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TECHNICAL REPORT  
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**PROCEDURES  
TO MINIMIZE MECHANICAL DAMAGE  
TO FREEZE-DRIED FOODS**

Rutgers University, Food Science Dept.  
Cook College  
New Brunswick, New Jersey 08903

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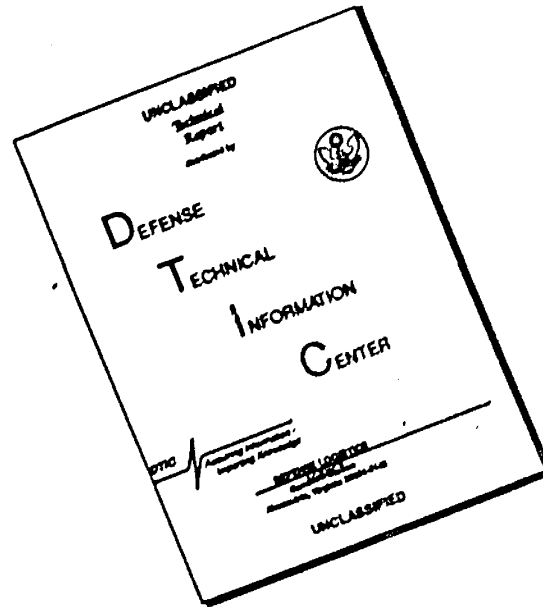
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The objective of the studies reported herein was to develop procedures principally to minimize mechanical breakage during handling and transport, while retaining or improving other quality characteristics of ten meat/seafood products. The effects of freezing rate prior to freeze drying, dehydration parameters, and various food grade additives were evaluated. Formulations have been developed which result in the products which are mechanically stable and exhibit superior texture, rehydration and organoleptic qualities. After being		

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Abstract (Cont.)

stored at 38°C, the products with newly developed formulations exhibited a favorable retention of all these qualities as well as a lower extent of rancidity and color change. The major advance discovered in this study has been the effectiveness of salt soluble meat proteins, dispersed by an optional incorporation of phosphates, as binder for the meat tissues, in a process sequence which includes lyophilization. The phosphate treatment is part of the additive system, the effectiveness of which is augmented by sodium chloride and in some cases by wheat gluten and meat emulsion. Thereby the major objective of minimizing mechanical damage appears to have been achieved along with the improvement in the various collateral quality factors.

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## PREFACE

Freeze dried foods are recognized as being far more susceptible to breakage and erosion during production, packaging, transport, and preparation for consumption than their fresh frozen counterparts. Particular criticism has been directed toward freeze dried meat items which are designed for portion controlled servings but frequently must be served as multiple pieces. Such breakage is inevitable with freeze dried fish squares, pork chops, and all patty items. Other freeze-dried products, such as shrimp and chicken pieces, undergo attrition. This study seeks to develop the technology needed to alleviate excessive fragility of specific freeze dried items which, unless corrective measures are forthcoming, may be eliminated from operational rations. The technology herewith developed should find application in upgrading other freeze dried products, including freeze dried compressed bars, in which a degree of fragility subtracts from potential performance.

The investigation here described was performed at The Department of Food Science, Rutgers University, New Brunswick, N.J. 08903. Funds were provided under Project Number 1J662713A034, titled: Military Food Service and Subsistence Systems.

Professor Edward Seltzer served as Principal Investigator and Dr. Shri Sharma as Collaborator. Dr. Maxwell C. Brockmann and Justin M. Tuomy were the Project Officer and the Alternate Project Officer, respectively, for the U.S. Army Natick Laboratories, now US Army Natick Research and Development Command.

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PROCEDURES TO MINIMIZE MECHANICAL DAMAGE TO FREEZE-DRIED  
FOODS

INTRODUCTION

Crumbliness or friability of freeze dried food products under mechanical stresses has been recognized as a major problem in these foods. Owing to mechanical abrasion the "unit" food items get partly crumbled into unusable powder during production and packaging, transit and preparation for consumption. Particular criticism has been directed toward a number of freeze dried meat/fish products which are designed for portion controlled servings but become damaged by breaking into pieces and, therefore, have to be served in multiple pieces.

