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To the Graduate Council:

I am submitting herewith a thesis written by Rong Cheng Lin entitled "Some Firming Agents on the Texture of Freeze-Dried Banana." I recommend that it be accepted for nine quarter hours of credit in partial fulfillment of the requirements for the degree of Master of Science, with a major in Food Technology.

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SOME FIRMING AGENTS ON THE TEXTURE OF FREEZE-DRIED BANANA

A Thesis

Presented to

the Graduate Council of

The University of Tennessee

In Partial Fulfillment

of the Requirements for the Degree

Master of Science

---

by

Rong Cheng Lin

August 1968

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ABSTRACT

The objective of this study was to compare the freeze-dried banana puree slices treated by firming agents with untreated banana puree slices to see if any particular levels of firming agents would produce a more desirable product texture.

In this study, an air-blast freezer (-30°F.) was used to freeze the rope of banana puree. Firming agents were selected to prevent damage from freezing, dehydration, and furthermore, to improve firmness of freeze-dried banana after rehydration. The frozen weiner casings with filled puree were removed from the air-blast freezer, and all subsequent work prior to freeze-drying was done inside a 0°F. still air freezer. The filled casings were removed with a sharp knife. When the trays of banana samples for freeze-drying were placed in the chamber (Del-Vac Freeze-Dryer, Model 11212 RVM), the platen temperature was approximately 10°F. while the samples were -20°F. The trays were placed on the shelf, a vacuum was applied, and the thermostat was turned to 150°F. After 4.5 hours the thermostat was adjusted (platen temperature) to 125°F. for the remainder of the drying time.

Both subjective and objective quality measurements were employed for evaluating the freeze-dried banana product. Randomized complete-block design was used for measuring the effect of kind and level of firming agents by taste panel test (both texture scoring and hedonic rating). Chi-square test was used for interpretation of difference-preference

paired comparison test for selection of type of puree sample). A 4 x 6 factorial design for computing the Shear-Press measurement was employed. Color measurement was carried out by the Color-Eye, interpretation of measurement was made on C.I.E. basis. An Allo-Kramer Shear-Press and recorder indicator was used to determine the texture of the banana product. In statistical analysis, BMD02S IBM Model 7040 Digital computer was used to compute Chi-squares. ANOVAR, with usage of the Fortran Library Input Subroutine and some Fortran IV subroutine subprograms, were used to perform the analysis of variance calculations. Duncan's Multiple Range test DUNMRT subroutine on the IBM Model 1640 Digital computer was used to perfrom this test; and it was used to determine which product mean scores were significantly different.

The following conclusions may be drawn from this study:

1. The freeze-drying cycle used in this study was found promising for the ultimate freeze-dried product.

2. Stability of color of freeze-dried banana slices which contained less than 5 per cent moisture was high.

3. Rehydration test showed the lower the pH of the medium the slower the water reabsorbed by freeze-dried bananas.

4. The four firming agents selected (MC, CMC, Kraystay, and Guar Gum) provided a higher finishing temperature in the freeze-drying operation with little or no damage of product quality. They improved the firmness of the product after rehydration.

5. Freeze-dried banana slices treated with firming agents rated significantly higher than the untreated freeze-dried banana product for both texture and preference tests.

6. Results of Duncan's Multiple Range test for the effect of levels of firming agents as measured by Shear-Press were all significantly different at 0.05 level of probability.

7. The effect of kinds of firming agents on the texture of freeze-dried banana as measured by the taste panel test showed that there were significant differences between each pair of mean score -- MC having the highest mean score, followed by CMC, Kraystay, and Guar Gum.

8. Results of Duncan's Multiple Range test on agents by levels interactions showed that 5 per cent CMC had the highest mean shear value, 5 per cent MC the next, followed by 4 per cent CMC, and 4 per cent MC.

9. The taste panel test score (both mean texture score and mean hedonic rating) revealed that firming agent levels from 1 to 3 per cent steadily increased mean score while higher levels decreased them. Thus the lower levels may be considered promising from the standpoint of the overall quality of the freeze-dried banana product. Conversely, the shear value of the product rose consistently with increased levels of the firming agents except for the Kraystay, in which case the value dropped sharply at levels exceeding 3 per cent.

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CHAPTER I

INTRODUCTION

The banana is a sweet, pulpy fruit, containing approximately 75 percent moisture. The greater part of the nutriment in the ripe fruit is sugar; that of the unripe is starch. The fruit is a source of easily digestible starch, approximately 21 percent carbohydrates. It is also a source of vitamins A, B, and C.

An interest in freeze-dried banana has been done by employing some different treatments in search of an improvement of the natural texture. Freeze-drying is an important method of preservation of foods. Basically, freeze-drying is changing frozen foods by sublimation. The ice is removed directly from the frozen state to the gaseous state, by passing the liquid phase. Freeze-dried banana slices could become popular as a component of cereal-based, ready-to-eat breakfast foods. Inadequate data are available concerning the effect of this process on the texture of the banana.

The main purpose of this work was to study the effect of various firming agents on the textural properties of freeze-dried banana slices which can be consumed with or without rehydration. The objectives of this study were to determine the:

1. Freezing dehydration condition
2. Effect between two types of samples
3. Kinds of firming agents

4. Effects among kinds and levels of firming agents

5. Relationships of rehydration, shear-press measurement, and sensory method of evaluation to the quality.

CHAPTER II

REVIEW OF LITERATURE

A. THE NATURE OF BANANA

The banana belongs to the family Musaceae. It is produced on a large tropical plant. The edible fruit occurs in hanging clusters, are usually yellow when ripe, and are about 6 to 8 in. long. The banana of commerce, Musa sapientum, was believed to have originated in the tropics, and it was one of the earliest fruits cultivated by man. The fruit cluster requires 75 to 150 days to mature and must be removed from the plant to ripen properly. Chilled banana fruits do not soften normally; hence for best edibility, the fruit is kept well ventilated at room temperatures of 60° to 70°F. and a relative humidity of 90 to 95.

The most important commercial variety in the United States is Gros Michel, which is one of several hundred varieties grown throughout the world. The Chinese Dwarf or Cavendishi banana, Musa mana, is grown widely in some areas, and is considered the most resistant to cold and wind (77)*. The Gros Michel, Cavendish, and Plantain are used in dehydration. The Lady Finger usually has a hard core and is not as suitable as the other varieties (99).

Research reported by the United Fruit Company revealed (94) that the banana contains sucrase, amylase, protease, catalase and other enzymes. The proximate chemical composition of the fully ripe banana was: 75.6 percent moisture, 19.9 percent total sugar (4.5 percent

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*The numbers in parenthesis will be similarly numbered references in the Bibliography.

dextrose, 3.5 percent levulose, 11.9 percent sucrose), 1.2 percent starch, 0.6 percent crude fiber, 1.3 percent protein (N x 6.25), 0.6 percent fat, and 0.8 percent ash.

The pH of the fruit varies from 5.02 to 5.6 in green bananas, to from 4.20 to 4.75 in the ripe fruit, although these limits will vary. This comparatively high pH would seem to preclude any safe method of canning bananas, except by heat processing, or by addition of an acid to increase the hydrogen ion concentration (108). Hall (45) stated that the marked astringency of the green fruit disappears during ripening, the tannin content decreases by about four-fifths, a decrease in pH from 5.0-5.5 to 4.2-4.8 and a twofold increase in titratable acidity.

Pollan (89) pointed out that the banana contains only seventy-five percent moisture, the lowest such content of any of the common fresh fruits or vegetables, that the principal solid constituents of the banana are easily digestible fruit sugars of high energy value, i.e., 445 calories per pound, or two to four and one-half times that of most other common fruits. In addition to a small percentage of fiber, fat, and protein and eleven nutritionally important minerals, ripe bananas contain vitamins A and C and small amounts of the B vitamins, and have proved of outstanding value for infant feeding, and for feeding the sick and the aged.

Miller et al. (82) reported that the predominant nonvolatile organic acids of the Dwarf Cavendish banana are malic and citric acids. Hultin and Proctor (56, 57) identified acetic acid, methyl alcohol, ethyl

alcohol, methyl acetate, ethyl acetate, 2-hexenal, 2-pentanone, and tentatively, isoamyl alcohol, isoamyl acetate, and 2-octanone as volatile constituents of the banana. They also indicated that pyruvic acid, valine, and oleic acid were shown to be the banana aroma precusors.* Issenberg and Wick's (59) work showed the following substances to be present in the Gros Michel banana concentrates: 2-pentanose, i-butyl acetate, i-butanol, n-butanol, i-amyl acetate, i-amyl alcohol, trans-2-hexenal, and i-amyl butyrate. i-butyl acetate, i-butanol, and i-amyl butyrate had not previously been shown to be components of banana. Grosbois and Mazliak (45) reported finding the fatty acid component of 18 different acids, from C_6 to C_{22} in bananas, four of which were unsaturated. Palmitic acid was the dominant acid.

According to the United States Department of Agriculture Handbook No. 8 (114), the composition of banana is shown in the table. From the table, it is easy to see that freezing-dehydration concentrates the nutritive components of bananas about four times. Ascorbic acid is the only component showing a decrease.

B. FREEZING AND CELL INJURY

Freeze-drying, as the name implies, involves two operations of equal importance. To many, freezing has been only incidental to the process and may be carried out in any convenient manner. A developing

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*Certain compounds added to the puree extrace mixture increased the rate of odor production; they were defined as banana aroma precusors.

literature on the subject, however, indicates that the freezing step may be as important as - or possibly more important than - the drying step. The most common complaints about freeze-dried foods such as raw beef, fish, and pork are of stringy texture and have a lack of juiciness. Products such as fruit juices, which are high in sugars, salts, acids, and flavor components (esters, ketones, essential oils, etc.), represent more sensitive materials and require greater care in freezing and drying in order to retain these desired organoleptic properties. Two questions to be answered with regard to the freezing step are the relation between the freezing and the eutectic temperature.

Luyet (73) studied the mechanism of ice formation and propagation in muscle and described forms of crystallization in both muscle and muscle extract. Many of their conclusions assumed that Huxley's interpretation of muscle structure was correct. In freezing-rate studies on tissue sections, Meryman (79) concluded that the main cause of tissue injury was a concentration of solutes rather than mechanical damage by ice crystals. It was claimed that rapid freezing, e.g., in an acetone-dry ice mixture, gave a product after drying that rehydrated slowly and was tougher and drier than comparable samples frozen at -10° to -20°C. In a discussion of products produced by AFD freeze-drying in 1958, it was stated that the fine pore structure obtained on quick freezing reduced the rate of drying. This opinion is supported by the observation that meat frozen slowly dried faster than meat frozen rapidly. The eutectic temperature is that temperature at which crystalliza-

tion of an aqueous solution is complete, and further cooling does not introduce new structural changes. Eutectic temperatures are also important factors in product quality. If a material is frozen slowly, large crystals of ice form, cell salts become concentrated, and cellular materials may be exposed to these materials for relatively long periods (21).

Greaves (42) suggested that properties of the final dried material can be appreciably altered by the method of freezing. In summary it appears that smaller ice crystals should produce a more rapid drying rate initially; however, in materials where vapor flow does not control, the ice crystal size would have little effect once the ice line had receded to the interior.

It is believed that the freezing rate not only effects the size of ice crystals but also effects the subsequent rate of drying. According to Gane, slow freezing has an adverse effect on the quality of freeze-dried meat and fish (38). Greaves (42) pointed out that sensitive proteins are partly denatured by long contact with the increasingly concentrated liquid phase as ice is frozen out.

With regard to the effect of different freezing methods, Marion and Stadelman found that liquid, plate, and air blast freezing gave no significant difference in the texture and drip of chickens (74). Barrie et al. (9) revealed the same conclusion for blast-frozen and liquid-frozen turkeys.

Karel (66) noted the following operational variables and possible changes during freezing:



Factors	Effects
Rate	Protein Denaturation and Cross-Linking
	Redistribution of Soluble Components
Temperature	Emulsion Destablization

Deatherage and Hamm (27) found that quick freezing caused a smaller decrease in the water binding capacity of meat than did slow freezing. Lee et al. (72) found no significant differences in the texture of strawberries frozen rapidly, slowly, and at intermediate rates. Glycerol in relatively large concentrations, permitted slow freezing and thawing by virtue of its ability to prevent the freezing of most of the water present, thus reducing the degree of dehydration and electrolyte concentration. The beneficial effect of glycerol was thought to be due to its action as a "salt buffer" (76).

From a physiological point of view, the salient feature of freezing appears to be that ice crystal formation occurs mainly in the intercellular spaces of the tissues. The formation of ice crystals effects the concentration of solutes in the liquid phase surrounding the cell, especially the increase in the hydrogen-ion concentration. This increased concentration disturbs the osmotic equilibrium of the cell, causing the removal of water from the cell contents into the intercellular spaces to form ice and thus causing death of the cell. Also, a dehydration of the protoplasmic colloids

is effected which may cause irreversible changes in structure and function of the cell, in turn reflected in gross effects in tissue quality. This may be traceable to an increase in the viscosity of the protoplasm to a point at which the cell cannot function. Even though water is present in the tissue in both the free and bound state, there seems to be little difference between the behavior of the two during the freezing process. The change in the tissues as a result of freezing may be considered analogous to "salting out" of proteins, also a dehydration phenomenon (60).

According to the crystal damage theory, ice crystal growth impairs food quality generally. Slow freezing permits ice crystal growth. Fruits and vegetables that are fresh, crisp, and firm before freezing, become soft and mushy after being subjected to slow freezing. The cells of meat, poultry, fish, shellfish, fruits, and vegetables all contain jelly-like protoplasm. To fix the original jelly-like mass, the rate of freezing must be such that minute crystals are formed uniformally, throughout the tissues. If such quick frozen tissue is thawed immediately, the water is reabsorbed within the tissues as the ice crystals melt. If the product is frozen slowly, or fluctuating conditions of temperature during storage permit ice crystal growth, cells become punctured and the thawed tissues can not return to their original jelly-like state. As freezing progresses through a substrate, there is an increasing concentration of electrolytes which may cause irreversible changes in colloidal structures. Fresh fish flesh is gelatinous, and

if its temperature is lowered rapidly to -40°F., when thawed the flesh loses little tissue fluid. On the other hand, if slowly frozen, the flesh may lose fluid as "drip" when thawed. There is a point of electrolyte concentration if attained in the tissues which may cause irreversible changes in colloidal structure (28). The quick-freezing process rapidly slows down chemical, microbial, and enzymatic reactions. A similar effect is produced by sharp or slow freezing, but with less rapidity. The physical effects of freezing are of great importance. There is an expansion in volume of the frozen tissue. Ice crystals form and increase in size. The crystals usually are larger with slow freezing, and more ice accumulates between tissue cells than with quick freezing and may crush cells. Water is drawn from the cells to form such ice, with a resultant increase in the concentration of solutes in the unfrozen liquor, which in this way has a constantly dropping freezing point until a stable condition is reached. It is claimed that the ice crystals rupture tissue cells or even microorganisms, but some workers minimize the importance of such an effect. The increased concentration of solutes in the cells, hastens the salting out, dehydration, and denaturation of proteins, and causes irreversible changes in colloidal system, such as the syneresis of hydrophilic colloids. Further, it is thought to be responsible for the killing of microorganisms. As was stated previously, bacteria, and probably other microorganisms, die most rapidly in the range -1° to -5°C.

