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## **Presentation of the Content**

In the first chapter we present, *Development of incubator for the preservation of endemic birds and reptiles of Quintana Roo* by CERECERO-NATALE, Luis Fidel, KAUIL-KAUIL, Jesús Alejandro, PUC-MAY, José Gerardo and CHAN-AC, Irvin Arlin with adscription in the Universidad Tecnológica de la Riviera Maya, as a second article we present *Proposal of Automatic Control System for the Pumping of a Shrimp Farm in the Town of Pimientillo, Nayarit* by MARTÍNEZ-AVIÑA, Salvador & SOSA-SALES, Jorge with adscription in the Universidad Tecnológica de Nayarit, as the following article we present *Retention capacity of maltodextrin antioxidants in cranberry juice by spray drying process* by SAAVEDRA-LEOS, María Zenaida, SILVA-CÁZARES, Macrina Beatriz, GONZÁLEZ-TREVIZO, Cynthia Lizeth and TERRONES-GURROLA, María Cruz del Rocío with affiliation in Universidad Autónoma de San Luis Potosí, as Next article we present *Surface tension of O<sub>2</sub>-Ar, N<sub>2</sub>-Ar and O<sub>2</sub>-N<sub>2</sub>-Ar mixtures* by GARCÍA-MARTÍNEZ, Mauricio, IBARRA-TANDI, Benjamín, LUIS-JÍMENEZ, Daniel Porfirio and LÓPEZ-LEMUS, Jorge with affiliation at the Universidad Autónoma del Estado de México and Centro de Ingeniería y Desarrollo Industrial

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## Development of incubator for the preservation of endemic birds and reptiles of Quintana Roo

## Desarrollo de incubadora para la preservación de aves y reptiles endémicos de Quintana Roo

CERECERO-NATALE, Luis Fidel †\*, KAUIL-KAUIL, Jesús Alejandro, PUC-MAY, José Gerardo and CHAN-AC, Irvin Arlin

*Universidad Tecnológica de la Riviera Maya*

ID 1<sup>st</sup> Author: *Luis Fidel, Cerecero-Natale* / ORC ID: 0000-0003-3012-3098

ID 1<sup>st</sup> Coauthor: *Jesús Alejandro, Kaul-Kaul* / ORC ID: 0000-0002-1811-8424

ID 2<sup>nd</sup> Coauthor: *José Gerardo, Puc-May* / ORC ID: 0000-0002-1449-7578

ID 3<sup>rd</sup> Coauthor: *Irvin Arlin, Chan-Ac* / ORC ID: 0000-0002-0876-8810

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### Abstract

This article presents the design, programming, control and monitoring of the temperature, relative humidity, angular position and aeration of eggs of an artificial incubator prototype for the preservation of birds and reptiles endemic to Quintana Roo. As an example, the incubation parameters of poultry (chicken) and an endemic species (macaw) are presented. A shield card is used to facilitate coupling on the Arduino Mega card with the electronic components. The programming routine performs the automatic incubation, using the LabVIEW software to tune the PID controllers (Proportional Controller - Integral - Derivative) by means of the Ziegler-Nichols first and second method.

**PID controller, Temperature, Humidity position**

### Resumen

En este artículo se presenta el diseño, la programación, el control y monitoreo de la temperatura, humedad relativa, posición angular y la aireación de los huevos de un prototipo de incubadora artificial para la preservación de aves y reptiles endémicos de Quintana Roo. A manera de ejemplo se presentan los parámetros de incubación de un ave de corral (pollo) y una especie endémica (guacamaya). Se emplea una tarjeta shield para facilitar el acoplamiento en la tarjeta Arduino Mega con los componentes electrónicos. La rutina de programación realiza la incubación automática, se utiliza el software LabVIEW para sintonizar los controladores PID (Controlador Proporcional – Integral - Derivativo) por medio del primer y segundo método de Ziegler-Nichols.

**Controlador PID, Temperatura, Humedad posición**

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\* Correspondence to Author (email: mecatronica.cerecero@gmail.com)

† Researcher contributing as first author.

## I. Introduction

At present there is a need to incubate eggs of birds and reptiles endemic to the state of Quintana Roo with the intention of preserving the greatest number of endangered species.

The fact that preservation talks are more constant reveals that the current situation is very worrying. The vast biological diversity developed on our planet through evolution over millions of years is in great danger. The rate of disappearance increases rapidly. It is estimated that between 17,000 and 35,000 species extinguish each year worldwide. It is expected that in fifty-one hundred years most species will have disappeared.

Endemic species are those species of flora and fauna typical of a geographical area, considered exclusive to a region, as they do not exist anywhere else in the world and represent one of the greatest riches of the landscape, as well as one of the best indicators of the biological heritage of a country. In general, they are usually classified as vulnerable species, since their restricted location and limited population make them susceptible to threats.

The state of Quintana Roo has a record of 483 bird species including two introduced species, from 71 families, representing 88% of bird species in the Yucatan Peninsula, 44% of birds in Mexico and 4.8% of birds in the world. It holds 23 families, 72 genera and 106 reptile species of the 140 reported for the Yucatan Peninsula 75%, which represents about 13% of the national total and 1.3% of the world. Table 1 shows the common name, genus and species, order and family. For more information see (Valdez, 2012).

Common Name	Genus and species	Order	Family
<b>Birds</b>			
Keel-billed toucan	Ramphastos sulfuratus	Pciciformes	Ramphastidae
Slaty-tailed trogon	Trogon massena	Trogonifor	Trogonidae
⋮	⋮	⋮	⋮
Black-and-white hawk-eagle	Spizaetus melanoleucus	Accipitrifo	Accipitridae
<b>Reptiles</b>			
Boa constrictor	Boa constrictor	Squamata	Boidae
Yucatan banded gecko	Coleonyx elegans	Squamata	Gekkonidae
⋮	⋮	⋮	⋮
Yucatsn white-lipped snake	Symphimus mayae	Squamata	Colubridae

**Table 1** Bird and reptile species in Quintana Roo included in NOM 059

The article is organized as follows: Section II contains information regarding artificial incubation, while the development of the prototype is described in section III. In section IV the proposed control system for the artificial incubation variables is presented; finally, in section V we present the conclusions.

## II. Artificial incubation

The incubation regime is defined as the set of physical factors present in the environment surrounding the egg. On the other hand, artificial incubation is a process by which conditions suitable for proper embryonic development are provided.

This type of incubation is possible because of a followed methodology, where the fertilized egg is taken and placed into an enclosure in a controlled environment; after a period of days, the embryo can develop until it hatches. This process involves variables that must be controlled to ensure its success.

Nº	Incubation (factors)	Artificial incubation (Variables)	Unit of measurement
1	Temperature	Temperature	[°C]
2	Humidity	Relative Humidity	[%]
3	Rotation	Angular position	[°]
4	Aeration	Aeration	[%]

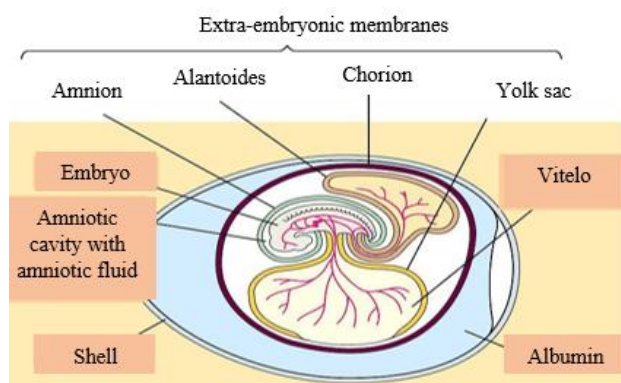
**Table 2** Factors and variables of an incubation and artificial incubation with its units of measurement

The efficiency of births is directly related to the control of the variables shown in Table 2. Excessive increase or decrease in temperature and/or humidity for a period of time causes a significant decrease in the number of births. For example, high temperatures can cause the birth of premature offspring and low temperatures a late birth. The angular position of the eggs is the variable which prevents the yolk from adhering to the shell, as it also helps the formation of the Vitelline vesicle membrane and blood vessels, the supply of nutrients to the extra-embryonic organs and to eliminate embryonic heat (Jiménez Rueda, 2008).

Finally, the last important manipulated variable is the air flow, better known as aeration, because while the embryo is developing, there is a constant flow of carbon dioxide and oxygen through the shell.

## Temperature

At the beginning of the incubation, the embryos are not functionally or organically prepared to emit heat. This is why they react like cold-blooded organisms, that is, when the air temperature raises, the metabolism of the embryos increases. If the temperature decreases, the metabolism decreases. Therefore, the increase in temperature favors cell multiplication, the formation of embryonic layers and membranes (allantoids, chorion, amnions and yolk sac are shown in Figure 1), as well as nutrition.



**Figure 1** Egg structure

## Relative Humidity

The humidity of the environment inside the incubator is lost due to evaporation and because water is absorbed by the embryo through the pores of the shell; therefore, it is necessary to restore that moisture. The highest humidity requirements are in the terminal phase of the incubation cycle, because the embryo needs to rotate in order to break the shell and if the surrounding membranes dry out due to lack of moisture, the embryo dies.

This is measured by evidencing broken eggs with dead embryos inside, due to insufficient humidity in the incubator. Viscous embryos have their lungs stuck due to a high humidity rate; abnormal, weak and small embryos develop due to insufficient humidity at the end of the incubation cycle; and embryos with small lungs form due to low humidity rates.

## Angular position

Embryos develop normally only when the eggs are turned, that is, a periodic angular rotation is performed during the first 18 days of incubation (in the case of chickens.) Varying the angular position improves oxygen utilization across the

entire shell surface. Both are reflected in better developed offspring.

For this reason, the embryo is exposed to sticking to the inner membranes of the shell, which can cause death, particularly during the first six days of incubation.

In addition to this, the fact that the specific weight of the embryo leads it to remain at the top of the yolk during the first days, below and very close to the shell in the air chamber.

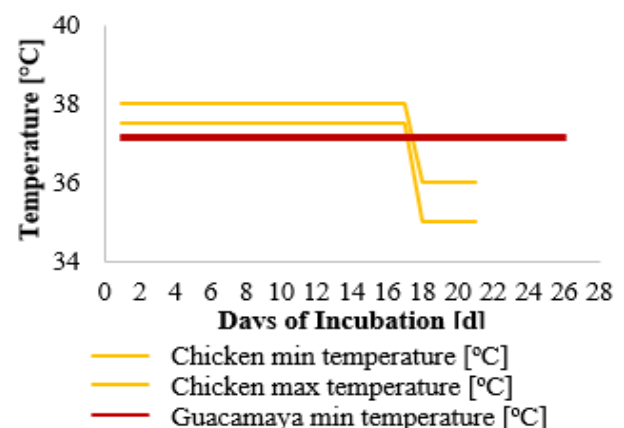
On the other hand, the position of the egg influences the position that the brood will adopt when preparing for hatching; this is of great importance to obtain a high percentage of births.