The investigations reported herein had to deal with the development of procedures for minimizing mechanical damage incident to handling and transporting of freeze dried meat products. Current Military Specifications for the designated products were used for general guidance in preparation. Eight of the following ten products were, under the terms of this contract, selected for development of one or more broadly applicable and commercially feasible methods to minimize the fragility without impairing their performance in operational rations:

- a. Beef patties, raw (MIL-B-43143A and Amend.-1)
- b. Beef patties, cooked
- c. Chicken, cooked, Type III, (MIL-C-0043134C and Amend.-1)
- d. Fish squares, raw, (MIL-F-43142B and Amend.-1)
- e. Pork chops, raw, (MIL-P-0043144C and Amend.-2)
- f. Pork chops, cooked
- g. Pork sausage, patties, cooked, Style I, (MIL-P-43383A)
- h. Shrimp, cooked, (MIL-S-43145C)
- i. Tuna, cooked, (MIL-S-43443)
- j. Scallops, cooked, (initial size conforming to Fed. Spec. PP-S-00120B, Scallops, Chilled and Frozen).

Wherever applicable and available, MIL Specifications were used in preparation (by freeze drying) of all the above items. After a preliminary investigation, the last two of the above ten products, namely, tuna and scallops were omitted in favor of emphasizing the first eight items. These two products were omitted because of their relative unavailability or unreliability at New Brunswick, N.J., where this study was carried out.

It is evident that the meat products described above represent a wide range of variability in terms of types of natural fibrosity, and some include as inherent variables the physico-chemical changes resulting from operations, like cooking and comminution prior to freeze dehydration. Also, since all products are destined for human consumption, all the additives and processes should meet the FDA standards as well as have good panel acceptance.



The objectives of the research reported herein were to:

- (a) investigate the nature of mechanical breakage in freeze dehydrated meat products;
- (b) develop the methods to evaluate the mechanical breakage;
- (c) develop broadly applicable and commercially feasible methods to reduce fragility in specified meat products without impairing their rehydration, texture and other organoleptic qualities;
- (d) evaluate response to storage of treated as well as control samples.

## EXPERIMENTAL EVALUATION OF FRAGILITY AND OTHER QUALITIES IN FREEZE DRIED FOODS

### Source of Meat

The raw meats/seafoods for all the eight products were obtained from USDA/FDA/PID inspected sources within easy reach from Rutgers University, New Brunswick, N.J. For some of the experiments on cooked chicken, diced frozen meat was also obtained from McCormick Foods, Inc., Bedford, Va., & Tip Top Poultry Co., Marietta, Ga. These samples were shipped by air in insulated containers.

### Sample Preparation

In general, the handling and processing techniques and the time-temperature limitations were adopted as outlined in Military Specifications for the appropriate product. Some salient features of the procedures will, however, be discussed briefly.

#### Comminuted meat products

For comminuted meat products (beef patties, raw and cooked; pork sausage patties, cooked) the sequence of operations involved in the preparation of the patties is given in Figure 1. Deboned and trimmed meat was comminuted in a Hobart meat grinder with plate having 1.9 cm r.p. openings. The meat was mixed with the desired additives (if any) at this stage and comminuted further through the plate with 0.48 cm. diameter openings. It was shaped into patties using a locally fabricated plastic mold having an opening size of 8.9 cm x 5.7 cm x 1.3 cm. The uniform sized patties thus obtained were held for a known period of time at refrigerated temperature before cooking or freezing. Cooking parameters for the different products are given in Table 1.

#### Non-comminuted products

Fresh, chilled haddock (Melanogrammus aeglefinus) samples were cleaned, filleted and the slices made into blocks. These blocks were covered with moisture barrier wrappers to avoid freezer dehydration and frozen to -34°C. Fish squares of size 8.6 cm x 8.6 cm x 1.3 cm were cut from the frozen blocks using an electric hack-saw. For a part of the experiments, fish squares of specified size were prepared from commercially available fish blocks.

