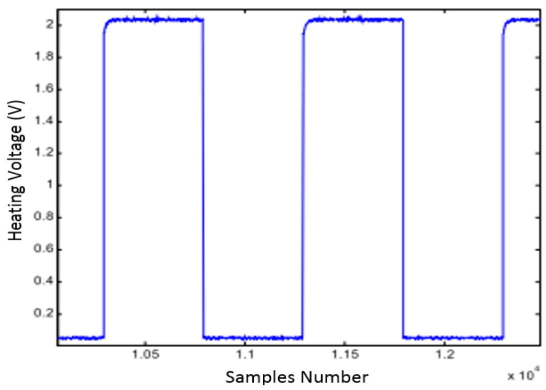

Figure 1. Voltage applied to the heating resistence

The time constant of first order marks the width of the frequency range to be modulated. Thus, the lower response time will imply a higher cutoff frequency of the system, which is directly generated by temperature modulation.

Figure 2 shows a bode diagram with an asymptotic module of this system, which the attenuation is above the cutoff frequency.

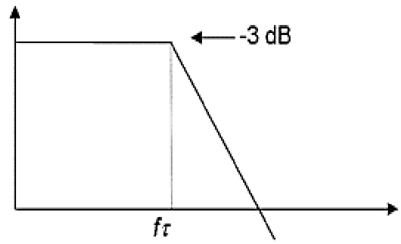

Figure 2. Bode diagram of the system

Several studies have used information from the dynamic response of a sensor thermally modulated to identify and quantify gases.
In our particular case, temperature modulation can alter the kinetics of the reactions occurring on the sensor surface in the presence of gases to be detected.

It is shown that using of dynamic response can reduce drift effects. Although the results are promising, only few studies have been done in selecting frequencies to be used in the sensors. A thorough study of the behavior of the sensors could be applied in a wide range of frequencies.

2. Materials and Methods

This study was made using a conditioning and measurement system, which were applied to develop an electronic nose, compounds by four commercial gas sensors. Each sensor is very sensitive to different volatiles; in this work, we have selected commercial gas sensors in order to make the system more robust and selective (i.e. capable of detecting any volatiles from the response of each individual sensor).

Figure 3 illustrates a block diagram of the classification process using the electronic nose, which was applied different modulation techniques.

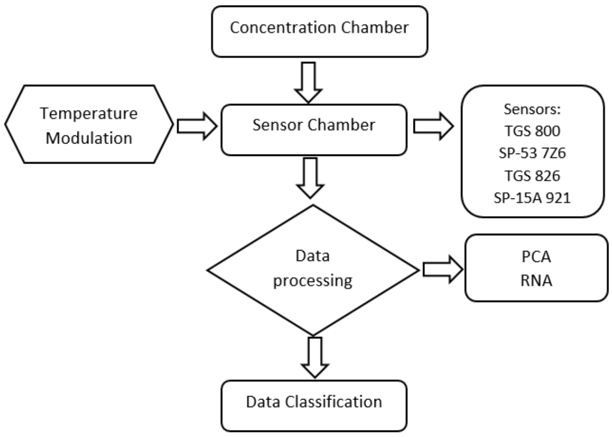

Figure 3. Scheme of measurement and data analysis

Figure 4 shows the experimental setup of the electronic nose and temperature modulation "home-made".

