NIGERIAN INSTITUTION OF AGRICULTURAL ENGINEERS (NIAE)

A DIVISION OF THE NIGERIAN SOCIETY OF ENGINEERS

PROCEEDINGS OF THE

18TH INTERNATIONAL CONFERENCE

AND

38TH ANNUAL GENERAL MEETING

UMUDIKE 2017

THEME:

DYNAMICS OF AGRICULTURAL ENGINEERING FOR FOOD AND AGRO-INDUSTRIAL RAW MATERIALS PRODUCTION FOR ECONOMIC RECOVERY IN NIGERIA

TUESDAY 3RD- FRIDAY 6TH OCTOBER, 2017

VENUE:

Senator Anyim Pius Auditorium,
Michael Okpara University Of Agriculture, Umudike

EDITED BY:

ENGR. PROF. I.E. AHANEKU
ENGR. DR. M. C. NDUKWU
ENGR. O.O. ONU
ENGR. G. INEKWE

Design and Construction of a Lyophilizer System Using Locally Available Material

[1]Bello, Ruqayyah. O., [2]Raji A. O. and [3]Oyefeso B. O.

[1]Department of Agricultural and Bio-Environmental Engineering School of Engineering, Yaba College of Technology, Lagos

[2]Department of Agricultural and Environmental Engineering Faculty of Technology, University of Ibadan, Ibadan

[3]Department of Post Harvest Engineering Oyo State College of Agriculture and Technology, Igboora

E-mail: robello59@yahoo.com[1], abdulganiyr@mail.ui.edu.ng[2], oyefesobabatunde@gmail.com[3]

Phone: 08034773704, 08035850005, 08025950339

Abstract: *A lyophilizer system was designed and constructed with a galvanized metal sheet to reduce the cost and to make it available to local and poor food processors. It consists of 330.2 mm × 508 mm × 457.2 mm rectangular shaped chamber with the inner and outer layer reinforced with a hollow mild steel square pipe of 1.7 × 1.7 mm² to prevent implosion of the lyophilizer during dehydration phase. The system was designed based on the specific heat capacity of Pears, Tomatoes and Carrot with values of 3.62 kJ/kg°C, 3.98 kJ/kg°C and 3.79 kJ/kg°C respectively. The results showed that dry-bulb temperature of 28°C at initial stage and 1°C after drying, while the dew point temperature of 20.8°C at initial stage and -6.1°C after drying were recorded. The result obtained also shows that the temperatures (dry-bulb and dew point) and relative humidity of the lyophilized product is lower as compared to the imported ones. A total of 6hours was used for freeze drying with 1/10 reduction in weight of the lyophililized product when weighed.*

Keywords: Lyophilizer, Preservation, Spoilage, Dehydration, Cooling, Post-harvest

1. Introduction

In Nigeria, agricultural sector constitutes the bulk of the informal economy apart from petrol. In fact, FAO (1995) reported that fruits and vegetables remain the core activities that cannot be left out in agricultural sector because they constitute the essential parts of balanced diet in human nutrition. Meanwhile, factors such as weak infrastructure, poor transport system, lack of storage facilities and the perishable nature of crops make farmers not to get enough value for their product (Ndukwu, 2011). This makes the loss to be enormous especially following the period after harvest from the farm, which results in spoilage of produce and hence lead to the product been fed to animals or dumped as refuse due to lack of suitable preservative system,. . In addition, due to the nature of some fruits and vegetables, such as tomatoes, banana that require cold storage, there is need for constant running of the freezer and or cold storage systems like refridgerator so as to keep the product fresh. In view of this, storage method of fresh fruits and vegetables needs special attention, although the storage condition varies depending on the type, variety or species and pre-harvest conditions of the products. The core factors that cannot be left out in the determination of storage life and quality of fruits and vegetables include respiration and transpiration.