(30.2° to 23°F.). Therefore, more cells should be killed by a slow-freezing process than by a quick one, but at the same time there would be more undesirable physical change with slow freezing. The advantages claimed for quick freezing over slow freezing are that (1) smaller ice crystals are formed; hence there is less mechanical destruction of intact cells of foods; (2) there is a shorter period of solidification and therefore less time for diffusion of soluble materials and for separation of ice; (3) there is more prompt prevention of microbial growth; and (4) there is more rapid slowing of enzyme action. Quick-frozen foods, therefore, are supposed to thaw to a condition more like that of the original food than slow-frozen foods. This seems to be true for some foods, e.g., vegetables, but not necessary for all foods. Research on fish, for example, has indicated little advantage for quick freezing over slow freezing (38).

Fennema and Powrie (34) stated that fruit and vegetable tissues are damaged appreciably by freezing. The change of the state of water associated with the change in volume. Water at 0°C. and 1 atmosphere of pressure expands nearly 9 per cent transforming into ice. The delicate cells in many fruits are very susceptible to rupture during freezing. Extracellular ice crystals which form during slow freezing presumably have a lower vapor pressure than does water present at cell surfaces. This vapor pressure differential allows water to migrate out of the cell where it can be deposited on the ice crystals. The slower the

freezing rate, the greater the opportunity for water migration. Cell shrinkage and a decrease in the freezing point of protoplasm accompany cellular "dehydration," perhaps to the extent that intracellular freezing never occurs. Conversely, rapid freezing apparently provides little opportunity for cellular dehydration; as a result, intracellular crystallization occurs.

According to the damage caused by the solute concentration, water in a suspension of cells begins to freeze at some temperature below 0°C. As it freezes, pure ice crystals separate out of solution; concomitantly, the solutes in the residual liquid solution become increasingly concentrated. With a continued drop in temperature, more and more water is withdrawn from the solution and deposited as crystals. The solution eventually becomes saturated with solute and a further lowering of the temperature causes all the solutes to precipitate, permitting all of the water to freeze. If only one solute is present, complete solidification occurs at a specific temperature, know as the eutectic point. If many solutes are present, each precipitates out at its particular eutectic point. However, some liquid water remains until the temperature has dropped below that of the lowest eutectic point for the solutes present (70).

In general, both frozen and freeze-dried foods appear to be of better quality following rapid freezing which results in small ice crystal formation. However, the rehydration of a dried product is better following either very rapid or very slow freezing. Luyet

has related these observations to the size of air bubbles trapped within the tissue. Very small bubbles are rapidly absorbed, very large cracks and cavities permit the immediate escape of air and lead to improved rehydration while intermediate sized bubbles tend to impede the entry of water. The current theories on injury from freezing and its prevention can be summarized in terms of two controllable variables, rate of freezing, and protective additives. Cellular materials frequently appear to be susceptible to injury from excessively high freezing rates which cause intracellular crystal formation, presumably destructive on a mechanical basis. There is no evidence that high rates of freezing are in any way injurious to non-cellular materials. Injury can also be produced by slow freezing. Here the damage is probably of a chemical nature resulting from the concentration of solutes, particularly electrolytes. As freezing progresses, more water is removed from the solution to form ice, solutes become more concentrated, and the potential for injury is increased. Simultaneously, the lowering of temperature reduces the reaction rate of chemical processes. This explains the sequence frequently seen in which a decrease in temperature after crystallization begins producing an increasing rate of injury as more water is frozen out and the concentration of solutes is increased. Although the lowering of temperature also reduces reaction rates, the rate of concentration increase vastly exceeds the temperature effect, leading to a net increase in injury with decreasing temperatures. Ultimately, when most of the ice has been formed, further concentration

becomes negligible and continuing temperature reduction then produces a reduction in the rate of injury. Since injury from solute concentration can occur regardless of whether or not the material is cellular, the effect of slow freezing, and particularly, prolonged exposure to the frozen state at moderate temperatures where reaction rates are appreciable, always must be taken into consideration. Alternate theories include pH change and others which explain injury on the basis of abnormal chemical bonding or even precipitation of proteins as a result of the removal of water. In all theories of biochemical injury, dehydration is the causative agent.

C. PROTECTIVE CHEMICAL ADDITIVES

Chemical additives can, in certain instances, minimize the deterioration of food materials during freezing, storage, and thawing. The ones in common use are sugar, salt, sulfurous acid and its salts, ascorbic acid, other edible acids, and calcium compounds.

It was first discovered that glycerol provides remarkable protection for biological materials which must pass through the freezing, storage, and thawing process in a living state. The mechanism of its protective action is still not fully known, but some or all of its properties as listed below are thought to be involved:

1. Nontoxic to host cell or tissue at the concentrations used
2. Highly soluble in aqueous solution in the range of 40° to -40°C.

3. Ability to penetrate cell membranes rapidly

4. Low cryohydric point

5. Ability to hydrogen-bond with water

6. Strong tendency to form amorphous structures at low temperatures

7. Ability to decrease the crystallization velocity of ice.

The last six properties have the effect of minimizing the amount of water that changes into ice, thereby minimizing volume and the concentration of solutes changes. The addition of glycerol also dilutes the solutes, thereby decreasing the maximum concentration attained during freezing (50).

Vos and Kaalen (80) investigated the effectiveness of twenty-six different compounds as protective agents for the slow freezing of cells in tissue culture. Ethylene glycol, diethylene glycol, propylene glycol, glycerol, DMSO, pyridine N-oxide and hexamethylene tetramine provided the best protection. Acetamide, dimethylacetamide, formamide, dimethylformamide, monoacetine, D-mannose, D-ribose, glucose, D-mannitol, sorbitol and inositol gave the least protection. Little or no protection was observed with dimethyl sulfone, PVP or serum. The addition of methanol, ethanol, phenol, resorcinol or sodium chloride reduced survival below that of controls.

Approximately 50 million pounds of gelatin are produced annually in the United States, with roughly the same figure for the rest of the world. About 65 percent of this goes into edible gelatin for use in

desserts, marshmallows, candy, jellied meats, consomme, bakery products, ice cream, and dairy products. Gelatin is used in emulsion polymerizations as a protective colloid to prevent premature precipitation of either the monomer or the polymer emulsion. Gelatin also prevents the coalescence of the viscous cohesive and adhesive particles that exist as an intermediate form in many polymerizations (58). Food is claimed to be preserved by coating with an aqueous solution of propylene glycol, gelatin, and gum arabic(36).

Glicksman (40, 41) reviewed the properties and applications of the natural polysaccharide gums used by the food industry, and covered the common seaweed extracts (agar, alginates, carrageenan, furcellaran), tree exudates (gum arabic, gum tragacanth, gum karaya), and plant seed gums (locust bean gum, guar gum). He also reviewed the use of synthetic gums in foods. The review not only includes the well-established cellulose derivatives and their modifications but also investigates the potential utility of newly available nontoxic gums that the author feels will eventually find a place on the grocery shelf.

Dehydrated food, such as fruit juices, vegetable powders, and dry soup powders, are more easily reconstituted in water and have a better texture and more palatable properties if sodium carboxymethylcellulose is added prior to dehydration. The primary function of CMC is to control ice crystal size and thereby maintain a uniform, smooth texture. In addition, the excellent water-retention properties of

CMC under drastic freeze-thawing conditions have led to its use in other prepared frozen food products (40). The addition of CMC to foods prior to dehydration is said to improve the retention of natural flavors (65).

According to Greminger and Savage (44), low viscosity methylcellulose (15-25 cps) at a concentration of 0.5-1.0 percent, based upon the weight of solids in the dehydrated fruit or vegetable products, aid in redispersion and provide better consistency in the reconstituted product. The reversible thermal gel point, together with the viscosity and suspending properties, improves product performance.

The properties of methylcellulose were used in an improved process for preserving foods by dehydration and freezing. This process was claimed to have advantages whereby the treated food products substantially retained their initial shape and appearance after freezing, thawing or cooking (40).

Glicksman (40) reviewed that the freeze-drying processing of certain aqueous food extracts by increasing the eutectic point of the extracts through the addition of PVP or some other hydrophylic materials. In frozen dessert preparations, such as simulated ice cream, a substantial amount of Avicel can be incorporated to reduce the formation of ice crystals, resulting in an ice cream of superior texture stability and control overrun properties.

Starvis is an oxidized pre-gelatinized wheat starch. Wheat starch is a polysaccharide made up of anhydroglucose units. Starvis is an edible adhesive as well as an instantly wettable precooked

starch. As a result of its high water-holding capacity, it has the ability to increase the absorption of bakery mixes and to standardize their consistency from lot to lot. It also aids in stabilization of fat emulsions (54).

Guar gum was introduced into the United States by the U. S. Department of Agriculture in 1903. The source of guar gum is the seed of the guar plant, Cyamopsis tetragonolobus, family Leguminosae. Guar gum, like locust bean gum, is a galactomannan, but there are significant differences in their chemical structure and properties. Guar, structurally, is composed of a straight backbone chain of d-mannopyranose units with a side-branching unit of d-galactopyranose on every other unit. The molecular weight was said to be about 220,000. The greater branching of guar is believed to be responsible for a hydration easier than that of locust bean gum. The most important property of guar is its ability to hydrate rapidly in cold water to an extremely high viscosity. A concentration of about 0.3 percent guar gum has been suggested for use in ice cream mix. Guar gum is often used as a thickener in salad dressings, usually at 0.2 to 0.8 percent of total weight. Guar gum-starch combinations has been used to prevent frozen-pie fillings from dehydrating, shrinking, and cracking (39, 40).

Amos' (2) work showed the following stabilizers to be effective in the production of formulated freeze-dried strawberry slices: Hercules CMC; Valatex Gelatin; Pad Guar Gum; Kelgin MV; Kelcoloid

LVF, dispersed in ethanol; Supercol F; and low methoxyl pectin (with no calcium chloride added).

The primary purposes of stabilizers can therefore be summarized as follows: (1) to produce smoothness in body and texture; (2) to retard or reduce ice crystal growth during storage; (3) to provide uniformity of product; (4) to provide resistance to melting (40).

D. DEHYDRATION OF FOODSTUFFS

Preservation by drying is probably the oldest method employed by man to keep supplies of foodstuffs for long periods. Methods of accomplishing drying have evolved different parts of the world, according to local conditions and traditions. The term dehydration has assumed the meaning in the food industry of the process of water removal by artificial drying. The dried fruits of commerce may be produced either by sundrying or hot-air drying by means of a mechanically ventilated dehydrator. Hot air dehydration room was introduced about 1795. The team of Masson and Challet in France developed the first vegetable dehydrator which consisted of hot air (105°E) flowing over thin slices of vegetables (28).

Harpter and Tappel (49) reported that a good review of the early development of the process was given by Flosdorf in 1949. He conducted some of the early exploratory work in food freeze-drying also. The early development of the freezing-dehydration was used for preparation of biological substances. A strong impetus was given

to the process during World War II by the demand for dried blood plasma. The wartime conditions also led to interest in freeze-drying as a method of preparing dehydrated foods of high quality, and improved storage stability. Studies were conducted during this period both in the United States and in Great Britain on freeze-drying of meats, vegetables, and fruit juices.

During and after World War II, individuals and governments of several European countries became interested in freeze-drying as a food-preservation method. Much basic work on freeze-drying of food was sponsored by the Danish Government. The British Government officially opened the Aberdeen Research Establishment in 1951 and much of the fundamental and development work of the 1950's was performed at this station. Many innovations that placed freeze-drying in a more favorable cost position were developed at Aberdeen. Work at that station was reported in The Accelererated Freeze-Drying Method of Food Preservation (22).

In 1952, research sponsored by the Quartermaster Food and Container Institute for the Armed Forces on freeze-dried meats was begun in the Department of Food Technology, University of California. A large portion of the data and conclusions reported herein are based on information developed in the course of this research (49).

The application of the freeze-drying technique to commercial products is relatively new. In 1960, only two major food processors were marketing freeze-dried food in the United States. In 1964, there were at least twenty. It has been estimated that sales of freeze-dried food could reach two billion dollars by 1970 (22).

Government agencies such as the Quartermaster Corps, U. S. Department of Agriculture, British Ministry of Agriculture, Fisheries, and Food, and many others have large exploratory programs of their own and have sponsored to Corridon in July 1963, the U. S. Department of Agriculture published a bibliography of selected references on the freeze-drying of foods; their library list abstracts from some 638 articles or books, most of which have been published in the past five years (26).

E. FREEZE-DRYING

The early application of freeze-drying were laboratory procedures, particularly in histological techniques and the preservation of biological materials and micro-organisms. The first reported use of freeze-drying in the laboratory was in 1890, when pieces of frozen tissue in a vacuum desiccator at -20°C. was dried for the preparation of histological sections. In 1935, the first real interest in the commercial uses of freeze-drying dates was reported; it described the first practical design of a cold trap cooled by methyl cellosolve containing dry ice. They also provided for the rapid freezing of the specimen in the same low temperature bath used to cool the condenser. Their analysis placed the process for the first time on a sound scientific and engineering basis and made the various parameters concerned with vapor pressure, vacuum, relative temperature and effects of mechanical obstruction to vapor flow self-evident. In 1949, the first book on freeze-drying was published. In 1951, the first scientific symposium on the subject of freezing and drying was held in

London under the sponsorship of the Institute of Biology. By this time, it was apparent that the importance of freeze-drying as both an industrial tool and a subject for laboratory investigation was well recognized. In 1954 "Biological Applications of Freezing and Drying" appeared, and in 1958 the second Institute of Biology Symposium was held in London (50, 70). In 1958 the first of a series of biennial courses on freeze-drying was held at Lyon, France, under the joint auspices of the International Institute of Refrigeration and the Institute of Applied Science, under the directorship of Professor Louis Rey. Three volumes containing lectures presented at the four courses held between 1958 and 1964 have been published. In 1959 a symposium on freezing and drying was sponsored by the New York Academy of Sciences (79).

There are four principal stages of the freeze-drying process during which injury can occur: during the initial freezing, during the drying, during the subsequent storage and on reconstitution (79).

Dehydration has long been employed as a means of food preservation but, in general, the quality and reconstitution has not been very satisfactory. Freeze drying represents a major improvement, both in quality and reconstitution. This is due to the fact that the moisture is removed as vapour directly from frozen material, leaving the spatial distribution of solubles and the basic structure unaltered (28).

