# Journal of

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### **Presentation of the Content**

In the first article we present, Synthesis of flexible polyurethane foam for applications in planar orthosis using the response surface methodology, by TORRES-OCHOA, Jorge Alejandro, OSORNIO-RUBIO, Nadia Renata, CORTAZAR-MARTINEZ, Orlando and MORALES-NIETO, Victor Alfonso, with ascription in the, Universidad Politécnica Juventino Rosas, Tecnológico Nacional de México-Celaya and CINVESTAV-Querétaro, as the nex article we present, Manufacture 4.0 with LabVIEW and Arduino Mega for distributed cooling control of the DeMag 250 Ton machine injection mold, by MORENO, José Miguel, MORALES, Victor Alfonso, RUIZ, Cesar Alejandro and PÉREZ, Guillermo Rubén, with ascription in the Universidad Politécnica de Juventino Rosas, as the next article we present, Ceramic densification effect on corrosion resistance and thermal conductivity, by ACOSTA-PEREZ, Emmanuel, SALAZAR-HERNÁNDEZ, Mercedes, MENDOZA-MIRANDA, Juan Manuel and SALAZAR-HERNÁNDEZ, Carmen, with ascription in the Instituto Politécnico Nacional and Universidad de Guanajuato, as the last article we present, Smart device development for cold chain control in biological materials, by RAMÍREZ, Mayra, LÓPEZ, Pedro, GONZÁLEZ, Juana and MENDOZA, Tania with ascription in the Universidad Politécnica del Bicentenario.

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Synthesis of flexible polyurethane foam for applications in planar orthosis using the response surface methodology

# Síntesis de espuma de poliuretano flexible para aplicaciones en ortesis planares utilizando la metodología de superficie de respuesta

TORRES-OCHOA, Jorge Alejandro†, OSORNIO-RUBIO, Nadia Renata\*, CORTAZAR-MARTINEZ, Orlando\*\* and MORALES-NIETO, Victor Alfonso\*

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### **Abstract**

In this work, the process for the formulation of flexible polyurethane foam is presented following a design of experiments for mixtures. The proportion of polyol, diisocyanate, and crosslinker was considered as factors. The response variables considered were foaming time and reaction temperature. The result of the experiments showed that there is an area where the foam formulation is better. This zone is closed with 5% crosslinker, 50% polyol, and 45% diisocyanate, in this formulation denser foams with more uniform bubbles were obtained.

### Polyurethane foam, Polyol, Diisocyanate

### Resumen

En el presente trabajo se presenta el proceso para la formulación de una espuma de poliuretano flexible siguiendo un diseño de experimentos para mezclas. Se consideraron como factores la proporción de poliol, diisocianato y reticulante. Las variables de respuesta consideradas fueron el tiempo de espumado y la temperatura de reacción. El resultado de los experimentos mostró que existe una zona donde la formulación de la espuma es mejor, esta zona está cerana al 5% de reticulante, 50% de poliol y 45% de disocianato, en esta formulación se obtuvieron espumas más densas con burbujas más uniformes.

### Espuma de poliuretano, Poliol, Disocianato

**Citation:** TORRES-OCHOA, Jorge Alejandro, OSORNIO-RUBIO, Nadia Renata, CORTAZAR-MARTINEZ, Orlando and MORALES-NIETO, Victor Alfonso. Synthesis of flexible polyurethane foam for applications in planar orthosis using the response surface methodology. Journal of Research and Development. 2020. 6-18:1-4.

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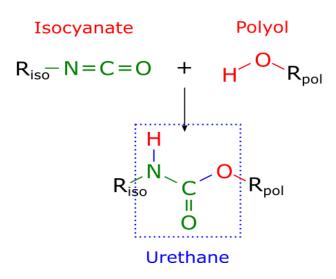
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### Introduction

Polyurethane foams are polymers with a high range of applications, such as packaging, automotive industry, electronics, footwear, construction, etc [1]. The formation of polyurethane foam is carried out when an isocyanate reacts with a polyol to form a urethane functional group, as shown in Figure 1.



**Figure 1** Reaction between isocyanate and polyol to form the urethane functional group *Source: own work* 

### **Experimental design**

To optimize the formulation of a chemical mixture, it is necessary to carry out a design of experiments for the mixtures. An important property of the design of experiments is the quantification of the change due to a response variable [3]. The design experiment for the mixture has some additional restrictions such as the sum of the proportions of all the factors is always equal to the total mixture, Eq (1).

$$x_1 + x_2 + \dots + x_n = 1 \tag{1}$$

To model the response surface it is necessary to adjust the experimental points to an already known mathematical model [4].

$$R(y) = \sum_{k=1}^{n} \beta_k \, x_k \tag{2}$$

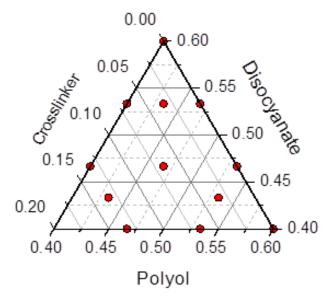
$$R(y) = \sum_{k=1}^{n} \beta_k \, x_k + \sum_{k < i} \beta_{ki} \, x_k \, x_i \qquad (3)$$

$$R(y) = \sum_{k=1}^{n} \beta_k x_k + \sum \sum_{k< j}^{n} \beta_{kj} x_k x_j + \sum \sum_{k< j< m}^{n} \sum \beta_{kjm} x_k x_j x_m$$
 (4)

In equations 2 to 4, the parameter  $\beta_k$  represents the linear contribution of each of the factors to the total mixture, the parameter  $\beta_{kj}$  represents the interaction of two factors after the final mixture, this can be synergistic, if the response improves, or antagonistic if the response worsens. The parameter  $\beta_{kjm}$  represents the interaction between of all components of the mixture.

### Methodology

A proportion of polyol was mixed with diisocyanate as shown in the design of experiments in Table 1, the precursors were vigorously mixed with a wooden mixer for 3 seconds. then the reaction temperature was measured and the time it took to start foaming.



**Figure 2** Ternary diagram of the feasible zone in the design of experiments, the red numbers indicate the ID of the experiment

Source: own work

The constraint for this experiment was 40 to 60% polyol, 40 to 60% diisocyanate, and 0 to 20% crosslinker. Figure 2 shows the experiments within the feasible zone. All experiments were carried out at room temperature, first activating the polyol with the crosslinker, and then adding the active polyol mixture to the diisocyanate.