However, apart from the cold storage system, other methods such as drying and dehydration of various forms are also common in food preservation techniques. Although, the latter seems to lasts longer in terms of shelf-life, but there is alteration in the composition of product due to its exposure to high temperature. On the other hand, cold storage is disadvantageous in that there is need for constant running of the refridgerator which is usually very expensive due to the cost of fuelling. The challenge of unavailability and expensive storage system leads to decrease in value of products thereby leading to alteration in physical properties, such as, changes in colour, reduction in weight, which in turn reduce the market value and result to the product being sold at lower price. (Shewfelt, 1994 and Olusunde et al. 2009). Lyophilization is a method to keep the food safe by slowing down the movement of molecules that causes spoilage and food borne illness causing it temporary in-active, thereby extending the shelf-life of food product for longer period. This method basically involves combination of both freezing and dehydration. The method is advantageous in that, after freeze drying, does not require any further, constant or stable power source to keep it safe for the period of storage. Rather, the lyophilized product is packaged in a moisture-proof material to prevent any form of absorption or release of moisture from its immediate environment.

Different designs of Freeze dryer have been reported in literature for food preservation. The design ranges from simple bench-top to sophisticated design. Freeze dryer, also known as a lyophilizer is a means of preserving products from spoilage to maintain and retain its nutritional values without changing its properties especially, colour, taste, texture among others. The storage span of this method is longer than other preservative method (as much as 3 to 5 times) NCHFP (2017). The principle of operation of this system includes cooling of product to the temperature of well below 0° C and dehydration of

the product by reducing the pressure within the chamber (Xiaolin and Michael, 2004). Usually, fruits and vegetables are the commonly used products that are widely processed with this type of method. This may be due to their susceptibility to spoilage. Meanwhile, lyophilization method is by far used beyond processing agricultural products. Other product, such as pharmaceutical products are also widely processed and preserved with this method (Xiaolin and Michael, 2004). This method might have been used for the preservation of these products due to their sensitive nature, which if any other method is used can disrupt their useful properties (chemical composition), making such product insignificant and render it useless due to its unacceptability from the market. Significance of freeze drying cannot be underestimated. Among the benefits are that the product is presented in a form that can easily and conveniently be packaged for market acceptability, reduces cost of transportation, product value addition which could open opportunity for foreign exchange earnings, its properties when reconstituted resembles fresh products as in taste, texture, colour, among others, and hence prolong the shelf life of the product more than other preservative method.
For a successful operation, lyophilization involves four main steps. The first method is the pretreatment of the product to preserve the product appearance, followed by freezing, then primary drying and lastly the secondary drying. Meanwhile, to achieve all this aforementioned, components such as refridgeration system, vacuum pump, control system and product chamber or manifold are of importance.

The study was based on the design of the lyophilizer using locally available material. This is due to the fact that, the existing imported ones are too expensive and at the same time, are out-of-reach for poor processors to afford. This system is so essential that it preserves many of the nutritional requirements by the food industry to meet the standard as stated by the World Health Organization. The lyophilizer system fabricated with galvanized metal steel will help to relief in product handling such system will be at reach, not sophisticated and affordable to local food processors. Such practice(s) will help in converting fresh farm produce into storable product in order to extend their shelf-life, add value to product for both local markets and exports, thereby reducing wastage to minimal level and hence ensuring food security in the country.

2. Materials and Methods

2.1 Principle of Lyophilization System

The principle of lyophilization involves both cooling and dehydration process. The product to be used is first frozen in the chamber under a very low temperature, followed by subjecting it to sublimation process through vacuuming (known as primary drying) and application of slight heat to remove the remaining bound water (secondary drying). The vapor pressure of the product to be dried should be below that within the chamber environment for the product to get dried. Also, freezing the product to temperature as low as below 0°C before dehydration helps to maintain the primary structure and shape of dehydrated product.

