4th International Student Conference on Food Science and Technology

Catering Global Demand: Enhanced Food Packaging and Marketing Strategy

The Committee of The 4th International Student Conference
on Food Science andTechnology

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The Characterization of Foods Before and After Freeze-Drying

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ABSTRACT

Freeze drying is a relatively recent method of preserving food. Freeze-dried foods can be easily transported at normal temperatures, stored for a long period of time and consumed with a minimum of preparation. The aims of this research was to study the freeze-drying process, to study the sensory characteristic of the final freeze dried product, and to study the changes that occur during the freeze-drying process. The sample used were banana, apple, kiwi, baked sweet potato, tofu, pudding, plain yogurt drink and brown rice milk. Physico-chemical quality parameters such as color values, texture, shape and taste profiles also moisture content were determined for the fresh and freeze dried samples. The moisture content value decreased during freeze drying process whereas color brightness values increased, the original color and the shape of the sample were maintained, the texture became crisp, spongy, soft, easily to destroy and crumbly when exposed with tongue.

Keywords: Freeze drying, Freeze drying methods, Lyophilization

INTRODUCTION

It is well known that food processing and preserving may partially or totally affect the quality of a food product. Various changes may occur in physical, chemical or biological characteristics of foodstuff during processing, storage and distribution (Duan*et al.*, 2010). The physical and biochemical changes of the foodstuff can reduce the product quality and the efficiency of the process, whereas the quality standard of food is importance to increasing the consumer's choice (Chuy&Labuza, 1994).

One of the oldest methods of food preservation is dehydration. Dehydration is a means of preserving foods in a stable and safe condition as it reduces water activity and extends self-life much longer than the food in fresh condition (Jiang &Mujumdar, 2010). There are so many conventional thermal methods, including airflow drying, vacuum drying, and freeze-drying, result in low drying rates in the falling rate period of drying (Wang *et al*., 2010).

Freeze-drying is not really a new process. Primarily it was developed for the pharmaceutical industry and drugs drying for nearly forty years before applied to the food industry. Freeze-dried foods have some high quality characteristic compared to products of alternative drying process such as aroma and shape retention, high porosity, good rehydration, superior taste and low bulk density. Freeze-drying is based on the dehydration by sublimation of a frozen product where the water or solvents are removed as a vapor from the frozen material in a vacuum chamber (Liapis*et al.*, 2007). Very good physical and chemicals properties of food and biotechnological products make this method the best for drying exclusive products.

Freeze-drying is an expensive process for food industry of dehydrated product. Equipment innovation and pre-treatment of raw material can reduce the time and energy that needed for this process. Nowadays, freeze-drying process is used to produce high quality value products for example coffee, crispy fruits and vegetables, ingredients to ready to eat foods (corn, flakes, cereal bars, ice cream or pastry making) and some aromatic herbs.

This research was conducted in the Faculty of Food Science, Fu Jen Catholic University, and took place 12^{nd}January to 11^{nd}March 2016. The general purpose of this research was to study about the freeze-drying process, to study the sensory characteristic of the final dried product, and to study the changes that occur during the freeze-drying process.

MATERIAL AND METHOD

Sample Preparation

Eight samples, namely banana, apple, kiwi, baked sweet potato, tofu, pudding, plain yogurt drink and brown rice milk were purchased from the supermarket in Taipei, Taiwan. For all the fruits were washed under tap water and peeled. Banana and kiwi were peeled first and cut into 0.3 cm thick slices, apple were peeled and cut into 6 slices of every half a loop, sweet potato were peeled and cut into (1 x 1 x 1) cm^3, tofu were cut into (1.5 x 3 x 1) cm^3, pudding were cut into 3 parts, then plain yogurt drink and brown rice milk were poured into the OPP plastics (± 200 ml).

Sensory Evaluation

The sensory evaluation of the samples was carried out by a taste panel of 5 untrained judges. The panelists were asked to indicate their preference for each sample, based on the quality attributes of color, shape, texture and taste before and after freeze-drying process.

Determination of Color

The samples will be used as much as 3 pieces each kind of samples, then measured the color by Chroma Meter one by one. It also conducted again after the freeze-drying process.

Determination of Moisture Content

All samples were weighed by Analytical Balance and determined the moisture content. The method to count the moisture content was shown in below:

$$MC = \frac{w - d}{w} \times 100$$

w = wet weight
d = weight after drying

Freeze-Drying
For the freeze-drying experiment the samples were spread out on the flat metal trays that cover with plastic wrapping, and frozen at -24 ± 1ºC for 3 days in the refrigerator. Then, the frozen samples were put in the vacuum drier at 27 ± 1ºC for three days until they were completely dried.