## Airation

The ventilation problem must be addressed in two ways: the circulation of air itself and the renewal or replacement of air. Temperature differences can be used to detect air flow problems.

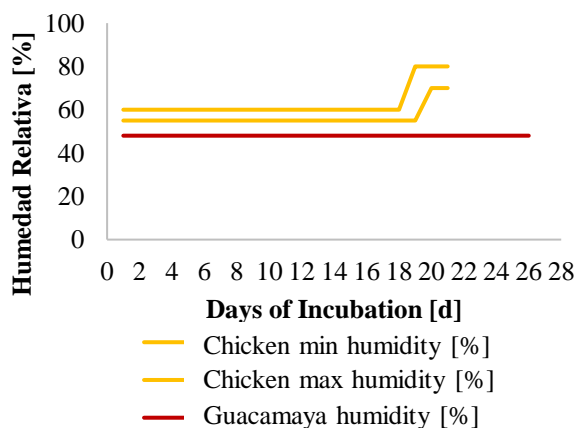
If the air is not being uniformly distributed throughout the incubator, the result is prenatal death of the embryo. During incubation the egg absorbs oxygen and removes  $CO_2$  in large quantities. Only an adequate air exchange guarantees good incubation results.

Figure 1 shows the temperature range comparison between a poultry (chicken) and an endemic species of Quintana Roo (macaw). On the Y axis the temperature range of each species is plotted, while on the X axis the incubation time is presented. It should be mentioned that the temperature range of the macaw is more precise, it only varies  $0.1^\circ C$ , while the chicken has a tolerance of  $0.5^\circ C$ .



**Graph 1** Incubation temperature comparison between a chicken and a macaw

Graph 2 shows the relative humidity variable in comparison between a chicken and a macaw, where the chicken has a range of 5% so that the embryo can develop, while for the macaw the control of relative humidity must be more subtle, since variations in their development are not allowed. The incubation of macaws is very delicate, 2 of 3 tend to die due to slight variations. The incubation period expressed in days is shown on the  $x$  axis and the relative humidity range on the  $y$  axis.



**Graph 2** Comparison of relative humidity to incubate a chicken and a macaw

Graph 3 shows the incubation time of the 28 days expressed in hours, in order to appreciate the change in the angular position. The rotation of the eggs helps the yolk not to adhere to the shell and thus the embryo develops correctly, even the turning of the eggs improves the distribution of heat and humidity, so the species has a better chance of developing in good conditions.

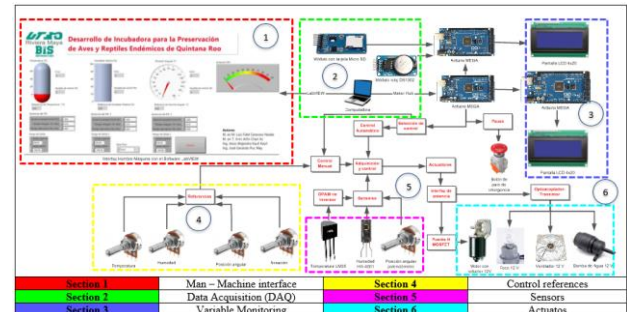
The  $x$  axis shows the hours of incubation and the  $y$  axis the degrees of rotation of the egg ranging from  $0^\circ$  to  $180^\circ$ .

It should be mentioned that the chicken must be rotated 5 times per day, while the macaw requires 6 rotations to ensure a successful incubation.

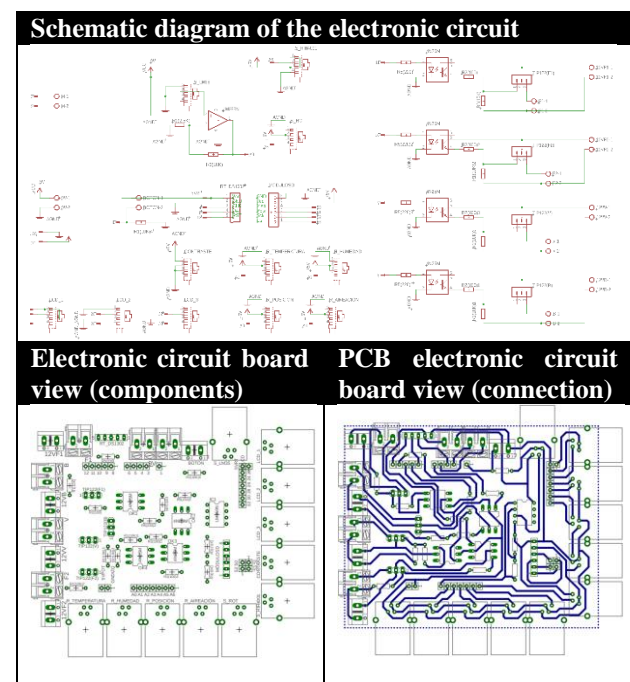
### III. Prototype development

In order to understand the operation of the proposed artificial incubator, a block diagram has been developed with the general components that have been classified into six sections, in which the Man-Machine interface, the data acquisition system (DAQ), the variable monitoring, the knobs to adjust the control references, the sensors and the actuators shown in Figure 2 stand out.

In order to observe in greater detail the components of the electronic instrumentation and the isolation for the power outputs by means of optocouplers, the electronic design of a shield card was made with an Arduino Mega 2560, in which the sensors, actuators and voltage sources are connected, as illustrated in Figure 3. Once the construction of the printed circuit board is completed, the actuators are assembled and the sensors are fixed to the incubator.



**Figure 2** Block diagram of incubator components



**Figure 3** Schematic diagram of the electronic circuit with two board views

### IV. Proposed Control System

To maintain the artificial incubation parameters of the prototype shown in Figure 4, a control system is proposed, which is composed of three PID controllers (Ogata, 2010), (Ziegler, 1942) and a conditioning controller.





**Figure 4** Prototype of the artificial incubator

The first controller serves to regulate the incubation temperature, where it is possible to vary the power of the actuator (four lights) to evenly increase the heat, the reading of this variable is done by means of an analog temperature sensor LM35DZ. The proposed structure of the PID controller to regulate the temperature using a bulb and a fan has been implemented for a closed thermal system, with experimental results in (Cerecero, Puc, Ceme, Kauil, & Chan, 2019).

The second controller serves to regulate the humidity of incubation, where it is possible to vary the power of the actuator (water pump with mist sprinkler) to increase the relative humidity inside the incubator, the reading of this variable is done by means of an HIH-4000-001 analog humidity sensor. The proposed structure of the PID controller to regulate humidity is based on the first controller, only the sensor and the actuator are modified.

The third controller serves to regulate the rotation of the egg, where it is possible to vary the angular position of the actuator (the axis of a CD motor) to turn the egg evenly as necessary for the incubation time, the reading of this variable is done by means of a logarithmic potentiometer configured as a voltage divider.

The proposed structure of the PID controller to regulate the angular position where a permanent magnet direct current motor is used has been implemented and compared in (Cerecero, Campos, Ramos, Marquéz, & Chan, 2017).

The conditioned regulator serves to induce forced aeration inside the incubator, as a secondary function, it controls the fan that serves to remove excess temperature or excess humidity in case any of these controllers exceeds the defined incubation parameters.

The visualization of the variables in the LabVIEW environment is very similar to that of the work shown in (Duran, Ramos, Cerecero, & Teran, SIMCI 2014).

### Acknowledgments

We thank the Technological University of the Riviera Maya, especially the Academic Directorate and the Deputy Directorate of Industrial Maintenance Engineering for the support provided for the registration of this article.

### V. Conclusions

It was possible to design and build a shield-type electronic card that integrates temperature, relative humidity and angular position sensors, signal conditioners, variable monitoring by means of 4X20 liquid crystal displays (LCD), the interface of power with optocouplers and transistors to manipulate the actuators which integrate the prototype of the artificial incubator.

Three PID controllers were implemented through the LabVIEW software using the Control Design & Simulation module, to send the data through the serial communication interface to the Arduino MEGA 2560 development card using the Maker Hub library. In the same way, a graphic programming interface was created with which it is possible to visualize, assign and control the artificial incubation parameters of temperature, humidity, position of the egg and aeration. So far it is possible to visualize and/or store the variables in a Datalogger for offline processing.

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## Proposal of Automatic Control System for the Pumping of a Shrimp Farm in the Town of Pimientillo, Nayarit

### Propuesta de sistema de control automático para el bombeo de una granja camaronera en la localidad de Pimientillo, Nayarit

MARTÍNEZ-AVIÑA, Salvador †\* & SOSA-SALES, Jorge

*Universidad Tecnológica de Nayarit*

ID 1<sup>st</sup> Author: *Salvador, Martínez-Aviña* / ORC ID: 0000-0002-5344-5324

ID 1<sup>st</sup> Coauthor: *Jorge, Sosa-Sales* / ORC ID: 0000-0002-7014-8209

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#### Abstract

The following project pretends to make a proposal to automate the pumping process for oxygenation in a shrimp farm of the company Loma de las cayotas, in order to reduce errors or accidents, which could generate product losses and overuse of energy, decreasing the income of the company. The oxygenation of water for shrimp farming is done by pumping water from the sea. For this process, a scale prototype was developed by automating the pump that will be used in the shrimp farm, which will act according to a selected sensor and the water level increase due to the natural tidal effect. The automation aims to reduce the uncertainty of incorrect oxygenation of the shrimp during its growth period. The process will be carried out with Arduino, a free hardware and software platform.

**Shrimp farm, Arduino, Automation**

#### Resumen

El siguiente proyecto pretende automatizar el proceso de bombeo para la oxigenación en una granja de crianza de camarón de la empresa Loma de las cayotas, con el fin de reducir errores o accidentes que se puedan presentar, generando pérdidas de producto, sobre uso de energía mermando los ingresos de la empresa. La oxigenación del agua para la crianza del camarón se realiza mediante el bombeo de agua proveniente del mar. Para este proceso, se desarrolló un prototipo a escala automatizando la bomba que se usará en la granja camaronera, que actuará de acuerdo a un sensor seleccionado y al aumento del nivel de agua debido a efecto natural de marea. La automatización pretende reducir la incertidumbre de una incorrecta oxigenación del camarón durante su periodo de crecimiento, la cual se llevará a cabo mediante Arduino que es una plataforma de hardware y software libre..

