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Design of a mechanism for applying sensitive films to gas sensors based on quartz crystal resonators

Diseño de un mecanismo para aplicación de películas sensibles a sensores de gas basados en resonadores de cristal de cuarzo

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Abstract

Gas sensors based on quartz crystal microbalance (QCM) are used to measure mass changes through frequency shifts, using the piezoelectric principle. These sensors are employed in odor detection and are essential for the development of electronic noses. For optimal performance, a sensitive film is applied to the surface of the crystal electrode, which is compatible with a specific compound. There are different methods of film deposition: ultrasonic atomization, spray coating, and casting. The casting method, which involves the manual application of sensitive films using a micropipette, is the most commonly used. However, it is not reproducible. To improve the casting method, a mechanism is proposed as a preliminary platform for a sensitive film application system. The objective is to achieve reproducibility in the construction of gas sensors by applying ethyl cellulose sensitive films onto the sensor surface, thereby reducing the margin of error. Tests were performed on the mechanism to verify the capabilities of the system for the deposition of sensing films using joysticks based on the resistance change principle.

Mechanisms, QCM, CAD

Resumen

Los sensores de gas de microbalanzas de cristal de cuarzo (QCM) son utilizados para medir cambios de masa a través de cambios de frecuencia, utilizando el principio piezoeléctrico, los cuales son usados para la detección de olores, por tanto, son requeridos para el desarrollo de narices electrónicas. Para un funcionamiento óptimo, se aplica una película sensible sobre la superficie del electrodo del cristal que sea afín a algún compuesto determinado. Existen métodos de aplicación: atomización ultrasónica, spray o casting. Este último es el más utilizado los otros métodos el cual, consiste en la aplicación manual de películas sensibles a través de una micropipeta. Sin embargo, no es reproducible. Para poder mejorar el uso del método casting se propone un mecanismo como plataforma preliminar de un sistema de aplicación de películas sensibles con el propósito de aplicar películas sensibles de etíl celulosa sobre la superficie de sensores de gas para lograr obtener una reproducibilidad en la construcción de sensores que sea capaz de reducir el margen de error. Se realizaron pruebas del mecanismo para verificar el alcance del sistema para la aplicación de las películas sensibles mediante el uso de palancas de control con el principio de cambio de resistencia.

Mecanismos, QCM, CAD

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Introduction

Quartz Crystal Microbalance QCM gas sensors have been widely used in the last decades, due to a wide field of applications, since these sensors are highly precise mass sensors, which are commonly used in the detection of analytes in gaseous and liquid phases, since they contribute with the detection of chemical and biological substances [1]. In order to obtain the optimal functionality of the Quartz Crystal Microbalance QCM gas sensors, a sensing film must be applied to absorb the gas molecules of a particular volatile organic compound for a proper reaction such as ethanol.

This reacts adequate with a dissolution of ethyl cellulose mixed in chloroform as sensing film. This film must be placed on the QCM sensor surface electrode, using several applying methods, such as, ultrasonic atomization, spray. Although, these methods are quite effective they tend to be complex in their application. On the other hand, the casting method is the approach must use for this purpose, which consist in applying two microliters of the sensing film using a micropipette. The inconvenience of this method is the low reproducibility. Therefore, it had been developed the design and manufacture of a mechanism to control automatically such task. (Alanazi *et al* [2023])

A mechanism is a mechanical device with the purpose of transfer a movement and/or a force from a source to an output, where under certain circumstances such transfer could be performed, however, during the development of any mechanism there are areas of opportunities to seek the best design to obtain the long-term device and a reduction of costs for the implementation that can make its manufacture profitable. The mechanism was designed using the software of design assisted CATIA computer (Torrecilla 2013).

In this case, the mechanism to develop is a sensing film sample injector of ethyl cellulose for the adsorption and absorption of ethanol molecules trough QCM gas sensors with the purpose of detect several parts per million (ppm) gases in a controlled environment.

The goal to achieve of the mechanism, is the precision, specifically in the deposition sensing films of ethyl cellulose, since this procedure was performed manually. Although, the results obtained were satisfying, there were a low reproducibility in the application of the sensing films in several sensors (Hu *et al* [2020]).

Experimental Setup

For the development and implementation of the system, the block diagram proposed is shown in Figure 1, which consist of the main mechanism and two sub-mechanism that are responsible of activate a micropipette and transport the QCM sensors through a displacement device.