RESULTS AND DISCUSSION

As Table 1. shows that all of the fruit after freeze drying become more white, spongy, smooth, firm, the original taste doesn't change and the shape is maintained. For the bake sweet potato and tofu the color become whiter, the texture more strong, crisp and the shape is maintained, but the taste become sticky and starchy. For the liquid sample, the shape become solid like crystal grains, the texture become smooth, very easy melt in the tongue, and has a strong original taste of each sample.

Table 2 shows the results of color measurements of fresh and dried samples. The L* color parameter indicates whiteness of the product. L* is a gradation of the skin brightness between the brightest white (high value) and midnight black (low value), with a stretch value of 0-100. Parameter a* show the gradations of color measurement spectrum of colors between green (-) to red (+), while the parameter b* show gradations of color spectrum between blue (-) to yellow (+). Each has its own stretch of values between -60 to +60. In general, the drying treatment resulted in significantly improved whiteness (increased L value, 45.09 until 94.45) while the value of a * and b * did not show too much difference. But on the tofu samples had decreased in L* value.

Drying removes the moisture from the food so bacteria, yeast and mold cannot grow and spoil the food, so can extend the shelf life. Drying also slows down the action of enzymes, but does not inactivate them. Because drying removes moisture, the food becomes lighter in weight. Low humidity allows moisture to move quickly from the food to the air. The result in table 3 depict the moisture content of the final samples depend on wet basis. Tofu was found to have the highest moisture content (0.909%) whilst sweet potato had the lowest (0.556%).

The application of freeze-drying process to foods is most important appears to be for meats (beef, pork, chicken, and fish). The second most interesting commodity group is fruits and vegetables. The functional properties of the freeze-dried milk are not as desirable as those of spray-dried milk. Nickerson ***et** al.* in Harper and Tappel (1957) found that overall flavor and storage characteristics of freeze-dried whole milk are essentially the same as spray-dried whole milk. Both milks have essentially the same flavor characteristics when fresh and tallow in storage. Mastenbroek(1954**)** in Harper and Tappel (1957) said that freeze-drying was found to be the preservation method of choice because complete pasteurization would cause denaturation of valuable antibodies and loss of some nutrients. Obviously the cost of production is very high and distribution is quite restricted.

Freeze-drying yoghurt may help maintain a sufficient quantity of viable probiotics (Capela, Hay & Shah, 2006). Previous research has found that certain strains of probiotics are better able to survive the freeze-drying process (Capela, Hay & Shah, 2006). The freeze-drying yoghurt also preserve the yoghurt in a high-quality shelf-stable powder form (Khumar& Mishra, 2004)). It would be beneficial if the yoghurt were concentrated before drying to increase its total solids, which improves the efficiency of the drying process. (Groux, 1973 in Khumar& Mishra, 2004). Since practically no further research has been done on the preservation of tofu, pudding and baked sweet potato by freeze-drying.

Raw Materials and Pre-treatment

All of the food that will be freeze-dried must be checked first for the contamination and the purity (Antolovich et al, 2000). Fruits, vegetables and some other edible foods are tested for bacterial counts and spoilage. Some drinks are purchased as a pre-brewed concentrated liquid. Unlike the water, the oil is not removed during the drying process (Burnett, 2001).Almost all fruits and vegetables can be freeze-dried.Liquids, thin portion of meat, and small fruits and vegetables can be freeze-dried easily. Fruits can be cut in half or sliced. Thin, uniform, peeled slices dry the fastest. Apples can be cored and sliced in rings, wedges or chips. Bananas can be sliced in coins, chips or sticks. Spray drying is a common method for producing powder from liquid, it is more simple and cheap, because actually freeze drying is a method of preservation of foods and biological materials (Jenkins, 1968).

Freeze-Drying Principle

The main principle in freeze drying is a phenomenon called sublimation. Sublimation is the transformation of ice directly into a gas without passing through a liquid phase (Nireesha*et al*, 2013). Sublimation occurs when the vapor pressure and the temperature of the ice surface are below those of the triple point (4.58 mm Hg, 0^{o}C), as shown in the pressure-temperature phase diagram of pure water (Figure 4) (Nireesha*et al*, 2013).

Freeze-drying separation method involves the following four main stages:

1. Sample preparation
2. Freezing stage
3. Primary drying stage, and
4. Secondary drying stage

(Nireesha*et al*, 2013).

Annealing is an optional step that used to crystallize the formulation component. If the solute separates out in crystalline form, it is known as the eutectic temperature. In contrast, if an amorphous form is formed, the temperature is referred to as the glass transition temperature, which leads to “meltback” or “collapse” phenomenon. In the majority of lyophilized functional properties and stability of the lyophilization product (Nireesha*et al*, 2013).

