**UNIVERSITÀ DEGLI STUDI DI ROMA**
**“TOR VERGATA”**

Facoltà di Ingegneria
Corso di Laurea in Ingegneria Elettronica

Tesi di Laurea

# “DESIGN AND TEST OF GAS SENSOR TEMPERATURE MODULATION FOR MEDICAL APPLICATION.”

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To my family.

# Gratitudes.

I would like to take this opportunity to express my gratitude to the people who has been with me until the end of this period.

First of all, thanks to my parents and my brother to teach me the values that makes me to arrive here, and also its unconditional support in all moment.

To my family for supporting me ever, when I was with a bad temper on the previous days of an exam or when it doesn’t come up how I have expected or simply for all the moments of happiness.

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# Index

# Introduction

Before entering deeper into the subject in question it is useful to make a summary of the principal topics in which is developed this thesis.

The first chapter, it is principally used to present the definitions, the classification and main characteristics that makes a sensor to be distinguishable from other devices. We would like to emphasize with all kind of details the intern structure of the chemical sensors of metal oxide and the mechanisms that are able to describe its principle operation. Finally, are introduced the techniques to make the temperature modulation.

The second chapter, it is however used to talk about the description of the system. It is defined the description of the 'self-adaptive' system and its operating function with feedback functions. We explain the different parts in which the circuit it is decomposed into, by emphasizing the sensor and its variable resistance.

The third and last chapter it is used to describe the experimental results, as well as, the devices used in the experiments and the way of acquire data. With this propose we have used different Matlab codes and its analysis it was completed with the Principal Component Analysis (PCA).

# Chapter I

## ***1.1 What a sensor is***

The sensor, in a general form, is defined like a device able to detect different kinds of materials, what means that it receives a signal or stimulus and respond to it with a electrical signal of output.

The transducer is defined as a device that transforms a physical variable (force, pressure, temperature, speed, etc) into another one.

Base on the two previous definitions and as we know that in practice these terms are commonly interchangeables what means that they are used as synonymous.

In fact we use a sensor as a transducer when we have a variable to measure and it requires a electrical signal in the output. Also these can be invasive, which means that, the sensor must be in direct contact to be able to detect or transform the physical variable, and no invasive that is when the sensor doesn't need to be in direct contact to do it.

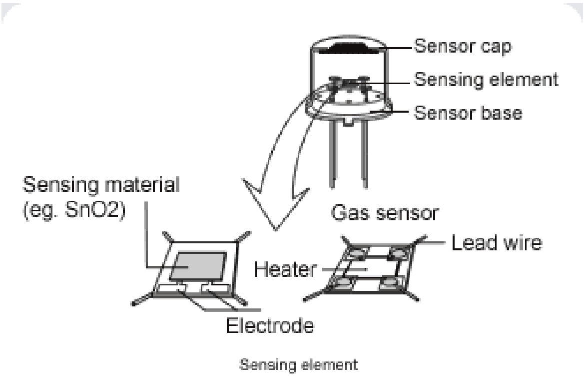



Fig. 1 - Sensor structure

## ***1.2. Operational or functional features***

They describe the response of the sensor as a function of the magnitude of the input and they are classified in:

*Statics*: Describe the response of the sensor in a permanent regime or slow changes of the input magnitude.

*Dynamics*: Describe the sensor response in front of significative changes of the input magnitude.

## ***1.2.1 Static characteristics***

a) Static Transfer Function

Relation between the input applied to the sensor and the output in a static regime.

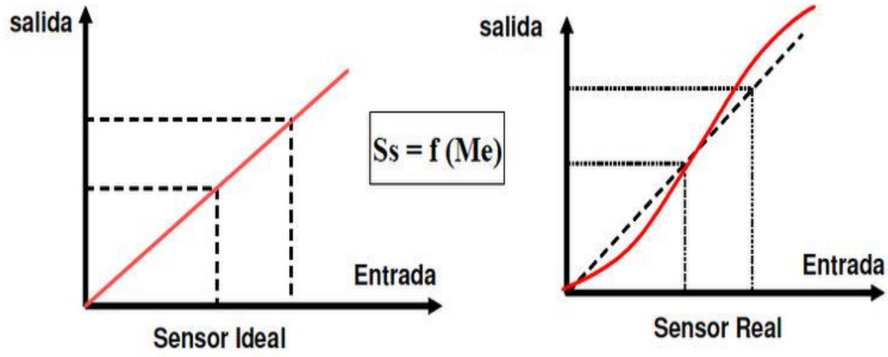



Figure 1.2: Static transfer functions.

b) Sensitivity

It is the variation that experiments the output signal when there is a little variation of the input signal.  It is the change of the output variable for each unitary change of the input variable. The sensitivity  is in fact the slope of the calibration's curve calculated in the working point. It is one of the most important parameters of a sensor along with linearity of the output, the range of measure and the accuracy.

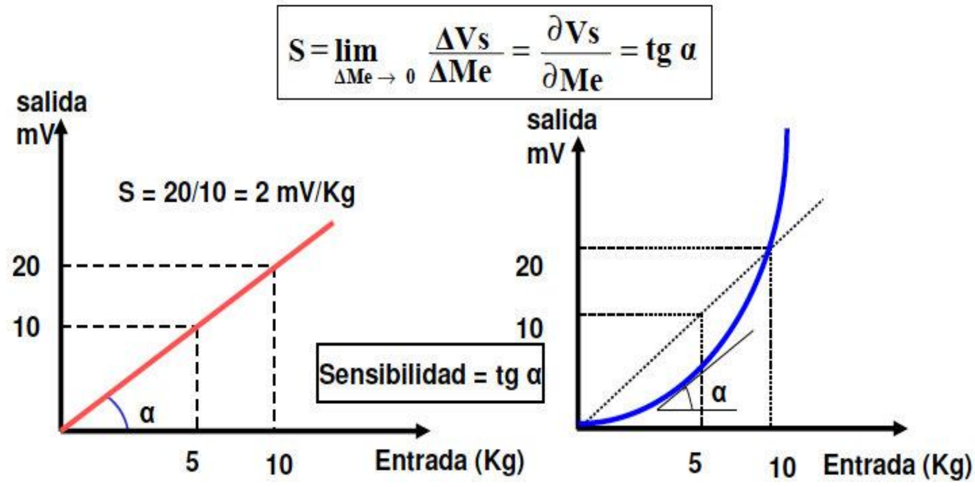



Figure 1.3: Sensitivity of a sensor

c) Linearity

Express the grade of coincident between the calibration's curve and one determined straight line. Can be expressed in three different ways which are:

*Independent*: The line it is defined by the method of least squares. This way the maximum positive error and the minimum negative are the same.

*Adjusted a zero*: the line it's defined by the same method but with the restriction of make it going through the zero.

*Terminal*: the line it is defined by the output without input and the theorical maximum output that corresponds to the highest admitted input.