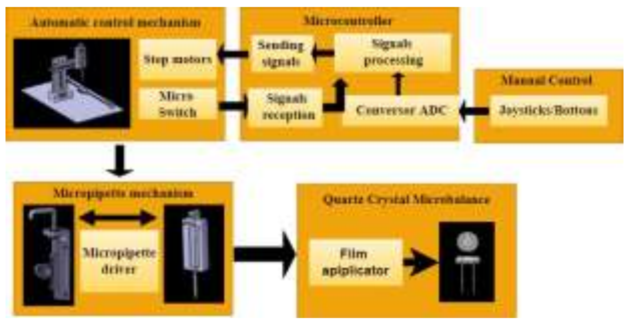

Figure 1 Block Diagram of the system
Source: Own

A cartesian mechanism was designed to be able to deposit a sensing fil if ethyl cellulose using a micropipette which it is moved by two axes and these are driven using stepper motors controlled by a joystick that use the principle of a resistance variation sending the correct signal sequence to move the motors clockwise or counterclockwise and this is possible developing a firmware (García 2009) within a microcontroller (PIC16f877A (Microchip 2002)) used as a processing programmable device to acquire and convert the analog signals from the joysticks to digital registers. In this case, a 10-bit resolution ADC were used.

The previous procedure it is necessary to obtain the desired operation of the mechanism. The motors were activated using electronic power devices. As final product all the components were assembled, resulting in a cartesian mechanism with precise movement using stepper motors, in such a way fulfills the desired task.

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Finally, in this work is presented the design, development and implementation of a two-axis mechanism with the purpose of activate the injection of a micropipette to deposit a solution on the electrode of a QCM gas sensor. In this particular case, the activation of the mechanism is activated manually using a microcontroller and a joystick proving that the system performance was satisfactory.

Experimental setup

Prototype: Sample injector on a QCM.

The mechanism has two degrees of freedom, since it has linear movements in the X and Z axis, these performed movements between one point an another is based on interpolations to ensure its location.

It was not necessary the addition of movement in Y axis, since the second mechanism is independent, described as linear conveyor of the QCM sensor, is in charge of move the components over the Y axis to facilitate the design in both mechanisms and to be able to manipulate the components as can be observed in Figure 2.

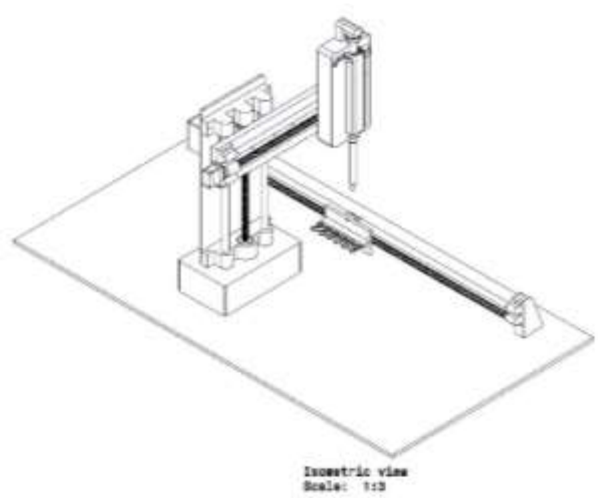

Figure 2 Sensing film depositor prototype
Source: Own Elaboration

Rack-Pinion Mechanism

To activate the micropipette which is the device that deposit the sensing film in the QCM sensor, the mechanism known as Rack-Pinion mechanism was designed. Figure 3, shows a pinion pitch relationship, number of teeth ant tooth module to be able to convert rotational in to rectilinear movements.

The following formula is used to calculate the linear displacement length as a result of the multiplication of the number of teeth (Zp) times the revolutions per minute (Wp) times step (P) of the gear per revolution obtaining:

$$L = Zp * Wp * P$$

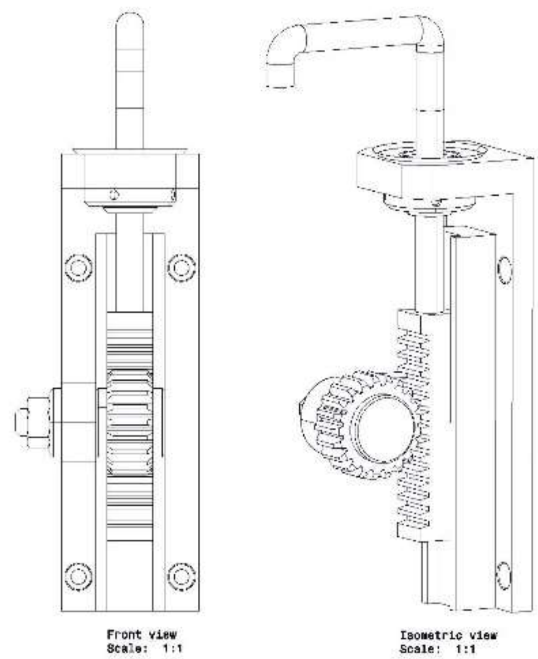

Figura 3 Rack-Pinion mechanism
Source: Own Elaboration

On the other hand, the sensing film will be deposited on the center of the electrode applying a sample of 2 µl of ethyl-cellulose solution. The crystal typically has a ratio of 4.3 mm, as can be observed in Figure 4 with the purpose of mentioned above, which will generate a frequency decrement in the fundamental frequency of the sensor, according of the film thickness, the QCM sensors use the piezoelectric principle.

In order to measure the sensor response, the use of a frequency counter is necessary to detect the frequency shifts when a sample of a volatile organic compound is applied. The Sauerbrey equation is used for this purpose (Sauerbrey *et al* [1959]).

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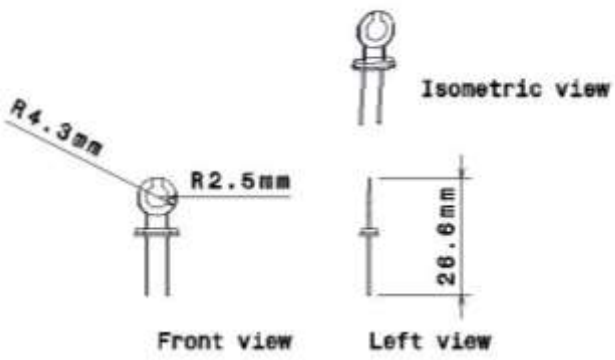

Figure 4 Quartz Crystal Microbalance (QCM)
Source: Own Elaboration

Linear QCM sensor transporter

For the development of this particular mechanism that performs along with the injection mechanism, it was necessary to calculate the design specifications such as the worm gear screw as is shown in Figure 5.

Calculations expressions for linear movements were used, however, the screw velocity must be expressed in revolutions per minute (rpm). The following expression is used for the unit conversion:

$$n = \frac{v}{p}$$

Being:
n: turning speed (rpm),
V: linear velocity (mm/min),
P: step (mm).

For the mean velocity, the following expression were used:

$$n_{mean} = \frac{n_{minimum} + 2 * n_{maximum}}{3}$$

Being:
n-minimum: minimum speed of the section (rpm)
n-maximum: maximum speed of the section (rpm)

Once these values were known, to mobilize the screw where the load is minimal the adequate motor calculation is required. Therefore, an 8 mm (commercial use) screw was selected using the following equation:

The performance η1, is in the range of 0.85-0.95.

$$n_1 = \frac{1 - \mu * tan\varphi}{1 + \left(\frac{\mu}{tan\varphi}\right)}$$

Being:
μ: friction coefficient, 0,003 ÷ 0,01.
P: step.
dn: Nominal diameter of the screw thread.
φ: helix angle.

For the helix angle the following relation is used:

$$tan\varphi = \frac{P}{\pi * d_n}$$

The applied torque TL (Nm) that must be used to the screw is:

$$T_L = \frac{F * P}{200 * \pi * n_1}$$

Being:
F: obtained linear load (N).
P: step of the thread (mm).

The power required to drive the screw PL (KW) is:

$$P_L = \frac{T_L * n * 2 * \pi}{60 * 1000}$$

Being:
n: revolutions per minute (rpm).

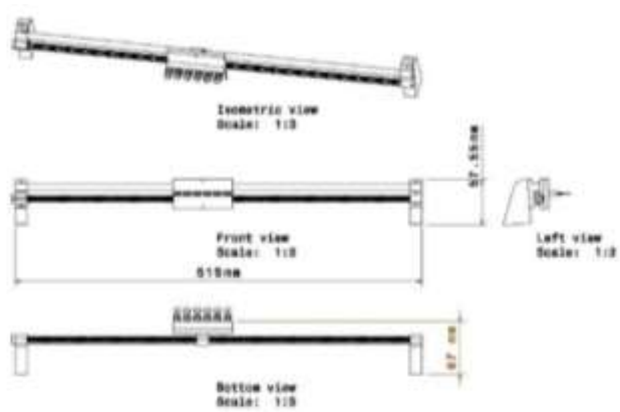

Figure 5 QCM Linear transportator
Source: Own Elaboration

With the screw characteristics obtained by the calculations, it was performed a search for the proper screw that accomplishes the specifications require.

