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LYOPHILIZATION OF SELECTED FRUITS IN EDE, OSUN STATE, NIGERIA.

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Abstract: Today, there is a drive towards consuming healthy food products, and fruits are one option for obtaining desirable nutrition in dehydrated products. Proper drying method selection is important for minimizing quality losses. Lyophilization is a technique that removes water by sublimation at low temperature, and can produce excellent dried products. The aim of this work was to lyophilize locally five different types of fruits namely; water melon, banana, orange, cucumber and pineapple at temperatures of -4^{o}C and -41^{o}C for 21 days interval. The lyophilization process consisted of the use of a deep freezer at -41^{o}C and after the stipulated time, the use of a microwave oven for 30 minutes. The results of the study revealed that 21 days' time interval was the required time to lyophilize fruit, when the fruits were brought out after 7 days, the fruits turned to black, at 15 days the fruits turned to brown and at 21days it turned back to normal i.e. its original color. Of the 5 selected fruits lyophilized, the greatest success was in this order; banana> pineapple>cucumber>orange>melon. The fruits were also edible after the process. The results revealed that local lyophilization could be done without the lyophilizer and it should be encouraged as a form of fruit preservation here in Nigeria.

Keywords*: Freeze-drying, fruits, microwave, dehydration, preservation methods.*

INTRODUCTION

Fruits are the means by which angiosperms disseminate seeds (Zhao *et al.*, 2014). In common language, fruit normally means the fleshy seed-associated structures of a plant that are sweet or sour, and edible in the raw state, such as apple, bananas, grapes, lemons, oranges, and strawberries (Lewis, 2012). In botany, fruits are the seed-bearing structure in flowering plants formed from the ovary after flowering. Fruits are the means by which angiosperms disseminate seeds (McGee, 2016).
Many African countries Nigeria included has witnessed voluminous increase in fruits production over the last few years according to WHO (2014). Significant progress has been made in area expansion resulting in higher production. Over the last decade, the fruits production area under horticulture grew by about 2.7% per annum and annual production increased by 7.0% (Steffen *et al.*, 2015). During 2014-2015 Nigeria produced 86,602 thousand tonnes of fruits. The highest annual growth of 9.5% is seen in fruit production during 2014-15 (Steffen *et al.*, 2015).

Fruits play an important role in agriculture and industrial economy. These crops, which are among the perishable commodities, are important ingredients of human diet. Fruits are one of the oldest forms of food known to human being (Kant, 2004). Moreover, in a country like Nigeria, where 20-40% of the population are eaten various kind of fruits like mango, orange, banana, and pineapple due to their nutritional values present in the fruits (Huang, 2010). Almost all fruits contain varying amount of food contents, such as carbohydrates, fats, proteins, vitamins, minerals, etc. The benefits of eating fruits are many.
Medically, fruits are essential source of vitamins, minerals, and energy for human health. Fruits are the excellent source of Vitamin A (which is good for hair and eyes), Vitamin C (helps in neutralizing free radicals), Potassium (which regulates blood pressure) and Magnesium (helps in relaxing muscles and protecting the heart against diseases) (Coyne, 2013). Different parts of fruits vary in their protein contents, vitamin, minerals and carbohydrate contents. Apart from the health improvements, the production of fruits improves the economy of a country as these are very good source of income and employment (Ali *et al.*, 2012). The contribution of fruits remains highest (59 – 61%) in horticulture crop productions over the last five years (Daramola, 2008). The nutritional intake from fruits is higher among urban population than that of rural population especially in Nigeria. Along with the urbanization, people are likely to increase their calorie intake at a higher pace through

fruits, the increase in calorie intake is more than 10% in urban area whereas it is merely 1.89% in rural area over the period from 2004-2005 to 2009-2010 (Jones, 2012).
Despite Nigeria blessed with different variety of fruits, but most of these fruits are not readily available all the time, because some of them are seasonable fruits in which they are abundant during their season and scarce during other season like dry season. Therefore, lyophilization process is needed to keep these fruits readily available at all time in Nigeria. This process will thereby fulfill the basic needs of all people concerning the consumption of fruits to satisfy diets. This study aimed to investigating the drying lyophilization of selected fruits (banana, water melon, cucumber, orange, and pineapples).