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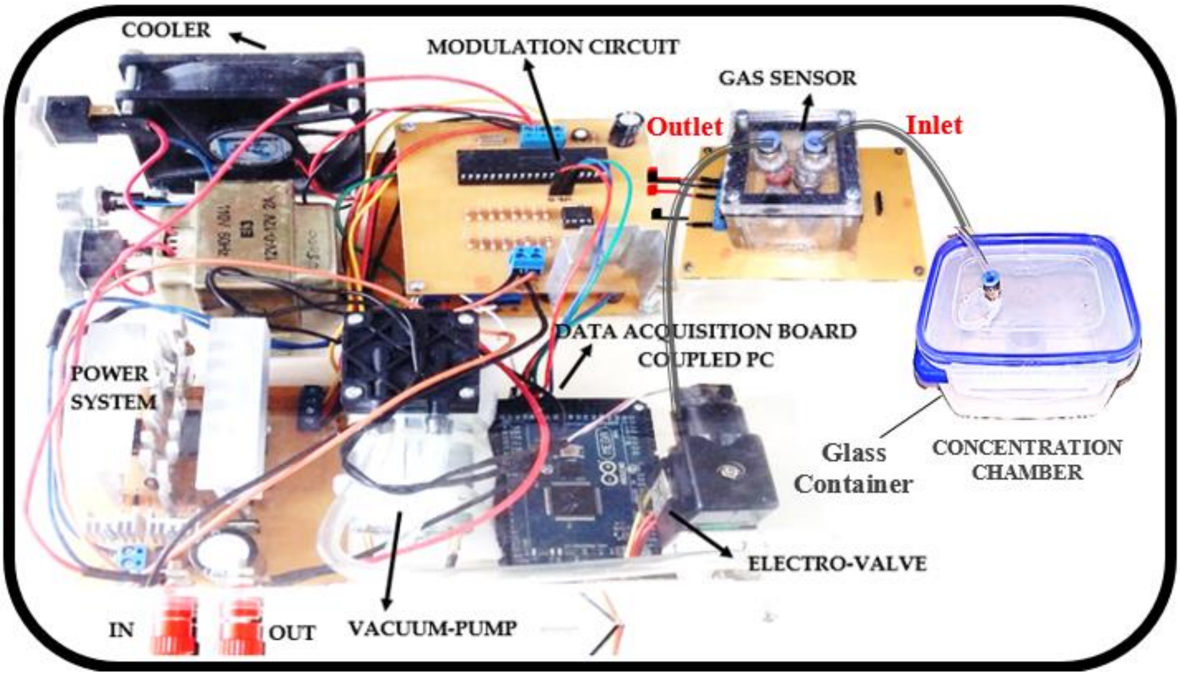

Figure 4. Experimental setup (home-made)

The components of experimental setup are shown below:

- Concentration chamber
- Electro-valve
- Vacuum-pump
- Sensor chamber (Gas sensor)
- Modulation Circuit
- Data acquisition board
- Power System

2.1. Concentration chamber

For the design of the concentration chamber a hermetic container of 200 ml of volume with a translucent plastic material was chosen and the sample was analyzed. The purpose of the concentration chamber is to be used for the volatile compound concentration released from the sample that is inside of the chamber, in order to make the best measurement protocol. For realizing the conditioning of the liquid sample within the concentration chamber, an uncovered container of glass of 4 ml was used to hold the sample. The concentration chamber is compound of one hole located on the top part of the cover, which was connected with the inlet orifice located on top side of the sensor chamber. The air outlet located in opposite side of the sensor chamber and vacuum-pump, shift the volatile compounds from the sensor chamber to the “Out” orifice of the system; in this stage the sample is measured. On the other hand, through “In” orifice it is take the air from outside in order to wipe the container as well as piping (i.e., avoiding condensation within the circuit) after the measurement. The concentration chamber was isolated from the measuring circuit thought of a two-way solenoid valve, in order to supply air from the outside at the time of wipe the sensors, obstructing the air flow of the chamber which brings a large number of concentrated volatiles while the sensors are cleaned. After the cleansing time of the sensors, a large volatiles compound was generated in the concentration chamber, whereas the solenoid valve position was changed, causing the gas to flow through an air pump with a flow rate of 500 ml/min to the sensor chamber. To deliver the gas to the sensor chamber, one-way of the solenoid valve and vacuum pump were activated to drag all compounds generated from the concentration chamber to the sensor chamber.

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2.2. Modulation System

Modulation circuits designed to vary the temperature of the heating resistor will be disclosed below:

2.2.1. Modulation circuit

Figure 5 provides the electronic circuit used to generate the square pulses applied to the heater coil of the sensor, in order to modulate the temperature.