Linear axis

The final component of the proposed prototype are the linear guides that are implemented along a worm gear with the help of a bench mobilize any object attached to it. The dimensions were calculated as well as the linear transporter of the QCM sensors. The design is shown in Figure 6.

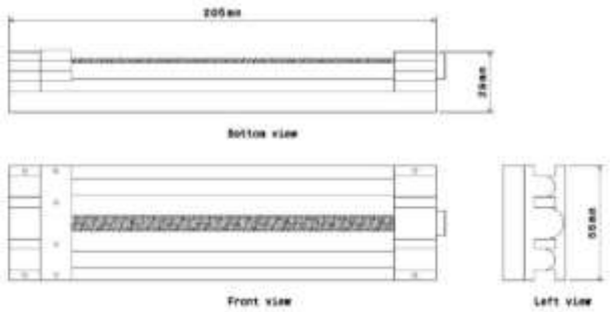

Figura 6 Linear axis of worm gear
Source: Own Elaboration

For the movement of the mechanism axis, stepper motors were used with a load capacity approximately of 3.5 Kg, which is the necessary torque to move the mechanism without causing an overexertion than could cause an overheating in the motors.

For the preliminary manipulation of the stepper motors, a firmware development was performed. In this case, a full step mode was used in order to observe the relation of the motor spin with the step of the worm gear, and thus, determine the exact distance that the axis will move. In order to obtain an accurate displacement of the axis a half step mode should be used, although in this case, is only a preliminary experiment.

Once the motor sequence is set, a firmware was developed using a microcontroller. The step number for the axis to travel is 200, since the motor step resolution is of 1.8°.

A joystick was used as a preliminary experimental setup, in order to manipulate manually the axis displacements of the system. The joystick is basically a set of two potentiometers, where each one of them move an axis. Therefore, an analog to digital converter (ADC) is used to process the signal from the joystick.

The ADC has a 10-bit resolution, which is considered in the programming for the manipulation of the motor to displace the corresponding axis. Furthermore, a speed control considered or the manual manipulation. (Technology Inc [2002])

Micropipette

The principal instrument of the system as shown in Figure 7 is the micropipette, this with the purpose of being certain of an adequate performance of the system. In this process the micropipette a high precision on the deposition of the sensing films as well as the position of application are imperative in order to increase the reproducibility of the QCM sensors. The sample for the deposition is approximately 2 µl. Furthermore, a sample of ethanol will be applied in a measurement chamber with a temperature controlled of approximately of 20 °C to reduce the error percentage, between applications, since the ethanol density is preserved at 0.797 g/cm^3.

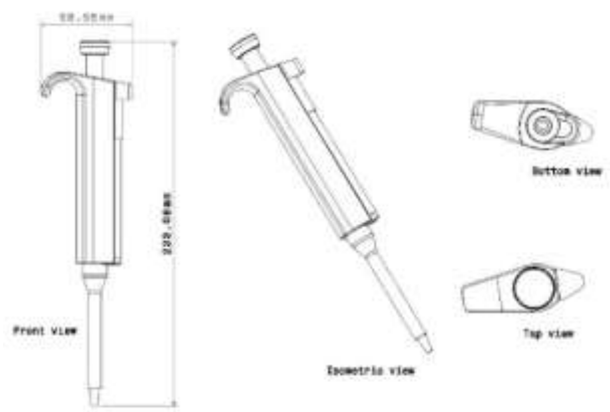

Figure 7 Micropipette p20 (0.5 - 10 µl)
Source: Científica Vela Quin S de R.L de C.V

For the development of the system an analogue micropipette was used, the developed mechanism presses the plunger to collect the sample solution from a bial and for the deposition of the sample. The collected volume is fixed.

Hardware and software development

For the microcontroller programming, a flux diagram was developed to show the interpretation of the firmware.

