

Design and Construction of a Prototype for the Lyophilization Process



**Erick Coronel M., Jessenia López O., Patricia Constante P.,
Cristina Sánchez L., Andres Ortega C., Andrea Tobar, Jines David E.,
and Andrea Lescano**

Abstract This study describes the design, construction and prototype control of a lyophilizer based on parameters of freezing temperature, heating and vacuum pressure, seeking the conservation of organoleptic properties of fruits and vegetables in the region. The prototype is designed with a drying chamber insulated with expanded polyurethane and is hermetically sealed with an approximate capacity of 10 lbm of fresh product distributed in 3 AISI 304 1 mm stainless steel trays. The prototype consists of 3 subsystems such as: the freezing subsystem that works at temperatures below $-35\text{ }^{\circ}\text{C}$, the heating sub-system that adds energy in the form of heat to the food, raising its temperature to $50\text{ }^{\circ}\text{C}$, and the vacuum subsystem in which it generates a pressure of -24 in Hg. Finally, the organoleptic properties of the freeze-dried beet were evaluated by duo trio analytical sensory tests and magnitude estimation, concluding that the color, flavor and smell were not modified; however, the texture of the fruit was altered, acquiring a crunchy composition.

E. Coronel M. · J. López O. (✉) · P. Constante P. · A. Ortega C. · A. Tobar
Universidad de las Fuerzas Armadas ESPE-L, Latacunga, Ecuador
e-mail: jelopez14@espe.edu.ec

E. Coronel M.
e-mail: ecoronel@espe.edu.ec

P. Constante P.
e-mail: pnconstante@espe.edu.ec

A. Ortega C.
e-mail: asortega3@espe.edu.ec

A. Tobar
e-mail: aytovar@espe.edu.ec

C. Sánchez L. · J. David E. · A. Lescano
Instituto Superior Tecnológico Tungurahua, Ambato, Ecuador
e-mail: [cgsanchez3@espe.edu.ec](mailto:cdsanchez3@espe.edu.ec)

J. David E.
e-mail: djines@institutos.gob.ec

A. Lescano
e-mail: alescano@institutos.gob.ec

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1 Introduction

Ecuador, being a tropical country, produces various fruits and vegetables; therefore, a method is sought to preserve its quality for long periods of time; due to its rapid deterioration, it is difficult to store the products. One of the methods used to solve this problem is freezing, but this can damage the organoleptic properties of the food due to the formation of crystals inside the membranes [1].

The use of new technologies is the right way to preserve food and one of them is freeze-drying; for this reason, this research describes the design of a dehydrator prototype with up to 3 trays of 1250 cm² area for fruit minced, which consists of the phases of freezing, primary drying and secondary drying inside a hermetic cabin that is subjected to vacuum in the second and third stages of the process; In addition, the prototype has an automatic work cycle.

2 Prototype Design

Lyophilization is a dehydration process by sublimation [2]. The process consists of freezing the product at very low temperatures, eliminating water to reach humidity levels of less than 5% of its initial content [3]. Initially the process this at environmental conditions, then the temperature is reduced to change the liquid to solid state, later is to freeze the food and when the pressure inside the chamber decreases and is reached to sublimation ice into vapor below the triple point of water; finally, the vacuum be maintained and the temperature of the food raised, which provides it with the energy necessary for the sublimation of the greater water amount [4]. For the design of the prototype, the freezing of the food below its freezing point was considered approximately $-40\text{ }^{\circ}\text{C}$, then the pressure is lowered below the triple point of the water and creates a vacuum with a temperature of -35 to $-40\text{ }^{\circ}\text{C}$; finally, is maintains the generated vacuum and raises the temperature of the product to $60\text{ }^{\circ}\text{C}$ to remove the partially bound water of the product.

2.1 Mechanical Design

For the mechanical design of the prototype a rectangular and simple chamber configuration was selected (see Fig. 1). For respect the sanitary and hygienic standards in the environment, materials must be selected that do not rust and that withstand the external load within the allowed range of deformation, without altering the workability of the machine [5], so AISI 304 1 mm stainless steel was selected, then an

analysis was carried out to verify the safety of the design, as can be seen in Table 1, the values of deformations and stresses that were generated inside the chamber do not exceed the established limits. Once the structure was designed, it was analyzed by CAE with finite element analysis with which the displacements, unitary deformations and stresses in the structure (see Table 1).

