EVALUATION OF FREEZE-DRYING CHARACTERISTICS OF PLANTAIN AND CARROT SLICES

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ABSTRACT
A study of the freeze-drying characteristics of, plantain and carrot chips was conducted. The process conditions were temperature of 32.1$\pm$ 0.98 *^oC and vacuum pressure of 67$\pm$2 cm-Hg at primary level and temperature of 55 ^oC for the secondary. The slice thicknesses were 0.5725 cm, and 0.2125 cm for the two crops. Logarithmic and Newton's thin layer drying models showed good predicting capabilities with high goodness of fit at 95% confidence interval. The drying constants were, 0.45 hr^{-1} and 0.33 hr^{-1} for, plantain and carrot respectively while moisture diffusion coefficients were,* 1.0643×10^{-06} *cm^2/hr* 9.2803×10^{-08} *cm^2/hr for, plantain and carrot respectively. It was also observed that the drying rate of the crops fell in the range of, -0.4 g_{water}/hr to 0.0 g_{water}/hr. Key words: Freeze-drying, Fruits, Plantain, Carrot, Drying Kinetics, modeling.*

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1.0 Introduction
Water loss and decay account for most of food losses, which are estimated to be more than 30% in the developing countries when added to inadequate handling, transportation and storage (Jayaraman and Gupta, 2006; kaya *et al.,* 2007). These losses cause serious gaps in the availability of the essential nutrients, vitamins and minerals which they supply to human diet. Large proportion of root crops in Nigeria is used for human consumption, therefore preservation of quality and wholesomeness of these crops are essential from the time the crop is harvested to when it is processed, stored and marketed.
Because of these considerations, drying of foods has been a major part of food industry for many years. One of the major ways of preserving wholesomeness in foods is by controlling the moisture content, which makes it unfavorable for micro organisms and enzymes that are responsible for spoilage of biomaterials.
The past few decades have witnessed some exponential rise in research and development in inter and multidisciplinary field of drying (Mujumdar, 2000). The focus of these researches are, freshly harvested crops which have relatively high moisture content that has to be reduced to a desirable storage moisture content level for safely stored. The quality changes such as physical alterations (size, color and shape) and changes in chemical and biochemical properties of the food and biomaterials may occur during drying. They may manifest in shrinkage, porosity changes, decreased ability to bond water and may damage the microscopic structures of the materials and consequently have effect on overall characteristics (Chrife and Buera,1995; Stapelfeldt *et al.,* 1997; Witrowa-Rajchert and lewicki, 2006). For instance, Moreira *et al.* (2008) showed that carrot slices dried over longer periods in conventional dryers demonstrated lower rehydration ratios. In their study, Sumnu et al (2005) reported that high internal pressure produced by microwave drying can cause the structure of material to expand and puff, resulting in a decrease in the density of the structure. They observed that the less dense structure has a higher capacity to absorb water and reconstitute.
Freeze drying as advancement in dehydration process involves the crystallization of the solvent or suspension at a low temperature and thereafter sublimating same to vapor phase and finally getting rid of the vapor. It has become one of the most important process for the

preservation of heat–sensitive biological materials (Geogre & Datta, 2002; Dincer, 2003; Liu *et al,* 2008). Some of the reasons for the popularity of freeze drying include sample stability at room temperature, easy reconstitution with water, retention of the product structure and surface (Dincer, 2003). The freeze-dried products are said to be much more appealing in sensory qualities than that of hot air drying (Pan *et al.* 2008). Alejanda *et al,* (2015) observed that freeze dried products kept their natural shapes, although might sometimes become fragile and develop noticeable yellowness in the final product, which was attributed to increase in concentration of nutrients. Litvin *et al.* (1998) dried Carrot slices by combining freeze drying with a short microwave treatment and used total drying time and quality parameters such as color, dimensions of slices and rehydration ratio as process parameters. The findings showed that freeze drying for 2 h at a plate temperature of 30 °C followed by 1.5 h at 55 °C was sufficient to remove all water by sublimation. XuDuan *et al.* (2010) studied microwave heated freeze drying of sea cucumber (*Stichopus japonicas*), and concluded that freeze drying (FD) yields the best quality of dried sea cucumber but at the cost of long drying time that increased the overall cost. Air drying (AD) on the other hand gives an unacceptably poor quality product. The freeze drying characteristics of foods are scanty in literature and require more work to provide the needed information in food process control.The objective of this paper is therefore to study the thin layer freeze drying characteristics of plantain and carrot slices.