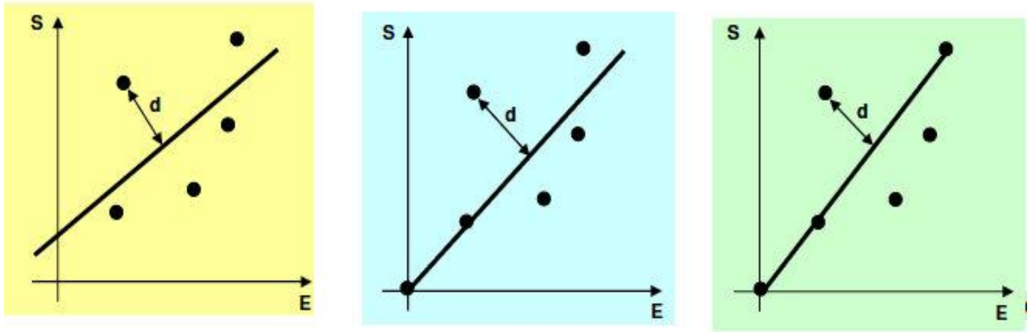



Figure 1.4: a) Linearity independent, b) Linearity adjusted a zero, c) Linearity terminal.

d) Non linearity

Maximum deviation of the calibration's curve regarding to the rect. The value of the non linearity it is used to be expressed in % respect to the scope (Span).

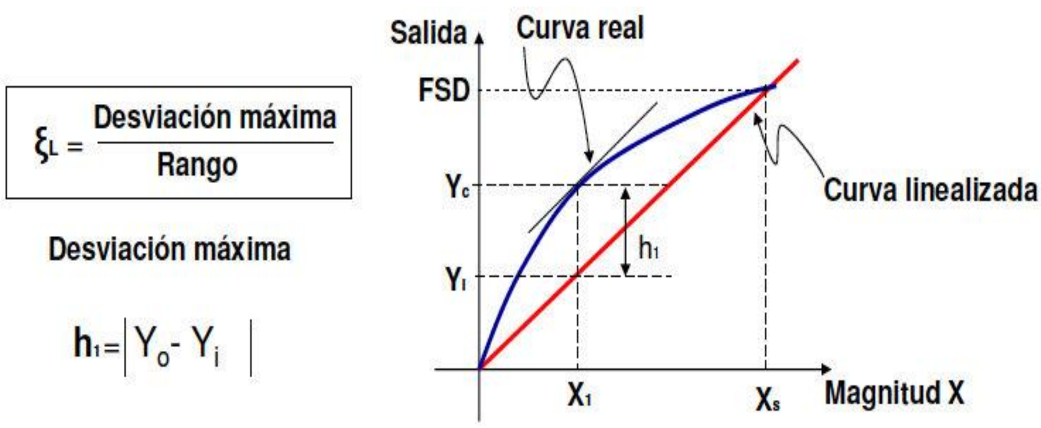



Figure 1.5: Non linearity of a sensor.

e) Hysteresis

Makes the difference between the values of the output that corresponds to the same value of the input, according to the reach, in one sense or another.

The hysteresis is used for removing the oscillations that appears at the output of the sensor, when the level of the input variable coincide with the one that makes change of state the output.

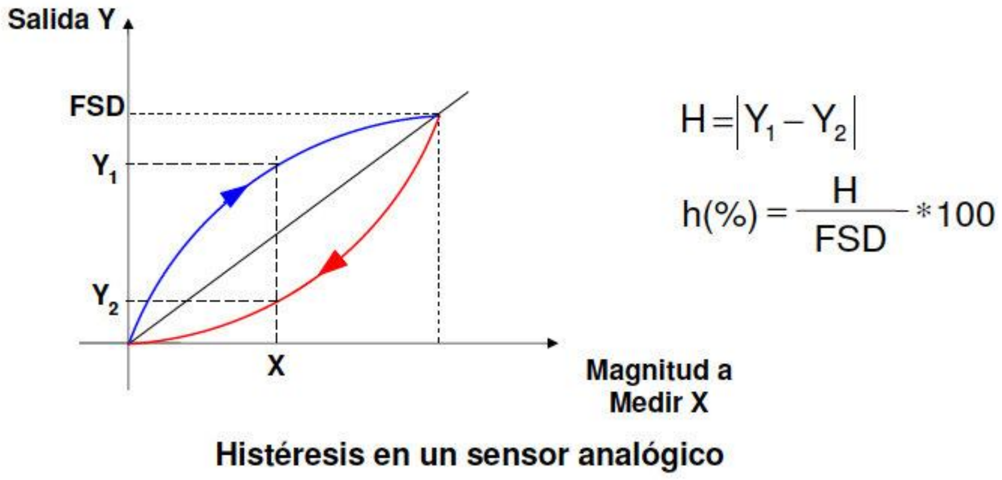



Figure 1.6: Hysteresis of a analogical sensor.

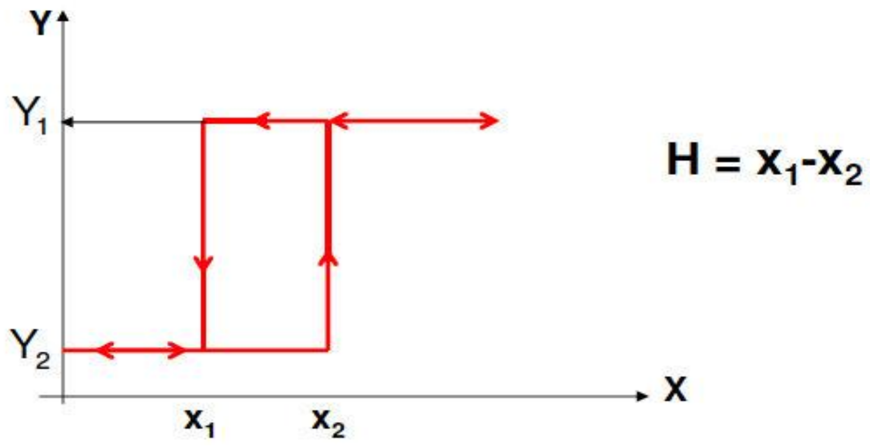
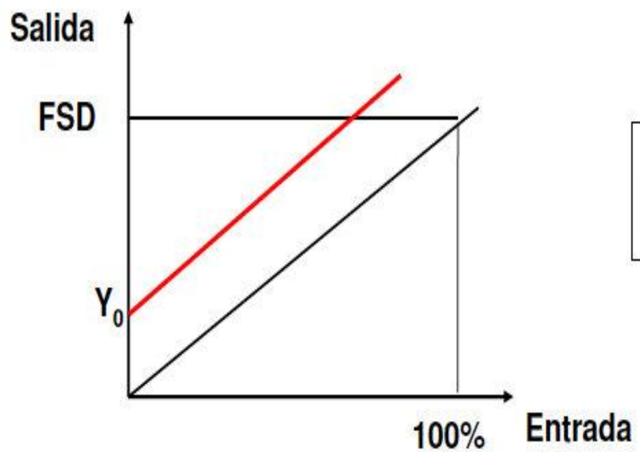



Figure 1.7: Hysteresis of a sensor.

f) Drift

Variation of any aspect of the calibration's curve regards to any ambient parameter (temperature, humidity, etc.) o regards to the time. The drift or the offset is used to be lineal with the measure and for that reason it is calculated as the output value with null input. It is expressed in % respect to the reach.




$$\boxed{\xi_D = \frac{Y_0}{FSD} * 100}$$

Figure 1.8: Drift of a sensor

<u>g) Resolution</u>

Minimum increment of the input variable which produce a measurable change at the output. It is the minimum increment measurable of the input variable. It is used expressed in % of the FSD value.