2.2 Design Consideration

The following factors were taken into consideration in the design:

i. The construction material for the chamber is a corrosive resistance material, locally made to reduce the cost.
ii. R134A refrigerant was used which is safe for human environment, economic, reliable and simple
iii. The sample product used for the freeze-drying process is ripe and safe from bruise

2.2.1 Design Calculation

Assumptions made in the design of the freeze dryer are as follow:

a. A one-dimensional heat transfer and mass flow which is normal to the interface and surface of the product was considered
b. The thickness of the interface was taken to be infinitesimal.
c. The freeze-dried products were of the same size and shape. i.e. homogeneous
d. The frozen region was considered to be homogeneous, i.e. of uniform thermal conductivity, density, and specific heat, and to contain a negligible proportion of dissolved gases.
e. Heat transfer is at constant temperature

2.3 Description of the Lyophilization system

The lyophilizer) designed in this study is of 15kg capacity, suitable to freeze dry fresh fruits and vegetables. The chamber consists of four evaporator trays on top of which the product trays were placed (plat 2). 7719.2 mm length of evaporator coil was used for the four trays with an expansion valve of length 2000 mm to control the flow of refrigerant and a compressor of 110 W capacity. The condenser used was 210 in long (53.4 mm) covered with its fins. The vacuum pump was operated by a 2 Hp electric motor.
The basic principle relies on firstly subjecting the product in a frozen state to temperature as low as below 0°C, after which it will be dehydrated through sublimation process by subjecting it to vacuuming

with a pressure gauge fixed to determine the pressure reduction in the chamber. The vacuum pump was powered by an electric motor, and heat was slightly applied at the commencement of the drying phase to energize the dehydration process. Plate 1 shows the side view of the lyophilizer developed.

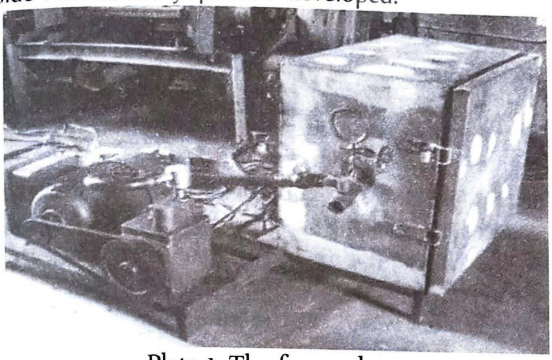

Plate 1: The freeze dryer

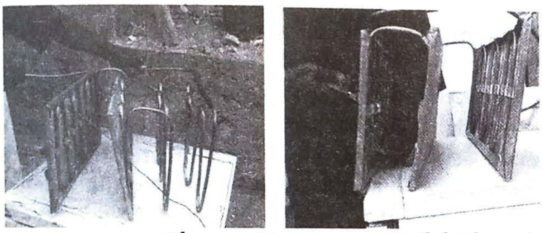

Plate 2: Evaporator Coil laid under its tray with 9inches spacing

2.3.1 Design procedure and the system development

Basically, the lyophilizer is made up of three main units, thus, the Cooling Unit, Dehydrating Unit and Heating Unit

The chamber design

The calculation to show the range of cooling efficiency of two sample fruits with higher and lower specific gravity to know the size of compressor suitable for this design is as calculated as follows

Rate of intake:

For temperature of product from harvest at 27°C, specific heat of 0.9*kcal/kg/°C* required to completely freeze-dry in 8hrs (working day period) at a temperature of -20°C. In freeze drying process, freezing temperature is within a temperature of -5 °C to -45°C.

$$Rate\ of\ energy\ intake = \frac{M \times T \times C \times 2}{t} \qquad 1$$

M is the capacity of the chamber (15*kg*)

T is the temperature of product from harvest (27°C)

C is the specific heat capacity of the product (0.9 *kcal/kg/°C*)

The factor 2, is a factor of safety.

Therefore the Rate of energy in take = 91.125 *kcal/hrs*

Note that: 4.186kJ = 1 *kcal*

This now implies that, 381.449 *kJ*/hrs= 105.9719J/s = 105.9719 *Watt*. Due to unavailability of 105watt compressor in the market, a 110 W rating was used.