For preparation of chicken, cooked, diced (Type III); shrimp, cooked; pork chops, raw as well as cooked, the Military Specifications MIL-C-0043135C-Amend. 1, MIL-S-43145C and MIL-P-0043144C-Amend. 2 were, respectively, followed. Cooking parameters for the appropriate products are given in Table 1.

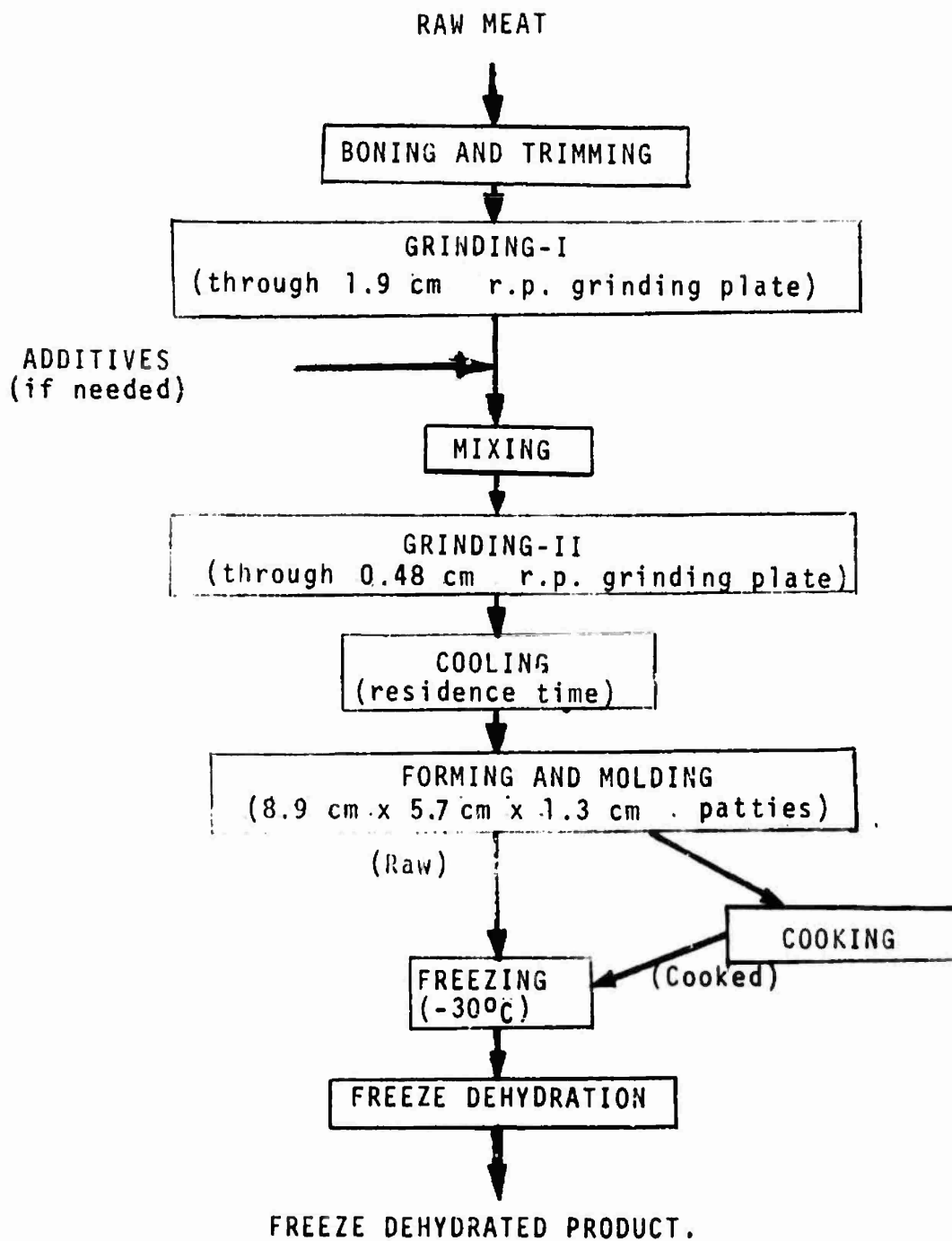


Figure 1. Flow sheet describing the steps involved in preparation of the comminuted meat samples

Table 1

Parameters describing techniques used for cooking of the various cooked-freeze dried products

Product	Mode of Cooking	Temperature Setting, °C	Duration of Cooking
1. Beef Patties	Pan-fried	182	2½ min. on each side
2. Chicken	Cooked in water	94-99	30 min.
3. Pork chops	Pan-fried	182	2 min. on each side
4. Pork sausage patties	Deep-fat-fried	176-190	2 min.
5. Shrimp	Cooked in water with 2% salt	74-85*	10 min.