Test	Polyol	Diisocyanate	Crosslinker
1	0.600	0.400	0.000
2	0.467	0.400	0.133
3	0.533	0.467	0.000
4	0.433	0.533	0.033
5	0.533	0.400	0.067
6	0.467	0.467	0.067
7	0.467	0.467	0.067
8	0.400	0.400	0.200
9	0.400	0.600	0.000
10	0.400	0.467	0.133
11	0.400	0.533	0.067
12	0.433	0.433	0.133
13	0.533	0.433	0.033
14	0.467	0.533	0.000

**Table 1** Composition of the mixture following a design of experiments for mixtures *Source: own work [Excel]* 

### **Results**

In each experiment, the room temperature and the temperature when the foaming reaction started were measured. Figure 3a shows the preparation of the mixture in the first block of experiments. The formation of cream is observed before the reaction and the start of the foaming reaction. In Figure 3b, the foams formed can be observed, considering that the foaming speed is correlated with the porosity of the material. It can also be observed that at a higher reaction temperature, there is a material with larger bubbles.



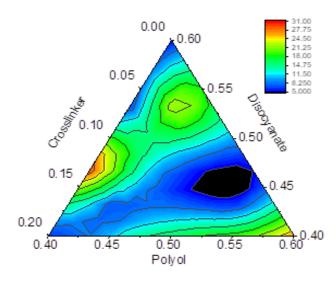
**Figure 3** a) Experiment where the measurement of reaction temperature and foaming time was carried out b) experiment selection with better foaming speed control

Source: own work

Tes t	Foaming Time (s)	Reaction Temperature (°C)
1	14	40.3
2	5	60.4
3	24	30.8
4	19	45.8
5	7	55.2
6	8	53.1
7	6	54.8
8	5	61.2
9	31	40.1
10	5	60.7
11	6	51.9
12	5	59.9
13	17	40.6
14	27	33.4

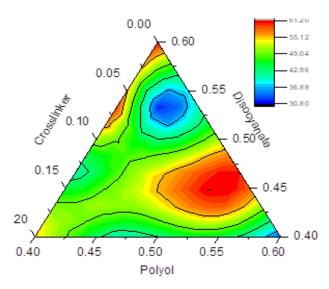
**Table 2** Result of the measurement of foaming time and reaction temperature

Source: own work [Excel]



**Figure 4** Ternary diagram of the feasible zone in the design of experiments, the red numbers indicate the ID of the experiment

Source: own work



**Figure 5** Ternary diagram of the feasible zone in the design of experiments, the red numbers indicate the ID of the experiment

Source: own work

TORRES-OCHOA, Jorge Alejandro, OSORNIO-RUBIO, Nadia Renata, CORTAZAR-MARTINEZ, Orlando and MORALES-NIETO, Victor Alfonso. Synthesis of flexible polyurethane foam for applications in planar orthosis using the response surface methodology. Journal of Research and Development. 2020

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Figures 4 and 5 represent the best response surface with the data obtained fitting to equation 5 correspondings to a special cubic model. In Figure 4 the black zone can be observed, which indicates that the foaming reaction time is short, while in Figure 5 it is observed in that same zone that the reaction temperatures are higher. Also, in that area, the best polyurethane foams were found.

### **Conclusions**

It was possible to formulate polyurethane foams employing a design of experiments for mixtures, finding that the foams with a percentage close to 5% of crosslinker, 50% polyol and 45% diisocyanate. In that area it was found that the reaction temperature is high, and the foaming time is short, it was also seen that the foams in this area had a better appearance.

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# Manufacture 4.0 with LabVIEW and Arduino Mega for distributed cooling control of the DeMag 250 Ton machine injection mold

# Manufactura 4.0 con LabVIEW y Arduino Mega para el control de enfriamiento distribuido del molde de inyección de la maquina DeMag 250 Ton.

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### **Abstract**

### Nowdays the injection plastics processes at insdustrial level have had a great development in the bajio region with the arrival of new enterprise suppliers of the automotive industry that work particuary de injection molding. The plastics injection molding is a semicontinuous process that consists of inject a polymer in the molten state into a mold closed under pressure, throught a small hole called gate, in the mole the material is solidifies, the piece or final part is obtained when the mold is opened and remove the piece molding from the cavity [1]. To monitor and control the temperature changes in the plastics injection cicles permite reduce errors and costs in the process. In this project we propose to apply manufacture 4.0 using the Arduino Mega microcontroller and LabVIEW to monitor part of the process of the injection molding of the DeMag 250 Ton Machine, specifically the coolding system adapting to control the injection molde temperature. The preliminare results show that is apropiate to use the LabVIEW an Arduino Mega combination to generation of innovation project applied to the plastic industry.

# Injection molding, manufacture 4.0, distributed cooling system

### Resumen

Hoy en día los procesos de inyección de plásticos a nivel industrial han tenido un gran desarrollo en la región del bajío con la llegada de nuevas empresas proveedoras de la industria automotriz que trabajan particularmente el moldeo por inyección. El moldeo por inyección de plásticos es un proceso semicontinuo que consiste en inyectar un polímero en estado fundido a un molde cerrado a presión, a través de un orificio pequeño llamado compuerta, en el molde el material se solidifica, la pieza o parte final se obtiene al abrir el molde y sacar de la cavidad la pieza moldeada [1]. Monitorear y controlar los cambios de temperatura en los ciclos de inyección de plástico permitirá reducir errores y costos en el proceso. En este proyecto se propone aplicar manufactura 4.0 utilizando el microcontrolador Arduino Mega y LabVIEW para monitorear parte del proceso del moldeo por invección de la máquina DeMag 250 Ton, específicamente el sistema de enfriamiento adaptado para control la temperatura del molde de inyección. Los resultados preliminares muestran que es viable utilizar la combinación de LabVIEW y Arduino Mega para la generación de proyectos de innovación aplicados a la industria del plástico.

Moldeo por inyección, manufactura 4.0, sistema de enfriamiento distribuido

**Citation:** MORENO, José Miguel, MORALES, Victor Alfonso, RUIZ, Cesar Alejandro and PÉREZ, Guillermo Rubén. Manufacture 4.0 with LabVIEW and Arduino Mega for distributed cooling control of the DeMag 250 Ton machine injection mold. Journal of Research and Development. 2020. 6-18:5-10.