Infiltration of External Air:

The ambient temperature is usually between 21°C to 37°C. Now, using an average temperature of 32°C to be the assumed atmospheric temperature, relative humidity of 70%, and vapor pressure of -33.76*mbars*, being the vapour pressure of the environment to calculate for product's property from harvest (i.e. external air). This will give the idea of what the physical properties of the surrounding environment where the lyophilization process will take. Such property when checked in the psychrometric chart will give insight to more properties, such as enthalpy. Now, from psychrometric chart, the enthalpy (ΔH) in cooling the product to temperature of 32°C gave enthalpy value H, to be 86.5 kJ/kg = 20.7 *kcal*.

Product Cooling:

The total amount of sensible and latent heat to be removed in cooling a product is given by:

$$H = M\,[(Ca\ \Delta Ta) + hl + (Cb\ \Delta Tb)] \qquad 2$$

H - Total quantity of heat, M - Mass of product (*kg*) = 15 *kg*, C_a - Specific heat capacity above freezing point (for apple fruit)= 3.65kJ/kg°C , C_b - Specific heat capacity below freezing point (for apple fruit)= 1.89 kJ/kg°C, h_l - Latent heat of freezing = 280 kJ/kg , ΔT_a - Temperature decrease above freezing point= 32°C, ΔT_b - Temperature decrease below freezing point= Let x represent this temperature.

Now, computing the values in the equation, we have:

H = 15[(3.6532) + 280 + (1.89 x)] = 1752 + 27xkCal.

Table 1 presents the combined heat and the parameters used in calculating the heat for different types of products.

Table 1: Required energy values for products to be dry in the lyophilizer

Product	Amount of energy required (KJ)
Apple	1752+27x
Celery	1915.2
Diary	1800
Meat, Bacon	1680+16.05x

Where 'x' is the temperature to be determined in the lyophilizer

From Table 1 , product with highest amount of energy will be required by the evaporator to cool the product and this will be determined from the result of the experiment.

$$C_p = \frac{Q}{M\Delta T} \qquad 3$$

C_p is the specific heat (kJ/kg°C), Q is the heat gained or lost from/to the environment (kJ), M is the mass of the product (kg), ΔT is the temperature difference between the ambient environment and the chamber (°C). Specific heat is an essential part of the thermal analysis of food processing or the equipment used in heating or cooling product. It is a function of the moisture content, temperature and pressure of the product being dried. Mykhailyk (2013) gave specific heat capacity of product as:

$$Cp = 0.837 + 3.349Xw \qquad 4$$

Where: X_w is the water content of product (in fraction), X_f is the fat content of product (in fraction) X_s is the solid content of product (in fraction)

From Table 1, water content (X_w) for fruits is between 87–95%. Now substituting the value of X_w = 0.95 into equation 4 . We have: = 0.837+3.349X_w. Note that the highest water content (95%= 0.95) will be used for this design, so that if product with lesser water content is to be used in the future, the design will be able to perform the expected function compared to when product of lower water content is chosen for the design. Therefore, = 0.837+3.349 (0.95) = 0.837+ 3.182 = 4.02 kJ/kg°C.

This implies that, the material of construction for the design should be of good thermal conductivity and that 4.01855kJ of energy will be required to remove every 1 kg mass of iced product by the condenser. Heat gained or lost, Q = mΔT = 4.01855 kJ/kg°C b,, m = 15kg, ΔT = 32-(x) = (32-x)°C

For Celery: Heat gained, Q = 1915.2, x = 0.227°C

For Diary: Q = 60.279, x = 2.1385 °C

For Meat, Bacon; Q = 60.279, x = = 3.26093 °C

This represents the heat gained or released to the environment by the evaporator

Where 'x' is as described above.

2.4 Experimental Methods

The research was conducted on three different fruits which were free from bruise, thoroughly washed with water to make them clean before making it into pulp. The first sample was peeled, seed was removed in the second sample and they were all grinded separately in a home type blender. The samples were identified based on their colour. Stainless steel material was used for the loading of the sample into the chamber, to avoid corrosion and contamination of the sample. Three uniform containers were used to prepare equal samples for each of the fruits. The samples are as shown in Plates 3a-c respectively.