When the increment of the input occurs from zero, it is denominated umbral (threshold).

h) Precision

It is the capacity of a system to provide the same result when the measure is repeated in a determined conditions. It's also named fidelity.

i) Accuracy

Characterize the correspondence of the result among themselves and with the true value, (precision and veracity). It's a concept that characterize the capacity of a system to provide the exact value of the variable that it's measured.

j) Repeatability

The capacity to get the same value of a magnitude when it's measured several times in a short interval of time in determined conditions.

k) Reproducibility

Capacity to get the same value of a magnitude when it's measured several times in a long interval of time, by different people or by the same person with different instruments or in different laboratories.

## ***1.2.2. Dynamic characteristics***

a) Delay time

It is the time that goes by from the application of the input signal and the output reach out a 10% of the final value.

b) Rise time

It is the time that goes by between the instant in which the output signal reach out a 10% of its final value and the instant in which reach out the 90% as a result of a change in the input magnitude. It is denominated $t_s$.

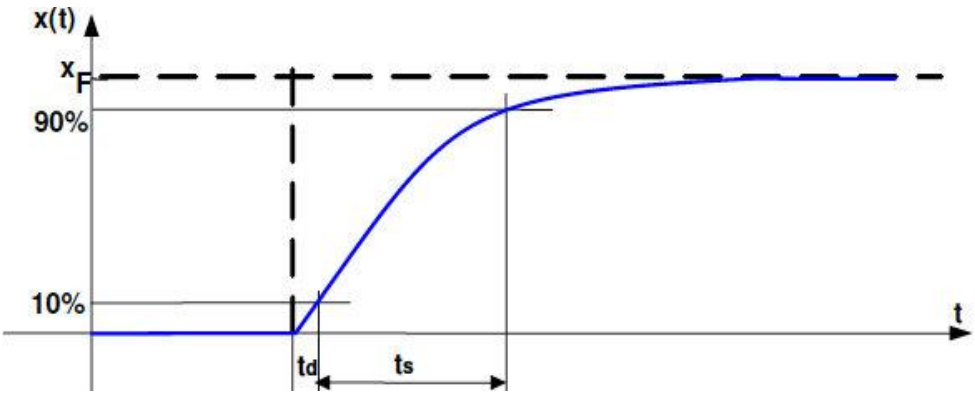



Figure 1.9: Rise time of a first order system

c)Response time

The time that pass from when a change of the magnitude measurable it is applied to the output when reach a determined percentage of its final value. When the percentage is of a 90% coincide with the delay time added to the rise time.

$$tr = td + ts$$

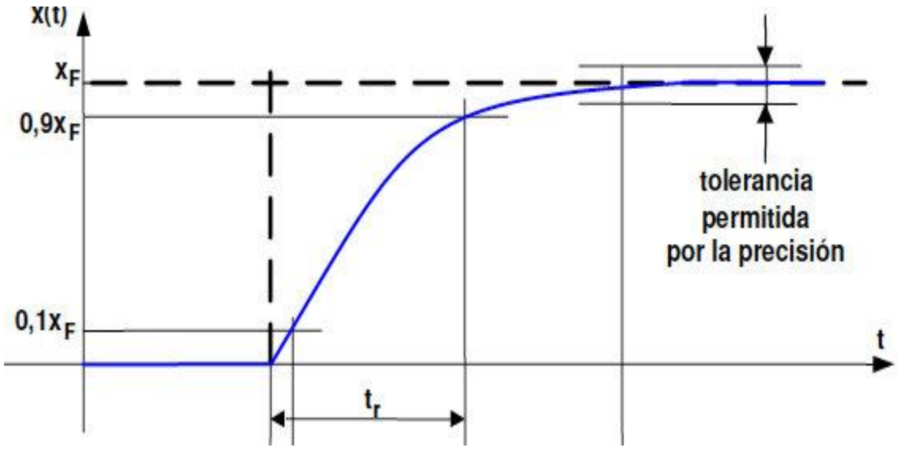



Figure 1.10: Response time of a first order system.

d) Time constant

It is the time that goes by from the application of a change of the magnitude we are going to measure to the 63.2% of its final value. Represents a particular case of his response time.

e) Settling time

It is the time that goes by from a application of a change in the input until the system provides an output inside the margin of tolerance defined by the accuracy. Sometimes it is considerate that its value is five times the time constant.

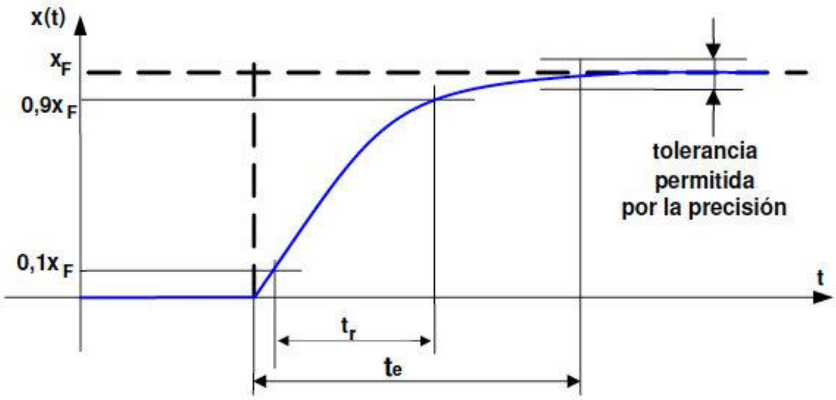



Figure 1.11: Settling time of a first order system.

## 1.3. Metal Oxide Sensors

### 1.3.1 Introduction

Without any doubt this is the most studied metal oxide in the gas detection because of it is a material that has a very low cost and it shows a high sensitivity to a great variety of gases. The realized studies include the use of a big number of different dopants that improve the response time, the operation time and the selectivity among other properties. The morphology of the surface of these types of sensors has been characterized using several techniques with the aim of explain the results that has been got the investigated that has worked with this material.

The tin dioxide has two stabile forms SnO and SnO2, in his pure state the Sno2 it's a semiconductor with a band-gap of 3.6eV and it presents a good sensibility to the gases in their non-stoichiometric, it means SnO2-x.