**Granja de camarón, Arduino, Automatización**

**Citation:** MARTÍNEZ-AVIÑA, Salvador & SOSA-SALES, Jorge. Proposal of Automatic Control System for the Pumping of a Shrimp Farm in the Town of Pimientillo, Nayarit. ECORFAN Journal-Ecuador. 2019 6-10: 7-11

\* Correspondence to Author (email: salvador.martinez@utnay.edu.mx)

† Researcher contributing as first author.

## Introduction

In many parts of the world, development is promoted through the implementation of technology that facilitates processes and activities, from the most basic ones, to the most complex and specialized. Shrimp production on farms is subject to different variables such as nutrition, food, oxygenation and biological variants which generate changes in the quality of water and pond soil.

The gaseous oxygen dissolved in water is vital to the existence of most aquatic organisms. The concentration of dissolved oxygen (DO) in an aquatic environment is an important indicator of environmental water quality (Galli Merino, 2007).

For intensive aquaculture, it is prudent to keep the incoming water as close as possible to the total DO saturation (100%), (Blanco, 2005).

Shrimp farms have presented good development and benefits for the owners and so, they are now an important part of the state of Nayarit, the process of water oxygenation being a fundamental part for quality production of shrimp.

Currently, the existence of farms with automated systems for the filling of shrimp farms and water oxygenation is practically null in the country, because the focus is on fish production. The prototype will mainly help facilitate the work of the person who performs the daily activity; this is an improvement for the process or operation of the farms through sensors and microcontrollers, benefiting from the physical phenomenon of tides.

The field trip provided first-hand information regarding the needs of the entrepreneur, which are: the rustic way of handling their shrimp farm network and the feasibility of carrying out the project. Practically, all the Pimientillo region of shrimp farms has electricity. According to the problems found, the proposal for a feasible or viable solution considering the investment and the benefit of the farms is the Arduino and its programming. Arduino is a free software platform, the use of labview is not considered necessary for the implementation of the project, but it was carried out to observe how this automation can be scaled.

## Methodology

A project for the automation and control of the filling of a shrimp pond was carried out in the town of Pimientillo, municipality of Tuxpan, state of Nayarit, Mexico, to reduce expenses from shrimp sales by paying the night shift to the operator and avoiding losses due to distractions and human errors that end in shrimp and income losses for the owner, for not performing the corresponding water oxygenation of the day.

According to the aforementioned, the automation of the pumping and drainage system for a shrimp culture will be carried out through circuits, level measurement sensors and actuators.

Having an automated system within the farm is very reliable and effective, since it would eliminate operator oversight when performing their pump on and off activities during the entire time the tide is high, as well as reducing labor costs because a person will no longer be needed for such activity.

Before describing the procedure, it is important to mention that the level or degree of oxygenation is not measured before or after the water pumping process. Everything is managed empirically and is knowledge passed from generation to generation, which determines the time of daily water pumping to the farms, hence no results regarding this aspect are shown.

The complete project includes measuring the degree of oxygenation and improvements in shrimp farming, the progress of the project and the article. It is limited to the automation of the process of pumping water for oxygenation, keeping in mind that it is of great importance to measure this variable.

Activities: Some of the activities that were proposed for the project were:

1. We analyzed a problem and looked for possible solutions.
2. We went on a field trip to learn the physical and environmental conditions where the problem was.
3. We proposed each of the stages that a project entails and we dedicated time for the elaboration of each one of them.



4. We designed the plans with the distribution and structures of the prototype, as well as the programming and facilities it will need.
5. We carried out the acquisition of the necessary material for the realization of the prototype; we also got some recycling materials.

Tools, equipment and machines used.

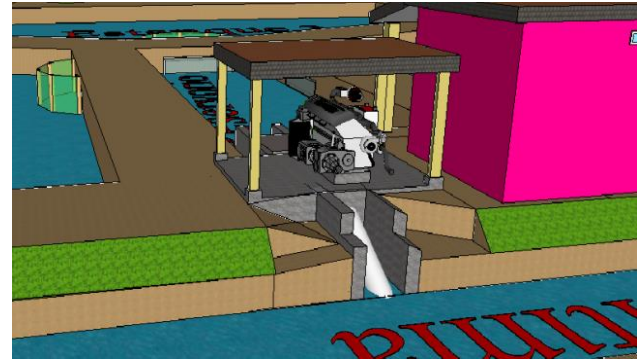
- Truper copper tipped soldering iron.
- Cross and blade screwdriver:  
Standard measurement, acetate plane measured 1/8x3.  
Adjustable wrench with 6" lining.  
1/2 type 58d34 high speed steel drill for general purpose.  
110V electric drill.
- Cutting, pressure and mechanical clamps.  
10" straight pressure clamps, standard mechanical 9" cutting clamps.
- 40 ounces curve hammer.
- Circular grinder with diamond disc (cricket).
- Flexometer.
- Protoboard.
- Cutter.
- Voltage source (5 to 24 V).

Figure 1 shows how the farm is currently working by means of a standard turbo diesel engine. We propose to implement an electric pump that activates automatically through an automated control, facilitating the filling process on the farm and avoiding hours of filling and manual labor.



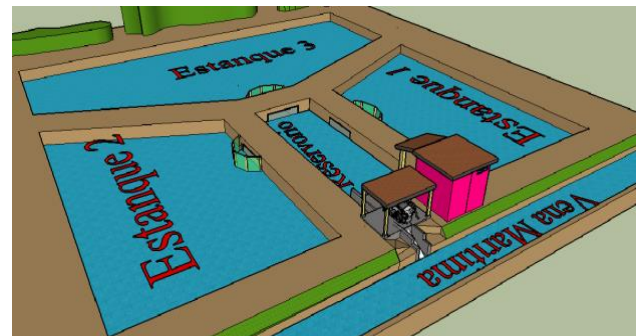
**Figure 1** Current pumping in shrimp farm

Figure 2 shows on a simulated scale the position where the turbodiesel engine is located in the shrimp farm, the change would be from the engine to an electric pump, automated by means of a level sensor.



**Figure 2** pumping structure

Figure 3 shows a preliminary design of the physical or flat distribution of the shrimp farm and its divisions, which are: 3 ponds, a reservoir and the sea vein, from which water is taken to fill the farm by means of the engine shown in figure 2.



**Figure 3** Shrimp Farm Distribution

Figure 3 shows the complete distribution of the farm, the area where the pump is located, as well as the house where the operator fulfills the function of turning on the pump when the tide rises and reaches a desired value, that is, to reach the hose or suction tube, while the tide is rising, which keeps the pump on and carries out the process of water oxygenation, once the tide drops, the operator will turn off the pump. The process usually takes place in the late evening.

Figure 4 shows the electrical connection, the part of the Arduino in connection with the protoboard and the ultrasonic sensor.

The sensor has the features of measuring range of 2 cm to 4 meters maximum, an accuracy of  $\pm 3\text{mm}$ , current consumption of 15 mA and operating voltage of 5V, which will be applied to measure the level of the tide, simulated with a vessel increasing the water level, as explained below.

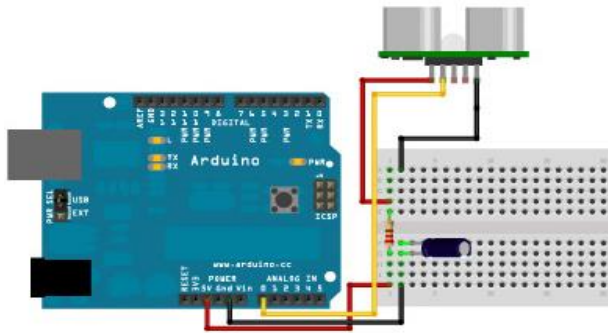


Figure 4 Arduino and ultrasonic sensor

Once the connections were made, the programming was only for the sensor and Arduino, through C language.

The routines were programmed so that the sensor acted by turning on a LED when it detected the level that was established in the programming and turned off when the level was below. Within the programming a time delay for the pump operation was placed, to avoid false level measurements by surge in the area, which in this case is a small channel where the suction tube is located.

Once the programming was completed, the Arduino plate was placed on a container and pumps were connected schematically, as seen in figure 5.

With this, the programming and the time delay were validated before a disturbance of the measured signal (level); the manual valves simulate the effects of the tide and the pump, like the one that injects water into the ponds, these being the models shown in figure 3.

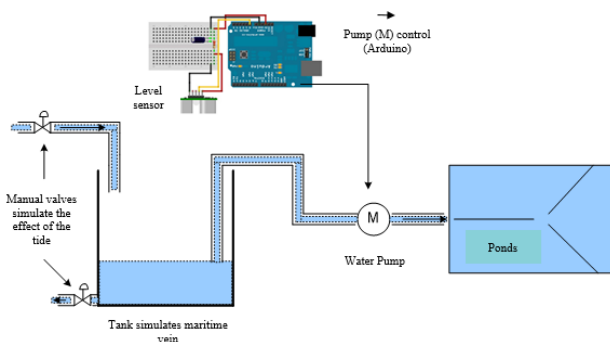


Figure 5 Programming Validation

Once this was done, the interface in the Labview program was programmed as a visualization and control system of the activation and levels.

Figure 6 shows that once the water reached its level, the tank is filled with the river level label. And the tanks that have the label major river level minor river level are those that adjust to the values of turning on and off (delay) the seawater suction pump to oxygenate the ponds.

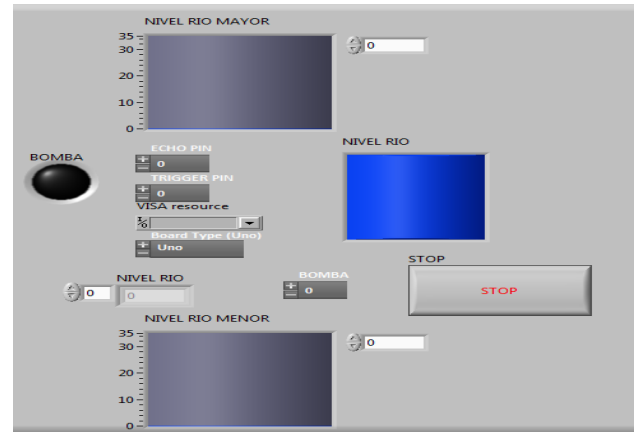


Figure 6 Simulation in Labview

Figure 7 shows the block diagram of the Labview program which performs the process of level comparison, as well as the visualization of the variation of the water level simulating the tide. When the LED on the display on the Labview front panel was turned on, the pump was turned on, indicating that the established conditions were met.

The conditions of the process are: with the sensor it will detect the presence of water at a distance of 5 cm; it will send a signal to the Arduino and through this, the pump will be activated and start filling the farm; as the farm was filling, the water of the container outside decreased until the distance of 2.5 cm is reached; the sensor detects the distance and also sends a signal to the Arduino, turning off the LED and deactivating the pump, stopping the oxygenation process.