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<sup>†</sup> Researcher contributing as first author.

### Introduction

Injection molding is one of the most significant material processing methods for mass production of plastic products. It is widely used in various industry sectors, and its products are ubiquitous in our daily life. The settings and optimization of the injection molding process dictate the geometric precision and mechanical properties of the Therefore, products. final optimization, and control of the injection molding process have a crucial influence on product quality and have become an active research field with abundant literature [2].

Because of low cost, complex molding ability, convenience, light weight, and corrosion resistance, polymer injection parts are widely used around the world. Currently, over 30% of all the plastic parts are manufactured by injection molding, and 80% of engineering plastic products are manufactured by injection molding [3].

The Society of the Plastics Industry (SPI) has reported that, in the United States, the plastics industry is the third largest manufacturing industry. In 2017, the US plastics industry accounted for \$432.32 billion in annual shipments and directly employed nearly one million people. Hence, the plastics industry has a large contribution to the nation's economy. Plastics are now among the most widely used materials and their use covers the entire spectrum of industries worldwide [2].

The growth of the automotive industry, as the strengthening of traditional sectors as the electrodomestics, has generated a sustained increase in the plastics industry in the Bajío region, with an advance of more than 100 percent in some states in the last years.

In Guanajuato, the production value of the plastics and rubber industry increased 123 percent in the last five years, going from an amount of 10 thousand 12.5 million of pesos in 2010 to 22 thousand 325.2 million in 2015, according to the data from the National Institute of Statistics and Geography (INEGI) [4].

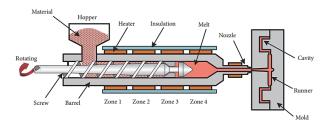
The injection molding process is achieved using an injection molding unit. The process is an unvarying, repetitive process that involves feeding plastic (pellets) to the barrel via a hopper, melting of plastic using heaters in a barrel, mixing the plastic using a rotating and reciprocating screw, injecting the molten plastic into a mold cavity, packing the molten plastic within the mold cavity, cooling of the molten plastic (in the mold cavity), and thereafter ejection of the solidified plastic part [5].

The novelty of the project is to use the Arduino MEGA Microcontroller as a data acquisition card in combination with an interface developed in LabVIEW to instrument the cooling system of a machine injection mold and create an advanced system for real-time monitoring of temperature, level flow and pressure of the cooling system, which can be seen on a web page from anywhere in the world.

### **Injection Molding process**

The typical structure diagram of an injection molding machine injection unit is shown in Figure 1. It mainly consists of a screw, a barrel, a nozzle, and a heating system. In general, the heating system has multiple heating zones. For each heating zone, there is a heater and a temperature sensor. In most cases, the temperature set for each heating zone varies according to the requirements of the process. In order to save energy, the heating system is covered with a layer of thermal insulation materials. The system has no cooler system. Therefore, the cooling rate of the system is very slow [6].

Injection molding machine (IMM) is the main equipment for plastic production, which maintains a complex system integrating mechanical, electrical, and hydraulic properties. Injection molding machine (IMM) as important manufacturing equipment has been widely used in many industries [7].



**Figure 1** Structure diagram of an injection molding machine injection unit [6]

Source: Y. Ruan, H. Gao, and D. Li, Improving the Consistency of Injection Molding Products by Intelligent Temperature Compensation Control, Hindawi Advances in Polymer Technology, China, 2020

As a highly complicated process, plastic injection molding can be divided into three stages: filling, packing, and cooling [2]. On the other hand, according with Saad M. S. Mukras the process can be divided into 4 phase which include filling, packing, cooling, and ejection. These phases determine the cycle time of the process, which is an important factor during production as it is directly linked to the cost of production. It can, therefore, be argued that reducing the cycle time (even seconds) within any of these phases will lead to time and cost-savings in the long run especially in the case of mass production [5].

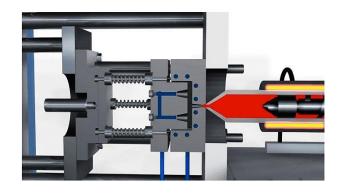
### Control temperature and cooling

Of the four mentioned phases, the cooling phase takes up more than two-thirds of the cycle time. It is also the most important phase in addition to productivity since the cooling has a significant effect on product quality. Inadequate cooling times will usually lead to defects on the product such as warpage and volumetric shrinkage. On the other hand, longer cooling times will generally lead to satisfactory product quality but result in low productivity (and consequently high production costs). \*is scenario has elicited research activities on ways to reduce the cooling time while ensuring acceptable product quality. Majority of these research activities have focused on conformal cooling channels within the molds to enhance and accelerate cooling. In conformal cooling, the cooling channels are designed to conform to the shape of the mold cavity. It has been demonstrated that this technique enables the mold temperature to reach the operating temperature faster than in molds with standard cooling channels [5].

In most of the existing injection molding processes, the temperature is usually controlled by a proportional integral differential (PID) algorithm. The PID algorithm has been widely applied in temperature control. It is easy to implement and can provide good robustness under static conditions. But the polymer injection molding process contains several iterative and repeated operations. Each operation is performed intermittently [6].

Therefore, the barrel is in dynamic during the injection molding process. Hence, the PID algorithm cannot provide a satisfactory barrel temperature control performance for the whole operation process. In the past decades, in order to solve the barrel temperature control problem, many advanced control methods have been proposed such as adaptive decoupling control, model predictive synchronous control, multivariable self-tuning predictive control, self-optimizing model generalized predictive control, adaptive predictive control, and response identification and internal model control [6].

Temperature control is an essential part for the correct plastic injection (figure 2).



**Figure 2** Plastic injection in the mold *Source: own elaboration* 

The main function of the cooling system is to minimize the time in the injection cycle, this has an impact on part quality and production. In this project we propose monitoring online and control the temperature of mold implementing a finite state machine to reduce the injection cycle on a 20 %.