The detection principle it's based on the conductance's variation, this variation depends on the extern gas and the operation temperature, this allows the detection of several toxic gases a low levels (parts per million). The conductivity of the tin dioxide it's denominated by the superficial conductivity.

Eventually the tin dioxide became the dominating gas sensitive material. The most significant contribution into the development of this technology was provide by Japanese scientist and engineers including Naayoshi Taguchi, who developed a series of ceramic sensors called Taguchi Gas Sensors (TGS)

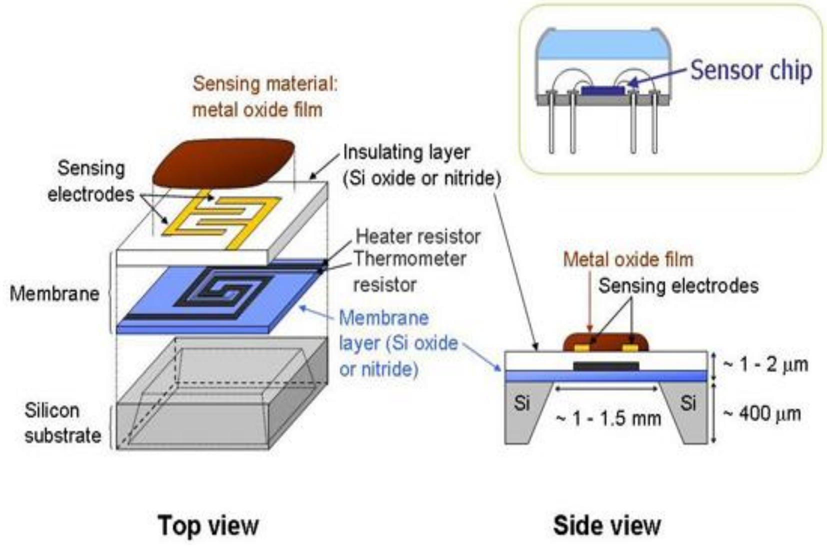



Figure 1.12 View of a TGS sensor.

### ***1.3.2. Operation principle***

When a metal oxide crystal such as SnO2 is heated at certain high temperature in air, oxygen is adsorbed on the crystal surface with a negative charge. Then the donor electrons in the crystal surface are transferred to the adsorbed oxygen, resulting in leaving positive charges in a space charge layer. Thus, surface potential is formed to serve as a potential barrier against electron flow. Inside the sensor, electric current flows through the conjunction parts (grain boundary) of SnO2 micro crystals. At grain boundaries adsorbed oxygen forms a potential barrier which prevents carries from moving freely. The electrical resistance of the sensor is attributed to this potential barrier. In the presence of a deoxidizing gas, the surface density of the negatively charged oxygen decreased, so the barrier height in the grain boundary is reduced. The reduced barrier height decreased sensor resistance.

The relationship between sensor resistance and the concentration of deoxidizing gas can be expressed by the following equation over a certain range of gas concentration.

$$Rs = A[C]^{-\alpha}$$

where: Rs = electrical resistance of the sensor
A = constant
[C] = gas concentration
$\alpha$ = slope of Rs curve

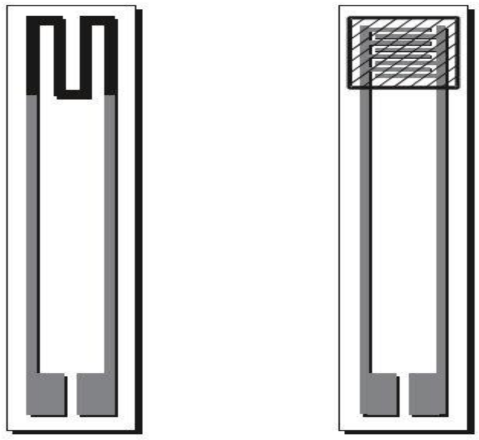


Figure 1.13: Thick film gas sensor layout. Platinum heater a) and gas sensitive layer deposited on interdigital type Au electrodes b)

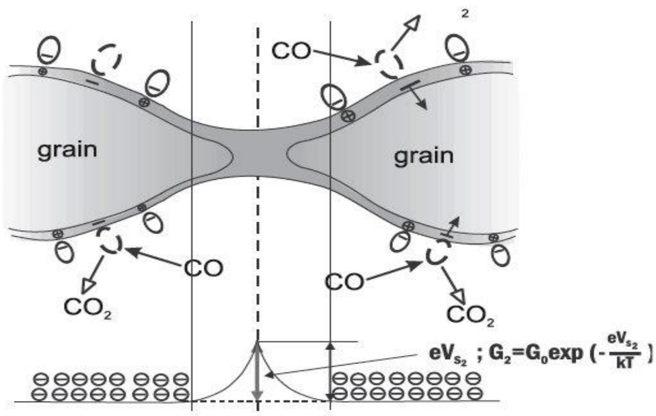



Figure 1.14: Model of inter-grain potential barrier in the presence of gases

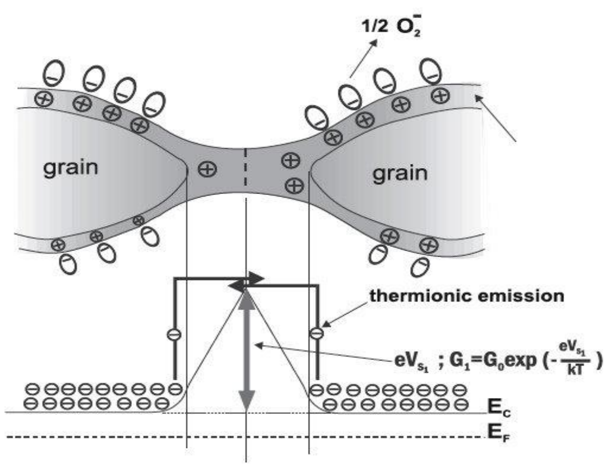



Figure 1.15: Model of inter-grain potential barrier in the absence of gases

### ***1.3.3 Temperature modulation***

In a review of semiconductor gas sensor selectivity in 1987, Morrison, concentrating on the use of catalysts, stated that “temperature programming of the sensor is not a common technique used for selectivity”. However, more recent work has demonstrated that temperature programming or modulation of a single sensor can achieve the type of selectivity that would otherwise require arrays of variously doped fixed temperature sensors.

Selectivity in semiconductor gas sensors can be obtained through a variety of methods which can be classified into four main groups: a) the use of filters or chromatographic columns to discriminate between gases on the basis of molecular size or other physical properties, b) the use of catalysts and promoters or more specific surface additives, c) the physical preparation of the sensor material, and d) the analysis of transient sensor responses to changes in analyte concentration or sensor temperature. Of the last category, the most commonly employed technique involves controlling the temperature of the semiconductor surface, whether by selecting a fixed temperature to maximize sensitivity to a particular analyte gas, or by programming or modulating the temperature.