Freezing, in which the liquid sample is cooled until pure crystalline ice forms part of the liquid and the reminder of the sample is freeze-concentrated into a glassy state where the viscosity is too high to allow further crystallization. The objective of the freezing stage is to freeze most of the water originally present in the product for its posterior sublimation, generally in a blast freezer at a temperature of about minus 40°F. (Nireesha*et al*, 2013).

Primary drying, wherein the ice formed during the freezing is removed by sublimation under vacuum at low temperatures, leaving a highly porous structure in the remaining amorphous solute that is typically 30% water. This step is carried out at pressures of 10-4 to 10-5 atmospheres, and a product temperature of -45 to -20^{o}C. Sublimation during primary drying is the result of coupled heat and mass transfer process. When the water molecules sublime and enter the vapor phase, they also keep with them a significant amount of the latent heat of sublimation (2840 Kj/kg ice) and thus the temperature of the frozen product is again reduced (Nireesha*et al*, 2013). The time at which there is no more frozen layer is taken to represent the end of the primary drying stage (Nireesha*et al*, 2013).

Secondary drying, the desorption of the remaining water from the solids is called secondary drying while maintaining low pressures. The bound water is removed by heating the product under vacuum. The following product temperatures are usually employed: (a) between 10^{o} and 35^{o}C for heat sensitive products and (b) 50^{o}C or more for less-heat-sensitive products (Nireesha*et al*, 2013).

Structural Changes

The sought after freeze-drying product are porous that maintain their volume, can have fast and nearly complete rehydration when water is added and do not shrink (Duan et al., 2010). However some freeze-dried products undergo undesirable structural changes. Microscopy can be used to study structural changes in freeze-dried fruits and to find a relationship to some physical properties (Yeom and Song, 2010). Some phenomenon that can occur during the freeze-drying process are:

1. Collapse = loss of structure, reduced pore size and volumetric contraction (Khalloufi et al., 2010).
2. Rehydration = the restoration of raw material properties when dried material is contacted with water, then thevolume changes(Yeom and Song, 2010
3. Shrinkage = Most of the shrinkage occurs in the early drying stages (Yeom and Song, 2010
4. Porous = Porous sponge-like structures are excellent insulating bodies and generally will slow down the rate of heat transfer into the food (Yeom and Song, 2010).

Physical Changes

Glass Transition

Absorption of additional moisture can lead to a state of amorphous disequilibrium, which brings with it a transformation from a glass solid state to a plastic fluid state when the glass transition temperature is reached (Duan et al., 2013). The glass transition temperature of dry solid would be an important optimization parameter can be used as useful tool for the choice of the most appropriate materials to be freezing-dried (Ratti, 2001).

Solubility

Many factors affect the solubility, including processing conditions, storage conditions, composition, pH, density, and particle size. It has been found that increasing product temperatures is accompanied by increasing protein denaturation, which decreases solubility. The heat treatments as well as the particle size must be considered when determining solubility.

Texture
Texture is one of the most important properties connected to product quality. Factors that affect texture include moisture content, composition, variety, pH, product history (maturity), and sample dimensions. The chemical changes associated with textural changes in fruits and vegetables include crystallization of cellulose, degradation of pectin, and starch gelatinization. High air temperatures (particularly with fruits, fish and meats) cause complex chemical and physical changes to the surface, and the formation of hard impermeable skin. This is termed “case hardening”.

Color
The conservation of color is considered an indications of quality in dried fruits given that non-enzymatic browning processes develop during the drying process (Ceballos et al., 2012). Freeze-dried fruits better maintain red and yellow colors than fruits dried using traditional methods (Shishegarha et al., 2002). The loss color in freeze-dried product is compared to the color loss in air-dried products. The color loss is noted, due to the decomposition of pigment (Guine and Barroca, 2012). Rapid freezing can produced a more intense white value.

Aroma Loss
Volatile organic compounds responsible for aroma and flavor have boiling points at temperatures lower than water. Volatiles, which have a high relative volatility and diffusivity, are lost at an early in drying. Fewer volatile components are lost at later stages. A second important cause of aroma loss is oxidation of pigments, vitamins and lipids during storage. The open porous of dried food allows access of oxygen. Most fruits and vegetables contain only small quantities of lipid, but oxidation of unsaturated fatty acids to produce hydro peroxides, ketones and acids, causes rancid and objectionable odors.

Chemical Changes
Chemical changes that occur during the freeze-drying is caused by some reaction like non-enzymatic browning reactions and lipid oxidation.