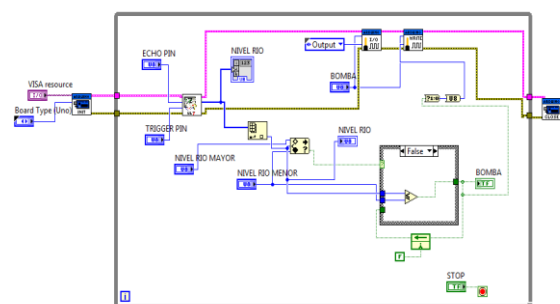


Figure 7 Labview block diagram

## Results

Figure 8 shows the complete and physical prototype with its electrical connections, which are not visible since it was part of the requirements for the presentation. The prototype was made to scale for the simulation of the main objective, that is, the automation of a pump. Figure 8 also shows the divisions of the ponds already filled with water, a drain pipe and the part of the pipe where the water flowed through the pump.

The pump used was of 24 V, so a power arrangement was made to condition the signal, since the Arduino handles 5V output voltages.



**Figure 8** Physical prototype

## Conclusions

After the installation and placement of the prototype, the electrical/electronic and structural part worked correctly. The initial test is only the filling control of the tank based on the level of the simulated tide with a container with the Arduino. Level sensor and pump functioned correctly, when adding the visualization and control by means of the Labview software the result was satisfactory, having the plus of observing the change of the level variables and the pump drive.

If more automation is required, Arduino has communication components that can extend the monitoring and control of the process remotely. As a functional prototype, the use of a platform with Arduino is justifiable due to its cost, but if greater certainty is required in the equipment, the use of more specialized and robust equipment in industrial settings such as the PLC is recommended.

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## Retention capacity of maltodextrin antioxidants in cranberry juice by spray drying process

### Capacidad de retención de antioxidantes de maltodextrina en jugo de arándano en polvo secado por aspersión

SAAVEDRA-LEOS, María Zenaida†\*, SILVA-CÁZARES, Macrina Beatriz, GONZÁLEZ-TREVIZO, Cynthia Lizeth and TERRONES-GURROLA, María Cruz del Rocío

*Universidad Autónoma de San Luis Potosí*

ID 1<sup>st</sup> Author: *María Zenaida, Saavedra-Leos* / ORC ID: 0000-0001-6721-7114, CVU CONACYT ID: 207550

ID 1<sup>st</sup> Coauthor: *Macrina Beatriz, Silva-Cázares* / ORC ID: 0000-0002-0277-0186, CVU CONACYT ID: 253090

ID 2<sup>nd</sup> Coauthor: *Cynthia Lizeth, González-Trevizo* / ORC ID: 0000-0002-2720-7242, CVU CONACYT ID: 252863

ID 3<sup>rd</sup> Coauthor: *María Cruz del Rocío, Terrones-Gurrola* / ORC ID: 0000-0002-2165-4674, CVU CONACYT ID: 448978

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#### Abstract

A group of four maltodextrins with different degrees of dextrose equivalent (DE) was used as carriers for the spray drying of cranberry juice, with the objective of evaluating the effect of drying temperature, DE grade and concentration of maltodextrin on the performance of cranberry juice powder and the ability to retain antioxidants. For this purpose, the conditions of the spray drying process were: inlet air temperature: 170-210° C, maltodextrin type (Mc, M10, M20 and M40) and maltodextrin concentration: 10-30% w/w. The different powders obtained from the spray drying process were analyzed by high performance liquid chromatography (HPLC) for the quantification of antioxidants (resveratrol and quercetin). The analysis of the data was carried out from a design of D-Optimal experiments. The results of the analysis showed that the highest yield of cranberry juice powder is achieved by using 30% w/w of M10 at a drying temperature of 193 °C. Such conditions allowed a retention of antioxidants in the powder of 20% and 30% of resveratrol and quercetin, respectively.

**Antioxidants, Cranberry, Spray drying process**

#### Resumen

Un grupo de cuatro maltodextrinas fueron utilizadas como agentes acarreadores para el secado por aspersión de jugo de arándano, con el objetivo de evaluar el efecto de la temperatura de secado y la concentración de maltodextrina sobre el rendimiento de jugo de arándano en polvo y la capacidad de retención de antioxidantes. Para tal efecto las condiciones del proceso de secado por aspersión fueron: temperatura de aire de entrada: 170-210°C y concentración de maltodextrina: 10-30% p/p. Los diferentes polvos obtenidos del secado por aspersión, fueron analizados mediante cromatografía líquida de alta resolución (HPLC) para la cuantificación de dos antioxidantes. El análisis de los datos se llevó a cabo a partir de un diseño de experimentos D-Optimo. Los resultados del análisis realizado mostraron que el mayor rendimiento de jugo de arándano en polvo se logra al utilizar 30% p/p de M10 a una temperatura de secado de 193 °C.

**Antioxidantes, Arándano, Polvo secado por aspersión**

**Citation:** SAAVEDRA-LEOS, María Zenaida, SILVA-CÁZARES, Macrina Beatriz, GONZÁLEZ-TREVIZO, Cynthia Lizeth and TERRONES-GURROLA, María Cruz del Rocío. Retention capacity of maltodextrin antioxidants in cranberry juice by spray drying process. ECORFAN Journal-Ecuador. 2019. 6-10: 12-20

\* Correspondence to Author (email: zenaida.saavedra@uaslp.mx)

† Researcher contributing as first author.



## Introduction

Antioxidants are bioactive compounds capable of neutralizing unstable free radicals that cause aging and chronic degenerative diseases, such as cancer, diabetes mellitus, cardio and cerebrovascular diseases, among others (Lee et al., 2015; Masisi et al., 2016; Polanski et al., 2015). From a nutritional point of view, the consumption of foods rich in antioxidants is one of the most effective ways to reduce the risk of chronic diseases.

Cranberries have been recognized for its high content of phenolic and flavonoid compounds, such as quercetin, resveratrol, myricetin and kaempferol (Parada-Caro, 2005). However, the use of this fruit as a source of antioxidants is restricted, as it is a perishable food with a high aqueous content of up to 85% (Hyun-Chun et al., 2012). In 2014, Mexico obtained a production of 5,529 tons of fresh cranberry (Sagarpa, 2014) of which 50% was processed in the form of various products, such as juices, nectars, yogurts, jams and semi-prepared foods, like powders, syrups and reconstitute concentrates (Skrovankova et al., 2015; Waheeda et al., 2015). Unfortunately, during the processing of the fruit, there are losses of the antioxidant compounds, associated with the heat treatments since the phenolic compounds are heat sensitive at temperatures above 60 °C (Wang et al., 2009).

The lack of adequate conservation methods that allow lengthening the shelf life, obtaining minimal losses of volatile components (phenolic and flavonoids) and maintaining the nutritional and organoleptic qualities during the processing and storage of the cranberry, has caused losses of 20% to 50% (Feippe-Fernández, 2013; Cano-Chauca et al., 2005). This leads to the search for more appropriate conservation methods. A viable alternative is spray drying, which has been preferred as an economical, flexible process, easy to operate and control, with high drying rates (Silva et al., 2008; Porras-Saavedra et al., 2015) and very short residence times, allowing heat-sensitive products to be dehydrated at relatively high temperatures, retaining some properties of foods such as taste, color, smell and nutrients (Masters, 1991; Mujumdar, 1998). As a result, a powder product is obtained with a longer shelf life and a reduced volume, a useful feature in terms of storage and transport (Cano-Chauca et al., 2005).

However, the difficulty of applying spray drying in products, such as fruit juices, is due to high water and sugar contents, which have a low glass transition temperature ( $T_g$ ) (Van Sleuwen et al., 2012; Saavedra-Leos et al., 2012; Collares and Kieckbush, 2002; Vanhal and Blond, 1999; Ruan et al., 1999). This causes low yields, operating problems and difficulty in predicting product quality. A practical solution is the use of high molecular weight additives (adjuvants) as carrier agents to modify the properties of the material in order to reduce the problem of sticking during spray drying (Saavedra-Leos et al., 2015; Ceballos -Peñalosa, 2008). The amount of carrier agent used ranges from 20% to 60% w/v for food dehydration (Masters, 1991).

In spray drying, maltodextrin is used as a carrier agent mainly because of its high molecular weight, low viscosity and glass transition temperature ( $T_g$ ) in a range of 100 to 188 °C, which depends on its dextrose equivalent (DE). This polysaccharide can have a DE value of 5-40 and depending on it, maltodextrin has different physicochemical and functional properties (Udomrati and Gohtani, 2015; Reineccius, 1991). Based on the above, it can be established that factors such as spray drying temperature and the type and percentage of carrier agent are determinants to improve the performance and quality of powdered juices. It is important to carry out systematic studies based on the use of statistical tools that allow evaluating the effect of these factors, in order to establish the optimal conditions for the processing of fruit juices guaranteeing a good efficiency.

That is why in the present work a design of D-Optimal experiments was used to evaluate four maltodextrins and concentrations of 10 to 30% w/w, as well as drying temperatures of 170 to 210 °C, to obtain cranberry juice powder and evaluate the performance and retention capacity of antioxidants.

## Methodology

### Spray drying

Dehydration was carried out in a Mini Spray Dryer B290 (Buchi, Switzerland). The samples were placed in the dryer at room temperature and a volumetric flow of hot air of 35 m<sup>3</sup>/h was used, adjusting the vacuum to 70% capacity.

A constant compressed air pressure of 1.5 bar was used. A total of 25 experiments were performed, where a range of inlet temperatures between 170 °C and 210 °C was handled, while the output was recorded between 70 °C and 90 °C. As the encapsulating agent, maltodextrin (MD) with different equivalents of dextrose (Mc, M10, M20, M40) was used at different concentrations as specified in Table 1.

Random	Run	Factor 1 T (°C)	Factor 2 C (g)	Factor 3 MD*
12	1	181.297271	30	M40
22	2	193.06311	30	M10
14	3	193.06311	30	M10
18	4	190	25.043167	M20
5	5	210	30	MC
11	6	170	10	M20
1	7	170	30	MC
16	8	175.00002	12.5	MC
19	9	209.375	24.1630577	M40
4	10	210	10	M40
21	11	170	10	M40
17	12	190	19.6296296	M10
8	13	170	10	M40
23	14	170	10	M20
20	15	210	20	MC
3	16	170	30	M20
24	17	181.297271	30	M40
2	18	186.541152	10	M10
10	19	170	21.6266586	M10
7	20	210	10	M20
13	21	189.99939	20.0003722	MC
6	22	210	18.3871388	M10
9	23	210	10	MC
25	24	170	21.6266586	M10
15	25	210	30	M20

\*MD= maltodextrin

**Table 1** Design of experiments for D-Optimal analysis  
Source: Prepared by the authors

## Design of experiments

A design of D-optimal experiments was used in Minitab 17, to evaluate the effect of the concentration of the carrier agent with different degrees of dextrose and different drying temperatures, in order to establish the optimal conditions for obtaining the best yields of cranberry juice powder. The 3 factors to be analyzed were classified as follows: 1) Temperature (T): 170, 175, 181, 187, 190, 193, 209 and 210 °C; 2) Maltodextrin concentration (C): 10, 12.5, 18, 20, 22, 24, 25 and 30% w/w; and 3) Type of maltodextrin (MD): Mc, M10, M20, M40.