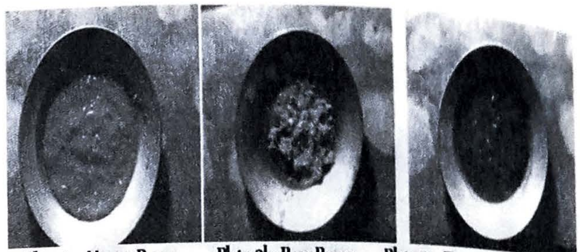

Plate 3a: Mango Puree Plate 3b: Pear Puree Plate 3c: Tomato Puree

The physical and mechanical characteristics measured include the weight of the sample, before and after freeze drying and the moisture content of the sample respectively.

The individual weight of the samples was measured using an electronic precision balance whose reading is to one decimal point (TL-5000 model, Japan). The initial moisture content of the samples was determined by oven method (ASAE, 1986) before the freeze drying. The sample was weighed before the commencement of the experiment and recorded as "wet weight of the sample", dried to a constant weight at a temperature of not greater than 103°C weighed and recorded as "dry weight of the sample".

2.5 Performance Evaluation of the Lyophilizer

Test were carried out to ascertain the performance of the freeze dryer. The evaluation was based on some properties such as dry bulb temperature, dew point temperature and relative humidity of the environment with in the chamber used for the experiment to ascertain the efficiency of the lyophilizer and the dryness of the samples used for the assessment. This was determined by the use of a data recorder which was placed on one of the evaporator trays inside the chamber. The temperature measurement ranges between -35°C to 80 °C and relative humidity of 0 to 100%.

3. Results and Discussion

3.1 Behavioral Characteristics of the Sample

The performance of the lyophilizer system loaded with three different samples was assessed at intervals of 5 minutes from 8:00am and 4:00pm. Within this period of evaluation, the paramater used for the assessement of the lyophilizer system include the dry bulb temperature, dew point temperature and relative humidity with in the chamber. The dry bulb temperature at the commencement of cooling was 28°C before loading of the samples. This was done to stabilize the condition in the lyophilizer chamber with that of the sample product. This temperature gradually increases and then reduces as cooling progresses, the cabinet experienced drop in temperature and thereafter maintain a temperature of about 2°C for the remaining cooling (freezing) period. Meanwhile, the product when assessed physically seems to be frozen.

On the other hand, dehydration phase commenced immediatelly after the product sample had reached the frozen state, and this was done with the use of a vacuum pump to reduce the surrounding pressure within the chamber. Dehydration process commenced with temperature in the chamber gradually increasing as time goes on, the chamber experienced drop in temperature and later increases. However, it was observed that there was drop in temperature and thereafter, it increases from 7.5 °C to 30.5 °C when heat was introduced. The result indicated that sublimation process actually took place with a pressure recorded as 100mmHg. Consequently, the relative humidity of 75% was recorded in the system at the commencement of the cooling while that of dew point temperature was 20 °C. Gradually, the relative humidity drops from 80% to 58% and later increased to 63.5% while on the other hand, the dew point temperature reduces from 23.3 °C to 10.7 °C and later increases to 13.4 °C then reduces to -3.7 °C. The increase in value of relative humidity might be due to the dehydration phase that was carried out, this indicates that, the vapour pressure within the chamber tends to be below that of the product that is being dry. This corresponds to the theory of Lide (1999) which stated that, the rate of drying is dependent on the extent to which the pressure in the chamber is below the vapour pressure of ice. It was therefore inferred from the result shown in figures 1 - 4 that, dew point temperature of 20.8 °C was recorded at the commencement of cooling which gradually drop from 13.4 to -3.7 °C as cooling continues tending towards the frozen state. Thereafter, dew point temperature increases to 8 °C and later drop to 1 °C. During the dehydration phase, it was observed that the dew point temperature increases from -3.7 °C to 4 °C, this gradually decreases as dehydration time extends at value of 1 °C and then increases to 4.6 °C before decreasing to 1.8 °C. However, there was instability in the values of the product at this stage, and this may be due to checking of the state of dryness of the product during the lyophilization process. Constant values of 4.7 °C, 4.3 °C, 4.4 °C, 3.9 °C, 4.0 was recorded before the values gradually increases to 28.1 °C, the constant values may be that the product is tending towards dryness, while the increase in the value till the end of the process may be due to application of heat to remove the remaining bound water from the product. The conditions within the dryer for the variation of dry bulb Temperature, dew point temperature and relative humidity for the freeze drying process as shown graphically in Figure 1. Temperature and dew point temperature are given in degree centigrade (°C), while the relative humidity was given in percentage (%).