Browning Reactions
Browning reactions are important in terms of the alteration of appearance, flavor, and nutritive value. Browning is undesirable for fruits, vegetables, frozen and dehydrated foods as it results in off-flavors and colors. The effect of browning is the lowering of the nutritive value of the food article. Rate of browning reactions depends on temperature of drying, pH and moisture content of the product, time of heat treatment, and the concentration and nature of the reactants. Browning reactions change color, decrease nutritional value and solubility, create off flavors, and induce textural changes. In freeze-dried, browning occurs because an enzymatic reactions.

Lipid Oxidation
Lipid oxidation is responsible for rancidity, development of off-flavors, and the loss of fat soluble vitamins and pigments in many foods, especially in dehydrated foods. Factors that affect oxidation rate include moisture content, type of substrate (fatty acid), extent of reaction, oxygen content, temperature, presence of metals, presence of natural antioxidants, enzyme activity, ultraviolet light, protein content, free amino acid content, other chemical reactions. The effect of oxygen on lipid oxidation is also closely related

to the product porosity. Freeze-dried foods are more susceptible to oxygen because of their high porosity.

CONCLUSION

Freeze-drying is suitable for the reliable preservation of a wide variety of heat-sensitive products likefruits, tofu, yoghurt, milk and tofu but not suitable for pudding. In the other hand, freeze-drying is suitable for products demand with the highest standards reliability control. The most important in this process are time, temperature and pressure. If they are well defined they may indeed affect the quality of the final product. An optimal drying system for the preservation of quality, is cost effective, eliminates or reduces the exposure to light and oxygen, and shorten the drying time, thus causing minimal damage to the product. Pre-treatment is very important to prevent browning reactions and also can limit the process time of freeze-drying.

The structural, physical, functional and nutraceutical effects of freeze-drying produce are dependent on intrinsic factors that are inherent to the samples and to extrinsic factors that are inherent to the process. Freeze-drying is an ideal method for heat sensitive fruits that require special care during processing. In many fruits, properties such as shape, dimension, appearance, flavor color, texture and nutraceutical ingredients are retained after freeze-drying, adding value of approximately 120%. Unfortunately, high porosity of dried foods has a negative effect on storage stability. The foods need to be stored in a hermetic package.

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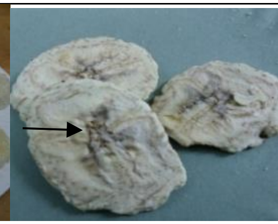
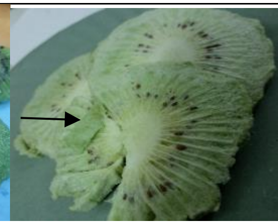
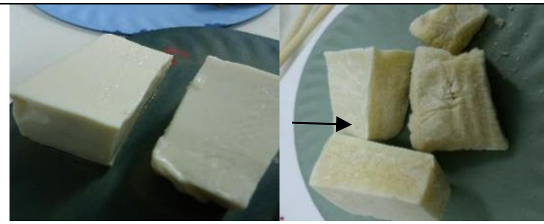
TABLE AND FIGURE

Table 1. Consensus Attribute Lists Before and After Freeze-Drying

Sample	Attribute	Before	After	Picture	
Banana	Color	Bright yellow skin, and creamy white flesh	Whitish yellow flesh		
	Texture	Soft, free from bruises or another injury	Crisp and soft texture, not easily destroyed		
	Taste	Sweet and creamy	Sweet inside, the original flavor is maintained		
	Shape	Long curving cylinder cut circle	The shape doesn't change just very little shrink	Before	After
Apple	Color	Red skin, white flesh and black seeds	Red, smooth skin, white flesh with a little browning in the surface		
	Texture	Smooth skin, firm flesh and crisp	Soft, smooth tough, firm, and spongy texture		
	Taste	Sweet flavor and juicy	Sweet inside, little sour and the original flavor is maintained	Before	After
	Shape	Rounded shape of flesh	The shape is maintained		
Kiwi	Color	Dull greenish-brown skin, bright green-golden flesh, and black edible seeds	Whitish green flesh and black seeds		
	Texture	Fibrous and fuzzy	Fibrous, easily crumble when exposed tongue, smooth and spongy		
	Taste	Sweet and citrusy	Very sour, citrusy and little sweet, but the original flavor still maintained	Before	After