\* This seemingly low cooking temperature is an intentional method for shrimp which toughens seriously at higher cooking temperatures (74°C for 10 min. ~ 94° for 2 min.).



## Freeze Drying

The products were completely frozen in advance of freeze drying thereby ensuring that all drying was by lyophilization. For freeze dehydration, an F. J. Stokes 42 square feet (4.8 m<sup>2</sup>) tray dryer was used. The following conditions were typical for a drying cycle:

Pressure = 50 to 200  $\mu$  Hg (0.05 to 0.20 mm)  
Shelf temperature = 49 to 60°C  
Condenser surface temperature = -40 to -54°C  
Product temperature at the conclusion of drying = 49  $\pm$  6°C  
Drying cycle time = 7 to 16 hours depending on the product.

The sample temperatures were recorded using a multi-point potentiometric strip chart recorder. The drying cycle was terminated by equalizing the drying chamber pressure to atmospheric with nitrogen. Immediately after their removal from the drying chamber, the products were enclosed in glass containers (Mason jars) impermeable to oxygen and moisture. By successive evacuation (to about 0.2 mm Hg) and flushing with nitrogen, the residual oxygen content of the interior environment surrounding the product was brought down to less than 0.1 percent.

## Evaluation of Mechanical Integrity

The mechanical integrity of the dehydrated products was evaluated by shear-compression and drop testing. The toughness of the fresh or rehydrated meat samples was also measured using the shear test. In general, the following procedure was used for experimentation and interpretation of the results from the two techniques:

### Shear-compression Tests

A single blade (0.32 cm thick) Kramer "Shear" cell, driven by an Instron system (Model TM), was used to evaluate the strength of the individual food units. After recording its geometrical parameters, a product sample was subjected to mechanical loading until the blade had completely pierced through it. Throughout the investigation, the rate of travel of the blade was adjusted to 2.5 cm per minute.

The nature of forces that produce failure of the specimen is highly complex and will not be discussed in this report. It may, however, be pointed out that the behaviors exhibited by the comminuted and non-comminuted products were distinctly different. Owing to the relatively poor cohesiveness between the comminuted meat particles, the freeze dried patties crumbled apart after only a slight deformation. The reinforced fibrous structure of the whole meat products (pork chops, fish squares, etc.) exhibited a mechanical response which was related to the direction of fiber orientation. The plane parallel to the

direction of fibers registered a much lower force value, which was also observed to be the plane along which the specimen fragmented in the drop test. Therefore, wherever unidirectional orientation of the fibers was apparent, shear values were recorded along the weaker plane, i.e., along the fibers only. In evaluating a sample, the shear-compression values were recorded at several positions and a mean value was considered an indicator of its overall cohesiveness. Force-deformation diagrams for two of the representative comminuted and non-comminuted meat samples are presented in Figure 2, indicating the salient force levels and the corresponding physical change exhibited by the sample.

Shear tests were also conducted on the prepared samples before freeze drying and after rehydration of their freeze dried counterparts to evaluate the relative difference in their toughness.

#### Drop Testing

A divided table type apparatus<sup>1</sup> conforming to the requirements of the ASTM D775-6i was used for conducting the drop tests. About 50 grams of the appropriate dehydrated product, consisting of at least three pieces, were enclosed in No. 10 (603 x 700) metal cans and dropped repeatedly on a smooth concrete floor from a height of one meter. The preliminary testing ranged from 60 to 150 cm in height of drop. The samples of products treated to reduce fragility and their respective control samples were subjected to an equal number of drops.