# Online Monitoring Polymer Injection Molding

The whole process is complicated because the process variables are strongly coupled and to analyze accurately. For manufacturing of qualified products injection molding still mainly relies on manual and trial-and-error operation Obviously, this traditional approach has disadvantages of low production efficiency. reliability and repeatability, dependence on prior experience. Therefore, it is imperative and crucial to develop an advanced injection molding method that is science based and technology oriented. Recently, much attention has been focused on intelligent (smart) manufacturing, represents an in-depth integration of nextartificial intelligence generation technology and advanced manufacturing technology. It runs through every link in the full life cycle of design, production, product, and service. Intelligent injection molding refers to the production process that employs ΑI technology—such as extracting information from production, computer optimization methods, and control strategies to develop an online production optimization system. -rough the comprehensive use of sensing, optimization, and control methods, the intelligent injection molding production process can increase production efficiency and product quality [2].

Quality control is a crucial issue in the injection molding process with target of obtaining a high yield rate and reducing production cost. Consequently, efective methods for monitoring the injection conditions (e.g., pressure, velocity, and temperature) in real-time and adjusting these conditions adaptively as required to ensure a consistent part quality are essential [8].

The main objective of this project is to apply manufacturing 4.0 through the design of a system based on LabVIEW and Arduino MEGA to monitor in real time the mold cooling system of a DeMag 250 TON injection machine (figure 3).



**Figure 3** DeMag 250 TON *Source: own elaboration* 

### LabVIEW + Arduino

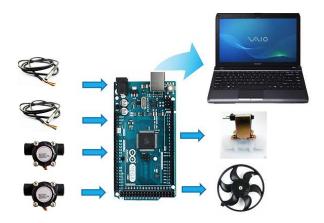
Different from amateur electronics hobbyists, lab automation applications want the system to be stable, reliable, and accurate enough and fast enough. Arduino is an open source system with hundreds of libraries and compatible hardware sets. Arduino makes the development of microcontroller-based systems much easier than with traditional register programming methods. Since Arduino lowers the barrier of embedded programming requirement, more and more students, engineers and scientists are employing Arduino in their applications [9].

Arduino microcontrollers are already established for use in low-cost high-quality scientific and engineering equipment. For example, they have been oceanographic research, mass spectrometry, microscopy, vision research, Skinner boxes, mechatronics, optics, electrophoresis, imaging. Most importantly for this sensor application, Arduinos have applied to systems for sensors for open hardware including colorimetry, nephelometry, pressure monitoring, smart plugs, and in vivo optimal imaging. In addition, Arduinos have been demonstrated for field sensing operations like soil moisture, developing a wireless sensor node to monitor poultry farms and an ambient sensor cloud system using OpenFS (open field high-throughput phenotyping. for Arduinos are also an established technology for controls, used as controllers on numerous challenging projects such as for aerial vehicles, robots, and microfluidics [10].

LabVIEW (acronym for Laboratory Virtual Instrument Engineering Workbench) is a platform and environment for graphic programming to design systems, used by engineers and researchers. Recommended for hardware systems and test, control and design software, simulated or real, using graphic and intuitive icons and cables that look like a flow chart. Offers an integration with multiple devices hardware and provides multiple libraries integrated for advanced data analysis, all to create virtual instrumentation [1].

LabVIEW is the de faro standard software for lab measurements and controls. It is easier to integrate or expand the embedded system with some other LabVIEW controlled system to build an even more complex system. There is an entry-level Arduino and LabVIEW toolkit called LIFA, for free download. But, with this toolkit, one cannot program the Arduino board, instead, one could only program in LabVIEW. For a complex task, one definitely should code his own Arduino applications [9].

The main objective is to apply manufacturing 4.0 through the design of a system based on LabVIEW and Arduino MEGA to monitor in real time the mold cooling system of a DeMag 250 TON injection machine, sensing the temperature and the flow in each one inputs of the injection mold (figure 4).



**Figure 4** Block diagram to monitoring in real time the mold cooling system *Source: own elaboration* 

### Methodology

The first step was to implement a cooling system adapted to the injection mold for the experimentation stage (figure 5).

The adapted cooling system has a pump that drives the coolant through a main duct and then passes through a manifold to distribute the coolant in each of the mold lines. Because the temperature in the mold is not distributed in uniform way, we put electrovalves and flowmeters in each one of the inlet and outlet lines of refrigeration in the injection mold to be able to monitor and control the flow independently.

We programmed the Arduino MEGA microcontroller to acquire temperature in one point of the lines and then we designed a code to sensing the flow in each point independently.



**Figure 5** Cooling system adapted to the injection mold *Source: own elaboration* 

The next step was to create in the LabVIEW software the interface and data logger to monitor and record the information obtained from the sensor (figure 6).

### Results

The first tests were done by monitoring the temperature and flow separately from LabVIEW, then both variables in a single program and so on. Once the monitoring was developed, a finite state machine was programmed using a case structure to sense a pair of temperature sensors and a pair of flowmeters to later control the opening and closing of the electro valves and in this form to control the distributed cooling.

The code in labview was complemented with the part of the data record, storing every second the values obtained from the sensors. For the online monitoring, the necessary stage was programmed to display the indicators regarding the temperature and flow at each point of entry and exit of the injection mold coolant lines on an internet page.

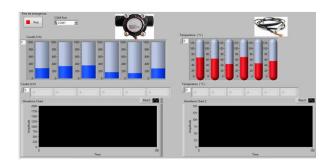


Figure 6 LabVIEW software running a data logger for monitoring

Source: own elaboration

### **Conclusions**

The objective was accomplished, it is possible to monitor the temperature and flow of the injection mold of the DeMag 250 Ton machine from anywhere in the world. The initial implementation of manufacturing 4.0 to the mold cooling system has proven to be a powerful combination for the development of projects related to the plastics injection industry. The future work is implementation of the system in other plastic injection machines, in companies in the region such as Vistamex and Mahle.

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### Ceramic densification effect on corrosion resistance and thermal conductivity

# Efecto de la densificación en cerámicos en la resistencia a la corrosión y conductividad térmica

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### **Abstract**

Thermal barrier Coatings (TBC) are a system of layers covering a surface with the aim to increase its insulation as well as protect the core of oxidation. In this paper the use of silica ceramics modified by polydimethylsiloxane (PDMS) as a new TBC for steel surface is proposed. The were obtained according to ceramics forming stable methodology a sol tetraethoxyetilsilicate (TEOS) at different percentage of PDMS (10, 20, 40 and 100%); the gelling was done using DBTL (dibutildilaurate tin) as a polycondensation catalyst. Therefore, the sol was applied on AISI-1018 steel surface by inmersion and a densification process is applied. The ceramic was characterized by infrared spectroscopy to observe its silica structure changes produced by the thermal treatment. Finally, the analysis the densification effect on properties on thermal conductivity and corrosion resistance was carried on.