The response variables were: 1) Yield of dust collected in % w/w; and 2) Retention of antioxidants from cranberry juice in µg/mL.

The design consisted of 25 experimental runs (Table 1), which were randomly selected in order to determine the response variables for each sample. To draw the contour curve graphs, a numerical assignment was made to the type of maltodextrin factor of 0, 10, 20 and 40 for Mc, M10, M20 and M40, respectively. Similarly, an ANOVA analysis was performed to estimate the effect of the factors evaluated on the performance of the powder juice obtained.

## Determination of antioxidant content by High Performance Liquid Chromatography (HPLC)

HPLC was carried out with a Waters chromatographic equipment (Waters Assoc. Milford MA, USA) composed of a Binary pump (HPLC model 1525), an autoinjector (model 717) and a double wavelength absorbance detector (model 2487).

The quantification of resveratrol and quercetin was performed under the following operating conditions: room temperature with a mobile phase of acetonitrile: formic acid 0.01% pH 3.0 (70:30 v/v) at a flow of 1 mL/min and a length of 306 nm wave. Chromatographic separation was carried out on a column of the Agilent C18 brand, (75 x 4.6 mm ID 3.5 µm) with an injection volume of 10 µL. All data was processed with the Empower Pro Version 4.0 program.

The calibration curves were made from stock solutions of resveratrol (1000 µg/mL) and quercetin (1000 µg/mL), prepared in HPLC grade methanol. The dilutions used were 0.01, 1, 5, 10 and 20 µg/mL, to prepare the six-point calibration curve. All solutions were prepared the same day as their injection.

Approximately 0.5 g in triplicate were weighed for sample extraction, to which a mixture of 0.01% acetonitrile and phosphoric acid (50:50 v/v) was added. Subsequently, it was vigorously stirred for 5 minutes and left in a dark place for 24 hours. Finally, the solutions were passed through Acrodisc filters of 0.45µm and were injected into the equipment.

## Results

Table 2 shows the yields of obtaining dried cranberry juice under the different established conditions.

MD*	MD* Concentration (% weight)	Cranberry juice concentration (% weight)	Drying temperature (°C)	Yield (%)
M40	30	70	181	2.02
M10	30	70	193	9.68
M10	30	70	193	11.87
M20	25	75	190	2.81
Mc	30	70	210	6.74
M20	10	90	170	0
Mc	30	70	170	6.37
Mc	12.5	87.5	175	1.82
M40	24	76	209	7.43
M40	10	90	210	0
M40	10	90	170	0
M10	20	80	190	8.15
M40	10	90	170	0
M20	10	90	170	0
Mc	20	80	210	7.68
M20	30	70	170	4.23
M40	30	70	181	1.25
M10	10	90	187	0.12
M10	22	78	170	9.48
M20	10	90	210	0
Mc	20	80	190	7
M10	18	82	210	5.45
Mc	10	90	210	0
M10	22	78	170	8.37
M20	30	70	210	3.03

\*MD= maltodextrin

**Table 2** Efficiency and drying conditions of maltodextrins  
Source: Prepared by the authors

The highest yield was obtained by using a 20% w/w concentration of maltodextrin at a process temperature of 210 °C, with a weight yield of 7.68%. When using a concentration of 30% w/w at 210 and 170 °C, a lower yield of 6.74% and 6.37%, respectively, was obtained.

In the case of a concentration of 12.5 and 10% w/w of maltodextrin, a collapse of the system associated with problems of bonding of the material in the walls of the drying chamber was observed. These results are similar to those reported by Saavedra et al. (2015) when using a 30% w/w concentration of carrier agent; however, there are no similar reports of system collapse when using 20, 12.5 and 10% w/w of maltodextrin.

In the case of M10 and based on experiment design, the concentrations handled were: 10, 18, 20, 22 and 30% w/w of carrier. Of the results analyzed in Table 2, the highest yield was obtained using a concentration of 30% w/w maltodextrin at 193 °C, with a yield of 11.87%. From the duplicate under these same conditions, a yield of 9.68% was obtained, with a general average of  $10.77 \pm 1.5485$ . On the other hand, at a concentration of 22% w/w and 170 °C, a yield of 9.48% and 8.37% was obtained in duplicate, giving an average of  $8.925 \pm 0.7848$ .

Likewise, when using a concentration of 20% w/w at 190 °C, a yield of 8.15% was reached. In the case of a concentration of 18% w/w and a process temperature of 210 °C, a yield of 5.45% was obtained. And finally, at 10% w/w of carrier agent the system collapsed, so the yield obtained was only 0.12%. These results are consistent with those reported by Caliskan and Dirim (2013), who studied the feasibility of a spray drying process of sumac extract (*Rhus coriaria* L.) with the addition of maltodextrin DE 10.

They used inlet/outlet temperatures of air of 160/80, 180/90 and 200/100 °C, and adjusted the total solids content of the extract to 10, 15, 20 and 25% w/w with the addition of maltodextrin DE10. The researchers obtained yields of 70.21%, 86.77%, 97.45% and 98.5% for extracts with 10, 15, 20 and 25% maltodextrin (ED 10) respectively, which allowed to conclude that increasing the concentration of carrier agent increased the yield of the powder obtained. On the other hand, Nadeem et al. (2011) obtained similar results to those reported in this work when using M10.

These researchers sprayed mountain tea (*Sideritis stricta*) using maltodextrin DE12 as a carrier at two concentrations (3 and 5% w/w) and three different drying temperatures (145 °C, 155 °C and 165 °C). They found that the increase in product performance was related to the concentration of carrier and not to the temperature process, which may be associated with a higher solids content of the feed solution. In this regard, Ameri and Maa (2006) point out that increasing the total solids content of the feed solution is a way to increase the recovery of powder in spray drying operations.

Regarding M20, concentrations of 10, 25 and 30% w/w of maltodextrin were used. When comparing the results in Table 2, it is observed that the highest yield was obtained by using a concentration of 30% w/w at a process temperature of 170 °C, with a weight yield of 4.23%. At this same concentration and at a temperature of 210 °C, a yield of 3.03% was obtained, while for a concentration of 25% w/w of maltodextrin at a process temperature of 190 °C a yield of 2.81% was obtained. In the case of a 10% w/w concentration of maltodextrin at temperatures of 170 and 210 °C respectively, the samples collapsed.

These results are similar to those reported by Bhusari et al. (2014), who studied the effects of different carrier agents, including DE20 maltodextrin, on the physical and microstructural properties of spray dried tamarind pulp powder. They observed an improvement in product recovery by increasing the concentration of the carrier. This increase in the percentage of powder recovery is attributed to the reduction in the adhesion and deposition of powder particles in the walls of the drying chamber, as well as to the increase in the glass transition temperature resulting from the mixing of the carrier agent and tamarind pulp.

On the other hand, Peng et al., (2013) showed similar results to those obtained in this work; they used maltodextrin DE20 when spray dried purple sweet potato flour with p/w percentages of maltodextrin from 10 to 40%, observing a greater increase with a 30% concentration of maltodextrin. However, they observed that as the percentage of maltodextrin increased from 30 to 40%, there were slight changes in yield, but a significant decrease in the quality of the powder obtained.

Finally, for M40 maltodextrin, concentrations of 10, 24 and 30% w/w were used. According to the results of Table 2, it was observed that the highest yield was obtained by using a concentration of 24% w/w at a process temperature of 209 °C, with a value of 7.43%. For a concentration of 30% w/w and a process temperature of 181 °C, a lower yield of 2.02% and 1.25% was obtained, with a general average of  $1.635 \pm 0.5444$ . In the case of a 10% w/w concentration of maltodextrin at temperatures of 170 and 210 °C respectively, said samples collapsed.

These results are similar to those reported by Saavedra et al., (2015) who analyzed the relationship between the degree of polymerization and the physicochemical properties of 4 samples of maltodextrin with different equivalents of dextrose, including maltodextrin DE40, under a spray drying process. In his research the performance of the product was not studied.

However, they mention that the lowest glass transition ( $T_g$ ) was presented by maltodextrin with a greater degree of dextrose equivalents (M40), so it can be assumed that the lowest yields were obtained when using M40, since the primary effect of the addition of adjuvants such as maltodextrins lies mainly in increasing the  $T_g$  of the system to avoid problems of sticking and collapse in the dryer chamber.

It can be observed in Table 2 that the highest percentage was achieved by using 30% w/w of M10 at a process temperature of 193 °C. With these results, it can be perceived that the use of the M10 carrier agent in the spray drying process of cranberry juice favors the obtention of higher yields. These results are consistent with those obtained by Saavedra et al., (2015) who proposes the use of M10 as a carrier agent in spray drying of sugar-rich systems such as fruit juices, due to their physical ( $T_g = 100$  °C and 90 °C,  $T_d =$  greater than 190 °C) and chemical properties (experimental DE = 1 and Degree of polymerization = 16).

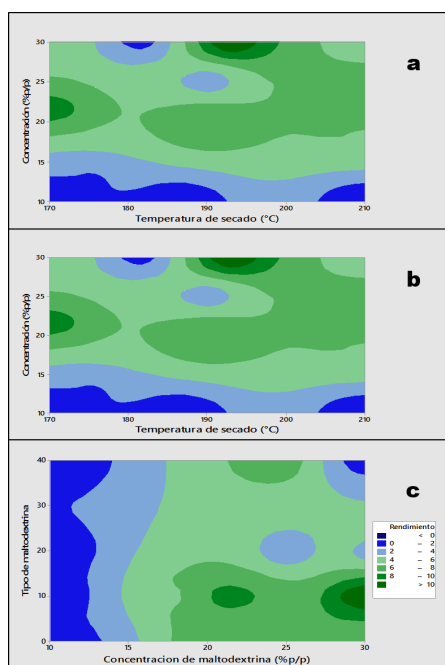
#### D-Optimal Analysis

Of the treatments proposed in the D-optimal design, a yield range of cranberry juice from 0 to 11% was obtained. From the combination of the three factors evaluated, we prepared the graphs corresponding to the contour curves (Figure 1). To identify the regions associated with the best yields, yield curves greater than 10% were analyzed, which are presented in an intense green color in Figure 1.