It is difficult to define the nature of forces which a product experiences as a result of drop-impact, but the forces possibly involve a combination of impact, crushing and shearing. If these forces experienced by the product matrix holding the meat particles (or fibers) are in excess of its maximum resistive strength, they result in the failure of cohesion of the matrix and production of an increased number of smaller particles of irregular size and shapes. If the control as well as treated samples of a product are dropped repeatedly until they assume a "steady" fragment distribution, the relative resistive strength of the two samples can be compared by analyzing their particle size distribution.

The distribution of the drop-fragmented samples were assessed by the following criteria:

- (i) Number and size of major fragments per unit in the initial sample;
- (ii) Sieve analysis, mostly using U.S. Standard Sieve Series: 1.90 cm, 0.95 cm, No. 3 (6.73 mm), No. 4 (4.76 mm), No. 5 (4.00 mm), No. 8 (2.38 mm), No. 16 (1.19 mm),

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<sup>1</sup>Manufactured by Gaynes Engineering Company, Chicago, Illinois for a design specification of Rutgers University.

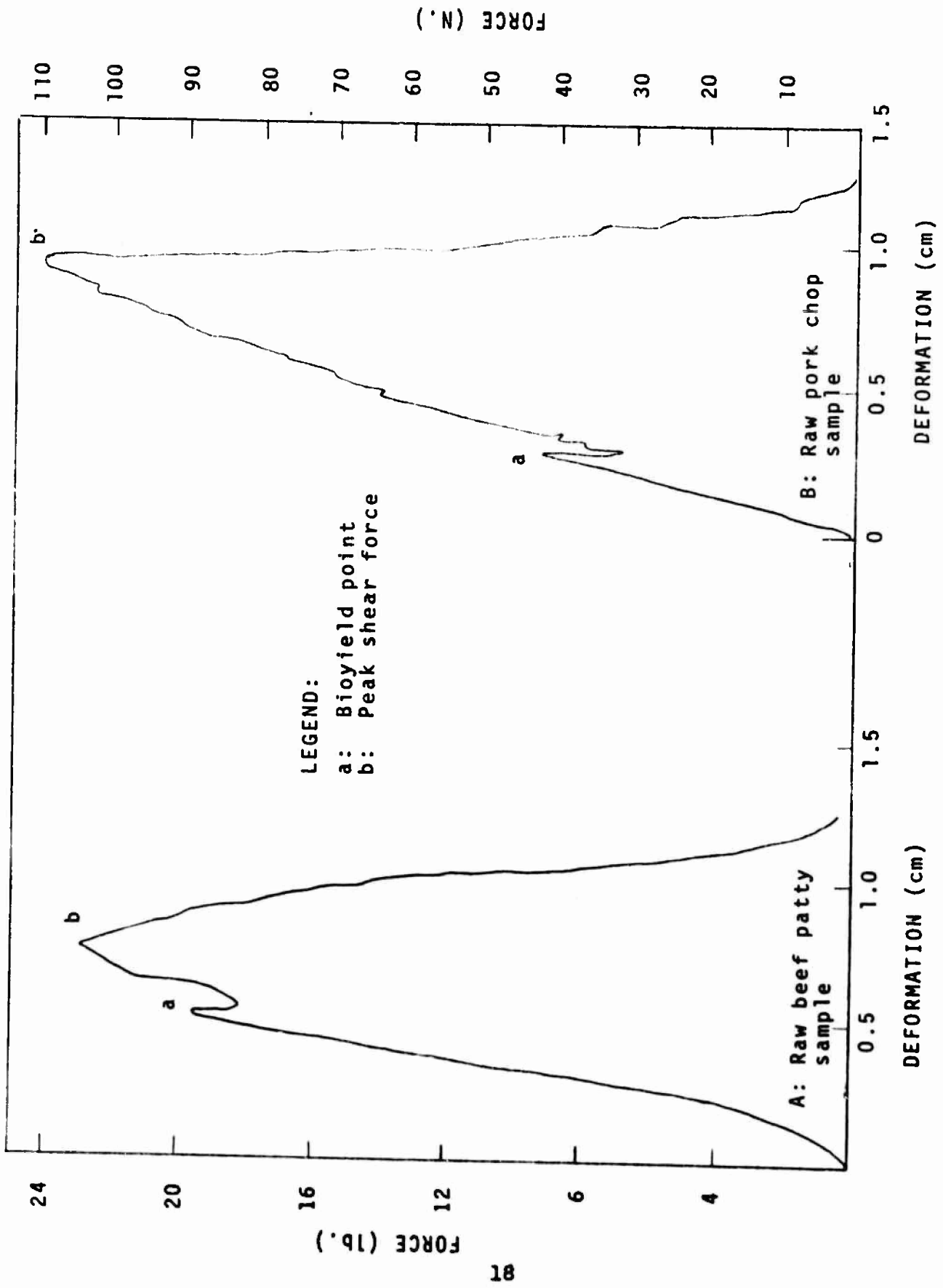


Figure 2. Typical force-deformation diagrams of: (A) a comminuted freeze dried product, (B) a non-comminuted freeze dried product

No. 20 (0.84 mm), No. 30 (0.59 mm), No. 50 (0.30 mm) and the pan arranged in a Ro-tap shaker. Weight of the samples retained on each of the sieves was recorded along with the number of fragments on the top two sieves.

A statistical interpretation of the sieve analysis data was sought by fitting the standard probability distribution functions. It was found that for most drop-fractured products (dehydrated) a log-normal distribution gives an adequate fit. There are various ways in which we can test whether an observed sample distribution fits an overall pattern of a normal curve. The one that is relatively easy to perform was used in this study, wherein