## SiO<sub>2</sub>, PDMS, Densification, Thermal conductivity, Corrosion resistance

### Resumen

Las barreras térmicas (TBC) son un sistema de capas que cubren una superficie con el propósito de aislarla térmicamente y proteger el material contra la oxidación. En este proyecto se propone el uso de cerámicas de sílice modificadas con polidimetilsiloxano (PDMS) como un nuevo TBC para superficie de acero. Las cerámicas se obtuvieron de acuerdo con la metodología sol-gel, formando un sol estable mezclando tetraetoxietilsilicato (TEOS) con diferentes porcentajes en peso de PDMS (10, 20, 40 y 100%). La gelificación se realizó empleando DBTL como catalizador de policondensación y las soluciones sol fueron aplicadas a superficies de acero AISI-1018 por inmersión; a estos recubrimientos se les realizó un tratamiento de densificación. Las cerámicas se caracterizaron por espectroscopía infrarroja para observar cambios en la estructura de la sílice modificada. Además, se analizó el efecto de densificación en propiedades tales como: la conductividad térmica y resistencia a la corrosión.

SiO<sub>2</sub>, PDMS, densificación, conductividad térmica, resistencia a la corrosión

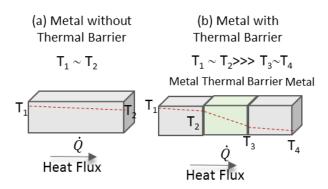
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<sup>†</sup> Researcher contributing as first author.

### Introduction

Thermal Barrier coatings (TBC) are a system of layers that cover a surface with the the aim to increase its insulation as well as protect the core of oxidation in chemically agresive environments and operating high temperatures. Thus, in a core without TBC and unidimensional heat transfer occurs, the temperature in both side are practically the same, Figure 1a shows the case of a heat source at 150°C where heat flows through a material core, the opposite temperature reaches 130°C without TBC. Appling a TBC on the surface of the core and using the same temperature of the heat source, the temperature at the opposite surface of core drastically decreases up to 90°C, Figure 1b, due by the thermal conductivity of the TBC and its thickness [1–3].



**Figure 1** Temperature profile (a) surface without TBC, (b) surface with TBC

Source: own work [Power Point]

TBC not only act as thermal insulators; they also reduce the wear of metal core due by corrosion caused by its exposure to high temperatures; for example, Amaya 2009 report that a turbine blade without TBC; after 2500hr of flight at low altitude over the sea showed several damage from pitting corrosion, meanwhile a blade with a Ni-Al TBC have no signal of wear or corrosion [3].

Among the most commonly materials used as a TBC are the ceramics shown in Table 1. These materials have low thermal conductivity (k); based on zirconium oxide (ZrO<sub>2</sub>) and TBC of iridium stabilized zirconium oxide (YSZ) which have an average k value of 2 W / (mK) [4-8]. TBCs are commonly formed by expensive metal oxides such as Ba, Zr, Ce, La, etc.

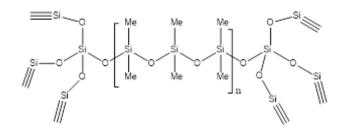
Material	Thermal conductivity (W/mK)	Material	Thermal conductivity (W/mK)
$ZrO_2$	2.17	CeO <sub>2</sub>	2.77
3YSZ	2.12	La <sub>2</sub> Zr <sub>2</sub> O <sub>7</sub>	1.56
Mullita	3.3	LaMgAl <sub>11</sub> O <sub>1</sub>	1.7
Al <sub>2</sub> O <sub>3</sub>	5.8	SiO <sub>2</sub>	2-0.4
BaZrO <sub>3</sub>	3.42	(Na/Ca) <sub>x</sub> (SiO <sub>4</sub> ) <sub>n</sub>	1.7

Table 1 Thermal conductivity for TBC and vitreous silica

Source: own work [Word]

Among the oxides with low conductivity with low cost are vitreous silica (K = 2 to 0.4 W / mK) and silicates (1.7 W / mK) however, these oxides have a low thermal expansion coefficient between 0.5 to 9  $\times 10^{-6}$  1 / ° C; that implies little capacity for thermal deformation. This complicates its use as TBC because its limited deformability at high temperatures compared to metals; for example, for steels the thermal expansion is ranging from 12 to 18  $\times 10^{-6}$  1 / °C.

Recently, Salazar-Hernández C et al. [9–12] have studied ceramic coatings based on polydimethylsiloxane-modified silica (PDMS) as anticorrosive coatings. In this project it is proposed to study the thermal properties of these coatings, considering silica with low thermal conductivity (1.48 W / m K). However, its coefficient of thermal expansion is different from a metal, thus, it is proposed to add an organic modifier to the ceramic such as polydimethylsiloxane (PDMS). PDMS is an elastomeric silicone with high thermal stability that could offer the ceramic structure greater flexibility as indicated in Figure 2.



**Figure 2** SiO<sub>2</sub>/PDMS ceramic structure *Source: own work [ChemDraw]* 

However, PDMS contains organic groups in its siloxane chain, thus, the thermal stability of material can be compromised at temperatures greater than 200 °C; Therefore, it is proposed a thermal densification treatment in order to increase thermal stability for the ceramic. Analyzing the effect of thermal densification treatment on thermal conductivity and corrosion resistance.

### MethodologySiO<sub>2</sub> / PDMS Synthesis

The sol was obtained according to the methodology proposed by Salazar-Hernández et al [7]; where the TEOS (99%, Aldrich) is mixed with different percentage of PDMS (Gelest, 16-32 sct) adding DBTL maintaining the ratio of 99: 1 TEOS: DBTL (Table 2). The mixture is heated during 30 min at 50 ° C before being applied to AISI-1018 steel surface.