It is widely accepted that the key process in the response of the semiconductor to a reducing gas involves the modulation of the concentration of adsorbed oxygen species such as $O_2^-$, $O^-$ or $O^{2-}$. By withdrawing electron density from the semiconductor surface, adsorbed oxygen gives rise to Schottky potential barriers at grain boundaries, and thus increases the resistance of the sensor surface. The surface oxygen concentration will decrease reducing gases  and thus decrease the sensor resistance. While the identity of the surface oxygen species remains slightly more controversial, it is clear that different gases have characteristic optimum oxidation temperatures, and therefore give rise to characteristic conductance–temperature profiles, which can be modified by doping the semiconductor with noble metals or other catalytic materials the figure 16 shows some typical responses of doped and undoped $SnO_2$ sensors to various analyte gases. In general, the temperature of the gas sensor surface is controlled by varying the voltage applied to the sensor heater.

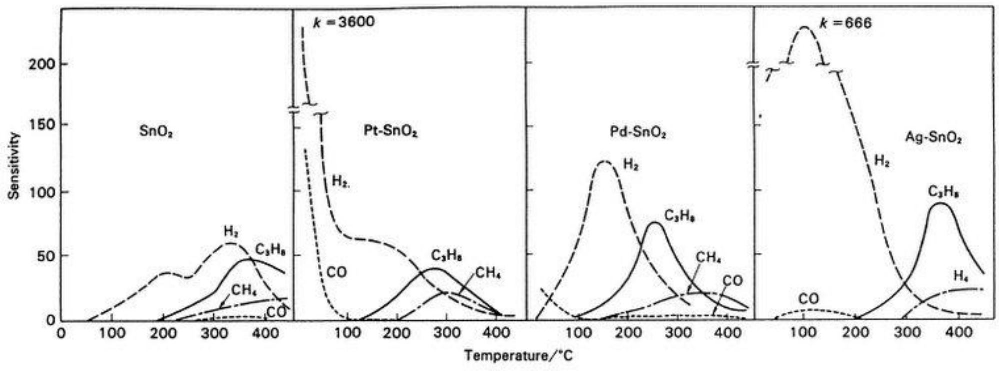



Fig. 16. Sensitivity–temperature profiles for doped and undoped tin oxide sensors in the presence of various analyte gases.

Other factors, both chemical and physical, which depend upon temperature and contribute to the sensor response, have been identified. These include rates of adsorption and desorption (of oxygen and reducing gases, or of oxidation products), the rate of surface decomposition of reducing gases, the charge-carrier concentration and the Debye length in the semiconductor. This means that the true relationship between the conductance of a semiconductor sensor in the presence of reducing gases and the temperature of the sensor surface is very complex. Another complicating feature of gas sensor operation is that the chemical reactions that give rise to the sensor response are usually exothermic, and thus make uncontrolled contributions to the sensor temperature. Furthermore, the actual measurement of the sensor conductance requires the flow of a small current through the sensor and thus causes Joule heating of the sensor, although the relative significance of this is questionable.

An important aspect of the temperature-dependence of gas sensor conductance is the hysteresis observed when the sensor temperature is continuously elevated and subsequently cooled over short time periods. It is found that hysteresis effects in sensor response to $O_2/N_2$ mixtures were minimized if the sensor temperature was varied slowly enough, that is, at around 20 K $h^{-1}$. This was done in an attempt to operate the sensor in a quasi-steady state at each temperature. It is attribute hysteresis effects to the slow attainment of equilibrium in the transfer of charge between adsorbed oxygen species and the semiconductor.

How it was said before the sensors of metal dioxide because its nature they are sensible to the temperature, this is as a result of how the temperature it is involved in the oxidation and reduction process. It is noted that a different temperatures the same gas interacts in a different way with the sensible layer. It is clear that the diverse value of the resistance when the temperature varies, fixed to the concentration of a kind of gas, it leads to take in consideration not only of a sensibility curve but in a surface of sensibility, where the independent variables are the concentration of the fixed gas and the temperature.

Making reference to the Figure 1.17 it is possible to detect that the times when the equilibrium it is get to are of milliseconds, also it is interesting take count that the curve in the atmosphere of CO changes from the transitory of heating to the transitory of the cooling. This phenomenon it is because of the latency with which the equilibrium is reach out. This phenomenon serves only for temperature variation with a speed lower than 20K/h, variations in this way with a little variation of temperature the sensor is always in equilibrium conditions.

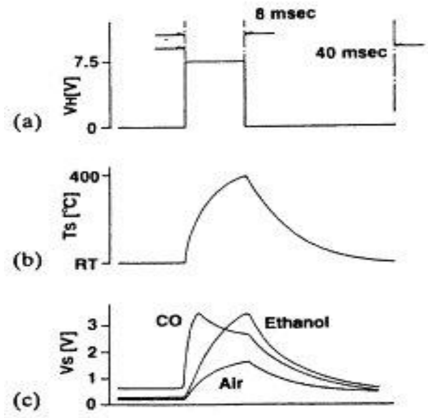



Figure 1.17: Operation of a "pulse-drive" sensor: (a) heater voltage; (b) sensor temperature; (c) sensor output voltage. Ethanol and CO concentrations are both 1000 ppm .

In summary, temperature modulation or control can be used to improve the selectivity and sensitivity of semiconductor gas sensors. This is possible because each analyte gas has a characteristic conductance vs. temperature (or applied heater voltage) profile for each sensor type. This means that if the response of one sensor is measured at n-temperatures, the sensor becomes analogous to an array of n “sensors”. Thus if m different actual sensors are used, m·n dimensional information is afforded for analysis, and herein lies the great potential for these techniques.

# Chapter II

## ***2.1. Electronic interface***

We are going to describe the operating principle of the blocks that are used. It will be also explained how form the response curve are taken the parameters used to describe the response of the modulated sensor.

The principal circuit can be represented by the next scheme of block where 2.1 the principal blocks are: the sensor, the multivibrator astable and the frequency divisor.

The operation of the circuit it is based on the astable circuit and the frequency divisor it is used to improve the discrimination features of the circuit.

In a multivibrator astable the duration of the impulses is regulated with a fixed parameter the threshold of commutation of the circuit and the value of the temporization product. During the operation the elements of temporization keep fixed but for us in this case the sensor resistance changes depending on the kind of gas and the concentration and for this the duration of the impulses depend on the gases that are presented on the atmosphere. Also for keeping the benefits of the thermal modulation it can be used the feedback the output signal of the multivibrator to the heating with the propose to make the thermal cycles. The feedback anticipate a decrease of the impulse number for minute in this way it is possible take a bigger interval of temperature, the interval of temperature depends on the gas and its concentration.