3.1.1. Dry Bulb Temperature

The result shown in Figure 1 indicated that the the dry bulb temperature at the commencement of cooling was 28°C and gradually decreases and tend towards zero as cooling progresses with the product physically observed to be frozen. This means the product attained a frozen state between 2°C and 0°C. Whereas, this is different from what (Rao *et al.,* 2005) stated, that frozen state of product other than water is below 0°C since it contain percentages of other solid content while that of water is 0°C since water is a pure substance. Therefore, the result shows that, the freeze dried sample attained a frozen state at below 0°C. Dehydration process commenced immediately after the cooling stage of the product with the use of vacuum pump to reduce the air pressure and water vapor within the chamber environment for sublimation to take place. This act of pressure reduction makes the vapor pressure in the system to be below that of the product in the chamber, which makes drying to take place. , since there is exchange of heat between the system and the product. This shows from the result that, the temperature within the system increases from 0°C to 9.5 °C when slight heating was introduce to trigger drying at the first 30 minutes of vacuuming. However, about 30minutes of the first vacuuming, the temperature dropped from 9.5 °C to 8 °C and later rise to 11.5 °C. This continued till the end of the dehydration when a temperature of 30.5 °C was finally recorded. This indicated that, sublimation process actually took place with a pressure of 100 mmHg recorded.

3.1.2. Relative Humidity

The relative humidity within the system at the commencement of freeze drying was obtained as 79% (Figure 2) and this progressively increased towards 100% and a final value of below 87% was recorded at the end of the process. The increment in value might be due to the cooling (frozen state) which the product had attained, while the fall in value was due to dehydration process. This indicates that, the vapor pressure within the chamber tends to be below that of the product that is being dry. Meanwhile, this corresponds to the theory of Lide (1999), which stated that, the rate of drying is dependent on the extent to which the pressure in the chamber is below the vapor pressure of ice.

3.1.3 Dew Point Temperature

The result for dew point temperature is as presented in figure 3. It can be inferred from the result that, 20.8°C temperature was recorded at the commencement of cooling. This temperature gradually decreases as cooling progresses, when the product was attaining frozen state. A value of -6.5 °C was finally recorded at the end of cooling stage (i.e. after about 2hrs). As dehydration process progresses, it was observed that the dew point temperature gradually increases from -6.1 °C to 28.1 °C. This shows that, there was condensation of the product during dehydration.

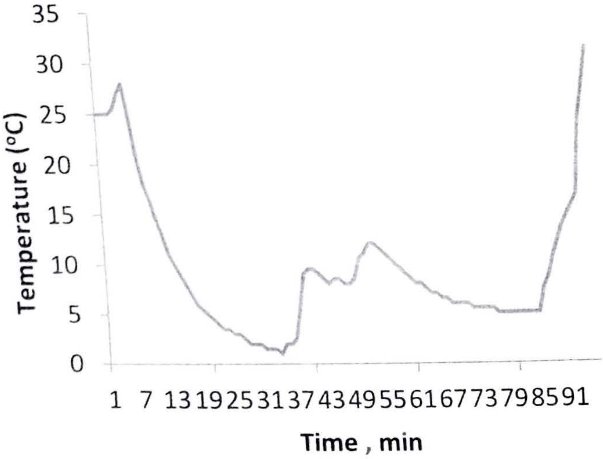

Fig 1: Periodic variation of Temperature (°C) for tomato puree

4.0 Conclusions

A lyophilizer system was developed with local available materials. It was able to cool the product to frozen state at a temperature of 2°C. Other properties recorded was the dew point temperature of 1 °C at the end of cooling stage while 28.1 °C was recorded at the end of dehydration stages. Thus, the lyophilize product after undergone the process of lyophilization does not give required end product as stated in literatures. This may be due certainly to the some deficiency that was observed from the system during the assessment. Part of it was the leaking