The factor of safety for average materials operating in ordinary environments, subjected to loads and stresses that can be determined with relative precision should be 2–2.5 [6]; based on the analysis, the Von Mises stress was obtained and the safety coefficient was calculated at the point of maximum stress of 6.5 indicates great safety in the face of failure.

Fig. 1 Prototype in CAD software

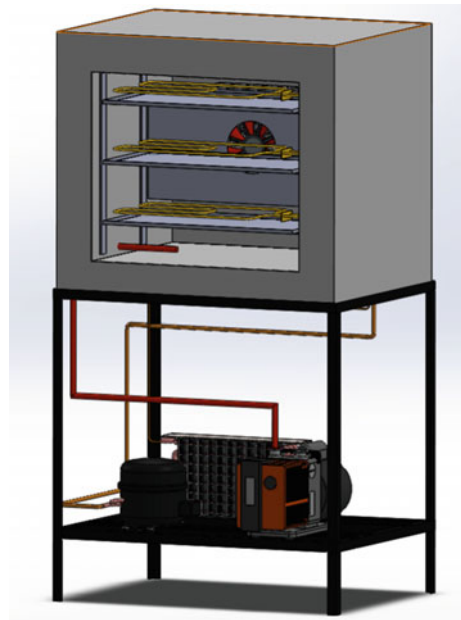


Table 1 Von Mises stresses, displacements and unit strains

Name	Type	Mín	Máx
Stresses1	VON: Von Mises stress	2.883e + 03 N/m ² Node: 14865	6.379e + 07 N/m ² Node: 4957
Displacments1	URES: Resulting Displacements	0.000e + 00 mm Node: 5493	4.755e - 01 mm Node: 5527
Unit strains1	ESTRN: Equivalent Unit strain	5.884e - 07 Item: 2034	6.810e - 04 Item: 3089

2.2 Sizing of the Freezing System

For the calculation of the thermal load of the product, the data of the average latent heat, specific heats and speed factor of “Heat and mass transfer fundamentals and applications” book [7], giving a cooling heat value of 456.327 [Btu], the latent heat of freezing point of 1097.67 [Btu] and the sensible heat below freezing point of 306.56 [Btu]. The cooling of the food inside the lyophilization chamber is carried out by circulating the cold air through a small fan that generates a power consumption of 27.5 [W]. The total sum of all the loads inside the lyophilization chamber is of 0.26 HP, the real value suffers losses due to factors unrelated to the design such as compressor efficiency or leaks, for which we add 30% of safety power, obtaining an approximate value of 0.35 HP. After the freezing process, the pressure must be lowered to -610 Pa in the center of the chamber; for this reason, the volume of the lyophilization chamber was calculated, obtaining a value of 4.65 ft³, for which the QVP-900 pump was selected.

2.3 Control System

The TC-900E electronic controller was selected for the refrigeration system, for controls temperature from -50 to 105 °C. The relay out-put directly controls the compressor up to 1 HP and the defrost output is up to 10 A. Activation of the refrigeration contactor occurs through the control relay output and is connected in series with a programmed timer to add freeze and preheat times. After this time, the chiller stops working for the final stage of production. When the freezing phase is complete to the temperature is reached, the vacuum system and the heating system are activated. The heating system activates or deactivates the resistances based on the readings of the sensors in the food at the desired temperature during the primary drying process. An Arduino Uno microcontroller was chosen to control these two systems, we also have a sensor (DS18B20) in contact with the food as input and a resistor and a vacuum circuit as output. For chamber cooling unit, the refrigeration unit through its TC-900 E controller drives the compressor nominal load amperage of 9.6 A with a voltage of 115 V and the condenser fan of 0.48 A with a voltage of 115 V at 60 Hz. With which the current that the conductor obtaining a value a current of the condenser of 12.48 A and of the fan of 24.48 A.

After sizing the electrical components, a force and control diagram was made in which we can see all the components (Fig. 2).

In the Fig. 3 shows the connection of the two solenoid valves considered, they will operate in parallel with each vacuum pump, the connection of the electrical control is also detailed of the refrigeration unit in series with a timer which allows us to deactivate the refrigeration system. The control scheme also includes a two-position ignition selector, the display shows the temperature of the chamber and the time that