cumulative percentage distribution is plotted on logarithmic probability paper illustrated in Figure 3 (Freud, 1967). It might be pointed out that an important feature of a normal distribution is that it can be completely described by its two parameters: (a) mean, and (b) standard deviation; expressed in the appropriate units. As an illustration, Figure 3, gives sieve analyses after drop shattering of a sample, which when plotted on the log-probability paper gave straight lines, from which means and standard deviations of the two distributions can be computed. The geometric mean particle size ( $\bar{d}_{gw}$ ) corresponds to 50 percent of the cumulative weight percent and the (log) normal standard deviation ( $S_{gw}$ ) can be computed as:

$$S_{gw} = \frac{d_{50}}{d_{16}} = \frac{d_{84}}{d_{50}}$$

To find the mean, we have only to observe that since the normal distribution is symmetrical, 50 percent of the area under the curve lies to the left of the mean. Hence, if we check the 50 percent mark on the vertical scale and go horizontally to the line fitted to the points, then the corresponding point on the horizontal scale provides an estimate of the mean of the distribution. To obtain an estimate of the standard deviation, let us observe that the area under the standard normal distribution to the left of  $Z = -1$  is roughly 0.16 and that to the left of  $Z = +1$  is roughly 0.84. Hence if we check 16 and 84 percent of the vertical scale, we can judge by the straight line we have fitted to the points (representing the cumulative distribution of the data) what values on the horizontal scale correspond to  $Z = -1$  and  $Z = +1$ ; their difference divided by 2 provides an estimate of the standard deviation. Since the scale on X-axis is logarithmic, mathematically we can write:

$$\begin{aligned} \log S_{gw} &= \frac{1}{2}(\log d_{84} - \log d_{16}) \\ &= \frac{1}{2}((\log d_{84} - \log d_{50}) + (\log d_{50} - \log d_{16})) \\ &= \frac{1}{2}(\log \frac{d_{84}}{d_{50}} + \log \frac{d_{50}}{d_{16}}). \end{aligned}$$