2.0 MATERIALS AND METHODS

2.1 Mode of Heat and mass transfer

The Heat transfer considered in this analysis is radiation and conduction since convection will be rare because very few fluid molecules are available under vacuum condition. The heat flux is given in equation 1;

$$q = \Delta H_s N_W \quad (1)$$

Where; N_w= sublimation rate of ice (g/h)

$\Delta H_S =$ Latent heat of sublimation (J/Kg),

Whereas latent heat of sublimation can be calculated with equation 2.

$$\Delta H_S = \Delta U_S + P_{chamber} \Delta V \quad (2)$$

ΔU_{vap} = increased internal energy of sublimation from solid phase to gas and ΔV = change in volume of product (equation 2).

Mass transfer is achieved by sublimation and desorption of moisture and the amount of mass transfer is given by equation 3.

$$M_w = \frac{M_g\,(w_1 - w_2)}{100 - w_2} \quad (3)$$

Where, M_w = amount of mass (water) transferred (Kg), M_g = initial mass of wet product (Kg), W_1 = initial moisture content (%), W_2 = final moisture content (%).

2.2 Sample Preparation

Fresh plantain and carrots were obtained from a grocery store in Awka North Local Government Area of Anambra State, Nigeria. The samples were thereafter washed in clean water, peeled and sliced into uniform thickness. To ensure uniformity, the crops were first marked out into equal lengths and cut by a clean stainless kitchen knife. The thickness was measured by a digital veneer calipers. Samples of the slices were tested for moisture content using official AOAC (1995) standard method.

The initial and final samples thickness and moisture contents of, plantain and carrot slices were presented in table1.

Table 1: Experimental Conditions and dimensions for the crops Samples

Samples' Properties	Test Samples	
	Plantain	Carrot
Initial chips thickness (mm)	0.5725	0.2125
Final chips thickness (mm)	0.3175	0.135
Moisture content after Freeze Drying (wb %)	2.3	2.4
Initial Moisture content (oven method) (wb %)	61.67	88.05

2.3 Drying Procedure

Weighed amount of the sliced samples were frozen and transferred to the primary drying compartment under vacuum pressure created by a vacuum pump to aid sublimation of the frozen water in the samples at reduced temperature (see Appendix). The entire process was set at temperature of 32 0.98 °C, pressure of 67 ± 2 cm-Hg and the same values for all the experimental runs to ensure common ground for analysis of the three selected crops. During drying, the sample weight was taken every 30 minutes using weighing balance (Model T503D) with sensitivity of 0.001g. The weight of the samples and their initial moisture content were used to determine the moisture content history of the drying samples. The moisture content (%wb) was converted to the dimensionless Moisture Ratio (MR) using equation 4.

$$MR = \frac{M_t - M_e}{M_o - M_e} \tag{4}$$

Where, Mt = moisture content at any time t in %wb, M_o = initial moisture content at t=o in %wb, and M_e = equilibrium moisture content in %wb

2.4 Shrinkage

The chips' thickness before and after the drying test was measured and recorded to determine the shrinkage ratio due to removal of water from the samples' pores and capillaries during drying. The shrinkage ratio was calculated by the expression given in equation 5.

$$\emptyset = \frac{t_o - t_t}{t_o} \tag{5}$$

Where, $\emptyset$ = shrinkage ratio, t_o = initial thickness in mm, t_t = thickness at any time t in mm.
The water extracted from the crop slices after condensation was tested for possible presence of volatile substances escaping from the drying samples. This was done by comparing it with standard natural water.
The experimental data was analyzed using MATLAB R2001b software. The curve fitting tool box was used to fit the drying data to two thin layer drying models (equations 6 and 7 namely:

Newton's model: $$MR = \exp(-kt) \tag{6}$$

Logarithmic model: $$MR = A * \exp(-kt) + c \tag{7}$$

Coefficient of determination (R^2) was used to determine the appropriateness of the model while the accuracy of fits was assessed using Sum of Square Error (SSE) and Root Mean Square Error (RMSE).

2.5. Thermal Efficiency of the Freeze Dryer

The thermal efficiency of the dryer is defined as the ratio of heat energy required for sublimation to the total heat energy supplied to the dryer, as expressed in equation 8.

$$\text{Thermal Efficiency } (\eta_0) = \frac{\text{Heat energy required for sublimation } (Q_a)\text{,Joules}}{\text{Heat energy supplied to the dryer } (Q_d)\text{Joules}} \times 100\% \tag{8}$$

Heat energy required is the heat needed to cause phase change from solid phase to gaseous phase called latent heat of sublimation (equation 9).

$$Q_a = h_l \times M_w \tag{9}$$

Where quantity of frozen water in grams can be calculated using equation 10.

$$M_w = \frac{M_g(w_1 - w_2)}{100 - w_2} \quad (10)$$

and latent heat of sublimation (h_l) is 2.83×10^6 J/kg. The thermal efficiency of the dryer was therefore calculated using equations 9 and 10.