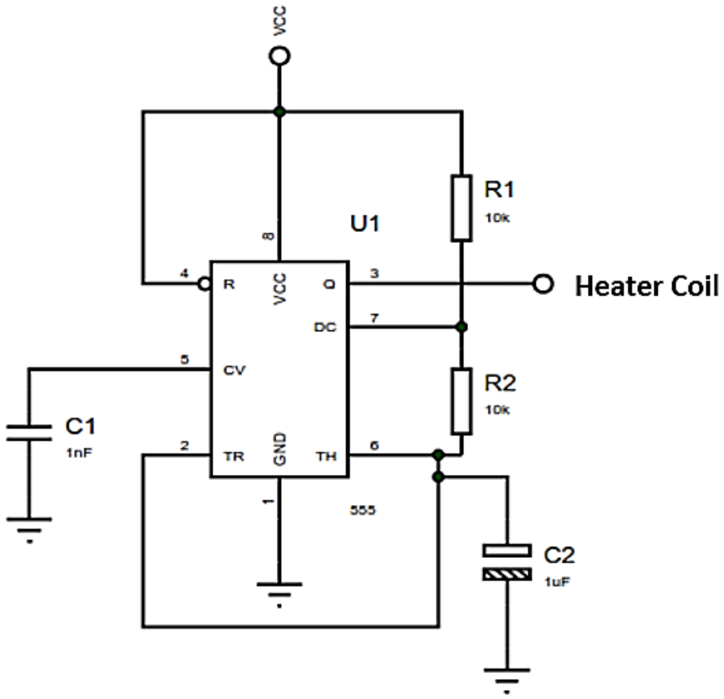

Figure 5. Generator Circuit PWM

For realizing the pulse modulation an integrated circuit (555 oscillator) was used in "stable mode", which can generate a square pulse; this simple circuit has the advantage because it is possible to know the period, frequency and the work cycle when it is having the resistance and capacitance. It should be noted that the period is the length of time; it takes ON and OFF cycles to repeat, while the duty cycle is the percentage of time that the output is on (i.e. T_1 / T).

The working cycle of this type of circuit cannot be 50% or less, therefore, to perform tests with a work cycle less than this value, it was necessary to use an Arduino 2560 card.

Figure 6 shows the output times of a PWM circuit. For the operation of the circuit, the following conditions must be taken into account: Increasing the capacitance will increase the cycle time, therefore the frequency will decrease.

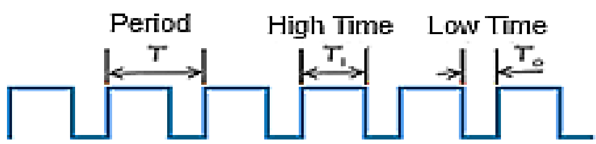

Figure 6. Generator circuit of a PWM signal

Also, increasing the value of the resistor R1 will increase the time within a high value (T_1) of the cycle, but the time in low (T_0) will not be affected.

Increasing the value of resistance R2 will increase the time in high (T_1) and the time in low (T_0) but will decrease the working cycle to a minimum of 50%.

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2.2.2. Generator circuit of a sawtooth signal

The electronic circuit to generate a sawtooth signal was composed of an integrated 555, which is low cost and very useful to give precision and highly stable delays of time or oscillation. This topology allows to vary the frequency of work, this can be carried out by simply varying the values of capacitance and resistance of the circuit.
To calculate the frequency generated by this circuit, the following equation was used:

$$\boldsymbol{f} = \frac{\boldsymbol{V_{CC}} - \mathbf{2.7}}{\boldsymbol{R} * \boldsymbol{C} * \boldsymbol{V_{PP}}} \quad (2)$$

where,
$\boldsymbol{V_{CC}}$ = Voltage Supply
$\boldsymbol{V_{PP}}$ = Peak to peak output voltage

2.3. Gas sensor chamber

The gas sensors was located within an airtight chamber with adequate conditions to work properly, preventing that other types of volatiles (from environmental air) can affect the measurements. It must be ensured that the chamber have not leak when the sample is introduced because it would lose the concentration of the volatile object of study. Table 1 describes the sensors which were used to be sure which sensors were more sensitive and selective to the different Volatile Organic Compounds (VOC's).
The sensor chamber (see Figure 7) was composed of four commercial gas sensors (Figaro and FIS SP) manufactured by (FIS Inc. Nissha Printing Co., Ltd., 1995) and (Figaro Engineering Inc. Figaro Gas Sensor Technical Reference, 1969). Each sensor has a different detection characteristic, because they are capable to generate a response with any type of volatile compound but will have a response when applied to a target gas for which it is more sensitive.