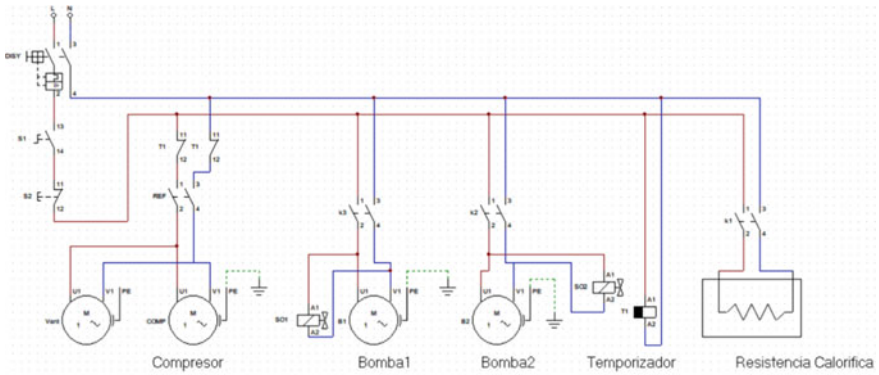


Fig. 2 Power circuit diagram

has elapsed in the process, an emergency stop button, the controller also activates resistance contactors, switching relays for pump 1 and 2.

For the power system in Fig. 4, the outputs of the Arduino Uno microcontroller are observed, they activate the coils with high or low pulses at 5 V dc of the relays, which allow the passage of the phase line at 110 V towards coil A1 of the contactors on the power side.

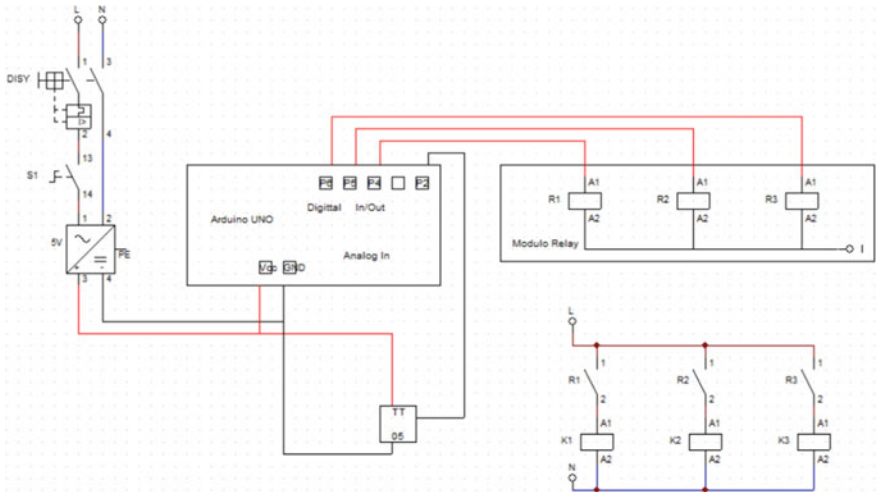


Fig. 3 Control circuit diagram

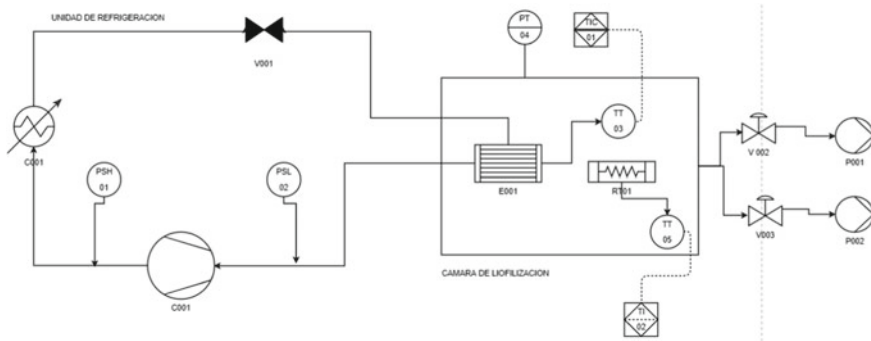


Fig. 4 P&ID piping and instrumentation diagram

3 Testing and Analysis of Results

3.1 Testing the System as a Whole

After calibrating the sensors and checking the operation of each subsystem, the prototype was tested in vacuum to verify the correct operation of the code implemented in the microcontroller, a screen is displayed to indicate whether the processes it is in freezing, pre-drying or secondary drying. The temperature measurement was recorded in an Excel file through the serial port of the controller and the vacuum level as a function of time through the analog reading of the vacuum gauge, the data obtained were plotted and it was verified that they comply with the curve of lyophilization. In the Fig. 5, the curve of the temperature of the food during the lyophilization process can be observed, the freezing reaches $-35\text{ }^{\circ}\text{C}$ (blue) and passes to the primary desiccation or sublimation in which heat is added and its temperature is maintained between -20 and $-25\text{ }^{\circ}\text{C}$ (red), in the secondary desiccation the refrigeration system is turned off and the temperature is raised between 45 and $50\text{ }^{\circ}\text{C}$ (green) (Fig. 5).