	% Components		
	$SiO_2$	<b>PDMS</b>	
TBC-10 PDMS	90	10	
TBC-20 PDMS	80	20	
TBC-30 PDMS	70	30	
TBC-40 PDMS	60	40	
TBC-PDMS		100	

**Table 2** Synthesis for ORMOSIL-UPIIG coatings *Source: own work [word]* 

### TBC application on AISI-1018 steel

The steel surface is pre-treated in order to remove impurities; This treatment consist of roughing the surface with abrasive paper to remove impurities, then ethyl alcohol was performed using an ultrasonic bath for 10 min (Ultrasonic Cleaner-Intertek Listed) and finally dried at room temperature. The application of the TEOS / PDMS sol solution to the steel surface was carried out with the dip-coating technique.

### **Densification Process**

To increase the thermal stability of the coatings, these were subjected to a thermal densification treatment indicated in Table 3.

	Temperature (°C)	Time (min)	%PDMS
	30	30	10 and
<b>T1</b>	80	60	10 and 20 %
	500	120	20 %
	80	30	20 and
<b>T2</b>	200	120	30 and 40 %
	400	120	40 %

 Table 3 Temperature ramps used for the densification

 process

Source: own work [Word]

### **Chemical characterization**

Infrared spectroscopy (IR-TF) was used to chemically characterize the coatings, with this technique the main components of these TBC were identified. The spectra were obtained with a Nicolet-iS10 ATR-TF spectrometer, with an average of 16 scans and a resolution of 4 cm<sup>-1</sup> with a spectral window of 4000 to 600 cm<sup>-1</sup>.

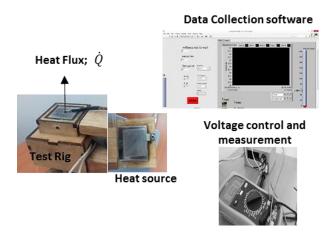
### **Corrosion Rate**

To perform this test, the Peak Tech corrosion test bench model DIT-105 was used, applying a constant current of 0.5A for 90 minutes. The corrosive agent used was sodium chloride with a concentration of 5% by weight. The corrosion rate ( $V_c$ ) was determined by gravimetry according to Equation 1; where  $\Delta m$  is the loss of mass in grams generated by corrosion, A is the exposed area in  $m^2$  and t the exposure time of the material in seconds.

$$V_c = \frac{\Delta m}{A*t} [=] \frac{kg}{m^2 \cdot s} \tag{1}$$

### **Measurement of Thermal Conductivity**

The thermal conductivity was determined using the measurement test rig shown in Figure 3. The test rig consists of a test section that is limited to cubic specimens with a length of 25.4 mm, the section is insulated with fiberglass to promote heat flow in the Z direction. As a heat source a resistance of 100W was placed at the bottom of specimen, thus, heat flux is controlled by an on / off control device based on Arduino; which is connected to a computer to record the temperature of the heat source and the top of the specimen.



**Figure 3** Test bench used to determine the thermal conductivity of materials and TBC *Source: own work [Power Point]* 

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The heat flux supplied was calculated by Ec. 2 for which the voltage (V) and current (i) were measured during the test. Because the contact area of the specimen is different from the total resistance area; Q is adjusted with the correction factor Fc, as shown in Equation 3.

$$Q = F_c(i \cdot V) \tag{2}$$

$$F_c = \frac{A_{probeta}}{A_{resistencia}} \tag{3}$$

Thermal conductivity was calculated using Fourier's law for composite walls; (Equation 4) where Q is the heat flux through the specimen, L is the layer length, K is the thermal conductivity for each section and A is the normal heat transfer area for each component.

$$Q = \frac{T_i - T_{i+1}}{\sum_{i}^{n} \left(\frac{L}{kA}\right)_i} \tag{4}$$

### Results

### SiO<sub>2</sub>/PDMS as TBC composition

Figure 4 shows the ATR-FT for these ceramics; where the 3600 cm<sup>-1</sup> corresponds to the terminal hydroxyl of the PDMS and there dissapear; on the other hand, the signals of the organic groups of TEOS (-CH<sub>2</sub>-CH<sub>3</sub>) are observed between 2900-300 cm<sup>-</sup> <sup>1</sup> and 1400-1600 cm<sup>-1</sup> but also not identified. This confirms that the DBTL promoted polycondensation between both reagents, forming the structure of the proposed TBC; where at 2900cm<sup>-1</sup> (signal 2) the -CH<sub>3</sub> linked to the polysiloxane chain "PDMS" embedded between the ceramic particles are observed; the silica network of the ceramic is identified with the signal 1 corresponding to the siloxane groups (-Si-O-Si) that were observed around 1017 cm<sup>-1</sup> as a broad band; that for CBT with 40% PDMS an intense shoulder is formed at 1192 cm<sup>-1</sup>; In addition, linear siloxanes from PDMS are identified with the fine signal between 780-796 cm<sup>-1</sup> [13].

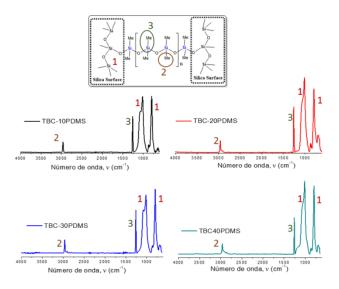
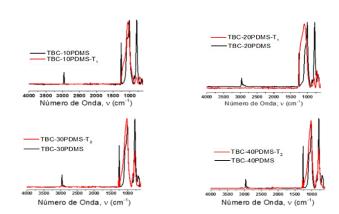


Figure 4 Characterization by ATR-FT for  $SiO_2$  / PDMS TBCs

Source: own work [Power Point and Origin]

### SiO<sub>2</sub>/PDMS densification

Figure 5 shows the graphs obtained with the infrared analysis for TBCs exposed to heat treatments; it can be seen that the ramps used achieve the decrease of organic matter on the surface of the coating by about 80%. The signals of the – CH<sub>3</sub> groups at 2900 cm<sup>-1</sup> and the Si-C signal at 1200 cm<sup>-1</sup> almost disappear after the densification treatment has been performed. In addition, the band corresponding to the siloxanes (silica network, -Si-O-Si) at 1100 cm<sup>-1</sup> is widened, which suggests that the linear chains of the PDMS condense forming a compact structure.



**Figure 5** Modification of SiO2 / PDMS after densification by ATR-FT Source: own work [origin]

Figure 6 shows scanning electron microscopy (SEM) for ceramics before and after heat treatment; where it is appreciated that by increasing the amount of PDMS precipitates in the silica network indicated by red circles. When densified; these groups generate fractures in the film formed probably because the increase in temperature causes their detachment generating an increase in stress that causes the fissure of the coating; which was major at TBC-100PDMS.