For the drying temperature-concentration ratio of maltodextrin (Figure 1a), there are two optimal regions, the first between 20-23% maltodextrin and 170-175 °C, and the second at concentrations of 28-30% maltodextrin in an interval from 190-200 °C. In Figure 1b where the contour curves of the drying temperature-type of maltodextrin ratio are shown, the optimal region for M10 is set at temperatures of 170 ° and 190-195 °C.

Finally, for the concentration-type of maltodextrin ratio (Figure 1c) the optimal region corresponds to M10 at a concentration of 30% maltodextrin. Based on the results obtained from the contour curves, it can be established that M10 maltodextrin is the carrier agent that allows obtaining the best yields at a concentration of 30% and temperatures between 190-195 °C.





**Figure 1** Contour graphs for the relations a) Drying temperature-concentration of maltodextrin, b) Drying temperature-type of maltodextrin, c) Concentration of maltodextrin-type of maltodextrin

Source: Prepared by the authors

Table 3 shows the results of the analysis of variance ( $\alpha = 0.05$ ), where the independent effect is determined. It shows that the concentration and type of MD are the only factors that have a significant effect on the yield of the powdered juice, while the temperature showed no significant effect. Based on these results, it can be concluded that to obtain the best yields the determining factors to consider are the concentration and type of MD.

Source	GL	SC	MC	F	p
Temperature	8	119.7412	14.9676	5.854	0.248
Concentration	6	190.0694	31.6782	1.303	0.011
Type of MD	5	9.1718	1.8344	2.578	0.000
Error	3	0.6160	0.2053		
Total	22	319.5985			

**Table 3** Analysis of variance for the performance of cranberry juice powder.

Source: Prepared by the authors

## Antioxidant retention

The results corresponding to the retention capacity of antioxidants in cranberry juice are shown in Table 3. It is observed that the sample AJ-M10 at 30% (w/w) and 193 °C was the only sample that managed to retain antioxidants. Regarding resveratrol, this varied from 0.662  $\mu\text{g}/\text{mL}$  in cranberry juice to 0.140  $\mu\text{g}/\text{g}$  powder with maltodextrin (AJ-Mc).

While for quercetin 3-D-galactoside the content ranged from 0.326  $\mu\text{g}/\text{mL}$  in cranberry juice, to 0.093  $\mu\text{g}/\text{g}$  powder in the AJ-M10 sample at 30% (w/w) at 193 °C. These results coincide with those reported by Robert et al., (2010), who encapsulated bioactive compounds of pomegranate juice and ethanolic extracts with maltodextrin and soy protein isolate by spray drying.

Bakowska-Barczak and Kolodziejczyk (2011) found that inulin was less effective in the encapsulation of blackcurrant polyphenols (*Ribes nigrum* L.) than maltodextrin. While Santiago-Adame et al. (2015) managed to retain about 60% of polyphenols by spray drying cinnamon infusions, using maltodextrin as an encapsulating material.

González et al. (2011) evaluated a series of treatments to conserve volatile watermelon juice compounds with spray drying, where they obtained a greater conservation of these compounds by using a mixture of maltodextrin DE10 and gum arabic 1:1 p/p and less conservation when using only maltodextrin or only gum arabic as carrier. Likewise, Quirino-Lacerda et al. (2016) mention in their work that when performing spray drying of jussara pulp, a higher anthocyanin content was obtained by using mixtures of maltodextrin and inulin as the wall material.

MD	MD Concentration (%)	Cranberry juice concentration (%)	Drying temperature (°C)	Efficiency (%)	Antioxidants $\mu\text{g}/\text{mL}$	
					Resveratrol	Quercetin
AJ-M40	30	70	181	2.02		
AJ-M10	30	70	193	9.68		
AJ-M10	30	70	193	11.87	0.140	0.093
AJ-M20	25	75	190	2.81		
AJ-Mc	30	70	210	6.74		
AJ-M20	10	90	170	0		
AJ-Mc	30	70	170	6.37		
AJ-Mc	12.5	87.5	175	1.82		
AJ-M40	24	76	209	7.43		
AJ-M40	10	90	210	0		
AJ-M40	10	90	170	0		
AJ-M10	20	80	190	8.15		
AJ-M40	10	90	170	0		
AJ-M20	10	90	170	0		
AJ-Mc	20	80	210	7.68		
AJ-M20	30	70	170	4.23		
AJ-M40	30	70	181	1.25		
AJ-M10	10	90	187	0.12		
AJ-M10	22	78	170	9.48		
AJ-M20	10	90	210	0		
AJ-Mc	20	80	190	7		
AJ-M10	18	82	210	5.45		
AJ-Mc	10	90	210	0		
AJ-M10	22	78	170	8.37		
AJ-M20	30	70	210	3.03		
					0.662	0.326

**Table 4** Resveratrol and quercetin antioxidant concentration of cranberry juice powder

Source: Prepared by the authors

Although maltodextrin has been one of the most used carrier agents for drying fruit juices, it is evident that it has a limited capacity to retain the antioxidants present in cranberry juice.

So it would be interesting and a better alternative to use mixtures of maltodextrin with other carrier agents to improve the retention of antioxidants.

## Conclusions

Cranberry juice powder was obtained by using Mc, M10, M20 and M40 at concentrations of 30, 20, 25, 24, 22 and 18 w/w and a process temperature of: 210, 209, 193, 190, 181 and 175, 170 °C.

The Mc, M10, M20 and M40 systems collapsed when using a concentration of 12.5, 10 w/w at a process temperature of 210, 175 and 170 °C.

The highest percentage of juice powder was obtained at 30% w/w of M10 at a process temperature of 193 °C, which favored higher yields.

Two optimal process regions are established: a) maltodextrin concentrations at 20-23 w/w and a range of 170-175 °C, b) concentrations of 28-30% maltodextrin in a range of 190-200 °C.

The optimal region is established for M10 at temperatures of 170 ° and 190-195 °C.

For the concentration-type of maltodextrin ratio it was determined that the optimal region corresponds to M10 at a concentration of 30% maltodextrin.

Based on the results obtained from the contour curves, it can be established that M10 maltodextrin is the carrier agent that obtains the best yields, it was also the only MD that presented resveratrol and quercetin antioxidant retention in an approximate percentage of 20% and 30%, respectively.

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## Surface tension of O<sub>2</sub>-Ar, N<sub>2</sub>-Ar and O<sub>2</sub>-N<sub>2</sub>-Ar mixtures

### Tensión superficial de las mezclas O<sub>2</sub>-Ar, N<sub>2</sub>-Ar y O<sub>2</sub>-N<sub>2</sub>-Ar

GARCÍA-MARTÍNEZ, Mauricio<sup>1†</sup>, IBARRA-TANDI, Benjamín<sup>1</sup>, LUIS-JÍMENEZ, Daniel Porfirio<sup>2</sup> and LÓPEZ-LEMUS, Jorge<sup>1\*</sup>

<sup>1</sup>Universidad Autónoma del Estado de México, Facultad de Ciencias, Instituto Literario 100, CP 50000, Estado de México

<sup>2</sup>CONACYT Research Fellow-Centro de Ingeniería y Desarrollo Industrial (Sede Campeche), Ave. Playa Pie de la Cuesta No. 702, Desarrollo San Pablo, Querétaro, Querétaro 76125, México.

ID 1<sup>st</sup> Autor: *Mauricio, García-Martínez* / ORC ID: 0000-0001-7490-5230

ID 1<sup>st</sup> Coautor: *Benjamín, Ibarra-Tandi* / ORC ID: 0000-0001-6405-8701, CVU CONACYT ID: 38320

ID 2<sup>nd</sup> Coautor: *Daniel Porfirio, Luis-Jiménez* / ORC ID: 0000-0003-4263-2789, CVU CONACYT ID: 161340

ID 3<sup>rd</sup> Coautor: *Jorge, López-Lemus* / ORC ID: 0000-0002-9486-6942, CVU CONACYT ID: 21571

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#### Abstract

The surface tension of some binary and ternary mixtures was calculated by means of molecular dynamics simulations in a canonical set. The analyzed mixtures were oxygen-argon, nitrogen-argon and oxygen-nitrogen-argon. The force field for argon was recalculated in order to reproduce the experimental surface tension. The corresponding force fields for O<sub>2</sub> and N<sub>2</sub> were taken from a previous work [Mol. Simul. 45 (2019) 958-966], where it was shown that such force fields reproduce the experimental surface tension curves, as pure fluids. The nitrogen-argon surface tension was calculated for several mole fractions of argon. The obtained curve was compared with those experimental data and a good agreement was found. The standard Lorentz-Berthelot combining rules were employed. For the oxygen-argon mixture it was necessary to modify the cross term of the combining rules in order to reproduce theoretical and experimental data. The surface tension of the ternary mixture was also estimated varying the mole fraction of argon at a certain concentration of oxygen and nitrogen, previously adjusted. Several temperatures were used in order to show a tendency mostly at relatively low temperatures. After comparing the available experimental data, which are scarce, a good agreement was observed.

**Surface tension, Molecular dynamics, Force field**

#### Resumen

La tensión superficial de algunas mezclas binarias y ternarias se calculó mediante simulaciones de dinámica molecular en un conjunto canónico. Las mezclas analizadas fueron oxígeno-argón, nitrógeno-argón y oxígeno-nitrógeno-argón. El campo de fuerza para el argón se re-estimó aquí para reproducir la tensión superficial experimental como fluido puro. Los campos de fuerza correspondientes para O<sub>2</sub> y N<sub>2</sub> se tomaron de un trabajo anterior [Mol. Simul. 45 (2019) 958-966], donde se demostró que tales campos de fuerza reproducen las curvas experimentales de tensión superficial, como fluidos puros. La tensión superficial de nitrógeno-argón se calculó para varias fracciones molares de argón. La curva obtenida se comparó con los datos experimentales y se encontró un buen acuerdo. Las reglas de combinación estándar de Lorentz-Berthelot fueron utilizadas. Para la mezcla de oxígeno-argón fue necesario modificar el término cruzado de las reglas de combinación para reproducir resultados teóricos y experimentales. La tensión superficial de la mezcla ternaria también se estimó variando la fracción molar de argón a una cierta concentración de oxígeno y nitrógeno ajustada previamente. Se exploraron varias temperaturas para mostrar una tendencia principalmente a temperaturas relativamente bajas. Luego de comparar con los datos experimentales disponibles, que son escasos, se observó un buen acuerdo.