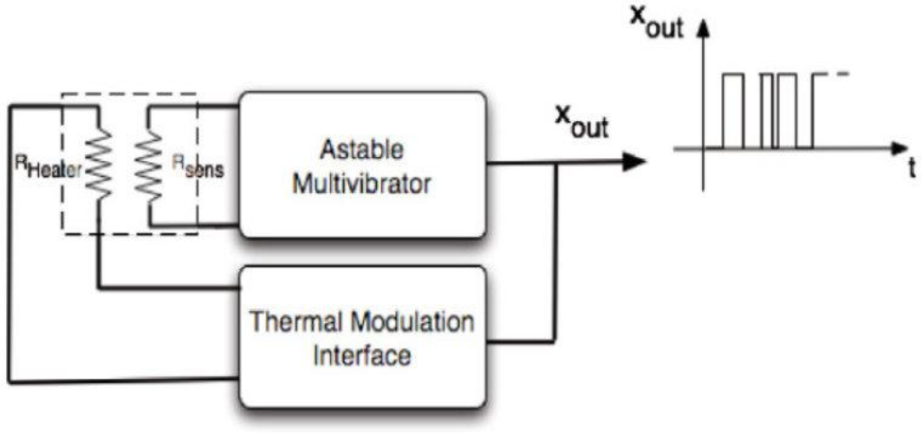



Figure 2.1: Block diagram of the performed circuit.

## ***2.2. Structure***

To make a periodic signal that depends on the resistance of the sensor it needed to use a multivibrator. We have used the integrated circuit NE555 in the astable configuration.

This device it is an integrated circuit very stabile which the principal function is to produce pulses of temporization with a high precision and, also, can be used as an oscillator. The two operation modes are monostable and astable the one that we are going to use in the astable mode.

On this configuration the device is used as a squared waveform generator in which the form of the output its regulated by the two extern circuits RC, one used for the load and the other one for the unload.

The times of load and unload are directly proportional to the value of the resistance and therefore the frequency of oscillation of the output signal of the multivibrator will depends on the instantaneous resistance of the sensor. The output of the multivibrator it is sent as an input to the second block which is the frequency divisor.

The frequency divisor through itself, the square waveform signal experiments a decrease of frequency. The obtained signal it will be adapted for being the input of the sensor (heating). With the aim of make the thermal modulation. The aim of making the frequency lower it is made because it is needed that the voltage variations applied to the heating must be compatibles with the time responses of the sensor.

The last block is the sensor with two different components: the sensor and the generator that introduces the time variable inside the system generating a proportional signal to the time that has occurred.

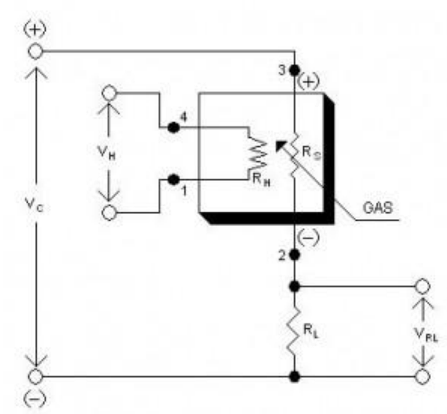



Figure 2.2: The following circuit shows about TGS2610-D00 Gas Sensors Circuit Diagram.

## CHAPTER II: ELECTRONIC INTERFACE

The sensor device is designed approximately for reproducing with the higher accuracy the variable resistance behavior of a tin dioxide sensor.

First of make any considerations we need to have clear some concepts which are:

- The tin dioxide sensors change its sensibility of a gas depending on the work temperature.

- The transient response of a sensor with a voltage applied to the heating varies in base to the composition of the atmosphere in which it is used.

- The temperature in the sensors reach out the equilibrium in a time of milliseconds.

- The tin dioxide sensors have hysteresis thermal if they are in quickly cycles.

- The temperature modulation improves the selectivity of the tin dioxide sensors.

The thermal modulations until now used are externs it means that they are chosen first of making the measure based on the precedent considerations.

Then, the characteristics that we are going to take considerations are: the dependence of the resistance to the temperature, type and concentration of gas and response time.

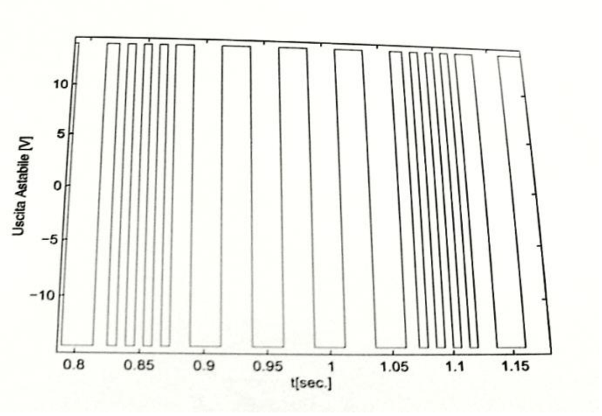



Figure 2.3: Output signal of the astable

# Chapter III

## ***3.1. Experimental results***

In this chapter it will be explained the way it were made the experiments and the results that we have obtained.

## ***3.2. Instruments***

To make possible the experimental verification of the circuit behavior it will be needed to heating the samples into water which it will make it to reach out the temperature of 37ºC. To make this happen we have designed a circuit scheme in figure 2.3. The system can be subdivided into two parts the part of controlled of the measure of the sample and the other of acquisition.

## ***3.3. Acquisition***

The most important signal of the circuit is the squared waveform signal produced form the astable, particularly the informative content of this signal it is in the temporally duration of the impulses. The acquisition, therefore, it shouldn't be needed to have a converter analogical to digital with a high accuracy in regard to the quantization, on the other hand it well be necessary that it will have a sample frequency pretty high to allow make difference the duration changes of the impulses and also to make the minimum error possible in the evaluation.

To make the acquisition we have used an USB-N16811 card of the National Instrument, and a code in Matlab which is required for the acquisition. The data acquired can be instantaneously treated with Matlab.

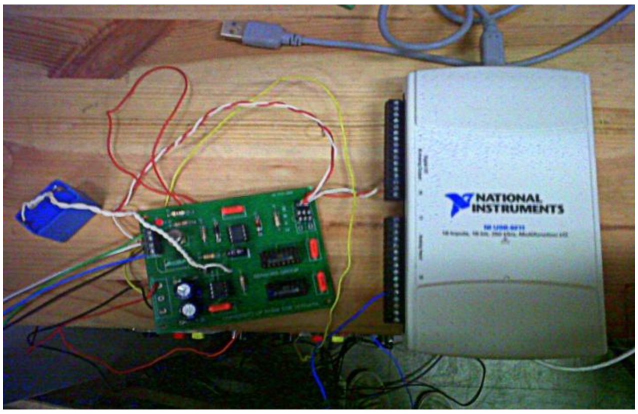



Figure 3.1: Circuit used and USB-N16811 card of the National Instrument.

## ***3.4. Measurement protocol***

In our experiment we have 3 samples of cells offered from the department of the University of Tor Vergata. They are 3 glass tubes filled with human cells that have been treated, cultured and were grown under standard conditions. Also we know that two of them are normal cells and the other one carcinogenic.