To freeze drying at an economic rate, the operation must be carried out at pressures of about 1 mm Hg (1000 μ) or less, and the temperature of the frozen material must be kept near 0°C. An efficient

method of transferring the heat of sublimation is necessary. In order to maintain the vacuum, a pumping system capable of handling very large quantities of water vapour is essential. Of the various pumping systems in general use, only steam ejectors or mechanical pumps and vapour condensers are suitable. The transfer of sufficient heat to ensure rapid sublimation can be achieved either by radiation or direct conduction to both top and bottom surfaces of the product. Maximum heat transfer is by double contact, which is the basis of accelerated freeze-drying. Various types of plants are available for production, ranging from single batch cabinets, processing about one ton of raw material per day, to continuous plants, capable of producing from 15 to 25 tons per day in one tunnel (22).

Luyet (73) described freeze-drying as a three stage process. The first stage, freezing, results in a separation of water from the tissue in the form of ice crystals; from the standpoint of the tissue this separation is, in reality, dehydration. In the second stage, ice crystals are caused to subline; this represents removal of water which has already been separated from the tissue and, in a strict physical sense, is not dehydration. The third stage is characterized by evaporation of residual water. Since residual water is from noncrystalline sources, its removal cannot be regarded as true freeze-drying. In specific cases shrinkage or collapse of structural components is associated with the withdrawal of this unfrozen or residual water.

Meryman (79) stated that the freeze-drying process can be subdivided into four distinct stages. The first of these is the transfer

of heat to the drying boundary. The second is the conversion of ice to water vapour at the surface of the ice crystal. The third stage is the transfer of this water vapour through the already dried shell of the specimen to the specimen surface. The fourth stage is the removal of this water vapour from the specimen surface.

According to Kuprianoff (70), on the basis of the biological and biophysical consideration, the following steps appear to be of specific importance in attempting to minimize product damage resulting from the freeze-drying process:

(1) With materials having structure, freeze before starting to withdraw water vapor; if foaming is wanted, freezing should be done under vacuum.

(2) Dry at a sufficiently low temperature until all free water is removed, only then may the temperature be raised carefully.

(3) To decrease the denaturation, withdraw water until a concentration of less than 20 per cent, relative to protein content, is reached. A value below 15 per cent may be better.

(4) Avoid marked differences in water content in different zones of the product. Since overdrying and insufficient drying are both deleterious, uniform drying to an optimal water content, even at microscopical levels, are of great importance.

(5) Storage with a water content of not more than 15 per cent; the optimal value must be determined for each item.

Karel (66) pointed out that operational parameters and changes during dehydration process are as follows:



Factors	Effects
Time	Protein-denaturation and cross-linking
Temperature	Crystallization of poly-saccharides
Total pressure and oxygen pressure	Loss of volatile components
Final water content	

F. FOOD TEXTURE

Texture has been defined by a committee of Institute of Food Technologists in terms of those properties apprehended by the eyes and by the skin and muscles of the mouth. Scott Blair (93) classified objective methods of texture measurement under the heading: fundamental, empirical, and imitative. Matz (75) defined the texture as the mingled experience deriving from the sensations of the skin in the mouth after ingestion of a food or beverage. It relates to density, viscosity, surface tension and other physical properties of the material being sampled. Kramer and Twigg (69) classified texture characteristics as finger feel (firmness, softness, or yielding quality) and juiciness.

Szczesniak (103, 104, 105) classified the texture characteristics of foods into mechanical and geometrical qualities and into properties related to the moisture and fat content. The primary parameters of the mechanical characteristics are hardness, cohesiveness, viscosity, elasticity, and adhesiveness; the secondary parameters were brittleness, chewiness, and gumminess. Texture can be regarded as a manifestation of the rheological properties of a food. It is an important attribute

in that it influences food habits, affects consumer preference of accepted foods, affects processing and handling; influences oral health and is often taken as a sign of spoilage or wholesomeness. Research indicates that the consumer is highly conscious of food texture and that in certain foods texture may be even more important than flavor. Objective measurements can be classified as fundamental, empirical, and imitative. Fundamental tests measure fundamental rheological properties, and relate the nature of the tested product to two basic rheological prototypes: a dashpot for a Newtonian liquid and a metal spring for a Hookean solid. The dashpots represent elastic moduli. The springs represent viscosities. Empirical tests measure characteristics related to textural quality. Instruments used to perform such tests include: penetrometers, consistometers, shearing devices. Imitative tests, such as, Massachusetts Institute of Technology denture tenderometer and the General Foods Texturometer. All texture measuring devices have five essential elements: driving mechanism; probe element in contact with food; force-direction, type, and rate of application; sensing element; and read-out system.

Degree of hardness, cohesiveness, and moisture content are associated most often with foods. Most people have a limited vocabulary with which to describe texture. "Crisp," "dry," or "juicy," "soft," "creamy," "crunchy," "chewy," "smooth," "stringy," "hard," are terms most often used (61).

Amerine et al. (1) classified foods into four groups: 1) liquids; 2) fruits and vegetables; 3) meat; and 4) other foods -- and described instruments that are used on each group.

Measurement of texture may be classified into two categories: 1) subjective measurement of texture; and 2) objective measurement of texture. The former belongs in the field of sensory evaluation; both ordinary sensory testing panels and special panels of experts have been used for the subjective evaluation of texture. The latter according to Bourne (17) can be considered to be divided into 9 groups based on the variable (or variables) that constitute the basis of the measurement. Group 1: force-measuring instruments. This is certainly the most common method used for measuring food texture. The measured variable is force, usually maximum force, and distance and time are held constant or at least replicatable. The force measurement has the dimensions mlt^{-2} (mass, length, $time^{-2}$). Examples of this instrument are the tenderometer, Magness-Taylor Pressure Tester, Warner-Bratzler Shear, Maturometer, Shear Press, Christel Texture Meter, and the Bloom Gelometer. Group 2: distance-measuring instruments. Examples: Penetrometers, Grawemeyer Consistometer, Seed Displacement, Succulometer. Group 3: time-measuring instruments, Ostwald Viscometer, Consistometer. Group 4: energy-measuring instruments such as Farinograph, meat grinder. Group 5: ratio-measuring methods. Group 6: multiple-measuring instruments. Examples of these instruments are the General Foods Texturometer, the Massachusetts Institute of Technology Denture Tenderometer and the Instron Universal Testing Machine. Group 7: multiple-variable instruments. These are instru-

ments in which more than one uncontrolled variable is present. An example of this is the Durometer. Group 8: chemical analysis. Probably the best known chemical measurement of food texture is the alcohol-insoluble solids test. Group 9: miscellaneous group and includes those devices and methods that cannot be classified under any of the other groupings above, such as Succulometer, alcohol insoluble-solids (they are indirect measurement of texture).

G. REHYDRATION

The term of rehydration refers to total water uptake by a product. Reconstitution refers to the actual water binding capacity to such extent that the product more closely resembles the original material (27).

In 1944 a study of rehydration described in the U. S. Department of Agriculture's Dehydration Manual was designed primarily to standardize the procedure for testing the rehydration properties of dehydrated vegetables. A rehydration test always comprises determination of the drained weight of a rehydrated sample, using specified procedures both for the rehydration by soaking or boiling and for the drained weight determination. The "rehydration ratio" is defined simply as the ratio of the drained weight after this treatment to the weight of the original sample (111).

Conditions of rehydration of importance to the final quality of the product include: (1) time; (2) temperature; (3) air displacement;

(4) pH; and (5) ionic strength. In 1960, the study of the rehydration characteristics of precooked ground beef and found optimal rehydration when the rehydration fluid was maintained at 34°F. and the rehydration ratio decreased with increasing temperature studied. Hanson (48) reported that rehydration of most freeze dried foods of plant origin can be accomplished satisfactorily in boiling water. The most important changes which can occur during rehydration are: changes in proteins, redistribution of soluble components and enzymatically caused changes. The pH and ionic strength of the water used for rehydration are important in rehydration of proteinaceous foods. This is due to the poly-electrolyte nature of the proteins, the structure of which, and therefore the water binding capacity, depends on electrochemical properties of rehydrating fluids. Hamm and Deatherage (47) observed that the water holding capacity of freeze dried beef was at a minimum at the isoelectric point of beef proteins. Lower or higher pH levels improved the water holding capacity. It was noted that rehydration of freeze-dried meats could be improved by rehydration in a solution containing 0.2 M NaCl, 0.01 M KCl and 0.05 M Na-ascorbate, without adversely affecting flavor. Rehydration of beef can be improved also by rehydration in water containing one percent table salt, without other additives. Low pH has been suggested as a means for improving the texture of rehydrated dried potatoes (44, 57).

Loss of water holding capacity is characteristic of deterioration of freeze-dried foods. It is usually associated with a decrease in organoleptic acceptability due to lack of "juiciness" and increase in "toughness". Karel (66) indicated that some of the possible mechanisms resulting in decreased water-holding capacity of foods are:

Cellulose, Starch —(Removal of Water; Hydrogen bonding)→ Increase in crystallinity; Increase in cross-linking

Proteins (Actomyosin) —(Removal of Water; Hydrogen Bonding, Salt Bridges)→ Cross-Link Protein Structure

Proteins —(Temperature; High Salt Concentration)→ Denaturation —(Removal of Water)→ Cross-Link Denatured Protein

The loss of water-holding capacity is generally attributed to an increased degree of association of food polymers which are responsible for the ability of food to hold water in a tight gel structure. In plant material these effects may be correlated with an increase in the crystallinity has been shown to be associated with the very nature of dehydration, that is, the removal of water which normally maintains a separation between the individual chains, thus preventing their cross-linking. The major mechanism of this increase in crystallinity in starch and in cellulose is thought to be hydrogen bonding.

Luyet (73) reported the following general conclusions on the mechanism of rehydration of freeze-dried muscle:

1. The process consists of two stages: (a) the penetration of water through the cavities in the tissues, either the large ones formed between the fibers in slow freezing or the minute ones formed within the fibers in rapid freezing; (b) the penetration of water through the solid parts of the tissues; either when the entire fibers consist of compact material as after slow freezing, or when only the framework of porous fibers constitutes the solid parts as after rapid freezing.

2. In both the slowly and the rapidly frozen tissues, under the conditions reported, water seems to penetrate more freely through the solid parts than through the cavities.

3. The mechanism by which water readily penetrates single fibers in the diameterial direction, and does not seem to be able to enter them longitudinally, calls for further inquiry.

CHAPTER III

MATERIALS AND METHODS

A. MATERIAL UTILIZED

1. Samples

Frozen banana puree, canned banana puree, and fresh bananas were the basic ingredients used as variants in this experiment. Frozen banana puree (made by the United Foods, Inc., Houston, Texas) was supplied by Winter Garden Corp., Knoxville, Tennessee, which was received on May 8, 1967. Kraft canned mashed banana (Kraft Foods, Dist., Div. National Dairy Products Corp., Chicago 90, Illinois) and fresh bananas from local super markets were used. Upon being received, the canned puree was placed in storage at room temperature or cool place until processed. The frozen banana puree was stored in an air-blast freezer room until processed. The fresh bananas were peeled and sliced with a sharp, stainless steel knife, then frozen and stored in the air-blast freezer until processed. The fresh bananas purchased at a super market were Chiquita Brand from Costa Rica.

2. Firming agents

In the preliminary experiment, Dow Company's MC and HG; Dycol Guar Gum EHV; Swift and Company's Pad Guar Gum HVI; Meer Gum Guar MM; Hercules CMC, grade 7HF; Du Pont CMC P-75-H; Hercules' Starvis, Lot 5087; soy protein binder; wheat flour protein binder; Fisher Science

Company's calcium chloride; Will Corporation's gelatin; and Kraystay were used. The 1.0 percent level by weight of banana puree of each firming agent was made for measuring the effect of types of the firming agents. The selection of some agents would be based on observations of firmness or disintegration of the freeze-dried banana after rehydration.

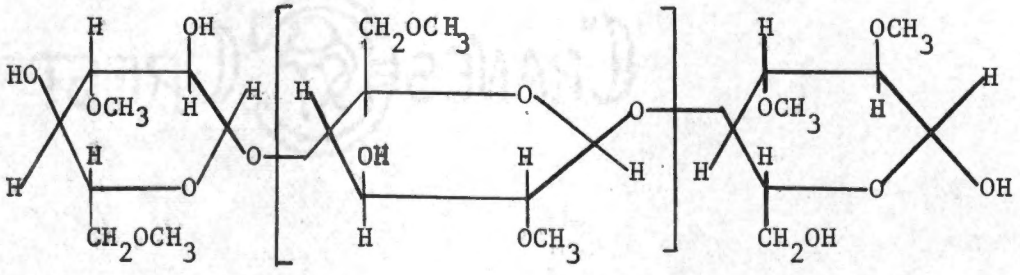
After the preliminary experiement, the selected firming agents were used for further work to determine the effect of levels among these firming agents on the quality of freeze-dried banana.

Firming agents thus chosen for extensive study were as follows:

a. Guar Gum. According to Dycol Chemical, Inc., Dycol guar gum EHV is a highly purified natural colloid for viscosity builder, stabilizer, suspending agent and binder in a wide variety of food products. Hy-Test Guar Dycol EHV is a cream white, free-flowing powder; 8.39 percent moisture content, 5.38 percent protein, 1.66 percent crude fibre, 0.79 percent ash, 1.29 percent ether extract, 82.49 percent of gum content, and has a pH of 5.6. It exhibits unusually high viscosities and fast swelling properties. After only 10 minutes of rehydration in cold water, Hy-Test guar yields 70 to 80 percent of its maximum viscosity. Guar gum has been declared by FDA to be generally recognized as safe (GRAS) under Section 121.101 of the Food Additives Regulations and is included as an optional ingredient in the standards of identity for various frozen deserts, cheeses, and salad dressings (40).

b. Methylcellulose and hydroxypropylmethylcellulose (MC). They are derived from and have the polymeric backbone of cellulose, a natural

carbohydrate that contains a basic repeating structure of anhydroglucose units. The basic structure for methocel MC methylcellulose may be shown as follows:



Cellulose fibers, obtained from cotton linters or wood pulp, are swelled by a caustic soda solution to produce alkali cellulose which is treated with methyl chloride, yielding the methyl ether of cellulose. The fibrous reaction product is purified and ground to a fine, uniform powder or granule, a Methocel MC product. The reaction may be represented as:

$$R\text{-}OH + NaOH \rightarrow R\text{-}ONa + H_2O$$

$$R\text{-}ONa + CH_3Cl \rightarrow R\text{-}OCH_3 + NaCl$$

In addition to this methoxyl substitution, the further reaction with propylene oxide will also give a hydroxypropyl substitution, thus yielding mixed ethers of cellulose; in the manufacture of Methocel HG products, propylene oxide is used in addition to methyl chloride to obtain hydroxypropoxyl substitution on the anhydroglucose units. This substituent group ($-OCH_2CHOHCH_3$) contains a secondary hydroxyl on the number two carbon, and may also be considered to form a propylene glucol ether of cellulose:

$$R\text{-}ONa + H_2C\text{-}CH\text{-}CH_3 \rightarrow R\text{-}OCH_2\text{-}CH\text{-}CH_3$$

(O bridge; OH)

Methylcellulose is nonionic, and therefore is not affected by salts or inorganic ions. The number of substituent group on the ring determines the properties of the various Methocel products. Methocel MC contains 27.5 to 31.5 percent methoxyl or a methoxyl D.S. of 1.64 to 1.92, this range yielding maximum water solubility. A lower degree of substitution gives products soluble only in alkali while higher degrees of substitution produce methylcellulose products soluble only in organic solvents. In Methocel HG products, the methoxyl substitution is still the major constituent comprising 84 to 93 percent of the groups attached to the ring.