The values of drying constants (k) were obtained by fitting the drying data models in the thin layer drying models of which Newton's and Logarithmic models were most suitable. To graphically estimate the moisture diffusivity (D), the natural logarithm (Ln MR) of the drying crop was plotted against drying time.

The value of k is factor of moisture diffusivity (D), and square of the ratio of pi (π) and half the slice thickness (r) as shown in equation 11:

$$k = D\frac{\pi^2}{r^2} = D\left(\frac{\pi}{r}\right)^2 \quad (11)$$

Therefore, the moisture diffusivity can be estimated using equation 12.

$$D = \frac{kr^2}{\pi^2} \quad (12)$$

3.0. RESULTS AND DISCUSSIONS

The graph of the moisture ratios against drying time is presented in figure 1. From the figure, there was little or no constant rate period, which suggests that drying took place in the falling rate period.

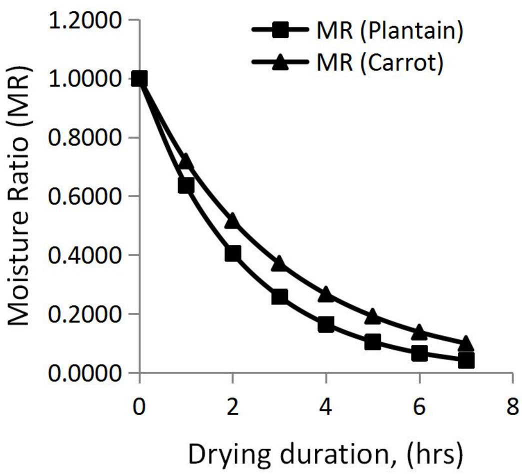

Fig.1: moisture ratio of plantain and carrot chips against drying time

The plot of natural logarithm of the moisture ratio against drying time is presented in figure 2. The slope of the plot gives the value of the drying constant (k). The drying coefficient is a temperature dependent coefficient that describes the rate of moisture removal from the materials. The average drying coefficients (k) are 0.45 and 0.33 hr^{-1} for, plantain and carrot slices respectively.

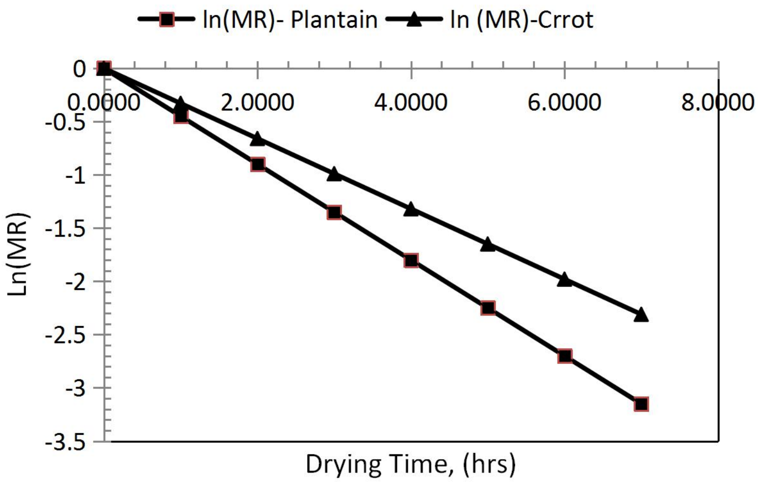

Fig. 2: Natural logarithm of moisture ratio of plantain and potato chips against drying time

They are within the published values for food and agricultural materials. The moisture diffusivities were 1.06428E-06 and 9.2803E-08 for plantain slices and carrot slices respectively.

The drying rates of the samples were plotted against time and presented in figure 3. As expected, the drying rate started increasing after the warming up period and began to decrease at about two hours of drying when it began to drop very gradually to equilibrium moisture content. This observation supports other reported works that drying of agricultural products takes place in the falling rate period.