With the use of the Gas Chromatograph on the headspace of this samples it can be detected volatile organic compounds (VOC's), this compounds could form part of the following groups :hydrocarbons, alcohol, ethers, ketones, esters, aldehydes and aromatic amines. In the 4 firsts groups the quantification of VOC's are increased and on the other three respectively the quantification is decreased.

Coming back to our topic apart of the 3 samples we are going to measure another glass bottle with treated and distilled water and other glass bottle with air. All the measurements are taken inside a vessel the glass bottle with air to see its behavior and each time a different glass bottle sample.

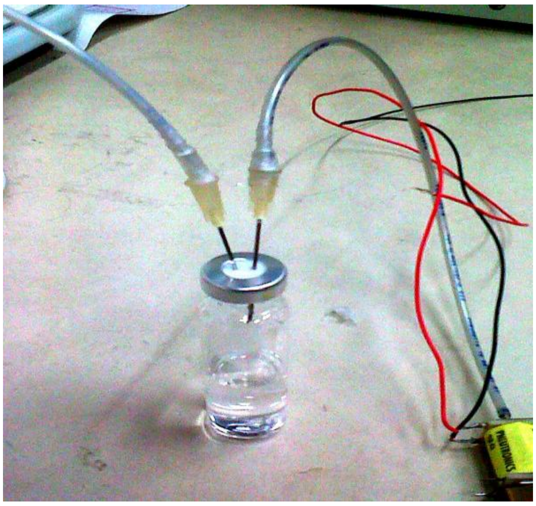


Figure 3.2: Glass tubes with water sample.

With the aid of a electro valve at which arrive the flow of air, it is able to separate two tubes ones that arrives to the sample and the other one to the air bottle. the two outputs arrive, in turn, to the sensor making a switch at a determined moment the outputs will be alternated and this ways it is possible to acquire the data for processing.

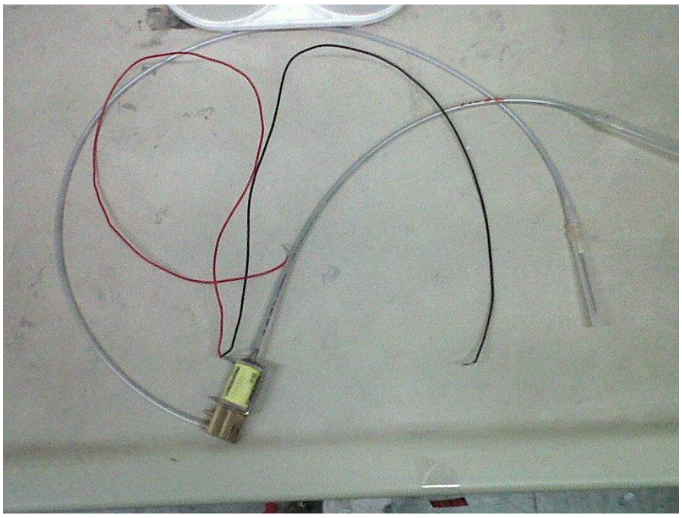


Figure 3.3: Electro valve used to make it switch.

# CHAPTER III EXPERIMENTAL RESULTS

To make the results obtained being reproducible, the measures must to be with the following process.

a) We must to leave the sensor in air for a big time allowing to the resistance of the sensor to be stabile.

b) First of doing the measure we must have the sensor under an air flow of 40 sccm[1] about 20 minutes and to allow the samples to reach the temperature adequate at 37ºC .

c) After reach the appropriate conditions we proceed to the measurement making for each time a measurement of a sample of air and then we switch with a power source of 5 volts. This way we can make a comparison between the two measurements.

d) Between two measurements it is needed to wait 20 minutes introducing air to make it possible the cleanup of the sensor.

[1]sccm: Standard Cubic Centimeters per Minute, a Flow measurement term

## ***3.5. Principal Component Analysis***

PCA is the simplest and most widely used method of multivariate analysis. However, sometimes because of its bad use the results could not be well interpreted. The peculiarity in a representation of the data set onto a subspace of reduced dimensionality where the statistical properties of the original data set are preserved.

PCA consists of finding an orthogonal basis where the correlation among sensors disappears. Its principal use it is to represent a set of data of a covariance matrix that it is not diagonal and has N dimension in a space of dimension inferior a N in which the same data are represented of a diagonal covariance matrix. The diagonalization it is obtained with a rotation of the coordinates in the base of the eingenvectors that constitute the principal components. The eigenvalues describes the elongation of the ellipse through the same principal component. It is important to realize that if the originary variables are partially correlated among them, some eigenvalues must have a value neglected and they could be eliminated so we can limitate the representation. Therefore considering the relative values of the eigenvalues ƛ (i) it is possible to reduce the representation to only those components carrying most of the information. Given a matrix of data PCA results in two quantities usually called scores and loadings. Scores are related to the measurements, and they are defined as the coordinates of each vector measurement (a row of matrix **X**) in the principal components basis. The loadings describe the contribution of each sensor to the principal components basis. A large loading, for a sensor, means that the principal component is mostly aligned along the sensor direction.

### ***3.5.1 PCA Application***

Assigned a matrix X of data, normalized to zero-mea, the principals components are the eigenvalues of the covariance matrix. The coefficients on the original base are called loadings, P. The data of the matrix X expressed on the reference system of the principal components, take the name of scores T, and they are calculated:

$$T=XP$$

The matrix it is the result of the product of the loadings and the scores:

$$X=TP^T$$

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Through the operation X·P it is made the base change and it is determined the coordinates of the singular data in the reference of the principal components. In this reference the covariance matrix has a diagonal form and the data are not correlated among them.

The graphical representation of the data in the base of the principal components it is called *score plot.* The analysis of the eigenvalues allows studying the distribution of the variance of the data in the different principal components and allows individualizing the eigenvectors most significantly.

## ***3.6. Results***

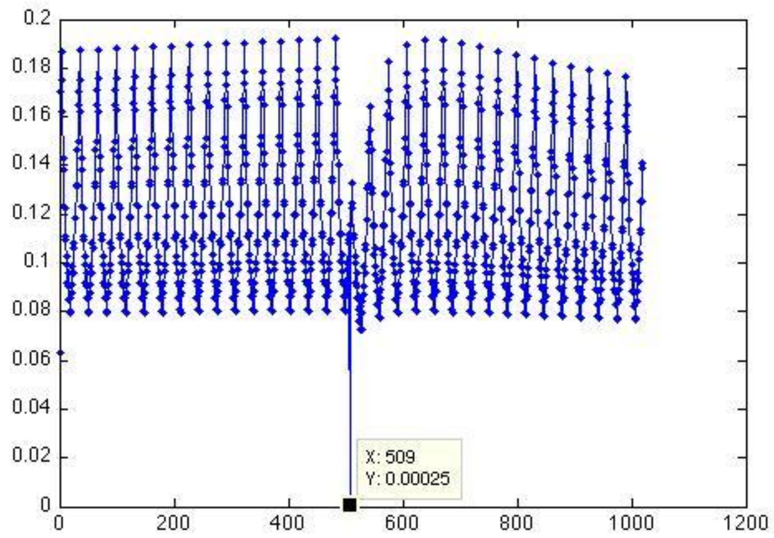


Figure 3.4: The first part of the graph goes until the switch that it is fixed at x:509. This first part corresponds to the air acquisition.