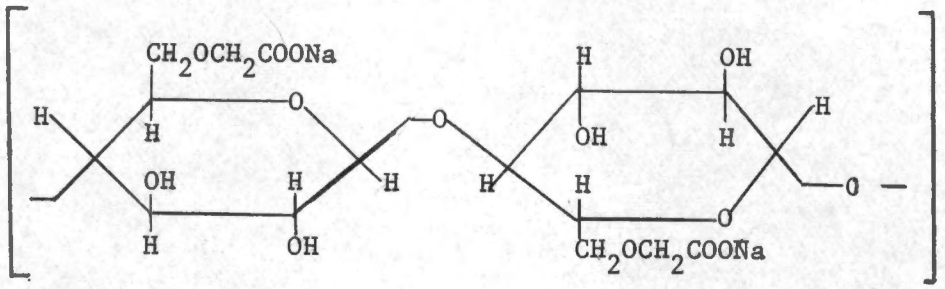
The methylcellulose products (Methocel, MC, U.S.P. series) have been approved by the FDA, and appears on the GRAS list as methylcellulose U.S.P. having a methoxyl content not less than 27.5 percent and not more than 31.5 percent on a dry-weight basis. Hydroxypropylmethylcellulose (methocel HG series) also is permitted as a food additive for an emulsifier, film former, protective colloid, stabilizer, suspending agent, or thickener in accordance with good manufacturing practice. Methylcellulose products have been used in dehydrated foods, frozen foods, ice cream and ice pops, milk stabilizer, pie fillings, preservative food coatings, etc. (28, 40).

c. Sodium carboxymethylcellulose (sodium CMC or cellulose gum). It is a cellulose ether produced by reacting alkali cellulose with sodium monochloroacetate under rigidly controlled conditions. The reaction may be represented as:

$$R\text{-}OH + NaOH \rightarrow R\text{-}ONa + H_2O$$

$$R\text{-}ONa + Cl\text{-}CH_2\text{-}COONa \rightarrow R\text{-}O\text{-}CH_2\text{-}COONa + NaCl$$

where R represents the cellulose structure. As with most chemical reactions, a side reaction competes for the basic raw material (sodium monochloroacetate is converted to sodium glycolate, $HOCH_2COONa$). The principal reaction by-products are sodium chloride and sodium glycolate. The structure of sodium CMC may be expressed as a chain composed of repeating cellobiose units, these are in turn composed of two anhydroglucose units. Each anhydroglucose unit contains three hydroxyl groups capable of reaction. By substituting carboxymethyl groups for some of the hydrogens of these hydroxyl groups, sodium carboxymethyl cellulose is obtained. The most widely used types of Hercules Cellulose gum have a degree of substitution of 0.7. This figure means that an average of 7 carboxymethyl groups are substituted per 10 anhydroglucose units. Higher D.S. yield products which exhibit improved compatibility with other soluble components. Cellulose gum are long-chain polymers. Their solution characteristics are dependent upon the average chain length or degree of polymerization as well as upon the degree of substitution. The average chain length and the degree of substitution determine the molecular weight of the polymer. As the molecular weight increases, the viscosity of cellulose gum solutions increases rapidly. The chemical structure of sodium CMC may be shown as follows:



The example shown has a D.S. of 1.0.

Sodium CMC is an anionic, water-soluble polymer derived from cellulose. First used as a substitute for natural gums, sodium CMC has developed markets based on its own unique properties. This protective colloid with its physiological inertness, ready solubility and film-forming ability can thicken, suspend, disperse, resist grease and stabilize. These properties suit it for a broad range of industrial and consumer applications in the food, pharmaceutical, cosmetic, laundry, textile, paper and many other industries. Cellulose gum is classified under "substances that are generally recognized as safe" (Gras) by Section 121.101, of the Food and Drug Regulations. The FDA defines cellulose gum as the sodium salt of carboxymethylcellulose, not less than 99.5 percent on a dry-weight basis, with a maximum substitution of 0.95 carboxymethyl groups per anhydroglucose unit, and with a minimum viscosity of 25 centipoises in a 2 percent by weight aqueous solution at 25°C. Definition and standard of identity Section 20.1. Ice cream -- Cellulose gum is included as an optional ingredient at a level not greater than 0.5 percent by weight of the finished ice cream (23, 29, 41, 53).

d. Kraystay. Kraystay is a purified, colloidal stabilizer obtained from the seaweed, Irish moss. It is an anionic polysaccharide with high power to modify positively charged colloidal systems. Kraystay Type K was made of carrageenan and of vegetable monoglycerides and diglycerides. It was dispersible in cold water, resulting in solutions of substantial viscosity (40).

B. DESIGN OF STUDY

1. Panel Test

Randomized Complete-Block Design (1, 97) was used for measuring the effects of kinds and levels of firming agents by panel test.

The experimental units can be meaningfully grouped in this design, the number of units in a group being equal to the number of treatments or some multiple of it (such a group is called a block or replicate). The object of grouping is to have the units in a block as uniform as possible so that observed differences will be largely due to treatments. Variability among units in different blocks will be greater, on the average, than variability among units in the same block if no treatments were applied. Clear variability among blocks does not affect differences among treatment means since each treatment appears in every block. Variation among blocks is arithmetically removed from experimental error.

In sensory comparisons where several judges at a single sitting taste and score each of various samples of a food product, the arrangement of scores is known as a randomized complete-block design. Each score (measurement) is fixed in the design; it belongs to one of the samples (treatments) and to one of the judges (blocks). It is, therefore, classified according to two criteria. To analyze such a design, the sum of squares as well as that between samples must be subtracted from the total to obtain the error (sometimes called inter-

action) sum of squares. The design is indicated in Table I, where k judges scored n products.

The mathematical model applicable to this design is $X_{ij} = m + \beta_i + t_j + e_{ij}$, where X_{ij} is the score of the i^{th} judge (block) assigned to the j^{th} product (treatment); m is the overall mean; β_i is the judge effect; t_j is the product effect; and e_{ij} is the residual error.

In such an analysis the total sum of squares based upon variation between judges; a sum of squares based upon the variation between products; and a residual sum of squares which is not the result of variation between judges or products. This last sum of squares is the remainder, or error, sum of squares. It is a measure of the unexplained variation in the experiment. The computations for this design are shown in Table I. From these sums of squares and degrees of freedom, three estimates of the population variance can be computed and the F-test applied. The Duncan's Multiple Range test was used to determine which product mean scores are significantly different.

a. Difference-preference test (1, 69). This was designed for measuring the effect between two types of puree samples. Two samples were compared 80 times by tasters. The Chi-square test was employed for interpretation of the difference-preference test.

Difference measurement: on the basis of the null hypothesis H_o: there is no difference between the puree samples--the probability, p, of a taster identifying by chance a particular sample in each of the several trials is one-half.

TABLE I

RANDOMIZED COMPLETE-BLOCK DESIGN

Judges (Blocks)[a]	Product (treatments)[b] 1	2	3	n	Totals[c]
1	X_{11}	X_{12}	X_{13}	X_{1n}	B_1
2	X_{21}	X_{22}	X_{23}	X_{2n}	B_2
3	X_{31}	X_{32}	X_{33}	X_{3n}	B_3
.	.	.	.	.	.
.	.	.	.	.	.
k	X_{k1}	X_{k2}	X_{k3}	X_{kn}	B_k
Totals	T_1	T_2	T_3	T_n	$G=\Sigma T=\Sigma B$

[a]The sum of squares and degree of freedom of Judges are $\sum_{i=1}^{k} B^2 i/n$-C.T. and k-1, respectively.

[b]Similarly, those terms for Products are $\sum_{j=1}^{n} T^2 j$-C.T. and n-1.

[c]Those for Total are $\sum_{i=1}^{k} \sum_{j=1}^{n} X^2_{ij}$-C.T. and nk-1.

Preference measurement: the null hypothesis H_o: there is no difference in preference between two puree samples by the group of tasters in taste test.

Let n represent the total number of trials, X_1 be the number of opinions favorable to F-D banana made from canned puree samples, and X_2 be the number of opinions favorable to the F-D banana of frozen puree sample. Then

$$X^2 = \frac{(|x_1-x_2|-1)^2}{n}$$

where $|x_1-x_2|$ indicates the absolute or positive value of the difference, and X^2 is based on one degree of freedom.

b. 4 x 6 Factorial design for computing the shear measurement. The mathematical model applicable to this design is $X_{ijk} = \mu + R_i + C_j + L_k + (CL)_{jk} + E_{ijk}$, where X_{ijk} is the quality indication-shear value of the i^{th} replicate assigned to the j^{th} chemical and k^{th} level; μ is the overall mean; R_i is the replicational effect; C_j is the chemical effect; L_k is the level effect; $(CL)_{jk}$ is interaction (or joined effect) by chemical and level; and E_{ijk} is the residual error.

In such an analysis (of variance) the total sum of squares based on variation between replicates; a sum of squares based on the variation among chemicals; a sum of squares based on the variation among levels; a sum of squares based on the i interaction between chemical and level; and a residual sum of squares which is the remainder, or error, sum of squares. It is a measure of the unexplained variation in the experiment. From these sums of squares and degrees of freedom, five estimates of the population variance can be computed and the F-test applied.

The Duncan's Multiple Range test was used to determine which product mean scores was significantly different.

2. Method of Preparation

The banana puree (frozen puree) was allowed to thaw for one hour at room temperature so that the experimental mixtures could be comminuted in a mixer (Sunbeam Mixmaster) with a capacity of three pounds (Sunbeam Mixmaster, Heat Resistant Bowl). Each experimental batch contained three elements: banana puree (two pounds or 907.2 g.); water (from 200 ml. to 700 ml. per batch to completely rehydrated the firming agents); firming agents (1.0 percent for measuring effect between two types of puree bananas and five levels of 1-5 percent for measuring effect of among kinds and levels) based upon the wet weight of banana puree.

A Sunbeam Mixmaster Mixer having 10 speeds and two blades, was selected in order to obtain good mixing and to prevent foaming of the product. The firming agents were added to the water in the mixer little by little until a well-rehydrated viscous solution was obtained. Then the banana puree sample was added. The mixer was set at the 2nd speed for four to fifteen minutes, and the puree formed was then poured into cellulose weiner casings with a 26 mm. diameter. The batch size used gave a rope of puree about five feet long. Each filled casing was hung vertically for 1/2 to 1 hour at room temperature to allow air bubbles to rise to the top. The filled casings of puree were frozen in an air-blast freezer at -30°F. and left there until slicing for freeze-drying. Each treatment was replicated two times.

The filled casings of puree were removed from the air-blast freezer, and all subsequent work prior to freeze-drying was done inside a 0°F. still-air freezer. The filled casing was sliced on a meat-type bandsaw with a new blade (BIRO, Model 22, The BIRO Mfg. Co., Marblehead, Ohio). Then the weiner casings were removed with a sharp knife. The slice-thickness guide was set at one cm. All the frozen banana puree slices were placed on a hardware cloth tray 15 by 15 cm. In order to get a uniform heat supply, only one layer of slices was placed on the drying shelf, since the uniformity of drying a load was desirable goal.

The freeze-dried untreated banana puree slices were taken as control. The subsequent experiments were conducted as follows:

Experiment one: the freeze-drying condition and estimation of freeze-drying time

Experiment two: moisture content of the puree and of the banana product

Experiment three: selection of banana puree sample

Experiment four: selection of possible firming agents

Experiment five: effects of kinds and amount of firming agents

Experiment six: rehydration

Experiment seven: subjective quality measurement

Experiment eight: objective quality measurement.

C. FREEZING

Prior to the dehydration operation, bananas were frozen by the following method: Air-blast freezer, sliced fresh bananas and filled casings of banana puree were frozen on a tray in an air-blast freezer (-30°F.).

D. FREEZING-DEHYDRATION

Freeze-drying, the sublimation of water from a frozen food by means of a vacuum plus heat applied at the drying shelf, was used. The temperature must, of course, be kept low enough to avoid melting any ice phase present. The rate of sublimation is greatly increased if the material is held under a vacuum, preferably with the total pressure well below the vapor pressure of the ice. In order to obtain maximum rates of drying it is also necessary to provide special means for supplying the latent heat of sublimation. Heat transfer, or more exactly energy supply, is important since each pound of water sublimed absorbs approximately 1200 Btu of energy, which must come from some external energy supply (22).

Del-Vac Freeze-Dryer, Model 11212 RVM, made by the American Sterilizer Company, was used in the freeze-drying operation. The area of the drying shelf was 12 x 12 sq. in., and the depth of the chamber was 6 in. A freon 22 coil was used as a condenser to freeze out water vapor. A vacuum pump for low pressures in freeze-drying was used to maintain a low partial pressure of water vapor at the surface of the product.

Frozen slices prepared were dehydrated in a freeze-drying unit (Del-Vac Freeze-dryer, Model 11212 RVM), a vacuum was applied and maintained at -30 psi. The surface of the shelf was covered with a layer of thin aluminum foil, and the hardware cloth trays were put on it.

When the trays of banana samples to be freeze-dried were placed in the chamber, the platen temperature was approximately 10°F. The temperature of sample was -20°F. After the trays were placed on the shelf, a vacuum was applied, and the thermostat controlling the platen temperature turned on mark 150. After four hours the thermostat was turned down on mark 110 for the rest of the drying time.

After completion of dehydration, the banana slices were stored promptly in glass jars with a desicant.

E. ESTIMATION OF PROCESS TIME AND END POINT DETERMINATION

The freeze-drying process is characterized by relatively high costs and a long final drying phase, the latter due mainly to the difficulty in supplying heat at the interface of ice with a dry layer. It is therefore desirable to stop the process when the moisture level is optimum, not drying unnecessarily (20).

Kan (64) pointed out that six methods may be used in determining the freeze-drying process end point. These are mass transfer, thermocouple measurements, and vapor pressure rise, vapor sample condensing, farvitron, infrared detection of product surface temperature. Burke and Decareau (22) stated that particle temperature method (thermocouple

and infrared methods were included), particle weight loss, particle density change, product moisture measurement (β-particle transmission and hydrogen recoil, NMR, capacitance, microwave, conductivity, X-ray), vapor moisture measurement (infrared, acoustic analysis, aluminum oxide, β-particle, absorption, ionization gauge, thermal conductivity, cold trap), and vapor-pressure rise methods were freeze-drying end-point determination methods.