	Shape	Like chicken's egg cut circle	Shape still maintained.	
Tofu	Color	White	More white	
	Texture	Soft, smooth, spongy, wet, firm and chewy	Crisp texture, very easy to destroyed crumbly when exposed with tongue	
	Taste	Subtle flavor, sweet taste and slight earthy	Creamy, has a strong soy bean flavor	
	Shape	Block	The shape is maintained just shrink	Before After
Bake Sweet Potato	Color	Clean orange flesh and bright, brown skin that easily removed from the flesh	Become whiter and orange	
	Texture	Easy to destroyed, smooth, moist and firm	Very crisp and slightly hard texture, not easily to destroy	
	Taste	Sweet taste	Starchy and little sweet	
	Shape	long non-symmetric cylinder flesh	Still maintained	Before After
Pudding	Color	Bright yellow color and shiny, brown on top	Whiter outside and yellow	
	Texture	Slightly thicker seeming broth, smooth and soft, moist, firm but not hard, and elastic	Very crisp, soft and dry	
	Taste	Sweet	Sweet and very sticky inside	
	Shape	Non-symmetric		Before After

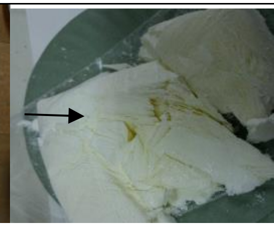
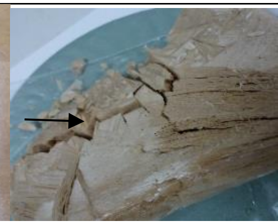
Plain Yogurt Drink	Color	Yellowish-white	Whitish yellow solid, like a crystal grains	 
	Texture	There is no award but there's bubbles, compact, smooth, little thick viscous	Smooth, melted and clot when exposed with tongue and very easy to destroy.	
	Taste	Sour, acid, little sweet, watery, buttery, creamy, fresh and plain	Little sour, buttery and creamy	
	Shape	Liquid	Solid like a crystal grains	
Brown Rice Milk	Color	Brown and clean	Brown	 
	Texture	Thinner in consistency than soymilk, smoothness, heterogeneous, and watery	Very easy to destroy and melted in the tongue	
	Taste	Sweet, creamy thick, firm, and milky also has a rice flavor	Sweet and has a strong rice flavor	
	Shape	Liquid	Solid like a crystal grains	Before After

Table 2. Colour Measurement Before and After Freeze-Drying

R		Banana	Apple	Kiwi	Tofu	Sweet Potato	Pudding	Yogurt	Brown Rice Milk
	*L** values								
	Before	65.29	74.35	64.21	90.15	56.69	78.32	80.49	53.45
	After	76.49	84.15	87.15	77.93	69.07	77.48	93.74	65.63
	*a** values								
1	Before	11.14	05.13	-00.13	02.45	04.07	06.93	10.19	04.82
	After	06.41	07.00	00.86	03.74	04.57	04.38	00.63	08.66
	*b** values								
	Before	37,91	23.07	21.07	12.71	35.20	37.63	11.16	12.59
	After	19.96	32.06	21.04	18.67	25.54	24.39	09.59	18.40
	*L** values								
	Before	50.93	71.08	70.30	88.45	54.49	71.14	73.71	61.13
	After	74.79	85.73	87.50	79.36	68.19	80.20	90.40	71.53
	*a** values								
2	Before	09.57	05.14	00.16	02.10	02.02	07.67	11.55	11.13
	After	06.78	07.91	00.58	03.82	05.67	08.39	00.86	09.06
	*b** values								
	Before	29.25	25.38	21.52	13.96	23.82	34.78	8.50	24.33
	After	19.37	30.58	19.47	18.59	27.86	29.68	11.53	19.26
	*L** values								
	Before	68.63	74.69	51.52	87.35	45.09	72.06	68.32	61.04
	After	75.80	86.41	85.14	84.23	73.25	83.21	94.45	68.74
	*a** values								
3	Before	10.40	05.17	-01.29	02.16	05.75	08.34	11.61	10.97
	After	07.58	07.21	00.09	05.02	06.05	06.63	00.58	09.67
	*b** values								
	Before	38.95	24.37	13.80	13.70	19.51	37.21	7.40	23.79

After	21.84	30.76	23.27	20.30	28.96	26.90	08.55	20.04

Table 3. Moisture Content of Final Samples (Wet Basis)

Parameter	R	Samples							
		Banana	Apple	Kiwi	Tofu	Sweet Potato	Pudding	Yogurt	Brown Rice Milk
Moisture Content (%)	1	0.763	0.834	0.860	0.906	0.653	0.773	0.848	0.852
	2	0.754	0.837	0.852	0.909	0.556	0.777	0.854	0.856
	3	0.751	0.836	0.847	0.900	0.696	0.712	0.852	0.856