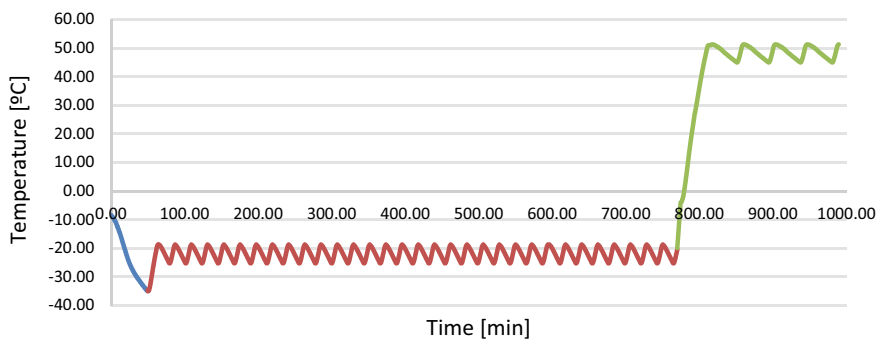


Fig. 5 Temperature versus real time of the process

3.2 Organoleptic Tests

Sensory tests were carried out with 10 judges to verify the conservation of organoleptic properties of freeze-dried beets (color, smell, flavor and texture), each judge was assigned a reference sample of fresh fruit and a freeze-dried fruit [8]. Once all the tests were finished, the ANOVA test was tabulated.

According to the results of Table 2, 3 and 4 we can observe that the critical value for F is greater than the value of F calculated, for which the null hypothesis (H_0) is not rejected, another factor to analyze is the value of P (probability), which confirms the hypothesis that the freeze-dried sample is the same as the fresh sample in terms of taste, smell and color; however, in Table 5 we can see that the critical value for F is less than the value of F calculated, for which the null hypothesis (H_0) is rejected. Another factor to analyze is the value of P (probability); therefore, the lyophilized sample is not the same as the fresh sample in terms of texture, this is due to process.

Table 2 Analysis of variance of flavor

Origin of variations	Average of the squares	F	Probability	Critical value for F
Between groups	0.002631281	0.2397	0.7884	2.5106
Within groups	0.010973524			
Total				

Table 3 Analysis of variance of odor

Origin of variations	Average of the squares	F	Probability	Critical value for F
Between groups	0.004581604	0.6355	0.53737	3.3541
Within groups	0.0072086			
Total				

Table 4 Analysis of variance of color

Origin of variations	Average of the squares	F	Probability	Critical value for F
Between groups	0.00951	3.36721	0.04947	4.004
Within groups	0.00282			
Total				

Table 5 Analysis of variance of texture

Origin of variations	Average of the squares	F	Probability	Critical value for F
Between groups	0.0660	9.42475	0.000785	3.354130
Within groups	0.0070			
Total				

4 Conclusions

The modeling of the prototype was carried out in a CAD software to establish the dimensions chamber of 60 cm wide, 50 cm high, 50 cm of deep and 3 trays with capacity of 10 lbm of fresh product. The design analysis using CAE verify that the prototype is safe; therefore, a dehydration system was designed of lyophilization, with three stages: freezing to $-35\text{ }^{\circ}\text{C}$ and atmospheric pressure, primary desiccation at $-10\text{ }^{\circ}\text{C}$ and a vacuum pressure of less than 610 Pa must be maintained and secondary desiccation to $60\text{ }^{\circ}\text{C}$ at a vacuum pressure of less than 610 Pa.

The electrical system was dimensioned, a Full Gauge TC-900E temperature controller was used, an Arduino Uno microcontroller was used for the vacuum and heating subsystems according to the programming for each of the freeze-drying stages. On the other hand, the result of the lyophilization process was evaluated by duo-trio analytical sensory tests and by estimation of magnitude with 10 judges and with three samples to evaluate one of fresh product and two of lyophilized product, in the beet the flavor, smell and color while the texture did change.

5 Future Works

For future projects, it is recommended to implement a steam trap, also called a condensation chamber, which prevents damage to the vacuum pump and prevents the product from being rehydrated by the circulation of steam and condensation inside the work chamber. In addition, the implementation of an HMI (Human Machine Interface) for the management of recipes and easy change of parameters should be taken as a new research work for the improvement of the prototype.

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