Each sensor has different sensitivity depending on volatile target, this selection was made in order to obtain an efficient and selective response at the time of making a discrimination of a volatile compound. The chamber was constructed with acrylic material of 1-centimeter thickness, and on top part there are two holes that allow the entrance and exit of air, through which the air of the sample should circulate with a constant flow.

Table 1. Metal oxide gas sensors

Type	Target Sensor	Heater Voltage	Circuit Voltage
TGS 800	Carbon monoxide, Isobutane, Hydrogen and Ethanol	≤ 5V ±0.2V AC/DC	≤ 24V AC/DC
SP-53 7Z6	Methane	≤ 5V ±0.2V AC/DC	≤ 24V AC/DC
TGS-826	Ammonia	≤ 5V ±0.2V AC/DC	≤ 24V AC/DC
SP-15A 921 F	Propane, Butane	≤ 5V ±0.2V AC/DC	≤ 24V AC/DC

To keep the chamber tightly sealed, 4 screws were used to generate pressure on the cover and the body respectively. It is covered rests on a rubber that ensures the tight seal and prevents leaks.

The sensors inside the chamber were operated with 5 DC Volts and the heating resistors were connected to the modulation circuits.

The camera was designed to change the configuration of the sensors, since these were connected with sockets, in order to be able to extract the sensors easily.

The load resistor connected to the output of the sensors has a fixed value of 4.7 kΩ, this resistance fulfills the function of voltage divider, which allowed to obtain a voltage variation when a volatile compound is applied to the sensor.

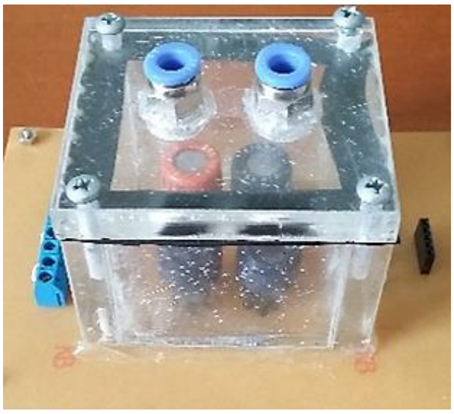

Figure 7. Gas sensor chamber

2.4. Data acquisition system

In order to make the acquisition of the signals coming from the gas sensors arranged in the matrix, an Arduino card 2560 was used to generate the modulated signals, data acquisition and process control and conditioning circuit.

The card has 8 analog inputs channels, which only 4 of them were used for the acquisition of the signals produced by the gas sensors and this was done with a sampling frequency of one second.

Two digital output channels were used for control the activation of a solenoid valve and air pump, therefore the system was totally automatic.

2.5. Data Processing

To make the discrimination of data set, we used the Principal Component Analysis (PCA) as a multivariate data analysis method that is widely useful in feature reduction, data compression, and variable selection and sometimes used for noise reduction [18]. This powerful tool for data analysis was selected in this application since it is an effective linear unsupervised and supervised method to extract the most relevant information and project data from several sensors to a two-dimensional plane using a scores plot. Therefore, it is possible to discriminate properly a measure set, finding the directions of maximal variance [19]. PCA returns a new basis which is a linear combination of the original basis. Each vector (orthogonal) has an amount of variance in the data set with a different degree of importance. The scalar product of the orthogonal vectors gives the value of the principal component.

3. Results

Figure 8 shows the response of the gas sensors TGS 800, SP-53, TGS 826 and SP-15A 921 F, this response was taken as a pattern or "fingerprint", when the heater resistance of the sensor was fed without temperature modulation. The figure depicts each response of the sensors when a sample of 2 ml of Ethyl Alcohol at 70% concentration was applied; in this way the gas sensor response can be determined by using a continuous voltage of 5 volts to the heater of the sensors, obtaining a very high percentage of sensitivity. Also, it responds instantly to the supply of the sample previously concentrated inside the chamber, where the sensors response is also determined by the type of compound, which it is sensitive, i.e. the TGS 800 is widely used to recognize gases like air and hydrogen, the TGS 826 is used to recognize ammonia, then they will have a response characteristic when these gases are supplied.