Thermocouple measurements were used in determining freeze-drying process end-point in this study. Thermocouples were inserted in three places: on the platen, inside a banana slice, and on the surface of a banana slice. This procedure measured the internal temperature of the freeze-dryer and the end-point temperature of the drying cycle in the product. When the product temperature approximated shelf temperature, this indicated in most cases, that the product was dry. The product was allowed to remain two hours longer as a safety factor. The process end-point was taken to be that time when the product temperature reached some predetermined point.

F. REHYDRATION AND ITS CONDITIONING

The freeze-dried banana slices were rehydrated in different liquids (from 50° to 55°F.) at 76°F. room temperature and a relative humidity of 79 percent. Approximately 10 grams sample was put into the rehydration medium. Floating of the product during the rehydration was prevented so that all of the solid material remained in contact

with the liquid. After rehydration to a specific time, the slices were removed from the liquid, and drained on a No. 8 screen, at an angle between 15° to 25° to facilitate complete drainage from the screen. The banana was weighed on a Mettler balance.

Effect of media on weight measurement after rehydration:

1. Rehydration with tap water, pH = 6.8

2. Rehydration carried out with milk (Pet, grade A, pasteurized, homogenized milk was used). The milk had a pH of 6.7

3. Rehydration carried out with orange juice, pH 3.7.

The results are expressed as follows:

(a) Rehydration rate in different media

(b) Rehydration ratio, sometimes referred to as rehydration factor or index, may be expressed as

$$\text{Rehydration ratio} = \frac{\text{Wt. of rehydrated product}}{\text{Wt. of dehydrated product}}$$

$$\text{(c) Degree of Rehydration} = \frac{\text{g. of water uptake by F-D banana}}{\text{g. of dry wt. of F-D banana}}$$

(d) Coefficient of Restoration =

$$\frac{\text{Moisture content of the rehydrated sample} + 1}{\text{Average moisture content of fresh foodstuff} + 1}$$

$$\text{(e) Drying Ratio} = \frac{\text{Rehydration ratio} \times 100}{\text{Coefficient of restoration of weight.}}$$

G. SENSORY METHOD OF EVALUATION

1. Taste Panel Test (1, 69, 78, 98)

Quality evaluation made by a group of 8 panelists by the repetition of the tests enough times to get a sufficient number of judgements.

Coding: combinations of numbers and letters were used (i.e., ME-5, LV-7, IN-3, etc.) in coding of the samples as to avoid giving information to the panel. In the difference-preference test, only one letter was randomizely selected (no A and Z letters).

Sample size and utensils: one slice of F-D banana of each treatment was provided to each panelist. 3/4 oz. squat souffle cups, beakers, timer, spoon, etc. were used.

Condition or environment: tasting tests were performed from 2:30 p.m. to 4:30 p.m. in a sequence of days. Regularity, quietness, comfortable surroundings, and air-conditioning were controlled in the test room.

Each replicate of each treatment was tested once. Two replicates of every treatment were tested (i.e., 1 sample x 8 panelists x 2 replicates in the same testing = 16 observations). 80 observations were obtained for both difference and preference tests. 16 observations of every sample were made for texture and hedonic rating test.

The panel was told to consider the texture evaluation after 3 minutes rehydration. Both rehydrated product and rehydration medium

(tap water) in a beaker were observed; score consisted of 20 percent as judged or compared by degree of disintegration in water, and 80 percent as sensed by mouth fell.

2. Scoring and Rating System (1, 78, 102)

A scoring method was used because of its simplicity, and ease of statistical analysis. A 7-point scoring scale was used. An evaluation sheet, illustrating the scoring method for judging quality factors, was used in the test. The descriptive terms used were as follows: very much worse than standard, worse than standard, slightly worse than standard, standard, slightly better than standard, better than standard, very much better than standard. Freeze-dried untreated banana slices were taken as a control (or standard) for the test.

The hedonic rating system was used to determine preferences. The essential features of the hedonic scale are its assumption of a continuum of preference and the direct way it defines the categories of response in terms of like and dislike. A numerical rating scale was used to obtain ratings of the affective values of texture. The descriptive terminology used was as follows: dislike very much, dislike moderately, dislike slightly, neither like nor dislike, like slightly, like moderately, like very much. A 7-point balanced scale was employed. For analysis of data obtained with the hedonic method, numbers from 1 to 7 were assigned to the descriptive categories.

H. OBJECTIVE TEXTURAL MEASUREMENT

1. Shear-Press Measurement

An Allo-Kramer Shear-Press, Model SP-12 IMP and Model E2 recorder indicator was used to determine the texture of the banana product (General Kinetics Incorporated, 2611 Shirlington Road, Arlington, Virginia 22206).

The 250 pound ring, and the shear and compression cell were employed. Instrument sensitivity was set to 100 percent range. The maximum force required to shear the sample and total area under the curve were recorded by means of the recorder indicator.

For the shearing test, 4 grams of F-D banana sample (4 to 5 slices) were rehydrated for 4 minutes, then placed into the cell. The rate of the stroke (or shear bar) was set at 19 seconds. The cell was placed in the test chamber and the instrument turned on. As the sample was being sheared, the instrument reading was recorded by recorder indicator, and then converted into the equivalent pounds force. After each measurement, the cell and blades were cleaned with water. Replications of 2 measurements were taken.

For stable operation, approximately 30 minutes warm-up is required. The electrical zero of the system was set by the displacement indicator zero control. The operation selector switch was then changed to the sensitivity range being used and the mechanical zero of the transducer was adjusted. The operation selector switch was next set at "calibrate" position and the sensitivity control adjusted to full scale deflection.

2. Color Measurement

Color measurement was carried out by the Color Eye (Model D-1, Instrument Development Laboratory, Division of Kollmorgen Corporation, Attleboro, Massachusetts).

Microdial measurement relative to white Working Standards (Absolute Standards) was used. In this method of measurement, one white glass reference standard was put in the "standard" reflectance port (Illuminant "C" - Barium Sulfate as a standard). The reflectance sample was put in the "sample" port. The procedure was as follows:

(a) Set selector switch on "Hi"

(b) Install "non-specular" insert

(c) Select "X" filter

(d) Turn Microdial until meter returns precisely to 100. The darker the sample, the more sensitive the Microdial adjustment. The Microdial must be turned opposite to the direction you wish the needle to move

(e) Read Microdial accurately. You have "forced" the white standard to match the sample under test in the red or "X" end of the spectrum, and the Microdial reading is directly the relative percentage amount of red in the sample as compared to the white standard. As we shall see later, this data may be converted to a definition of the basic color of the sample under test.

(f) Repeat for "Y" and "Z" filters.

Interpretation of measurement on C.I.E. basis. The x, X, Y, and Z value measured were converted to C.I.E. system based on the following equation (for tristimulus values and chromaticity co-ordinates):

$$X_{CIE} = 0.783\ X_{color\text{-}eye} + 0.197\ x_{color\text{-}eye}$$

$$Y_{CIE} = Y_{color\text{-}eye}$$

$$Z_{CIE} = 1.180\ Z_{color\text{-}eye}$$

then

$$x = \frac{X}{X + Y + Z}$$

$$y = \frac{Y}{X + Y + Z}$$

were calculated, and hue, purity, dominant wave length, and luminosity were obtained by reference of the C.I.E. Chromaticity Diagram.

3. Moisture Content of Raw Material and of the End Product

The proportion of the total moisture removed was closely related to texture and most other properties. It is believed that bacteria do not grow below 18 percent available moisture; yeasts require 20 percent or more and molds require 13 to 16 percent. A level of 5 percent moisture or lower is effective in preventing brown discoloration of bananas and peaches stored at 28°C. for at least 6 months (30,37). Excessive heating of banana product (high in sugar) may cause carameli-

zation or decomposition of the sugar with the loss of the elements of water. The moisture content of freeze-dried banana was determined by measuring weight loss on heating the sample in a vacuum oven where the temperature was maintained at a lower figure and the pressure reduced to facilitate loss of moisture. Procedure of measuring moisture content is as follows:

Approximately 10 g. of sample were weighed to the nearest mg. in a tarred and covered weighing dish, then placed in a vacuum oven at 60°C. for 36 hours with the cover removed. When the samples were removed from the oven, they were replaced in the desiccator until cool, and weighed.

The percent of moisture content was expressed by the formula:

$$\text{Percent total moisture on freeze-dried banana} = \frac{\text{g. of total moisture loss}}{\text{g. of freeze-dired banana}} \times 100 .$$

I. STATISTICAL ANALYSIS

BMD02S IBM Model 7040 Digital computer was used to compute chi-squares. This test was used in interpretation of the difference-preference test.

ANOVAR or BMD02R system (17) on the IBM Model 7040 digital computer (The University of Tennessee Computer Center) with usage of the Fortran Library Input Subroutine and some Fortran IV subroutine subprograms were used to perform the analysis of variance calculations. Effect of amount and various firming agents were tested by means of the ANOVAR.

Duncan's multiple range test DUNMRT subroutine (88) on the IBM Model 1460 digital computer was used to perform this test at the 0.05 level of significant. It was used in determining which product mean scores were significantly different.

CHAPTER IV

RESULTS AND DISCUSSION

A. FREEZE-DRYING CYCLE AND ESTIMATION OF TIME

Temperature change during freeze-drying cycle is shown in Figure 1. The initial temperature of the platen was 10°F.; while the temperature of the frozen banana slices was -20°F. At the start of the freezing-dehydration operation, the thermostat was set at 150°F. The surface temperature of platen increased very fast; the temperature dropped a little bit and maintained that figure for 4.5 hours, then the thermostat was adjusted to 120°F.

The critical temperature for banana tissue is 140°F. During the first stage of dehydration, the separation of water from the banana tissue in the form of ice crystals was very fast; because of the high temperature difference between platen and sample, the temperature of thermostat was set for 150°F., but the platen temperature only reached 140°F. The product showed no shrinkage of the tissue. During the second stage of freezing-dehydration, the water vapor was transferred through the already dried shell of the sample to the surface; consequently, the dehydration rate was a little slower. The final stage of dehydration was characterized by evaporation of residual water (bound water), and the dehydration rate proceeded very slow. This period occupied the largest part of dehydration time (approximately 13 hours).

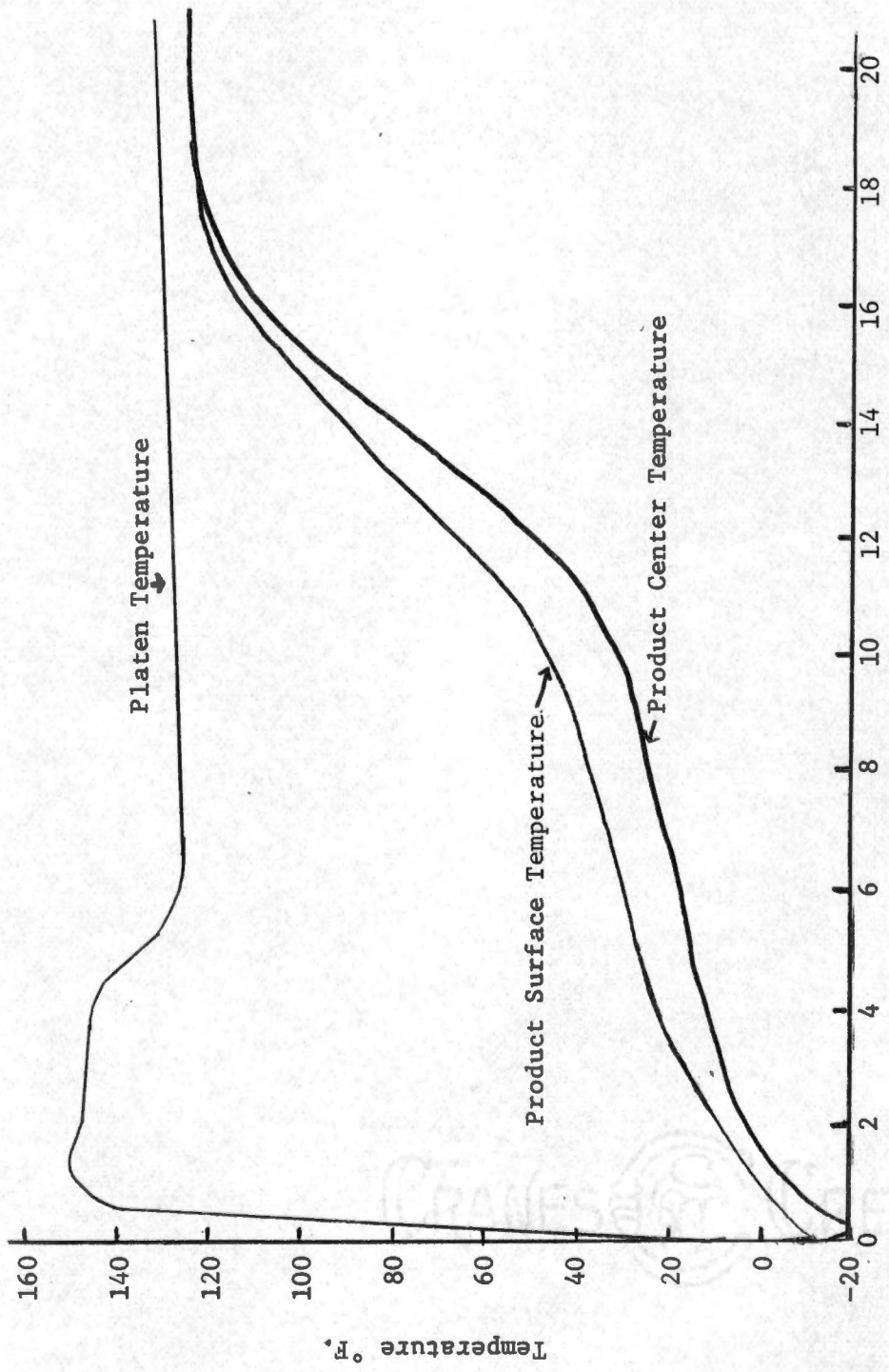


Figure 1. Temperature change during freeze-drying cycle.

In order to minimize the shrinkage or damage to the texture of the sample, the temperature was maintained under 125°F. It was thought that the selected firming agents not only improved the firm texture after rehydration but also permitted to use the relatively high finishing temperature.

The complete cycle of freeze-drying required 20 hours. The process end-point was taken to be that time when the product temperature reached a predetermined point.

B. MOISTURE DETERMINATION

The initial moisture content of frozen banana puree was 78 percent while the canned puree contained 76 percent moisture. The former was a little bit easier to fill into the weiner casing than the latter using the gravity method.