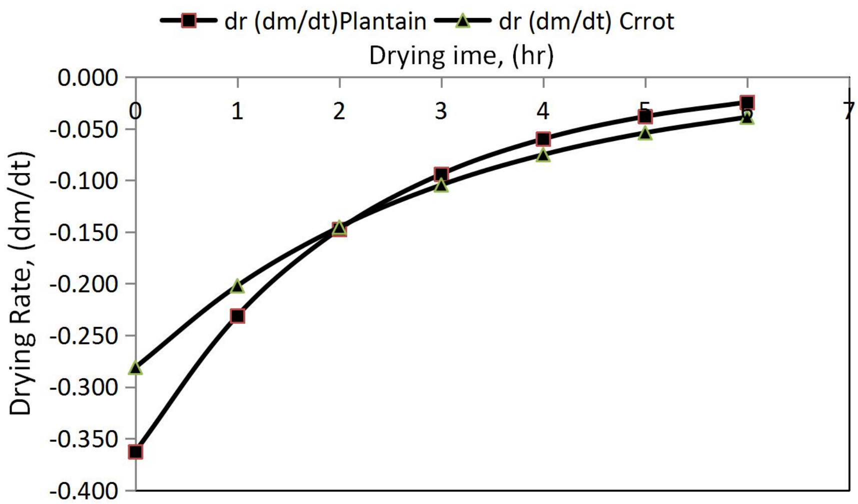

Fig. 3: plot of drying rate against time

The curves met at drying rate of -0.150 g/hr and drying time of two (2) hours after which they started diverging to their final points of equilibrium. The drying conditions at which this happened were vacuum pressure of 67 ± 2 cm-Hg, wet bulb temperature of 29.4 ± 0.49 °C and dry bulb temperature of 32.1 ± 0.98 °C.

The logarithmic and Newton's models in terms of moisture ratio and drying time for plantain slices are presented in equations 13 and 14 respectively. The logarithmic drying model at 95% confidence bound is as follows:

MR = 0.9987*exp(-0.4469*t)+ 0.0012 (13)

The goodness of fit are as follows: SSE=1.668e-005, R2: 1 and RMSE= 0.001826.

Using Newton's model at the same 95% confidence bound the following expression was obtained using plantain slices:

$$MR = e^{-0.45*t} \tag{14}$$

The goodness of fit are shown as follows: SSE= 3.383e-005, R2 = 1 and RMSE: 0.002198.

The predicted data was plotted against the observed in figure 4. It can be seen in the figure that the scater points were closely lying in both sides of the trend line. This indicates that the model prediction was good.

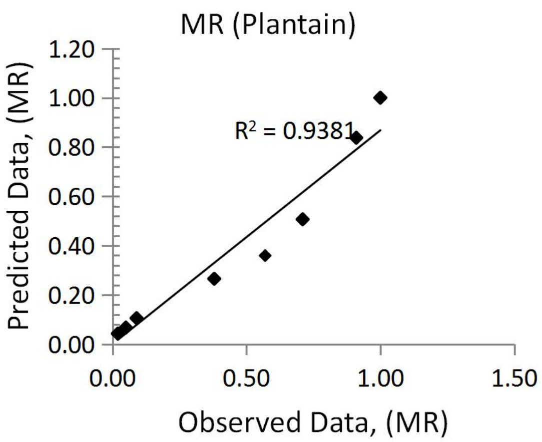

Fig. 4: predicted moisture ratio against observed moisture ratio using Newton's model for plantain slices

The logarithmic model for carrot slices at 95% confidence bound is shown in equation 15:

MR = 0.9975*exp(-0.3291*t)+ 0.0024 (15)

The goodness of fit parameters are, SSE= 4.927e-005, R-square: 0.9999, RMSE: 0.003139

Using Newton's model at the same 95% confidence bound the following equation was obtained (equation 15):

$$MR = e^{-0.3275*t} \tag{16}$$

Goodness of fit parameters are, SSE= 5.168e-005, R2 = 0.9999 and RMSE= 0.002717. The plot of predicted moisture ratio against observed is presented in figure 8. This shows scattered points around the line of fit thereby indicating good prediction.

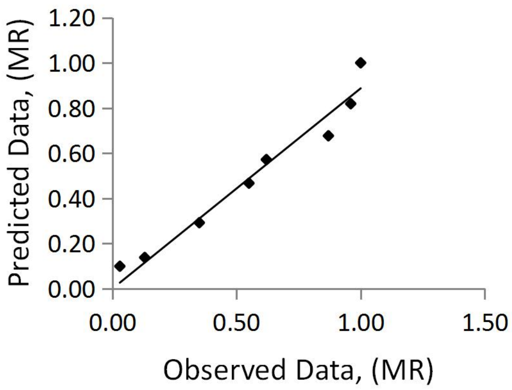

Fig. 5: predicted moisture ratio against observed moisture ratio using Newton's model for carrot slices

3.6 Shrinkage Ratio

The shrinkage ratio is the term used to estimate the extent the material reduces in size when undergoing drying. From equation 9, the shrinkage ratio (ϕ) of potato was 0.37, plantain was 0.45 and carrot was 0.36. From these analyses, carrot lost 37% of its size, while plantain lost 45% of its size in the drying process.