But since the curve is symmetrical about the mean

$$d_{84}/d_{50} = d_{50}/d_{16}.$$

therefore,

$$S_{gw} = d_{84}/d_{50} = d_{50}/d_{16}.$$

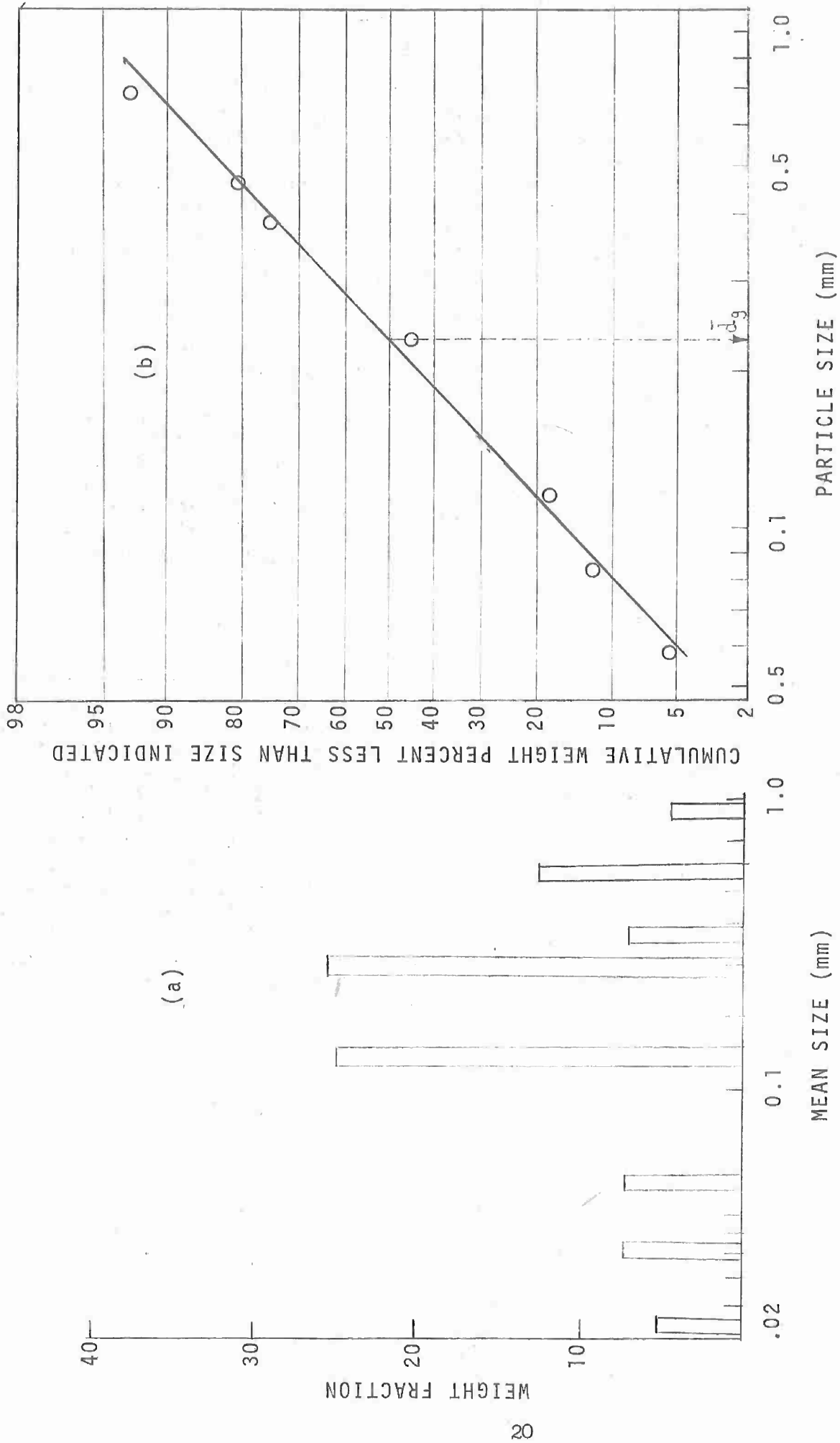


Figure 3. An illustration of drop tested sample fitting log normal distribution. The histogram in (a) gives the size distribution of a cooked beef patties sample after drop testing. The same observations are plotted on a log probability paper in (b) above.

It may be pointed out that if the observed cumulative weight percentages lie very close to a straight line, it should be considered as evidence that the distribution follows the general pattern of a normal distribution (on a logarithmic scale). Only large and obvious departures from a straight line are real evidence that the data do not follow the pattern of a normal curve. Once the normality of distribution is established for the fragmented control and treated samples of a product, the mean sample sizes can be taken as their relative indices of breakage from the initial sample size, and their respective standard deviations are indicators of the uniformity of distribution.