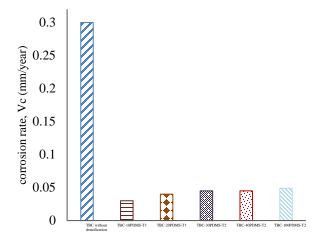
# Before Densification TBC-10PDMS TBC-10PDMS-T1 TBC-20PDMS TBC-20PDMS TBC-20PDMS-T1 SEC 13VV WOTINNESSED 50°9 1450 50°0 50

**Figure 6** SEM for SiO2/PDMS TBC *Source: own work [PowerPoint]* 

### **Corrosion test**

Figure 7 shows the corrosion rate determined in mm of penetration per year for the TBC of SiO<sub>2</sub>/PDMS with and without densification treatment; where the coating without densification presented a corrosion rate of 0.27 mm / year.

When densifying the SiO2/PDMS, the anticorrosive activity in the ceramic is increased by 94%. The corrosion rate for these materials was about 0.02 mm / year.



**Figure 7** Corrosion rate for TBC SiO<sub>2</sub>/PDMS *Source: own work [Excel]* 

### Thermal conductivity for SiO<sub>2</sub>/PDMS

The thermal conductivity for the AISI-1018 ranges from 50 to 56 Wm-1K-1 in the temperature range of 50-150  $^{\circ}$  C; for this metal there is no considerable effect of temperature on the thermal property. However; different values were determined for the TBC-SiO2 / PDMS. According to Figure 8, no significant effect was observed in the PDMS content on the conductivity of the TBCs. Calculating an average value of 4.19  $\pm$  0.16 Wm-1K-1 at 50°C, 15.96  $\pm$  2.12 Wm-1K-1 at 100°C and 42.38  $\pm$  4.09 Wm-1K-1.

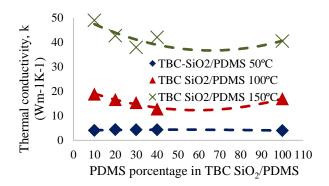


Figure 8 Effect of PDMS concentration on termal conductivity

Source: own work [Excel]

According to the increase in temperature, an increase in conductivity was observed and a greater deviation between the values of K. This is because the thermal stability of the TBC depends on the PDMS content. Then, to improve this property, the coatings were densified to obtain the conductivity values indicated in Figure 9.

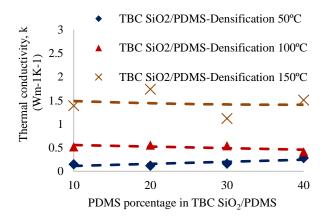
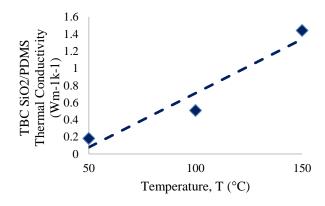


Figure 9 Effect of densification on thermal vonductivity

Source: own work [Excel]

After the densification treatment, the conductivity decreased to observe an average of 0.179-0.071  $Wm^{-1}K^{-1}$  at 50 °C, 0.507  $\pm$  0.065  $Wm^{-1}K^{-1}$  at 100 °C and 1.44 to 0.025 $Wm^{-1}K^{-1}$ . Observing that the content of PDMS in the structure of the TBC has no effect on the value of K. However, the temperature modifies the K linearly for the TBC-SiO<sub>2</sub> / PDMS (Figure 10).



**Figure 10** Temperature effect on TBC-SiO<sub>2</sub>/PDMS termal conductivity *Source: own work [Excel]* 

### **Conclusions**

The ORMOSIL-UPIIG ceramic coating has a good performance as an anticorrosive coating for aluminum surfaces reducing the corrosion rate by 93%.

However, densification increases the properties of the material by eliminating the microporosity formed in the ceramic structure. Therefore, this reduces the corrosion rate of aluminum by 99.5%.

### Acknowledgments

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### Smart device development for cold chain control in biological materials

# Desarrollo de sistema inteligente para el control de la cadena de frío de materiales biológicos

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### **Abstract**

In Mexico, controlling and monitoring the cold chain in vaccines is one of the biggest problems nowadays. Vaccines are biological products sensitive to variations in light, humidity, time, and mainly temperature. When some of those parameters get lost because of an external situation, consequently, these vaccines lose their functionality, that is why is important a portable device adapted to the design is developed according to the software-hardware prototype, because of this, different variations in those vaccines can be monitored, temperature and humidity for example. According to this, is possible to transfer biological samples and medicines, without forgetting of course to apply the rules in handling the control of the cold chain, guaranteeing the safety of those products. The purpose of this is prevent that the cold chain in medicines and biological samples within health institutions get lost. This make possible to achieve a better control and monitoring applying a versatile system, which is based according to the norms and safety standards.

### Biological samples, Cold chain, Temperature

### Resumen

En México, controlar y monitorear la cadena de frío en vacunas es uno de los mayores problemas hoy en día. Las vacunas son productos biológicos sensibles a las variaciones de luz, humedad, tiempo y principalmente temperatura. Cuando algunos de esos parámetros se pierden debido a una situación externa, en consecuencia, estas vacunas pierden su funcionalidad, por eso es importante que se desarrolle un dispositivo portátil adaptado al diseño de acuerdo con el prototipo de software-hardware, debido a esto, diferentes variaciones en esas vacunas pueden controlarse, por ejemplo, la temperatura y la humedad. De acuerdo con esto, es posible transferir muestras biológicas y medicamentos, sin olvidar, por supuesto, aplicar las reglas en el manejo del control de la cadena de frío, garantizando la seguridad de esos productos. El objetivo es evitar que se pierda la cadena de frío en medicamentos y muestras biológicas dentro de las instituciones de salud. Esto permite lograr un mejor control y monitoreo aplicando un sistema versátil, que se basa en las normas y estándares de seguridad.

### Muestras biologicas, Cadena de frío, Temperatura

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### Introduction

The cold chain systems for biological materials, vaccines and medicines is a topic of great importance since it directly impacts the user's health, the correct storage, transport and distribution of these substances prolongs their useful life, on this way guarantees efficiency of its operation [1] and [2]. However, we know that limited resources and lack of adequate technology for the cold chain in the transfer of these substances is an area of opportunity [3]; that would bring great benefits to the health sector in our country [4] and [5].