3.7 Nutrients Loss Analysis

The water from the drying crop slices collected after condensation was analyzed chemically to compare it with normal water to know if there were nutrients escaping from the products during water removal. The results of the analysis are presented in table 5.

Table 5: Comparative analysis of desorbed water and normal water constituents

Components	Plantain	carrot	std water
B P (^{o}C)	105.5	108	100
Cond. (μs/cm)	57.3	59.2	55
F P (^{o}C)	-2	-5	0
Ph	5.79	5.44	7.25

The results did not show sufficient changes in the normal values of water to warrant the assumption of significant nutrient ingress into the removed water.

4.0 Conclusions

The laboratory size freeze dryer was successfully developed for use in the study of the freeze-drying characteristics of plantain and carrot chips. The data from the experimental investigations were subjected to statistical analysis using Matlab toolbox and Excel spreadsheet. The primary drying temperature was 32.1 ± 0.98 ^{o}C while vacuum pressure of the chamber was 67 ± 2 cm-Hg. This condition was kept constant throughout the primary drying period for the three crops. The secondary drying temperature was 55 ^{o}C for the whole experiments. Other variables include the slice thickness, initial and final moisture contents.
Logarithmic and Newton's thin layer drying models were used to fit the experimental data of this study because of their wide acceptability. The two models fitted the experimental data well and were used to carry out the characterization and kinetic study of the drying. They showed good predicting capabilities with high goodness of fit at 95% confidence interval. These results further confirmed the adequacy of the two models in predicting the drying data.

The drying coefficient (k) and moisture diffusion coefficient (D) calculated by the two models were the same: 0.45 hr^{-1} and 0.33 hr^{-1} for plantain and carrot respectively while moisture diffusion coefficients were 1.0643×10^{-06} cm^2/hr , 9.2803×10^{-08} cm^2/hr for plantain and carrot respectively. It was observed that after two hours of drying, the two crop slices attained the same drying rate of -0.150 g_{water}/hr.

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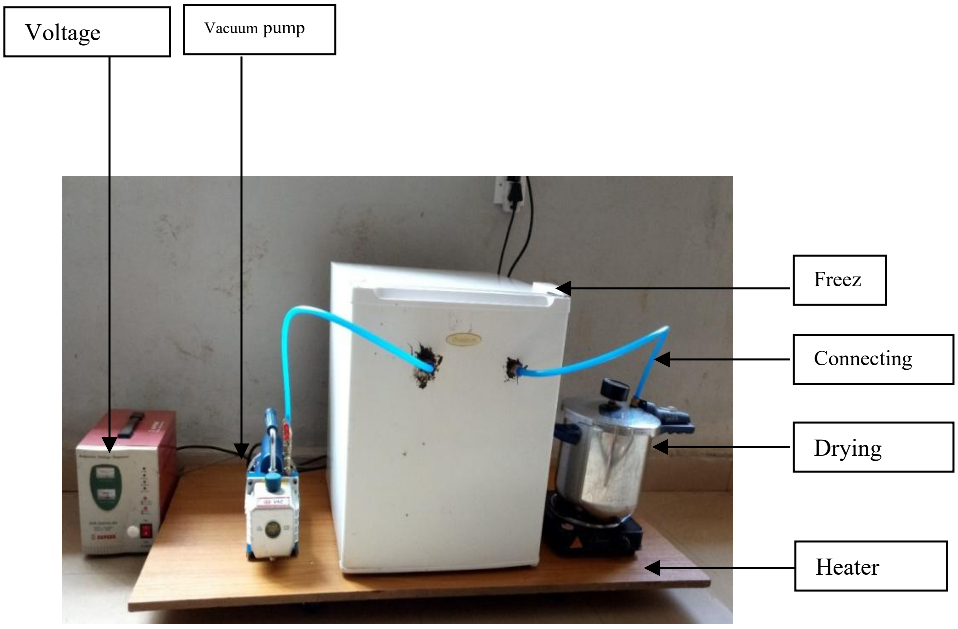

Fig. 14: The freeze dryer assembly and components.