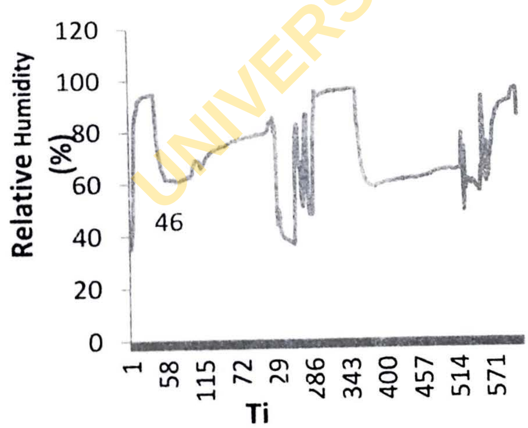

Fig 2: Periodic variation of humidity (%RH) for tomato puree

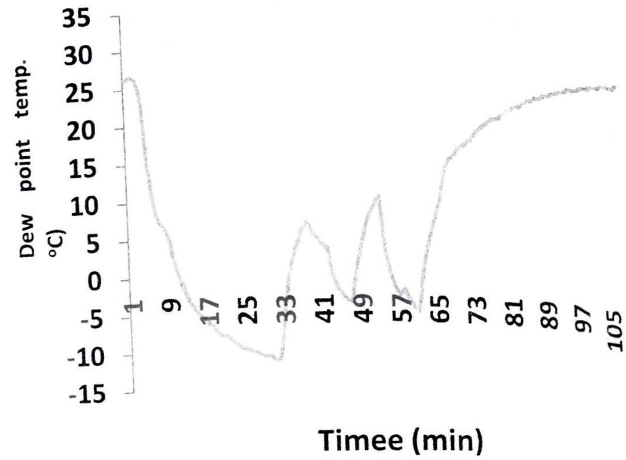

Fig 3: Periodic variation of Dew Point Temperature (°C) for tomato puree

of the treaded path where the system was welded. A total of 6hrs was used to freeze dry the product sample that was used to test-run the lyophilizer. The freeze dryer that was designed and fabricated proved to be easy to operate and maintain. The cost of ₦21, 910 used in developing the freeze-dryer indicates that freeze dryer should be affordable to small-scale food processors and for house hold use.

References

Beals J. M., M. J. Edwards, M. J. Pikal, and J. V. Rinella, Jr. (1997). Formulations of obesity protein, *Eur. Pat. Appl.* (Eli Lilly and Co., USA). EP, pp. 48

FAO. 1995. Small-scale Post-harvest Handling Practices -A Manual for Horticulture Crops. Food and Agriculture Organisation of the United Nations (FAO), Rome. 3rd edition. No. 8. P.80

Ndukwu, Macmanus Chinenye (2011). Development of Clay Evaporative Cooler for Fruits and Vegetables Preservation. Agric Eng Int: CIGR Journal, 13(1): CIGR MS No.1781

Mykhailyk, V (2003). Specific heat capacity of apple at different moisture content. *Journal of Food Engineering* 25(1):1-19

Olosunde William Adebisi, J.C. Igbeka and Taiwo Iufemi Olurin (2009). Performance Evaluation of absorbent materials in Evaporative Cooling System for the Storage of Fruits and Vegetables. *International Journal of Food Engineering* 5:33

Pikal, M. J. (1990). Freeze-drying of proteins. Part I: process design. *BioPharm* **3**: 18 - 28.

Rao M.A., Syed S.H. and Rizvi Ashim K. Datta (2005): Engineering Properties of Foods. Third edition. 153-155.

Shewfelt, R. 1994. Quality characteristics of fruits and vegetables.

Xiaolin Charlie Tang and Michael J. Pikal (2004). Design of Freeze-Drying Processes for Pharmaceuticals: Practical Advice. *Pharmaceutical Research 21:2*