A bad process in the cold chain could have serious consequences, for example in 2015 an adverse accident occurred in Chiapas, due to the failure in the process of the cold chain of vaccines reported by the IMSS, this cause 31 minors, 29 required hospitalization and two died, therefore it is important to note that a cold chain guarantees to the costumers that the product they get has been kept within a temperature range during transport, storage, sale or distribution.

According to the data obtained in an investigation in the health units in Silao town in Guanajuato city, the vaccination transport protocols in vaccination campaigns are not very automated, it consists of an ice maker that has a refrigerant attached at the top and in the inside of it a hole to insert a mercury thermometer.

The process to verify that the cold chain is maintained in vaccination campaigns is to open the cooler every 2 hours to record the temperature, since it does not have a system that allows them to verify the temperature of the container without the need to open the lid, which causes loss of the cold chain.

That is why it is necessary to develop an intelligent temperature and humidity monitoring system for the refrigeration equipment that stores and transports such substances; which is considered to be an integrated system that allows to record a graphic history of the cold and humidity chain, with the objective of efficiently controlling the parameters set by OMS and the adjustment of Mexican regulations for each substance.

The system is considered as an intelligent red neuronal that selects the established parameters for blood, organs, vaccines and medications.

It will also have a system that alerts audibly and visually in case of the parameters detected by the sensors were not correct, and location that can be tracked by GPS, will also improve the quality in the cold chain process and bring great benefits that could help to improve the attention to the needs of each costumer.

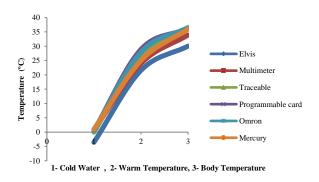
It is expected that the implementation of this system in Health Facilities in Silao Guanajuato solves the current cold chain issue, improving their processes and providing a safe service that will benefit the users directly.

### Methodology

An investigation was carried out to condition the sensors to be used, subsequently the different types of analogic and digital sensors was used to quantify the measurement of temperature. Next, we programmed the Data Acquisition Card, and designed an algorithm that al lowed us to analyze and put the collected data in a chart. This information was then processed so as to elaborate a database and a platform. We also incorporated an auditive and visual alarm system. Finally, we used analyzers to verify the measurements obtained.

### **Results**

A comparison of the different analog and digital sensors response-time, in order for us to be able to choose the most appropriate one. Table 1 and graphic 1 show the difference in the response of the 4 sensors detected in the test (cold water, ambient temperature and body temperature), as well as the mercury thermometer and Omron taken as control due to their standards are established.



Graphic 1 Characterization graph of temperature sensors

Source: own work [Excel 2013]

	Cold Water °C	Warm Temperature °C	Body Temperature °C
Elvis	-3.567	21.57	30.03
Multimeter	0.7	24	33.9
Traceable	0	27	36
Programmable card	0.5	28.87	36.06
Omron	0.5	27.9	36.5
Mercury	1	25	36

**Table 1** Characterization of temperature sensors Source: own work [Excel 2013]

Using the given sensor, a code in Arduino was created so as to get the temperature data of 5 DS18B20 sensors, which were strategically located within the cold chain device.

The chosen sensor for the development of the device, due to its response time, was a digital sensor, which happens to be humidity resistant, and it shows an optimal temperature range for the task.

The data obtained by the temperature sensor is shown in the serial monitor of the Arduino, it is also to be linked to a MySql server via internet connection and then is saved in a text file, recording date and time.

The database that was developed requires Log ID, user and password to log in. (Figure 1), sticking to the security and dataencryption protocols.

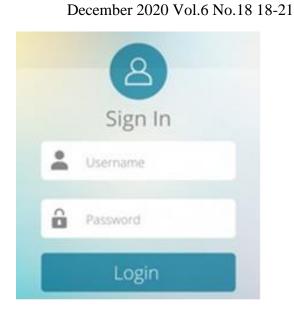


Figure 1 Login into the database Source: own work [PHP]

Data monitoring is done through a sensor network, located strategically (sample, room and device temperature) in order to control the biological samples cold chain. These sensors are monitored in real time, locally, through the design of a graphic interface (Figure 2).

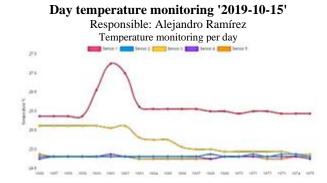


Figure 2 Graphs of the database Source: own work [PHP]

regarding "Dynamic appendix search" is considered, to analyze the Daily Temperature Logs, showing them on a PDF file (Figure 3).

D	DATA	SENSOR 1 °C	SENSOR 2 PC	SENSOR 3 PC	SENSOR 4 °C	SENSOR S
1656	15/10/19	25.87	24.81	25.62	24.75	24.87
	13 36 11					
1657	15/10/19	25.87	24.81	25.62	24.81	24.81
	13 36 22					
1658	15/01/19	25.87	24.81	25.62	24.81	24.81
	13 36 33					

Figure 3 PDF format of report Source: own work [PHP]

Finally, the platform was moved to a Raspbian system to have a more ergonomic design and installed on a computer board (SBC) (Figure 4).



**Figure 4** Visualization of the database in the Raspbian System

Source: own work [PHP]

A prototype was made (Figure 5), for the implementation of the control system for the acquisition of physical measurement parameters for the biological samples contained in the device.



**Figure 5** Visualization of the database in the Raspbian System

Source: Own work [Ramírez, 2020]

### Annexes

We are grateful with "SICES" because of the economic support in the young researchers program which it was necessary to develop the prototype, also we must say thank you to the Universidad Politécnica del Bicentenario educational program and because we could use the university infrastructure.

### **Conclusions**

In Mexico there is a lack in applications to technologies focused on the transport of vaccines, so an intelligent system was developed that allows monitoring measurement parameters for the conservation of the cold chain.

The implementation of this system in hospital institutions will optimize times, processes, resources and above all it will guarantee safety and conservation in the transfer of biological samples by applying the regulations in force in our country.

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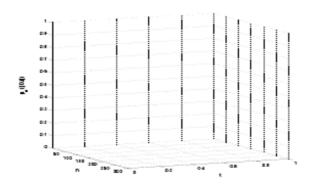
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