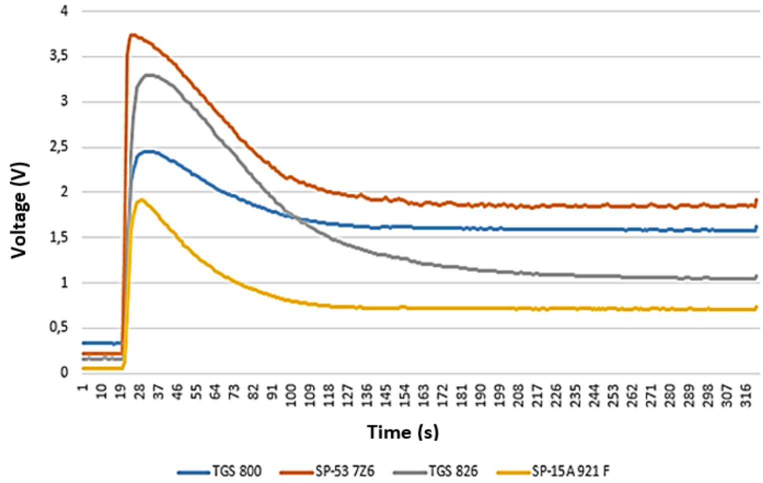

Figure 8. Response of the sensors without temperature modulation, exposed to a sample of Alcohol (70%)

The response of the sensors can be observed in Figure 9, when a PWM modulation using a DC voltage of 0 – 4.5 Volts was applied to the heater resistor with a frequency of 490 Hz and a 33.3% duty cycle, which was generated using the same concentration parameters of the sample. From this point of view was evaluated the behavior of sensors with modulation and with constant voltage of 5 volts, determining a low energy consumption through this method, good accuracy and repetitiveness.

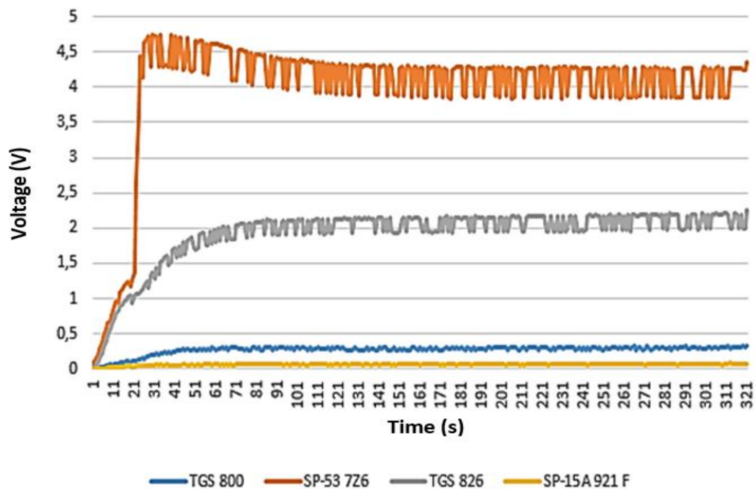

Figure 9. Response of the sensors to a PWM signal with a 33.3% duty cycle, on the heater resistor (Alcohol (70%))

Deducing the graph of the voltage response of the sensors by applying a PWM modulation with a duty cycle of less than 50%, it can be seen that the response when the sensor is feed tends to a maximum amplitude value. Thus, it is very similar to a response without modulation in the heater resistor, but with the difference that the response with the modulated signal in the heater tends to be kept longer with the maximum value, whereas the response when a continuous signal in the heater has a slope and tends to remain at a half value approximately of the maximum value obtained, when the sample is applied.

It can also be noticed that the response of the SP-53 7Z6, TGS 826 and SP-15 921 F sensors change completely since none of the sensors reaches the maximum value obtained, when the signal without modulation was applied to the heater. In this case, we could increase the sensitivity of the sensor SP-53 7Z6, applying PWM modulation.

TGS 826 and SP-15 921F sensors obtained a very small voltage response, such as can be seen in the figure, the maximum voltage reached did not exceed 0.5 volts, but nevertheless, it can be said that these sensors responded to the moment that was applied the sample of alcohol previously concentrated in the chamber. Figure 10 illustrates the response of the sensors subjected to the continuous airflow of a sample of 2 ml of 1% of *Moscatel* wine made in Colombia.