At the end of 4.5 hours the moisture content of the banana slices was 26 percent, after 8 hours it was 10 to 12 percent, after 18 hours it was 4 percent, and after 20 hours freezing dehydration operation, only 3 percent moisture in the freeze-dried product. The moisture removed as a function of time is shown in Figure 2. It was noted that most of the drying time was spent in removing the last 11 percent of the moisture.

C. SELECTION BETWEEN TWO TYPES OF PUREE SAMPLES

Results of the difference-preference test showed that 75 observations out of a possible 80 indicated there was a difference between

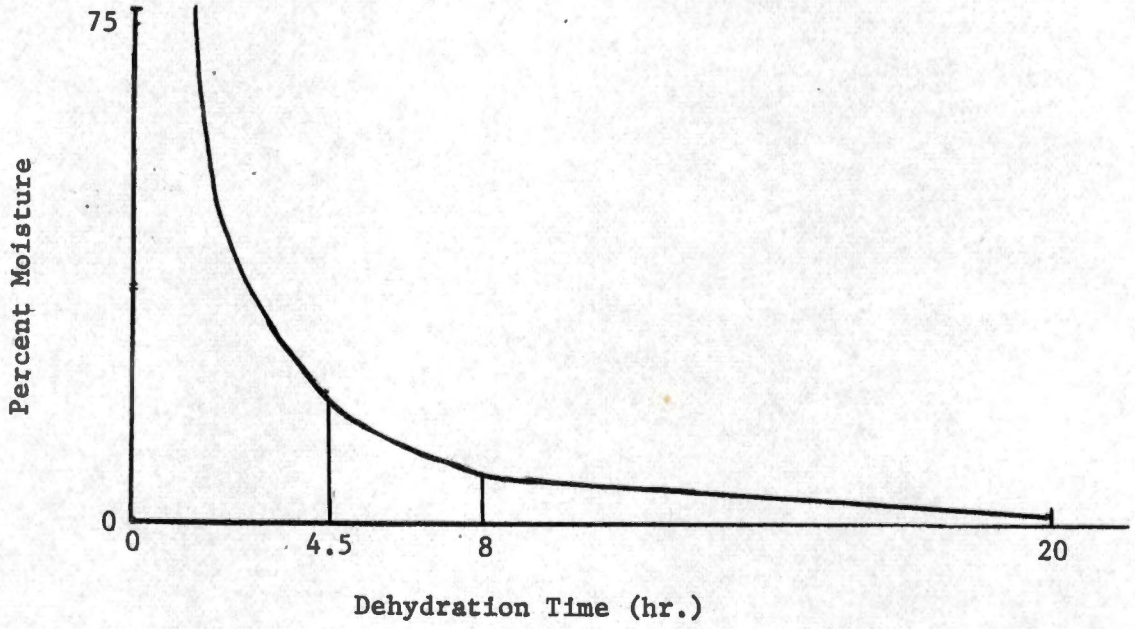


Figure 2. Moisture and time curve at different stages of the freeze-drying operation.

freeze-dried banana slices, after rehydration, prepared from the two types of banana puree. 5 out of 80 observations indicated that there was no difference between them. 51 out of a possible 80 preferred the freeze-dried slices prepared from canned puree, while 29 judges stated that they preferred the freeze-dried product prepared from frozen banana puree.

The result of Chi-square test ($X_1^2 = 59.5$) of a paired comparison (difference) test is significant at 0.01 level of probability. The conclusion is that there is a significant difference between the two types of banana puree.

The result of Chi-square test ($X_2^2 = 5.5$) indicated that, on the basis of the taster's performance, canned banana puree is significantly better than frozen puree at 0.01 level of probability.

D. EFFECT OF KINDS AND LEVELS OF FIRMING AGENTS

1. Preliminary Experiment

Observations of freeze-dried banana slices after rehydration in the preliminary experiment showed that soy protein binder, wheat flour protein binder, Hercules starvis lot 5087, Dow Company's HG, Fisher Science Company's calcium chloride, and Will Corporation's gelatin were weak and/or had no firming function when added to banana puree before freeze-drying. After rehydration, the freeze-dried banana slices lost their original shape or disintegrated and dispersed in water at room temperature. The observations were taken at 1.0 percent treatment level in the preliminary experiment.

Hercules CMC 7HF, Dycol guar gum EHV, Dow Company's MC, Kraft Foods' Kraystay, Du Pont CMC P-75-H, Swift and Company's pad guar gum, and Meer gum guar showed a possible utility as firming agents for freeze-dried banana slices. After rehydration the freeze-dried banana slices retained at least a degree of their distinct shape. These compounds were selected for further test (of the various guar gum only Dycol guar gum EHV was employed in these later studies).

2. Results of Taste Panel Test

Descriptions of the treatments corresponding to the treatment numbers used in this part of study and the results of the taste panel test are presented in Appendix, Table XI , page 93. Means of texture scores and hedonic rating on the quality of freeze-dried banana slices as measured by the taste panel test are shown in Appendix, Table XIII, page 95, as well as in Figures 3 and 4.

The summary of the analyses of variance for the kind and level of firming agents used, as measured by panel test, may be found in Tables II and III. The texture freeze-dried banana slices is shown to be significantly related to level, and highly significant related to agents, and agents by levels interactions. Similarily, changes in agents, levels, and agents by levels interactions were significantly related to the hedonic rating of freeze-dried bananas.

Results of Duncan's multiple range test for the effect of kind and level of firming agents on the texture of freeze-dried banana as measured by panel tests are presented in Tables IV and V. It was

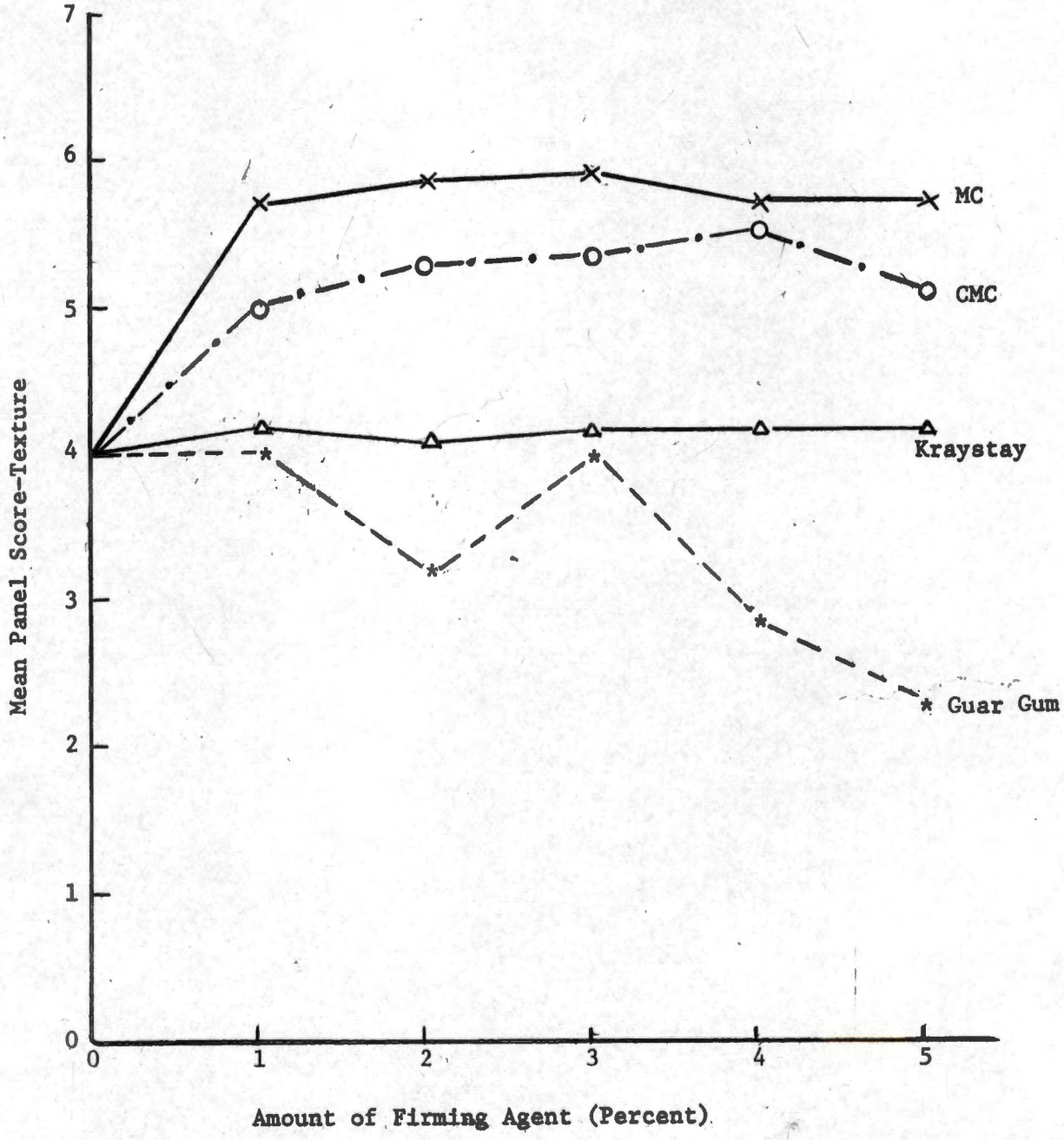


Figure 3. Relationship between mean texture score and amount of various firming agents as measured by taste panel test.

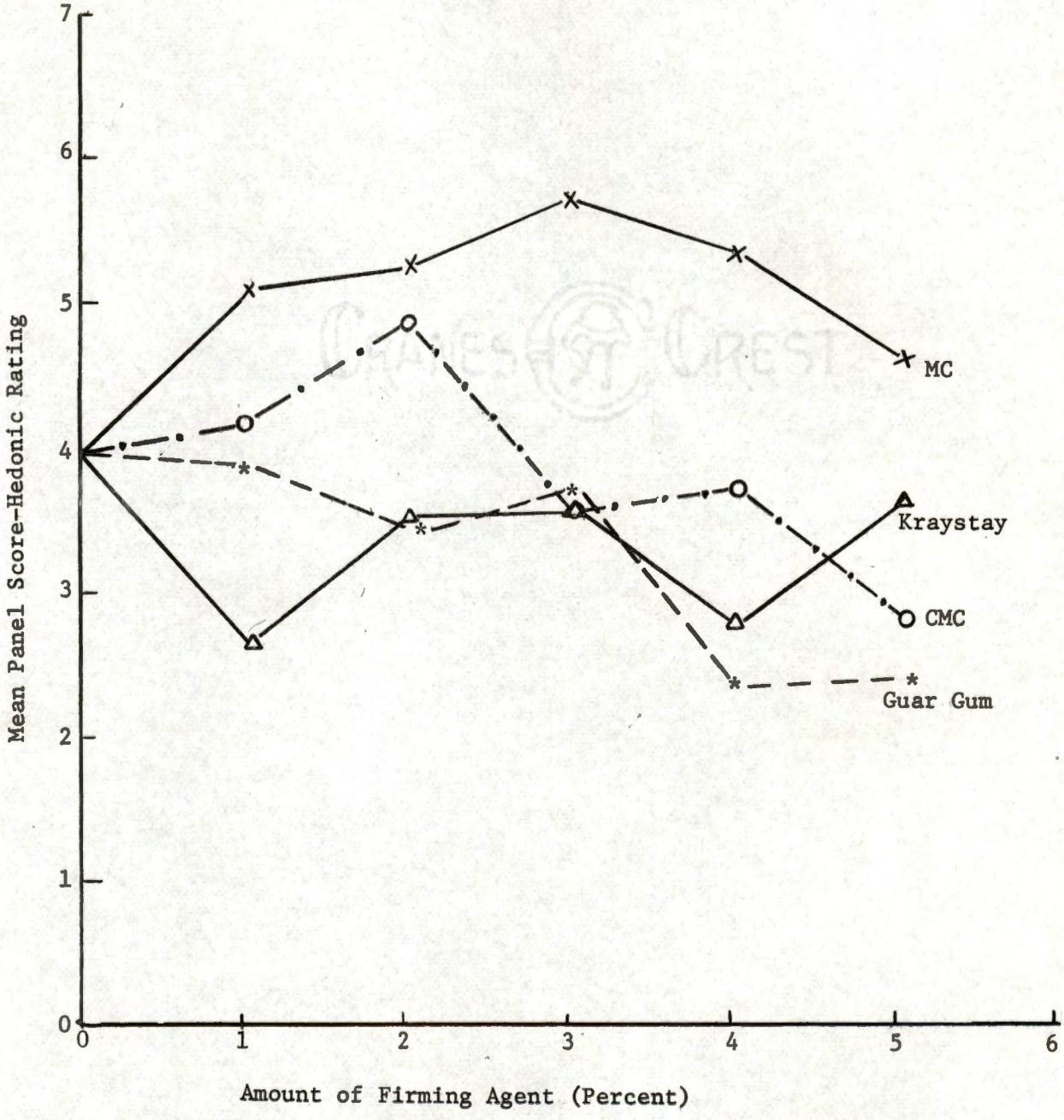


Figure 4. Relationship between mean hedonic rating and amount of various firming agents as measured by taste panel test.

TABLE II

SUMMARY OF THE ANALYSIS OF VARIANCE FOR THE EFFECTS AMONG KINDS AND LEVELS OF FIRMING AGENTS ON THE TEXTURE OF FREEZE-DRIED BANANA SLICES AS MEASURED BY PANEL TEST

Source	D.F.	Sums of Squares	M.S.	F-Ratio
Agent	3	232.6000	77.5333	36.809***
Level	5	29.9333	5.9867	2.842*
A x L	15	75.3333	5.0222	2.384**
Error	336	707.7333	2.1063	
Total	359	1045.6000		

*** Statistical significance at the 0.001 level of probability.

** Statistical significance at the 0.01 level of probability.

* Statistical significance at the 0.05 level of probability.

TABLE III

SUMMARY OF THE ANALYSIS OF VARIANCE FOR THE EFFECTS AMONG KINDS AND LEVELS OF FIRMING AGENTS ON THE PREFERENCE OF FREEZE-DRIED BANANA SLICES AS MEASURED BY PANEL TEST

Source	D.F.	Sums of Squares	M.S.	F-Ratio
Agent	3	164.4750	54.8250	25.193***
Level	5	36.6917	7.3383	3.372**
A x L	15	101.0083	6.7339	3.094*
Error	336	731.2000	2.1762	
Total	359	1033.3750		

*** Statistical significance at the 0.001 level of probability.

** Statistical significance at the 0.01 level of probability.

* Statistical significance at the 0.05 level of probability.