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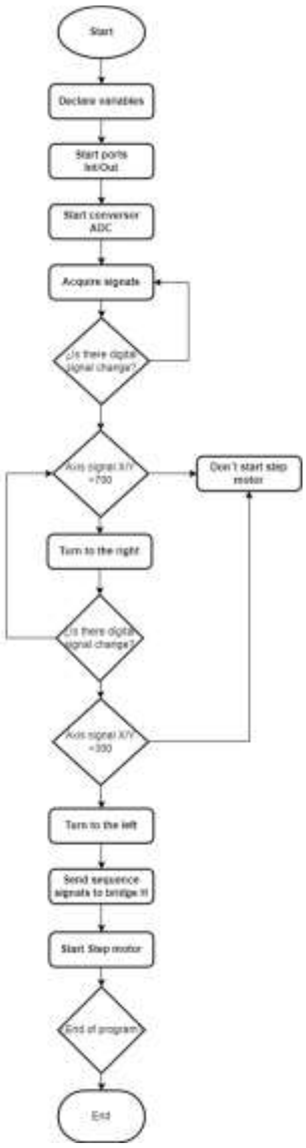

Figure 8 Flux diagram of the firmware developed
Source: Own Elaboration

The diagram describes the logic programming, in this case the commands that are executed according to the input conditions. When the joystick is turned to the right the stepper motor is displaced over the X axis using the full step mode to the right. If the joystick is moved to the left the stepper motor displace the X axis to the right. On the other hand, if the joystick is turner up or down the other stepper motor activates the Z axis displacing them in the corresponding case. Figure 8 shows the flux diagram of the firmware development (Tojeiro Calaza [2009], Schneider Electric Motion [2011]).

The electronic diagram used for the electronic system is shown in Figure 9. Potentiometers were used as the joystick. It can be observed the connections used at the mechanism. An LCD display is used to visualize the movement of the mechanism axis.

To activate the stepper motors, L298 power driver modules were used and these could manipulate the axis X and Z as described above (García [2009]).

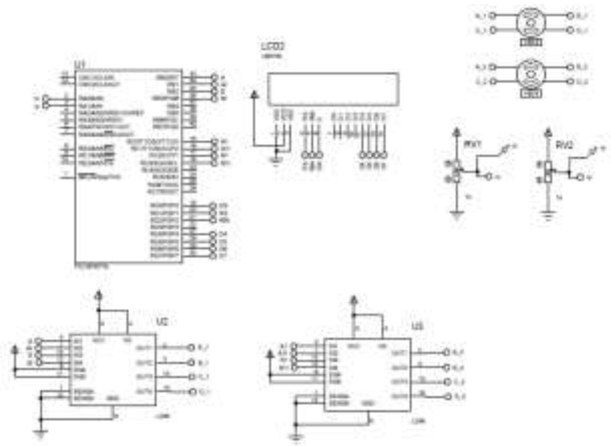

Figure 9 Electronic diagram
Source: Own Elaboration

In addition, to manufacture the mechanism based on the CAD designed in CATIA, a 3D printer was used in order to reduce costs. In Figure 10, a preview can be observed. Using the CURA software the pieces were manufactured using ABS filament due to the physical characteristics as mechanical resistance (Pérez [2018], Palacios [2019]).

Figure 10 Analysed rowlock in Ultimaker Cura
Source: Own Elaboration

Results

Once the manufacture of the 3D printer pieces was finished, the assemble was performed, as well as some elements that were acquired commercially. In this case, aluminum screeds were used due to the mechanical resistance for better support of the axes of the worm gears, as well as the lightness to avoid additional load on the stepper motor.

The ABS material offers more benefits compared to other materials and the result in this system assembled are shown in Figure 11.

Figure 11 Final assembly of linear axis
Source: Own Elaboration

As we can observe in Figure 12, we obtain the final assembly of the quartz crystal microbalance transporter, which operates on the same principle as the "Z" and "X" axes for crystal displacement. The position of the QCM carrier is related to the position of the micropipette, which is displaced by the aforementioned axes. The crystal's position is determined by the formulas discussed in the linear transporter section, which consider the angular velocity of the screw and its thread pitch.

Figure 12 Final assembly of linear axis of worm gear
Source: Own Elaboration

Once the linear axis was assembled the complete mechanism was integrated having as support the aluminum screeds previously welded to warranty a better fastening of the components as well as the printed components added to the aluminum using 4mm Allen screws as can be observed in Figure 13.