**Tensión superficial, Dinámica molecular, Campo de fuerza**

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\* Correspondence to Author (email: jllemus@uaemex.mx)

† Researcher contributing as first author.

## Introduction

Molecules such as nitrogen, oxygen and argon are important air compounds. The surface tension of these relatively simple fluids has been subject to theoretical, experimental and computer simulation studies. From the point of view of molecular simulation, we can see in the literature that there is more than one model for each, but not all of them reproduce the experimental surface tension. Garcia et al. (2019) have proposed force fields for oxygen and nitrogen that reproduce the experimental data as pure fluids, where the interaction function is composed of the potentials of Lennard-Jones (L-J) and Colulomb.

In the case of argon, Goujon et al. (2014) proposed a correction to the L-J function to reproduce the experimental surface tension as a function of temperature, that is, they added an extra term to the interaction potential. We believe it would be convenient to use the L-J and Coulomb potentials in all molecular models to calculate the surface tension of the mixture of these same fluids.

The surface tension of the binary mixtures oxygen-argon and nitrogen-argon has been estimated with a theoretical procedure by Kim et al. (1969). They compared their results with the experimental data generated by Blagoi (1960), and by Blagoi and Rudenkov (1959), as a result, they found a good degree of coincidence. This interfacial property was estimated at  $T = 83.85\text{K}$  for all mixtures. Eckert and Prausnitz (1964) have estimated by theoretical calculations the surface tension of the same binary mixtures, and as a result they found a good agreement. However, there is no data of the same interfacial property at different temperatures. We believe that with molecule models that reproduce the experimental surface tension as pure fluids, it is possible to obtain good results when calculating the same interfacial property of the mixtures at  $T = 83.85\text{K}$  and for a wide range of temperatures.

On the other hand, Pandey et al. (2004) have estimated the surface tension of the ternary mixture Ar-O<sub>2</sub>-N<sub>2</sub> at different temperatures, from 90K to 110K, and at different pressures. They developed a theoretical approach using the Brock-Bird relation, where critical properties, such as pressure, volume and temperature of pure fluids, were considered in the calculations.

These authors compared their results with experimental data and observed a good agreement. Experimental data were obtained using the capillary ascent method estimated by Kai et al. (1994), where they considered temperatures from 90.1K to 114.8K, which are equivalent to those used in the aforementioned contribution. However, they do not do a systematic study where the concentration of each species in the mixture is fixed and the temperature is varied. We believe it is convenient to have a database that can serve as a reference.

In this paper, the surface tension of the binary mixtures O<sub>2</sub>-Ar and N<sub>2</sub>-Ar, as well as the ternary mixture O<sub>2</sub>-N<sub>2</sub>-Ar, was calculated by molecular dynamics simulations using appropriate force fields that reproduce the surface tension of each species as pure fluids. We determined the numerical values of the L-J parameters for argon that reproduce the experimental surface tension as a pure fluid. We assume that it is necessary to have good force fields for pure components. The force field for oxygen and nitrogen was taken from the work of García et al. (2019).

As far as we know, there is no database of such interfacial property covering a wide range of temperatures, in fact, a similar comment was made by Pandey et al. (2004). An immediate extension of the present work is the analysis of the influence of the air in the measurement of the apparent surface tension of aqueous solution of hydrophilic solutes. Docoslis et al. (2000) analyzed the case of sugars, where they showed that the experimentally measured surface tension did not correspond to the cohesion-free energy of the aqueous solution. The rest of this paper is distributed as follows: Section 2 is dedicated to explaining the molecule models and the relevant equations used. Section 3 contains the details of the simulation. Section 4 shows the relevant results and Section 5 presents the final conclusions.

## Relevant equations

The force fields used for nitrogen and oxygen were taken from the work of García et al. (2019), which consists of models of three sites, these interaction sites are along a given axis. For binary molecules, two atoms are attached at a certain separation distance  $L$ , and a virtual site is included between the two atoms.

The experimental quadrupole moment was used as an input data to adjust the partial charges, the relation between both properties is quite simple due to linear symmetry,  $\Theta=Lq/2$ . A partial charge  $+2q$  is located in the virtual site in the center of the molecule and two partial charges,  $-q$ , are located at the ends of the molecule. The L-J and Coulomb potential make up the law of interaction between molecules.

$$u=4\varepsilon \left[ \left( \frac{\sigma}{r} \right)^{12} - \left( \frac{\sigma}{r} \right)^6 \right] + \frac{1}{4\pi\varepsilon_0} \frac{q_i q_j}{r} \quad (1)$$

$\varepsilon$  and  $\sigma$  are the depth of the attraction well and the diameter of the atom, respectively.  $\varepsilon_0$  is the vacuum permittivity,  $r$  is the separation distance between two interaction sites, and  $q_i$  is the partial charge related to the  $i$ -site. The L-J parameters were adjusted to recover the experimental surface tension as a function of the temperature of each fluid. Particularly, the diameter of each atom  $\sigma$  was used to modify the slope of the surface tension curve and the intensity of the attraction well  $\varepsilon$  was used to raise or lower the entire curve. This last procedure was followed to reproduce the experimental surface tension of argon.

The surface tension is calculated using the difference between the components of normal and tangential pressure with respect to the interfacial area found in the  $x - y$  plane as follows:

$$\gamma = \frac{L_z}{2} \left[ \langle P_{zz} \rangle - \frac{1}{2} \langle P_{xx} + P_{yy} \rangle \right] \quad (2)$$

$L_z$  is the longest side of the simulation cell, and the angular brackets denote a set average  $\langle \cdot \cdot \cdot \rangle$  in the thermodynamic property. Equation (2) is divided by 2 because two interfaces are considered, as detailed by Allen and Tildesley (1987).  $P_{ii}$  are the pressure components estimated using virial expression

$$VP_{\alpha\beta} = \sum m_i (v_i)_\alpha (v_i)_\beta + \sum \sum (r_{ij})_\alpha (f_{ij})_\beta \quad (3)$$

where  $v_i$  and  $m_i$  are the velocity of atom  $i$  and the mass of the same atom, respectively.  $\mathbf{r} = \mathbf{r}_i - \mathbf{r}_j$  is the distance vector between two atoms.  $\mathbf{f}_{ij}$  is the interaction force between atoms and  $V$  is the volume occupied by the fluid.

Atom	$\sigma(\text{\AA})$	$\varepsilon(\text{kJ/mol})$	$L(\text{\AA})$	$q_i(e)$
O	3.11	0.4015	1.23	-0.1101
N	3.375	0.29508	1.098	-0.482
Ar	3.484	0.9556		0

**Table 1** Parameters of the potentials of L-J and Coulomb. The charges are presented in units of the electron charge. The oxygen and nitrogen data were taken from the work of García et al. (2019).

## Methodology

Molecular dynamics simulations were performed in a canonical set as explained by Allen and Tildesley (1989).  $N = 8000$  molecules were placed in a cubic crystalline arrangement centered on the faces, which forms a liquid plate that is surrounded by vacuum, forming a parallelepiped cell. The free GROMACS-5.2.1 software was used for all our simulations. Typical dimensions of the simulation box used here were  $L_x = L_y = 5.0\text{nm}$  and  $L_z = 30\text{nm}$  for argon, with  $L_z = 40\text{nm}$  for the binary mixtures and  $L_z = 50\text{nm}$  for the ternary mixture.

The algorithm called *frog jump* was used to predict the trajectories of all molecules. Periodic boundary conditions and minimum image criteria were considered in all three directions. The passage time was 1fs in all our simulations. The Particle mesh Ewald method was used to estimate Coulomb interactions using a 0.2nm grid for reciprocal vectors with a spline of 4, as explained by Essmann et al. (1995). A cut-off distance of  $R_c = 2.5\text{nm}$  was used in real space for Coulomb and the dispersive term of the L-J potential. The LINCS method (LINear Constraint Solver) proposed by Hess et al. (1997) was used to keep diatomic molecule bonds rigid.

To reach equilibrium,  $1 \times 10^5$  steps were performed and  $1.9 \times 10^6$  additional steps were performed to estimate the averages. A similar procedure was followed to simulate the binary mixtures, but  $N = 7000$  molecules were used instead; the concentration of both species varied, keeping the total number of molecules constant. For all cases, the Lorentz-Berthelot combination rules commented by Allen and Tildesley (1987) were used

$$\varepsilon_{ij} = \eta \sqrt{\varepsilon_{ii} \varepsilon_{jj}} \quad (4)$$

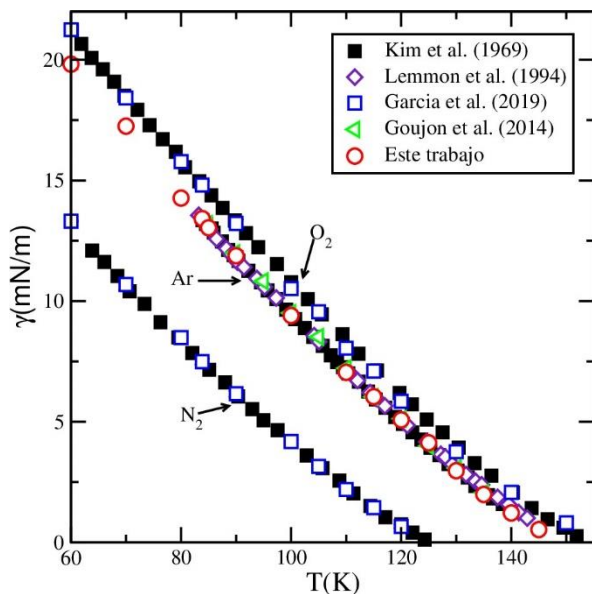
$$\sigma_{ij} = \eta \frac{\sigma_{ii} + \sigma_{jj}}{2} \quad (5)$$



$\varepsilon_{ij}$  and  $\sigma_{ij}$  are the cross terms of the L-J potential and model the interaction of different species.  $\eta$  is an external parameter that allows to modify the cross terms and in the case that said parameter is equal to the unit, the standard combination rules will be used.