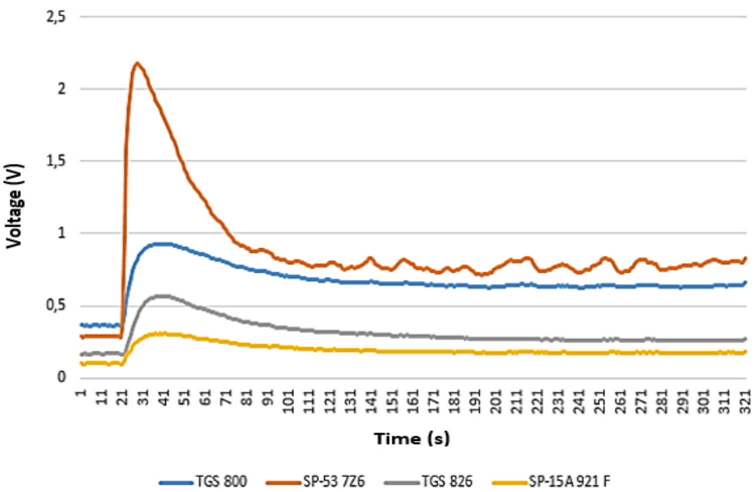

Figure 10. Response of the sensors without temperature modulation, subjected to a sample of 2% (Moscatel wine)

The behavior of this signal was determined when a sample of 2 ml of Moscatel wine at 2% of Alcohol was supplied to the sensor chamber. In this case the amplitude of the signal changes, being smaller when a sample of wine is applied. The sensitivity of sensor SP 53 was good, giving a voltage response almost instantaneously when the wine sample was applied. Figure 11 illustrates the response of the sensors exposed to the continuous airflow of a sample of 2 ml of Moscatel wine to 2% by applying a PWM modulation with a frequency of 490 Hz and a duty cycle of 33.3%. In this experiment the voltage applied to the heater resistance was 0 - 4.5 volts, since the temperature modulation was applied. Therefore the sensor response (i.e, selectivity) was better unlike when was applied only 5 volts using constant voltage.

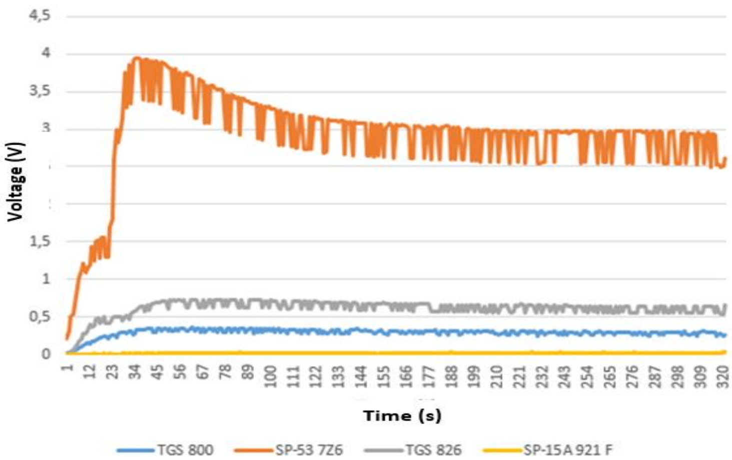

Figure 11. Response of the sensors subjected to temperature modulation with a sample of Moscatel wine Alcohol (2%)

Despite to reduce the amplitude of SP-15A and TGS 800 with temperature modulation, was possible improved the selectivity and sensitivity of SP-53 and TGS 826 sensor, achieving an excellent amplitude response and it is noteworthy that they are still sensitive when the temperature modulation was used. The amplitude of the signal tends to remain stable after reaching the maximum amplitude, this was a satisfactory response being for analysis and data processing. In this preliminary study it can be said that the response of the sensors can reach a good performance when modulation is applied, whereas the data processing can be easily at the moment to obtain the data discrimination and/or succes rate of classification. The changes of voltages produced by the sensors and each response varies the amplitude according to the type of volatile compound. To get the discrimination of data set, a modulated sawtooth signal was used to the heater of each sensor. Then a data matrix (16 measurements: 12 *Moscatel Wine* (**W**) with 2% of alcohol and 6 measurements with 70% of *Ethyl Alcohol* (**A**)) was calculated and auto-scaled before applying the PCA, in order to make all the sensors with equal weigh. Figure 12 shows the PCA analysis where an excelent classification was achieved.