MATERIALS AND METHODS

Collection of Sample

The fruits sample (cucumber, banana, water melon, orange, and pineapple) required for this experiment were collected from local market (Oja-Oje market) in Ede, Osun State.

Apparatus, One big Perforated Tray, Knife, Paper thin, deep Freezer, microwave oven, Baking rack.

Sample Preparation

The samples collected were washed to remove all foreign materials from their surface. Then, the fruits' peels, cores, and stems were removed with the use of knife and were sliced into small particles for quick freeze drying. It was ensured that the slicing was not bigger or thicker than one another. All was exactly 2.5mm in thickness then the fruits were layered side by side on a perforated tray in which one does not lay on the other. After cutting, the fruits was then covered with an aluminum foil which was taped at all edges and labeled correctly with the name of fruits that are inside. After covering, it was taken into the freezer at 4^0C and 41^0C of temperature which is said to be a suitable temperature for lyophilization process. It was ensured that the freezer is not frequently opened in order not to disrupt the reaction. The glove was used during these processes for proper hygiene.
After a week in the freezer, the fruits samples were brought out of the freezer and were allowed to thaw, then change in color and taste were observed but unfortunately their color changes to black but the taste remains the same. This shows that seven days was not suitable for lyophilization process. The remaining fruits were returned back into the freezer and rechecked after 21 days. Changes in color and taste were observed again, this time around the color does not change to black after thawing. That means, 21 days interval was the accurate time for lyophilization and the fruits were ready for further process which is micro-waving. The fruits was micro-waved for 30mins, test for change of color and taste were observed again but now the color changes to brown like chips while it tasted nice when eaten. Shortly for the process, it was observed that the fruits used are able to undergo lyophilization procedure at freezing point of 4^0C and 41^0C for 21 days.