TABLE IV

RESULTS OF DUNCAN'S MULTIPLE RANGE TEST FOR THE EFFECT OF KIND OF FIRMING AGENT ON THE TEXTURE OF FREEZE DRIED BANANA AS MEASURED BY PANEL TEST

Mean No.		Mean		Mean	Test	Rp	
Large	Small	Large	Small	Difference	Difference	At .05 Level	At .01 Level
1	2	5.500	5.056	0.444	4.212	4.023*	5.287
1	3	5.500	4.156	1.344	12.750	4.235*	5.509**
1	4	5.500	3.422	2.078	19.714	4.379*	5.660**
2	3	5.056	4.156	0.900	8.538	4.023*	5.287**
2	4	5.056	3.422	1.634	15.502	4.235*	5.509**
3	4	4.156	3.422	0.734	6.963	4.023*	5.287**

* Statistical significance at the 0.05 level of probability.

** Statistical significance at the 0.01 level of probability.

TABLE V

DUNCAN'S MULTIPLE RANGE TEST FOR THE EFFECT OF LEVEL OF FIRMING AGENTS ON THE TEXTURE OF FREEZE-DRIED BANANA AS MEASURED BY TASTE PANEL TEST

Mean No. Large	Mean No. Small	Mean Large	Mean Small	Mean Difference	Test Difference	R_p at .05 Level	R_p at .01 Level
3	1	4.883	4.750	0.133	1.030	4.023	5.287
3	2	4.883	4.617	0.266	2.060	4.234	5.509
3	4	4.883	4.600	0.283	2.192	4.379	5.660
3	5	4.883	4.350	0.533	4.129	4.483	5.773
3	6	4.883	4.000	0.883	6.840	4.566*	5.863**
1	2	4.750	4.617	0.133	1.030	4.023	5.287
1	4	4.750	4.600	0.150	1.162	4.235	5.509
1	5	4.750	4.350	0.400	3.098	4.379	5.660
1	6	4.750	4.000	0.750	5.801	4.483*	5.773**
2	4	4.617	4.600	0.017	0.132	4.023	5.287
2	5	4.617	4.350	0.267	2.068	4.235	5.509
2	6	4.617	4.000	0.617	4.779	4.379*	5.660
4	5	4.600	4.350	0.250	1.937	4.023	5.287
4	6	4.600	4.000	0.600	4.648	4.235*	5.509
5	6	4.350	4.350	0.350	2.711	4.023	5.287

*Statistical significance at the 0.05 level of probability.

**Statistical significance at the 0.01 level of probability.

found that there were significant differences between agents 1 and 2, 1 and 3, 1 and 4, 2 and 3, 2 and 4, and 3 and 4 at the 0.01 level of probability. There also were significant differences between 1 and 6. 2 and 6, and 4 and 6, but not between 5 and 6 (the latter being the control) at 0.05 level of probability. One may conclude that the firming agents used are superior to the control, and with 95 per cent confidence, that all levels of all treatments are significantly better than the control. There was also a significant difference between levels 1 and 6, and 3 and 6 at 0.01 level of significance, from which one may conclude that 1 to 3 per cent levels of firming agents are significantly better than control. Results of Shear-Press measurements of the treatments corresponding to the treatment numbers used in this part of the study, results of shear measurements, and means of shear values are presented in Appendix, Table XIV, page 96.

The summary of the analyses of variance for the effect of kind and level of firming agents as measured by Shear-Press are shown in Table VI. The relationships between mean shear values and levels of kinds of firming agents are presented in Figure 5. Results of the application of Duncan's multiple range test for the effect of kind and level of firming agents, and agents by levels interactions on the texture of freeze-dried banana as measured by Shear-Press are presented in Tables VII, VIII, and IX.

The summary of analysis of variance showed that, on the basis of the shear test, replications were not significant, but agents, levels, and agents by levels interactions were highly significant.

TABLE VI

SUMMARY OF THE ANALYSIS OF VARIANCE FOR THE EFFECTS AMONG KINDS AND LEVELS OF FIRMING AGENTS ON THE TEXTURE OF FREEZE-DRIED BANANA SLICES AS MEASURED BY SHEAR

Source	D.F.	Sums of Squares	M.S.	F-Ratio
Rep.	1	6.2368	6.2328	0.888
Agent	3	15862.1858	5287.3953	753.152**
Level	5	29041.1433	5808.2286	827.341**
AxL	15	27910.0952	1860.6730	265.040**
Error	23	161.4683	7.0204	
Total	47	72981.1289		

**Statistical significance at the 0.01 level of probability.

TABLE VII

RESULTS OF DUNCAN'S MULTIPLE RANGE TEST FOR THE EFFECT OF KIND OF FIRMING AGENT ON THE TEXTURE OF FREEZE-DRIED BANANA AS MEASURED BY SHEAR

Mean No. Large	Mean No. Small	Mean Large	Mean Small	Mean Difference	Test Difference	R_p
2	1	72.775	63.817	8.958	31.031	7.734*
2	3	72.775	33.567	39.208	135.820	8.206*
2	4	72.775	31.483	41.292	143.040	8.452*
1	3	63.817	33.567	30.250	104.789	7.734*
1	4	63.817	31.483	32.334	112.008	8.206*
3	4	33.567	31.483	2.084	7.219	7.734

*Statistical significance at the 0.05 level of probability.

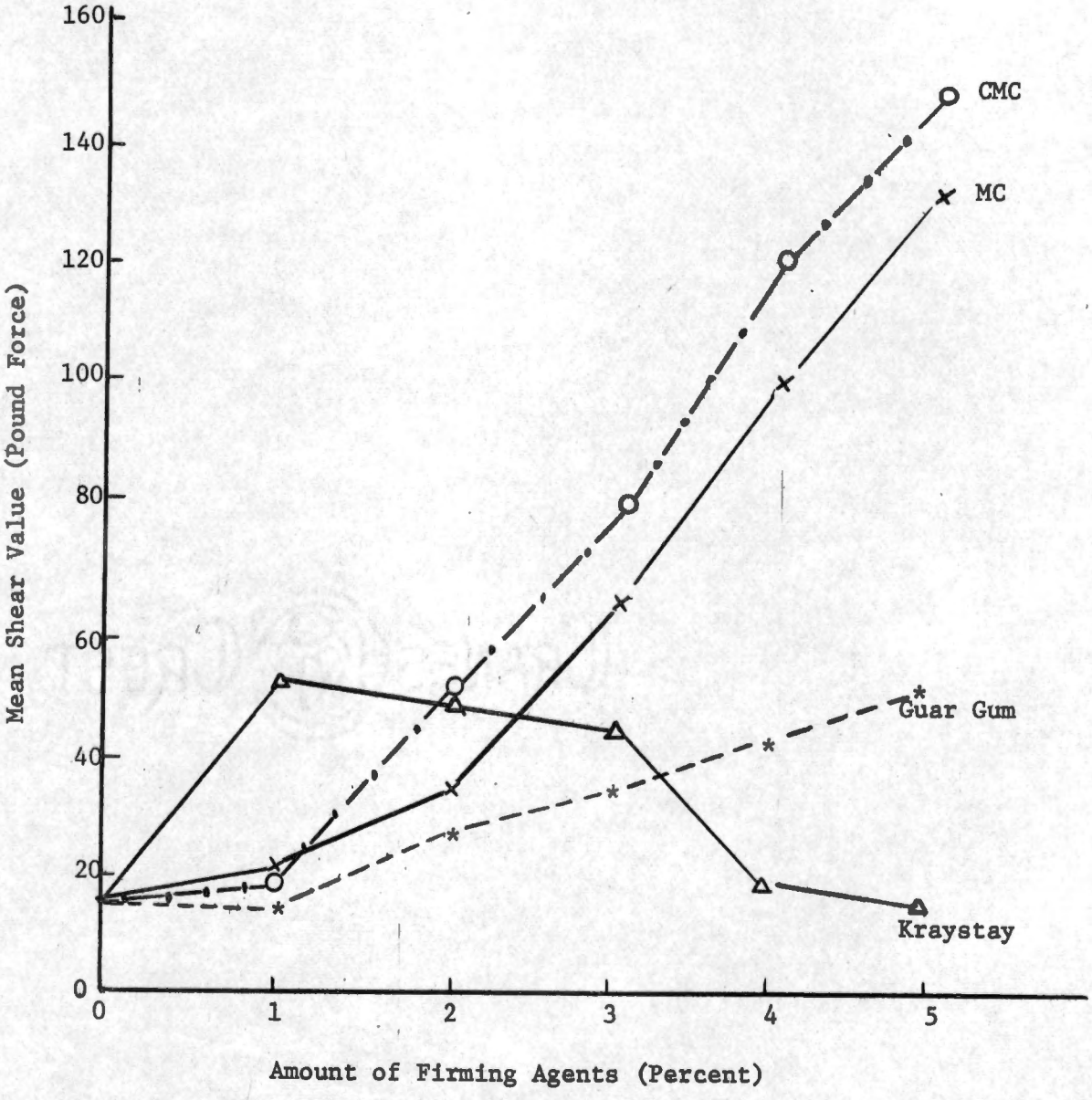


Figure 5. Relationships between mean shear value and levels of various firming agents.

TABLE VIII

RESULTS OF DUNCAN'S MULTIPLE RANGE TEST FOR THE EFFECT OF LEVEL OF FIRMING AGENT ON THE TEXTURE OF FREEZE-DRIED BANANA AS MEASURED BY SHEAR

Mean No. Large	Mean No. Small	Mean Large	Mean Small	Mean Difference	Test Difference	R_p
5	4	87.688	72.063	15.625	44.194	7.734*
5	3	87.688	56.500	31.188	88.213	8.206*
5	2	87.688	42.250	45.438	128.518	8.206*
5	1	87.688	27.063	60.625	171.473	8.625*
5	6	87.688	16.900	70.788	0.000	8.752
4	3	72.063	56.500	15.563	44.019	7.734*
4	2	72.063	42.250	29.813	84.323	8.206*
4	1	72.063	27.063	45.000	127.279	8.452*
4	6	72.063	16.900	55.163	0.000	8.625
3	2	56.500	42.250	14.250	40.305	7.734*
3	1	56.500	27.063	29.437	83.260	8.206*
3	6	56.500	16.900	39.600	0.000	8.452
2	1	42.250	27.063	15.187	42.955	7.734*
2	6	42.250	16.900	25.350	0.000	8.206
1	6	27.063	16.900	10.163	0.000	7.734

*Statistical significance at the 0.05 level of probility.

TABLE IX

RESULTS OF DUNCAN'S MULTIPLE RANGE TEST FOR THE EFFECT OF AGENTS BY LEVELS INTERACTIONS ON THE TEXTURE OF FREEZE-DRIED BANANA AS MEASURED BY SHEAR-PRESS*

Mean No. Large	Mean No. Small	Mean Large	Mean Small	Mean Difference	Test Difference	R_p
8	13	54.500	53.500	1.000	1.414	7.734
8	23	54.500	51.500	3.000	4.243	8.206
8	14	54.500	50.000	4.500	6.364	8.452
13	23	53.500	51.500	2.000	2.828	7.734
13	14	53.500	50.000	3.500	4.950	8.206
23	14	51.500	50.000	1.500	2.121	7.734
15	22	43.500	42.250	1.250	1.768	7.734
2	21	36.750	34.500	2.150	3.182	7.734
16	1	22.000	20.250	1.750	2.475	7.734
16	7	22.000	18.500	3.500	4.950	8.206
16	24	22.000	16.900	5.100	7.212	8.452
16	18	22.000	16.900	5.100	7.212	8.625
16	12	22.000	16.900	5.100	7.212	8.751
16	6	22.000	16.900	5.100	7.212	8.847
16	19	22.000	16.900	6.000	8.485	8.924
1	7	20.250	18.500	1.750	2.475	7.734
1	24	20.250	16.900	3.350	4.738	8.206
1	18	20.250	16.900	3.350	4.738	8.452
1	12	20.250	16.900	3.350	4.738	8.624
1	6	20.250	16.900	3.350	4.738	8.751
1	19	20.250	16.900	4.250	6.010	8.847
1	17	20.250	15.500	4.750	6.718	8.924
7	24	18.500	16.900	1.600	2.263	7.734
7	18	18.500	16.900	1.600	2.263	8.206
7	12	18.500	16.900	1.600	2.263	8.452
7	6	18.500	16.900	1.600	2.263	8.625
7	19	18.500	16.000	2.500	3.536	8.752
7	17	18.500	15.500	3.000	4.243	8.847
24	18	16.900	16.900	0.000	0.000	7.734
24	12	16.900	16.900	0.000	0.000	8.206
24	6	16.900	16.900	0.000	0.000	8.452
24	19	16.900	16.000	0.900	1.273	8.625
24	17	16.900	15.500	1.400	1.980	8.752

TABLE IX (continued)

Mean No. Large	Mean No. Small	Mean Large	Mean Small	Mean Difference	Test Difference	R_p
18	12	16.900	16.900	0.000	0.000	7.734
18	6	16.900	16.900	0.000	0.000	8.206
18	19	16.900	16.000	0.900	1.273	8.452
18	17	16.900	15.500	1.400	1.980	8.625
12	6	16.900	16.900	0.000	0.000	7.734
12	19	16.900	16.000	0.900	1.273	8.206
12	17	16.900	15.500	1.400	1.980	8.452
6	19	16.900	16.000	0.900	1.273	7.734
6	17	16.900	15.500	1.400	1.979	8.206
19	17	16.900	15.500	0.500	0.707	7.734

*The chemicals by levels interactions results presented here are all not significantly differences; the rest of the combinations of pared comparison are all significantly differences at the 0.05 level of probability.

Results of Duncan's multiple range test for these differences showed that there were significant differences between agents 1 and 2, 1 and 3, 1 and 4, 2 and 3, and 2 and 4, but not between 3 and 4 at the 0.05 level of probability. The conclusion from this phase of the study is that agents 1 and 2 are significantly superior to agents 3 and 4. Results of Duncan's multiple range test for the effect of levels of firming agents on the texture of freeze-dried banana as measured by Shear-Press showed there were significant differences between levels 1 and 2, 1 and 3, 1 and 4, 1 and 5, 2 and 3, 2 and 4, 2 and 5, 3 and 5, and 4 and 5, but not between 1 and 6, 2 and 6, 3 and 6, 4 and 6, and 5 and 6 at 0.05 level of probability.

E. REHYDRATION

The rate of water uptake by freeze-dried banana slices was relatively high. In the rehydration test, the relationship between weight gain and rehydration time are presented in Figure 6. Results showed that most of the rehydration medium was absorbed in the first 5 minutes, and lower the pH of the medium the slower the rehydration rate. Results of rehydation tests are shown in Appendix, Table XV, page 97.

F. COLOR MEASUREMENT

Results of color measurement are shown in Table X, and Appendix, Table XI, page 93. No significant differences were found among the test results.