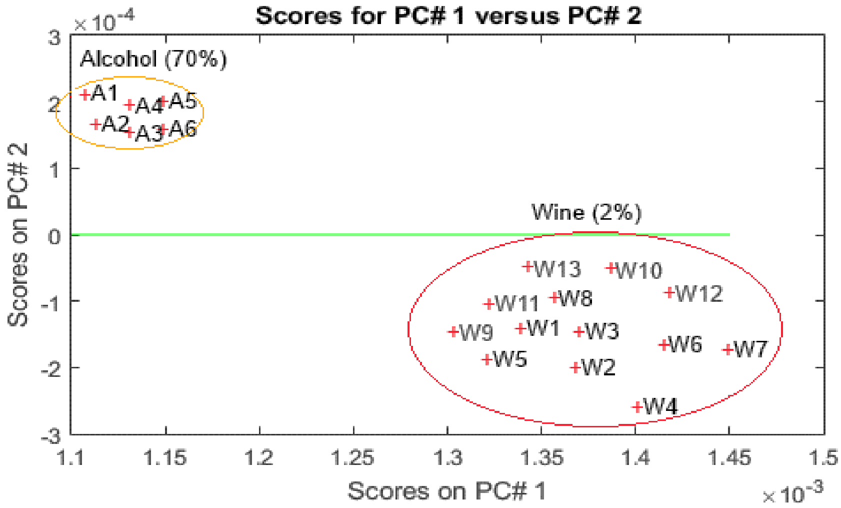

Figure 12. PCA to discriminate the measurements of alcohol (70 %) and wine (2 %).

The PCA was performed with all sensors since the features extracted and responses of them were suitable. This figure depicts an excellent discrimination using the two PC's reaching a 94.5% of variance and 100 % of accuracy the data set. It is important to note that these results showed a good sensitivity, selectivity, and repeatability.

In this study, the main purpose of this test was tried to discriminate between two compounds with different degrees of alcohol, in order to obtain an approximation of the performance of sensors when they are subjected to this type of modulation. With these preliminary results would be possible to implement this system in food applications to evaluate the different degrees of alcohol in several types of wines.

4. Conclusions

In this study a multisensorial device was developed and implemented, in order to obtain electrical signals produced by the gas sensors when a volatile compound was supplied and the temperature of the heater was applied to change the response through modulation techniques. The signals from the gas sensors were acquired by an data acquisition card of low cost and then were stored in a computer to perform the data processing and classification.

According to the measurements obtained with the electronic nose system, it was possible to determine which sensors vary their response according to the type of modulation that is useful to the heater.

The performance of the electronic nose system was improved when a temperature modulation was applied to each sensor, it could be noticed since the sensors generated a voltage output, whereas was possible to identify the final voltage to be applied to each sensor. This promising behavior of the sensor allowed to implement a processing and/or discrimination method for volatile compounds discrimination; for example, to obtain the relevant information of data set we used the static parameter $G = G_{max} - G_{min}$, whereas was possible to extract and feed to a pattern recognition techniques like PCA analysis.

The sensitivity of sensor SP 53 was better unlike other when temperature modulation was used, giving a voltage response almost instantaneously when the wine and alcohol samples was conditioned.

An excellent discrimination using two PC's with 94.5% of variance and 100 % of accuracy of data set was reached, when the sensors were exposed to a continuous flow of a wine sample (i.e, 2 ml of wine to 2 % and 70 % of ethil-alcohol) by applying a PWM modulation with a frequency of 490 Hz and a duty cycle of 33.3%.

Applying another type of data processing method a better comparison could be made to perform the discrimination or classification of measurements, and with this result, to validate the response of the sensory system through the temperature modulation.

The results with PCA Analysis could be obtained in fast and easy form since was possible to acquire the information in "on-line" mode and realize the processing instantly.

Conflicts of Interest: The authors declare no conflict of interest.

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