RESULTS AND DISCUSSION

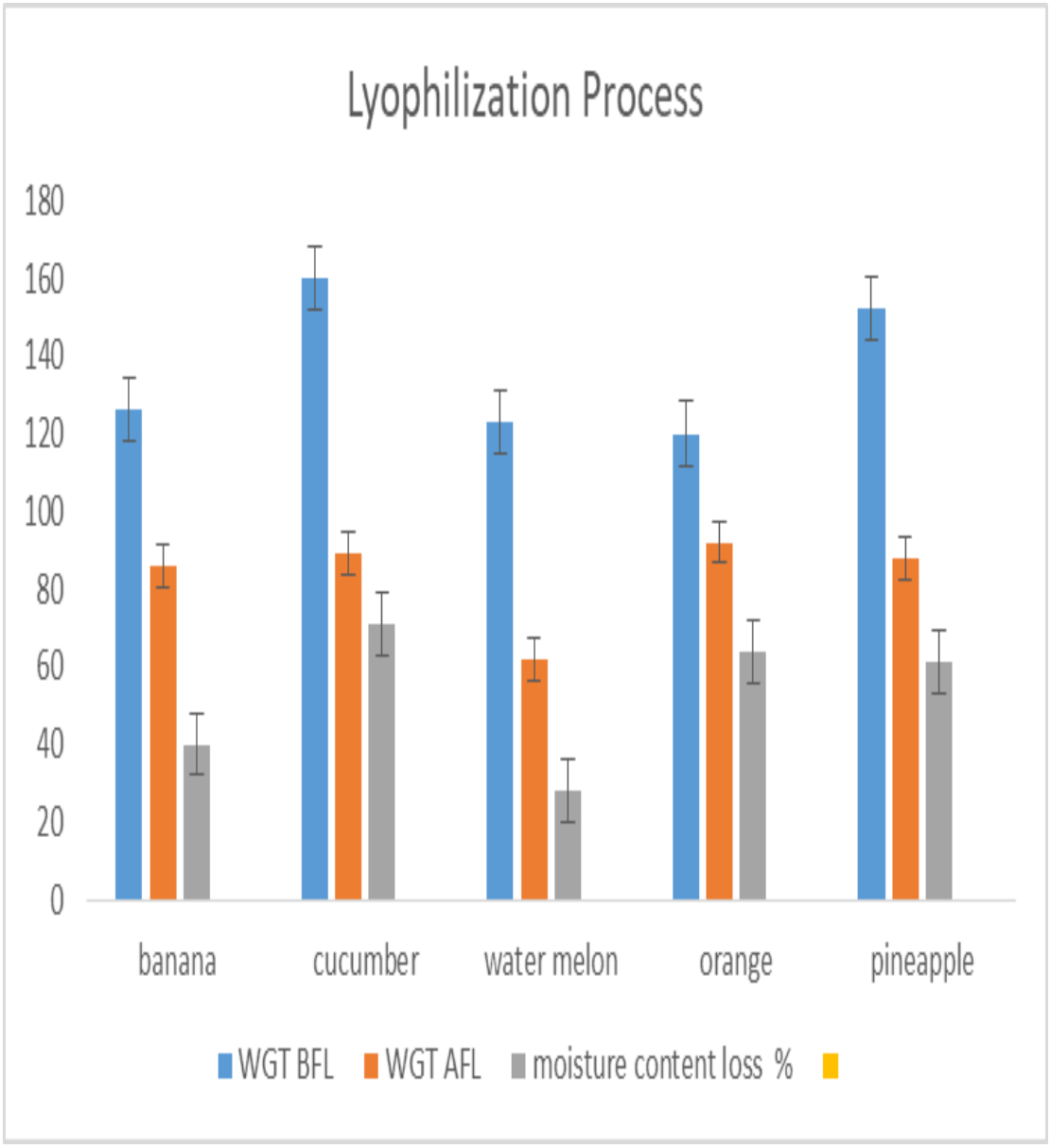

Fig 1.Weight of fruit samples before and after lyophilization.

Key: WGT BFL- weight of fruit before lyophilization**, WGT AFL-** weight of fruit after lyophilization.
Fig 1 below shows the weight of the fruits used for the experiment such as banana, pineapple, water melon, cucumber, and orange before and after lyophilization process. The weight of banana sample before undergoes lyophilization process was 126g and dropped down to 86 g after the lyophilization process, while the water melon sample was 123 g before and 62 after the process, and pineapple has 152 g before and 88 after. Cucumber weight before the put it into lyophilization techniques was 160 g and after techniques the weight decreased down to 89 g and the last selected fruit (i.e. orange sample) has weight of 120 g before the lyophilization process and dropped to 92 g after the process. The result shows that all selected fruits' sample undergoes freezing process by losing water of crystallization.

Fig 1 shows the moisture content of selected fruits after lyophilization process, banana fruit sample used had moisture content of 40 g, while moisture content in pineapple was 64g and in water melon, it was 61g. The moisture content of cucumber was 71g and that of orange was 28g. This result showed that cucumber fruit had

the highest moisture content followed by pineapple and water melon. The fruit with the lowest moisture content was orange with 28g. However, the result indicated that cucumber fruit had high water holding capacity than pineapple, water melon, banana, and orange fruit in the following order; Cucumber > pineapple > Water melon > Banana > orange as shown below.

Percentage water loss = Weight before lyophilization process – Weight after lyophilization process
100

% water loss of Banana = 126-86/100 = 40%

% water loss of Pineapple = 162-88/100 = 64%

% water loss of Watermelon = 123-62/100 = 61%

% water loss of Cucumber = 160-89/100 =71%

% water loss of Orange = 120-92/100 =28%

After the lyophilization process for 21 days, the fruit samples were tested for taste, odor and color. It was observed that after at 4^oC and – 41^oC of temperature, the color, odor, and the taste of banana, cucumber, pineapple, water melon, and orange remain unchanged but when dried with microwave for 30 minutes, only color changed and both odor and taste remain unchanged. The color changed was due to loss of water of crystallization in fruits which make them look like chips. This result showed that the banana, cucumber, orange, pineapple, and water melon can be lyophilized at both 4^oC and – 41^oC for future use.

However, when the fruit sample particularly banana fruit was removed from lyophilization this process at 7 days, the color of the fruit was changed into black but when the time of lyophilization extended to 21 days, the black color disappeared and turn back to normal banana color. This results shows that 7 days' time series was not enough to lyophilize the banana fruits and other type of fruits. Also, the result indicates that 21 days was an adequate time interval to lyophilize different kind of fruits.

In Fig 1 above, it was discovered that the weight of all fruit samples used dropped down as the fruits after the lyophilization process. Banana fruit lost 40t, pineapple lost 64g , water melon lost 61g, and cucumber lost 71g, while orange lost 28g of weight. The result shows that all fruit samples lost their water of crystallization during the lyophilization process.