Next it will be shown the PCA of the 3 samples and the water where we can easily make differences among them on their situation after three followed repetitions.

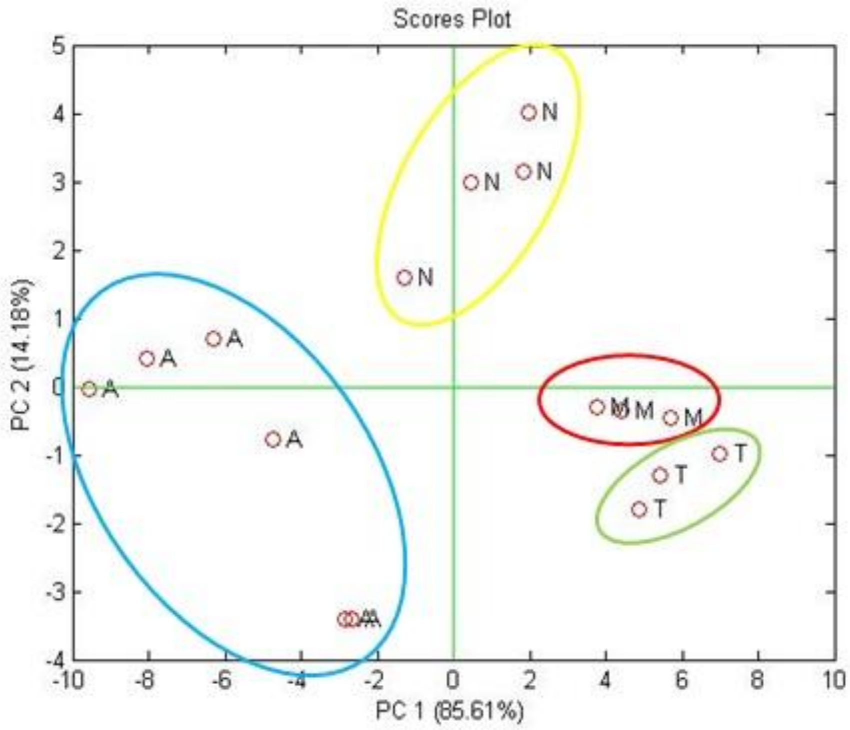


Figure 3.5 PCA results a)blue circle: water b)yellow circle N sample, c) red cicle: M sample and d)green circle: T sample.

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LIST OF FIGURES

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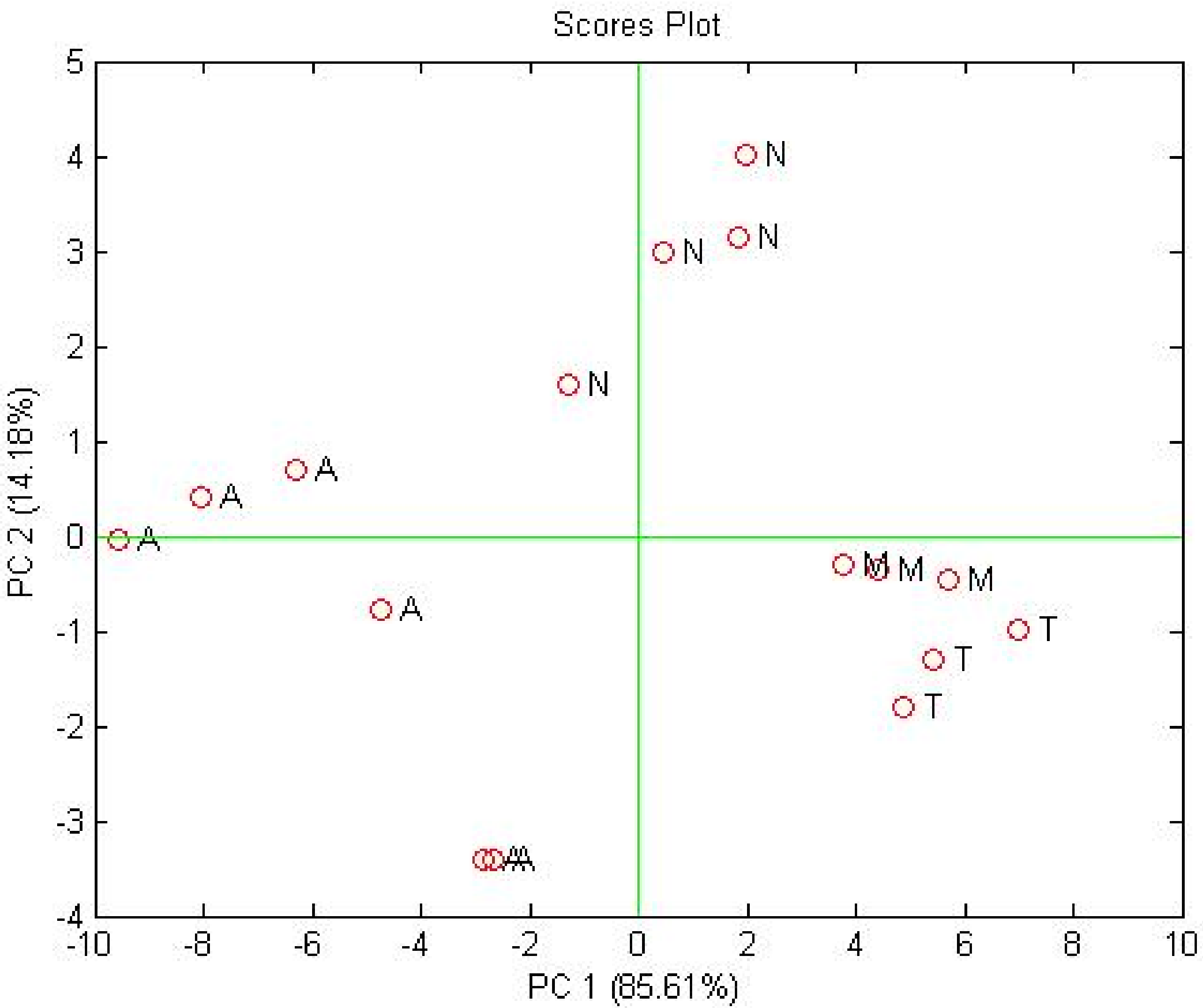
Scores Plot
PC 1 (85.61%)
PC 2 (14.18%)
N
N
N
N
A
A
A
A
AA
M
M
M
T
T
T
-10
-8
-6
-4
-2
0
2
4
6
8
10
5
4
3
2
1
0
-1
-2
-3
-4