## Results

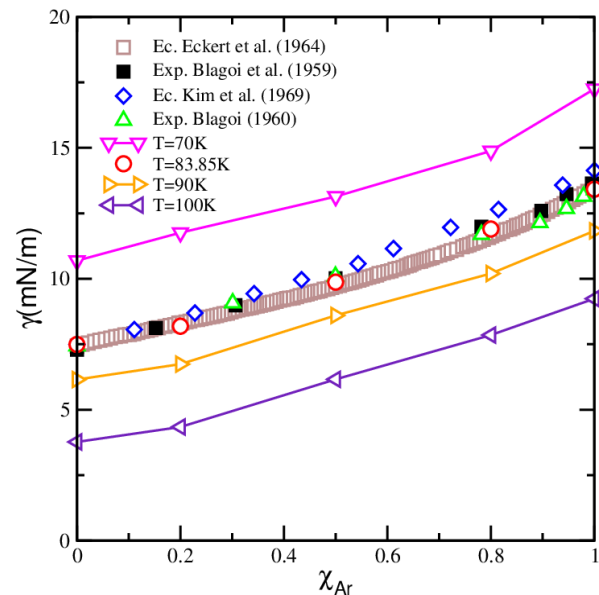
The surface tension of argon as a function of temperature was calculated using the parameters  $\varepsilon=0.9556\text{kJ/mol}$  and  $\sigma=0.3484\text{nm}$  to reproduce the experimental data, see Figure 1. The data of Goujon et al. (2014) were included for comparison, and it can be seen that the agreement is excellent. The surface tension of both pure fluids, oxygen and nitrogen, was also included, these data were taken from the work of García et al. (2019).



**Figure 1** Surface tension as a function of temperature. The symbols are explained in the box in the figure

The parameters of the interaction potential that characterize the nitrogen and argon molecules are contained in Table 1, these data will be used in the molecular simulation of the binary nitrogen-argon mixture. In Figure 2, the data obtained of the surface tension of the mixture already described as a function of the mole fraction of argon in a temperature range of 70K to 100K is shown. In the literature, we found 83.85K temperature experimental data compiled by Kim et al. (1969). These same data coincide with those that were generated by theoretical calculations reported by Eckert and Prausnitz (1964), as well as the theoretical calculations reported by Kim et al. (1969).

Our results are identified with the empty circles at  $T = 83.85\text{K}$  and, as observed, the agreement with the experimental and theoretical data is excellent. We also included predictions of surface tension as a function of the concentration of argon at temperatures 70K, 90K and 100K. Unfortunately, we did not find experimental data with which a comparison could be made.

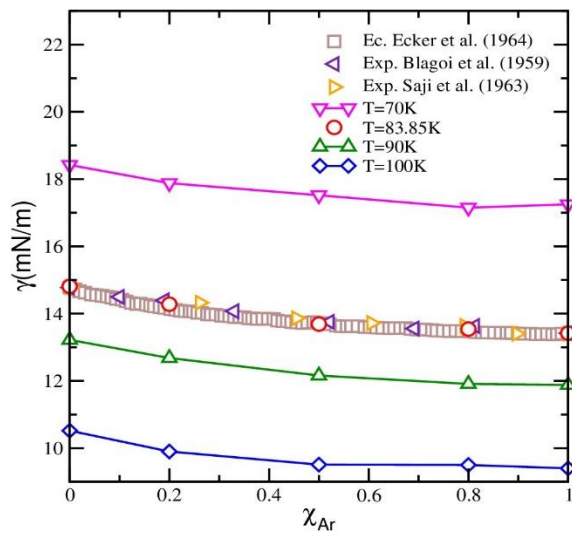


**Figure 2** Surface tension of the binary nitrogen-argon mixture as a function of the concentration of argon, at different temperatures. The experimental and theoretical data were obtained at  $T = 83.85\text{K}$ . The symbols that identify the data are explained in the same figure. The empty circles at  $T = 83.85\text{K}$  are results of this work, as well as the data shown for temperatures 70K, 90K and 100K

Figure 3 shows the surface tension of the binary oxygen-argon mixture in a temperature range [70--100] K. Experimental data are included at  $T = 83.85\text{K}$  reported by Saji and Okuda (1963), and by Blagoi and Rudenkov (1959). Theoretical data are also included in the same figure as those reported by Eckert and Prausnitz (1964). The data generated in this work are shown with empty circles at  $T = 83.85\text{K}$ , and when compared with the aforementioned data, we found good agreement. It is important to note that the cross terms of the combination rules were modified with the intention of reproducing the experimental data, so the values used were  $\eta_\varepsilon = 0.979$  and  $\eta_\sigma = 1.0193$ . This means that the van der Waals type attraction between species is weakened and the size of the interaction between different species grows slightly. This indicates that the oxygen-argon mixture deserves separate attention to analyze its L-J cross-interactions.



The corresponding data generated for temperatures 70K, 90K and 100K have been included in Figure 3. So far, we have not found experimental data with which to compare.



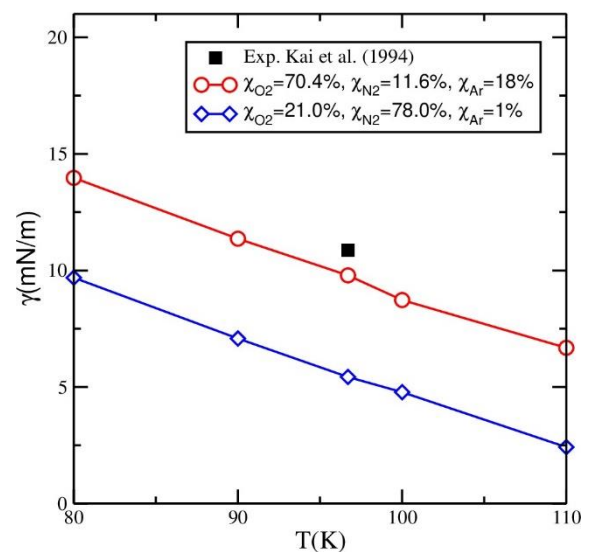
**Figure 3** Surface tension of oxygen-argon at different temperatures. The symbols are identified in the figure. Empty circles are data generated in this work at  $T = 83.85\text{K}$ . The data corresponding to 70K, 90K and 100K temperatures were also generated in this work.

Figure 4 shows the surface tension of the oxygen-nitrogen-argon ternary mixture at different temperatures for various concentrations of each species. As far as we know, there is no systematic study of the dependence of the surface tension of the ternary mixture at the same concentration and at different temperatures. When comparing with data available in the literature, we found data of the surface tension of the experimentally measured ternary mixture,  $10.87\text{mN/m}$ , where they used mole fractions of  $\chi_{\text{O}_2} = 70.4\%$ ,  $\chi_{\text{N}_2} = 11.6\%$  and  $\chi_{\text{Ar}} = 18\%$ , that we will call mixture 1, at  $T = 96.7\text{K}$  and at a vapor pressure of  $2.3\text{atm}$ .

The data obtained in this work for the same interfacial property at the same conditions was  $9.80\text{mN/m}$ , which is 9.8% below the experimental. It is important to mention that the vapor pressure we obtained was  $2.8\text{atm}$ , which is 18% above the experimental measure. This deviation may be due to the force fields used and/or the combination rules. In this calculation we maintained the modification in the cross-interactions of oxygen-argon, that is, we included the external parameters in the Lorentz-Berthelot combination rules to calculate the cross terms  $\eta_{\epsilon} = 0.979$  and  $\eta_{\sigma} = 1.0193$ , because with these modifications we could reproduce the experimental curve of the surface tension of the same binary mixture at  $T = 83.85\text{K}$ .

In the nitrogen-argon and nitrogen-oxygen interactions we used the standard Lorentz-Berthelot combination rules.

Additionally, we include a surface tension curve for another species concentration, different from the previous case,  $\chi_{\text{O}_2} = 21\%$ ,  $\chi_{\text{N}_2} = 78\%$  and  $\chi_{\text{Ar}} = 1\%$ , which we will call mixture 2. It is mentioned in the literature that this concentration of species corresponds to that found in the troposphere, which is the “breathable” layer, Gálvez et al. (1996). Unfortunately, we found no experimental data in the literature with which we could make a comparison. Particularly, we obtained a surface tension of  $5.43\text{mN/m}$  at temperature of  $96.7\text{K}$ . On the other hand, if we compare the surface tension obtained at  $T = 96.7\text{K}$  for mixtures 1 and 2, we find that the data for the second mixture is 50% below the data corresponding to the first mixture. This is because the highest concentration in the first mixture is nitrogen, which has a lower surface tension than oxygen, which is the majority species in the second mixture.



**Figure 4** Surface tension of the oxygen-nitrogen-argon ternary mixture at different temperatures. Concentration of the different species  $\chi_{\text{O}_2} = 70.4\%$ ,  $\chi_{\text{N}_2} = 11.6\%$  and  $\chi_{\text{Ar}} = 18\%$ , the full table is an experimental data and the empty circles are data generated in this work. Empty diamonds are data generated in this work for the concentration  $\chi_{\text{O}_2} = 21\%$ ,  $\chi_{\text{N}_2} = 78\%$  y  $\chi_{\text{Ar}} = 1\%$ .

### Acknowledgments

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## Conclusions

The L-J potential parameters determined allowed us to reproduce the experimental argon surface tension curve.

The surface tension curve is shown as a function of the temperature of the ternary mixture that can be considered as an initial or controlled model of air.

The force fields that reproduce the surface tension of pure fluids provide the opportunity to obtain good results in binary and ternary mixtures. In addition, surface tension curves of binary and ternary mixtures at temperatures where we have not found experimental data were calculated and presented.

Some mixtures are difficult to model because cross interactions introduce inaccuracies. This is the case of the oxygen-argon mixture, where the cross term of both combination rules had to be modified. Even with all the inaccuracies found, computer simulation is a useful tool for generating reference data where there is no existing data.

We believe that a simple air model will allow a good analysis of its influence on the apparent surface tension of aqueous solutions of hydrophilic components.

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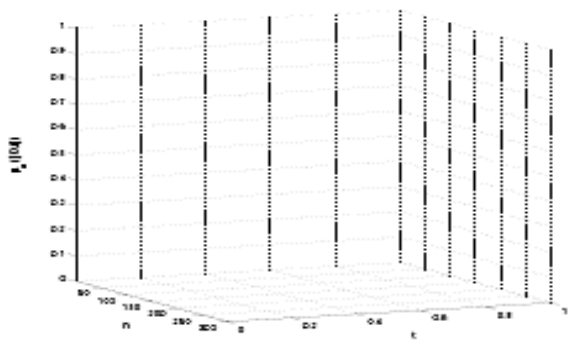
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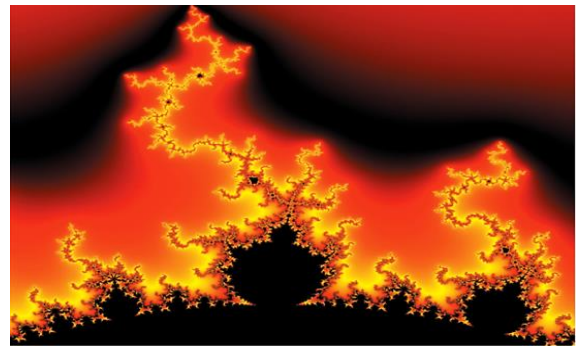
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