The result was similar to Kunnal *et al.* (2015) study who stated that the weight of samples lyophilized decreased compared to the initial weight of sample due to sublimation reaction which involves the direct transition between the solid state and the gaseous state without passing through the liquid phase.

Frank (2008), stated that to achieve effective lyophilization process of fruits, the frozen fruits is dried under vacuum, without being allowed to thaw out to reduce the moisture contents of the fruits for proper preservation for future. However, lyophilization, is the most common unit process for manufacturing drug products too unstable to be marketed as solutions.

According to Smith (2008), annealing is commonly performed during the freezing step and it not only crystallizes the crystallizing recipients in the formulation matrix, but also removes freezing heterogeneity and reduce the primary drying time. However, if annealing is applied, selection of the annealing temperature and time is critical. Annealing can potentially lead to product instability due to conformational change in protein structure or amorphous phase separation. Hence annealing should be performed with caution.

Chen (2008) also stated that insufficient drying time results in product collapse or melt back due to premature progression into secondary drying, whereas a prolonged drying time results in an unnecessarily long cycle time for lyophilization. However, during the observation period on the lyophilized fruits, it was discovered that the fruit was lyophilized at 4^oC and -41^oC in 21 days period. This shows that the fruits can be lyophilized using 21 days and temperature of 4^oC and -41^oC. The taste, odor, and color of fruits lyophilized remain unchanged after lyophilized process. This means that the vitamin, minerals, and generally nutritional properties of lyophilized fruits remain intact after lyophilization process.

According to Francis (2009), formulation and process development challenges need to be considered during 7 days development to ensure product quality, the time must increase to a month or 3 weeks. Smith (2008) confirmed that the lyophilization process is a technique to preserve and assure desired product quality of fruit.

Bankers *et al*. (2010) described lyophilization as a method of preservation of food which has been used in sample pre-treatment, particularly for sample preservation, but also for pre-concentration. Its use depends on the

type of analyte (organic or inorganic) to be assayed after restitution of the sample, which, in summary, determines the usefulness of the pretreatment step. Each group of analytes has been further divided according to the nature of the sample matrix (inorganic and organic, both from plants and animals).

Ali *et al*. (2006) opines in his study that lyophilization process is very cost effective when compare to freezing drying is a relatively expensive process. The equipment is about three times as expensive as the equipment used for other separation processes, and the high energy demands lead to high production costs. Furthermore, freeze-drying also has a long process time, because the addition of too much heat to the material (to speed the process) can cause melting or structural degradation. The low operating temperature of the process leads to minimal damage of these heat-sensitive products – in our case the anthocyanins. Therefore, freeze-drying is often reserved for materials that are heat-sensitive, such as proteins, enzymes, microorganisms, and blood plasma.
Chen (2008) conducted an experiment on the lyophilization of fruits and discovered that lyophilization of fruits successfully yielded pure and stable anthocyanin product. Anthocyanins extracted from elderberry stored as a powder are protected against degradation. This product could be used for many different purposes in dietary supplements.

CONCLUSION AND RECOMMENDATION

Based on the results and observation obtained from this study, it showed that the lyophilization process is an effective technique to preserve and ensure desired product quality of fruits for future use. It can also be concluded that lyophilization technique is an economical method of preserving food with minimum effort. 21 days and above is the required period to lyophilize the fruits, time taken lower than this may change the taste, color, and nutrients present in the fruits.

Recommendations

Due to the findings above, the study hereby recommends the following:

- Since freeze drying is a rather expensive process of food preservation, therefore lyophilization is recommended to preserve fruits for future use especially to make different kind of fruit available in all season.
- Freeze-drying is applicable for all fruits; especially fruits with low moisture content that required being in dry state.
- Further research should be carried out to know different parameters such as require temperature, pressure, pH, and nutrients contents in the fruits after lyophilization process.

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PICTURE GALLERY

ORANGE BEFORE FREEZE

SLICED CUCUMBER BEFORE FREEZE

SLICED WATER MELON BEFORE FREEZE

SLICED BANANA CHIPS BEFORE FREEZE

LYOPHILIZED BANANA

SLICED PINEAPPLE BEFORE FREEZE

LYOPHILIZED PINEAPPLE