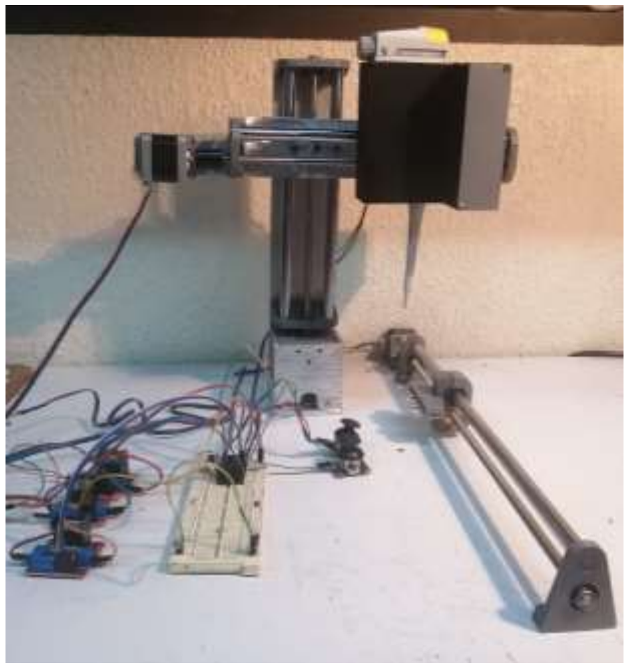

Figure 13 Final assembly
Source: Own Elaboration

Finally, the complete assembled system is shown in Figure 14 with the electronic control circuit to activate the actuators through the microcontroller. Since the system is manipulate manually there are no a proper control on the manipulation of the mechanism at the axis displacements. As future work a vision system will be implemented to control the complete system.

Nevertheless, with a manual control the designed mechanism shows an effective displacement with the design and materials used for the implementation of the system. Therefore, the systems show satisfactory results.

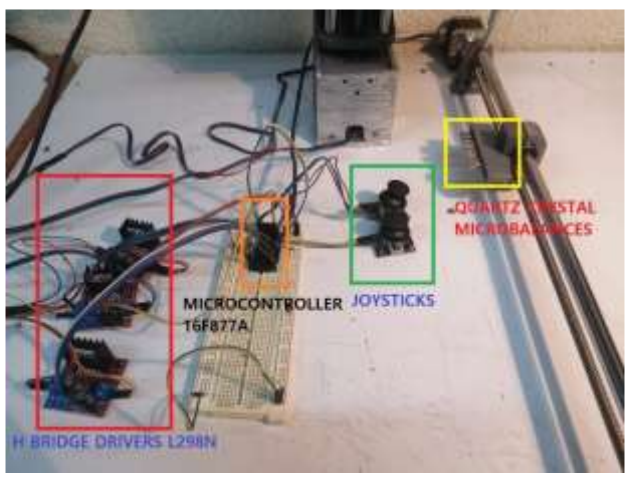

Figure 14 Control system
Source: Own Elaboration

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Conclusions

A robust mechanism had been implemented, the system is capable to perform a casting deposition of sensing films more accurately, which will be used as a platform of applications for such purpose.

According with the forecast if mechanical resistance of the printed pieces of the prototype, they perform the desired tasks, although further tests need to be performed to complete the full cycle of work.

During the assembly of the mechanism, we found areas of opportunity, such as to improve the balance of the system and avoid operation errors.

The design of some components was improved in order to increase the loading support performed by the mechanical elements.

An open loop control was developed for operation tests to obtain a better programming of the actuators to scale the system for a fully automated system to reduce errors under several circumstances in order to achieve a high reproducibility of the QCM gas sensor, as well as to avoid the waste of solution used form the deposition of sensing films.

Future work

Using the current results, it will be possible to continue the improvement of the system to a fully automated using a LabVIEW interface capable of decide the movement of the mechanism using a vision system through a web cam. Using this vision technique, the system will have the ability to generate a coordinate to move the mechanism to the corresponding QCM sensor and deposit the sensing film. With the development of the complete system the reproducibility of the QCM will increase, generating a reliable system.

Once the casting method was automated it could be developed a system robust enough to perform such activity and thus, reduce the percentage of human error. Since the mechanism will work automatically, a user interface to control the movements of the mechanism and execute efficiently the deposition of the sensing obtaining a high reproducibility in such deposition. In order to prove de efficiency in the reproducibility of the sensing films deposited on the sensor substrate using the casting method, a fundamental principle in statistics is contemplated, such as the Gaussian distribution, which lets us know that there are variables in the way in which the sensitive film is distributed, as well as its location for what it is. that the purpose of the mechanism is justified in a better way so that this variety is reduced as much as possible.

Currently several experimental setups had been performed in order to improve the mechanism of the activation of the micropipette.

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