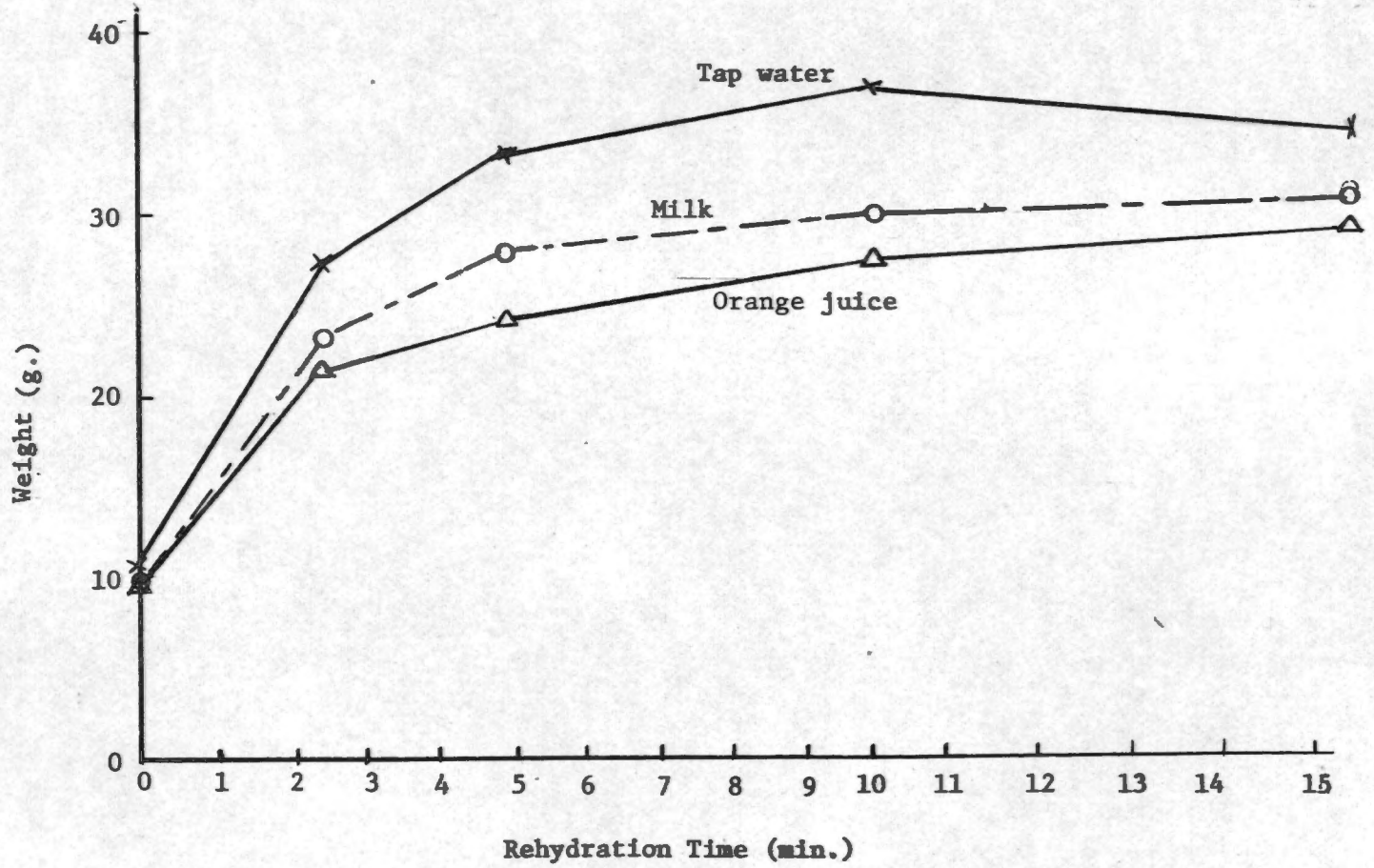


Figure 6. Relationships between weight gain and rehydration time at room temperature.

TABLE X

RESULTS OF COLOR MEASUREMENTS

Terms	Data and Results / Treatments		2%MC	4%MC	2%CMC	4%CMC	2%Kr.	4%Kr.	2%G.C.	4%G.G.
Color-Eye Reading		X'	35.1	44.9	35.4	28.6	41.9	39.5	27.1	34.2
		X	60.2	70.0	60.0	52.7	66.6	64.8	52.4	58.1
		Y	55.5	65.6	55.6	48.2	62.3	60.5	47.2	54.0
		Z	36.3	45.9	36.7	29.7	43.0	41.2	27.9	35.3
C.I.E.	Chromaticity Co-ordinates	x	.395	.381	.393	.405	.384	.387	.415	.393
		y	.364	.357	.364	.370	.359	.361	.374	.365
	Hue		white	white	white	white	white	white	white	white
	Purity (%)		35.8	30.9	35.3	33.4	31.0	33.9	43.9	36.3
	Dominant Wave Length (mμ)		584	585	584	584	582.5	584.6	584	583.8
	Luminosity		55.5	65.6	55.6	48.2	62.3	60.5	47.2	54.0

although slices treated by Kraystay showed a somewhat deeper color than the others. The stability of freeze-dried banana slices which contained less than 5 per cent moisture was high. As stated previously, a level of 5 per cent moisture or lower is effective in preventing brown discoloration of banana stored at 28°C. for at least 6 months (30).

CHAPTER V

SUMMARY

The objective of this study was to compare the freeze-dried banana puree slices treated by firming agents with untreated banana puree slices to see if any particular levels of firming agents used led to a more desirable product from the standpoint of texture.

Freeze-drying, as the name implies, involves two operations of equal importance. A developing literature on the subject, however, indicates that the freezing step may be as important as - or possibly more important than the drying step. Products which are high in sugars, salts, acids, and flavor components, represent more sensitive materials and require greater care in freezing and drying in order to retain these desired organoleptic properties.

The change of state of water associated with the change in volume, water at 0°C. and 1 atm. of pressure expands nearly 9 per cent, transforming into ice. Protein denaturation, redistribution of soluble components, emulsion destabilization, and rupture of cell wall were effected by freezing. The current theories on the injury from freezing and its prevention can be summarized in terms of two controllable variables, rate of freezing and, protective additives. Dehydration might cause protein denaturation, crystallization of polysaccharides, loss of volatile components, case hardening, and deterioration of freeze-dried foods. So, control dehydration time, temperature, vacuum, and final water content were very important

factors. In other words, careful control of the freeze-drying cycle is necessary.

In this study, an air-blast freezer (-30°F.) was used to freeze the rope of banana puree. Firming agents were selected to prevent damage from freezing, dehydration, and furthermore to improve firmness of freeze-dried banana after rehydration. The frozen weiner casings with filled puree were removed from the air-blast freezer, and all subsequent work prior to freeze-drying was done inside a 0°F. still air freezer. The filled casings were then removed with a sharp knife. When the trays of banana samples to be freeze-dried were placed in the chamber (Del-Vac Freeze-Dryer, Model 11212 RVM), the platen temperature was approximately 10°F. while the samples were -20°F. After the trays were placed on the shelf, a vacuum was applied, and the thermostat was turned to 150°F.; after 4.5 hours the thermostat was adjusted (platen temperature) to 125°F. for the rest of the drying time.

Both subjective and objective quality measurements were employed for evaluating the freeze-dried banana product. Randomized complete-block design was used for measuring the effect of kind and level of firming agents by taste panel test (both texture scoring and hedonic rating). Chi-square test was used for interpretation of difference-preference test (paired comparison for selection of type of puree sample). A 4 x 6 factorial design for computing the Shear-Press measurement was employed. Color measurement was carried out by the Color-Eye; interpretation of measurement was made on C.I.E. basis.

An Allo-Kramer Shear-Press and recorder indicator was used to determine the texture of the banana product. In statistical analysis, BMDO2S, IBM Model 7040 Digital computer was used to compute Chi-squares. ANOVAR, with usage of the Fortran Library Input Subroutine and some Fortran IV subroutine subprograms, were used to perform the analysis of variance calculations. Duncan's multiple Range test DUNMRT subroutine on the IBM Model 1460 Digital computer was used to perform this test; it was used in determining which product mean score were significantly different.

The following conclusions may be drawn from this study:

1. The freeze-drying cycle used in this study was found promising from the standpoint of the ultimate freeze-dried product.

2. Stability of color of freeze-dried banana slices which contained less than 5 per cent moisture was high.

3. Rehydration test showed the lower the pH of the medium the slower the water reabsorbed by freeze-dried banana.

4. The four firming agents selected (MC, CMC, Kraystay, and Guar Gum) provided a higher finishing temperature in the freeze-drying operation with little or no damage of product quality. They improved the firmness of the product after rehydration.

5. Freeze-dried banana slices treated with firming agents rated significantly higher than the untreated freeze-dried banana product in both texture and preference tests.

6. Results of Duncan's Multiple Range test for the effect of levels of firming agents were all significantly different at 0.05 level of probability as measured by Shear-Press.

7. Results of Duncan's Multiple Range test for the effect of kinds of firming agents on the texture of freeze-dried banana as measured by the taste panel test showed that there were significant differences between each pair of mean score - MC having the highest mean score, followed by CMC, Kraystay, and Guar Gum.

8. Results of Duncan's Multiple Range test on agents by levels interactions showed that 5 per cent CMC had the highest mean shear value, 5 per cent MC the next, followed by 4 per cent CMC, and 4 per cent MC.

9. The taste panel test score (both mean texture score and mean hedonic rating) revealed that firming agent levels from 1 to 3 per cent steadily increased mean score while higher levels decreased them. Thus the lower levels may be considered promising from the standpoint of the overall quality of the freeze-dried banana product. Conversely, the shear value index of the product rose consistently with increased levels of the firming agents except for the Kraystay, in which case the index dropped off sharply at levels exceeding 3 per cent.

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APPENDIX

TABLE XI

TREATMENTS USED IN THE TEST FOR EFFECT OF KIND AND AMOUNT OF FIRMING AGENTS ON THE TEXTURE OF FREEZE-DRIED BANANA

Treatment No.	Chemical Treatment	Levels
1	Methylcellulose	From 0 to 5 per cent*
2	Sodium Carboxylethyl-cellulose	From 0 to 5 per cent*
3	Kraystay	From 0 to 5 per cent*
4	Guar gum	From 0 to 5 per cent*

*1 indicated 1 per cent level, 2 indicated 2 per cent level, 3 indicated 3 per cent level, 4 indicated 4 per cent level, 5 indicated 5 per cent level, and 6 indicated 0 per cent level or control.

TABLE XII

RESULTS OF EFFECT OF KINDS AND LEVELS OF FIRMING AGENTS ON THE TEXTURE OF FREEZE-DRIED BANANA SLICES AS MEASURED BY TASTE PANEL TEST

No. of Treatment*	Texture Score							Hedonic Rating						
	1	2	3	4	5	6	7	1	2	3	4	5	6	7
1			1	1	4	4	5		1	3	2		6	3
2			1	1	3	4	7		2	2	2	2	5	3
3				2	3	4	7			1	2	3	5	4
4			2	1	2	4	7	1	1	1	2	3	4	4
5			2		3	5	5	1	2	1	3	5	3	1
6				15							15			
7		1	2	2	4	3	3		2	4	2	3	4	
8	1	1	1	1	5	4	3		2	3	2	4	5	1
9			2	2	5	4	2	1	2	5	4	2	1	
10			3	1	1	4	6	2	2	3	3	2	2	2
11		2	1	2	2	4	4	3	5	2	2	3		
12				15							15			
13	1	1	3	4	3	1	2	7	1	7	2			
14	1	3	1	1	7	2		2	3	2	2	5	1	
15	1	2	2	2	5	2	1	2	3	2	2	4	2	1
16		3	3	3	2	2	2	5	2	4	1	1	2	
17		3	3	2	3	3	1	2	2	5	1	1	3	1
18				15							15			
19	3	3	1	2	1	4	1	2	2	3	2	1	4	1
20	4	3		2	6			2	3	3	3	2	2	
21	1	3	3		4	3	1	2	3	2	2	4	1	1
22	6	2	1	2	3		1	5	4	1	4	1		
23	6	3	3	1	2			6	4	1	3	1		
24				15							15			

*Numbers 6, 12, 18, and 24 indicate controls. Numbers 1 to 5, 7 to 11, 13 to 17, and 19 to 23 indicate percentage levels of MC, CMC, Kryastay, and Guar Gum respectively

TABLE XIII

RESULTS OF MEANS OF TEXTURE SCORE AND HEDONIC RATING ON THE QUALITY OF FREEZE-DRIED BANANA SLICES AS MEASURED BY TASTE PANEL TEST

No. of Treatment	Means Texture Score*	Means of Hedonic Rating*
1	5.733	5.067
2	5.867	5.133
3	5.933	5.733
4	5.733	5.333
5	5.733	4.600
6	4.000	4.000
7	5.000	4.200
8	5.267	4.933
9	5.333	3.600
10	5.600	3.733
11	5.133	2.800
12	4.000	4.000
13	4.200	2.267
14	4.133	3.533
15	4.200	3.733
16	4.200	2.800
17	4.200	3.667
18	4.000	4.000
19	4.067	3.933
20	3.200	3.467
21	4.067	3.667
22	2.867	2.333
23	2.333	2.467
24	4.000	4.000

*Mean value of 15 observations.

TABLE XIV

RESULTS OF EFFECT OF KINDS AND LEVELS OF FIRMING AGENTS ON THE TEXTURE OF FREEZE-DRIED BANANA SLICES AS MEASURED BY SHEAR-PRESS

	Levels of Chemicals											
	MC						CMC					
	Shear Values (# Force)											
Rep.	1%	2%	3%	4%	5%	Std.	1%	2%	3%	4%	5%	Std.
1	19.5	37.5	70.0	100.0	135.0	17.0	21.0	57.0	80.0	122.0	150.5	17.0
2	21.0	36.0	65.0	108.0	140.0	16.8	16.0	52.0	81.0	118.0	142.0	16.8
Mean	20.25	36.75	67.50	104.00	137.50	16.90	18.50	54.50	80.5	120.00	146.25	16.9

	Kraystay						Guar Gum					
Rep.	1%	2%	3%	4%	5%	Std.	1%	2%	3%	4%	5%	Std.
1	57.0	50.0	45.0	20.0	15.0	17.0	16.0	27.5	34.0	42.5	51.0	17.0
2	50.0	50.0	42.0	24.0	16.0	16.8	16.0	28.0	35.0	42.0	52.0	16.8
Mean	53.50	50.0	43.50	22.0	15.50	16.90	16.00	27.75	34.50	42.25	51.50	16.90

TABLE XV

RESULTS OF REHYDRATION TESTS

	Tap Water	Milk	Orange Juice
Initial weight of F-d banana	10.02 g.	8.91 g.	9.33 g.
2.5 min.	27.46 g.	23.14 g.	21.56 g.
5.0 min.	33.08 g.	27.89 g.	24.14 g.
10.0 min.	36.63 g.	29.74 g.	27.42 g.
15.0 min.		30.81 g.	28.67 g.
Rehydration ratio	3.68	3.46	3.08
Degree of rehydration	2.60	2.46	2.04
Coefficient of restoration	0.97	0.92	0.86
Drying ratio	3.80	3.76	3.58

*Based sample treatment No. 1.

VITA

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