#### Evaluation of the Rehydration Characteristics

Simple gravimetric technique was used to measure the ability of the product to rehydrate under specified conditions. The sample was immersed in excess of water (or rehydrating solution) for a specific time and the amount of water uptake was measured by the net gain in weight. The results were expressed as a ratio of the rehydrated weight to the weight of the fresh sample (i.e., prior to any processing operation), or as Rehydration Ratio defined as:

$$\text{Rehydration Ratio} = \frac{\text{Weight of the rehydrated sample}}{\text{Dry weight of the sample}}$$

Table 2 gives the time-temperature conditions used for rehydration of the various products.

#### Organoleptic Evaluation

The evaluations were carried out in a Sensory Evaluation Laboratory equipped with 11 independent booths and facilities for preparation, rehydration and maintaining the product at a desired temperature. The samples used for organoleptic evaluation were rehydrated as described in Table 2. After removal from the pan, the samples were covered with a moisture-barrier foil to avoid any moisture loss and held in an incubator during serving. The specimens (control as well as treated ones) were individually placed in separate cups, coded randomly and presented to a panel of 15 to 25 judges. The panel was comprised of individuals having undergone a formal training in sensory evaluation. They were not given any information regarding the history of the meat product and were asked to evaluate a batch (two to three) samples in terms of their appearance, flavor/aroma, texture/consistency and an overall impression on a subjective scale as illustrated in Table 3. Separate score sheets were supplied for each sample, and the use of intermediate ratings on the scale and comments was encouraged. Most of the panel tests were carried out either at or about 11:00 A.M. or 3:00 P.M.

The qualitative ratings received from panelists were converted to numerical equivalents on a 1 (unacceptable) to 9 (excellent) scale.



Table 2. Standard conditions for rehydration of various products

Product	Rehydration Medium	Temperature of the Medium, °C	Rehydration Time, min.
Beef patties	water	21-38	10
Chicken	water	82-93	20
Fish squares	water	10-24	10
Pork chops	2% salt water	32-38	20
Pork sausages	water	82-99	0.5
Shrimp*	water	32-38	20

\* After draining, the samples were held for 4 hours at about 4°C in covered containers and weighed thereafter.

Table 3. A specimen sheet for panel tests

PREFERENCE EVALUATION

Name: \_\_\_\_\_ Brand: \_\_\_\_\_

Date: \_\_\_\_\_ Product Flavor: \_\_\_\_\_

Descriptive Product Rating

	Appearance	Aroma/Flavor	Texture Consistency	Overall
EXCELLENT				
GOOD				
FAIR				
POOR				
UNACCEPTABLE				

Comments: (Use other side if needed).

The means and standard errors of the ratings for appearance, flavor/aroma, texture/consistency and overall impression were estimated so that a comparison of the various treatments with the control and within themselves could be carried out. Probability levels of 0.05 or 0.01 were used for ascertaining the differences in the responses for the different samples.

### General Analytical Tests

During the course of product development as well as the evaluation of the stored products, several analytical tests were carried out. A brief description of these tests will follow.

#### Moisture Determination

A representative product sample was pulverized with mortar and pestle, accurately weighed (about 5.0 g.) and dried in either:

- (a) a vacuum oven at 65°C for 18 hours, or
- (b) an air oven at 100 to 105°C for 24 hours.

For most of the experiments, the vacuum oven technique was used (AOAC).

#### Fat Determination: (By Soxhlet Method)

A dehydrated product sample weighing 10.0 grams was extracted with 125 ml of  $CCl_4$  for a period of 6 hours using the Soxhlet extraction apparatus. The fat was extracted in a previously weighed flask ( $W_1$ ). At the end of extraction, the flask was cooled and  $CCl_4$  was removed by distillation. Subsequently, the flask was kept in a vacuum desiccator so that the traces of  $CCl_4$  were removed. The flask was weighed ( $W_2$ ). Fat content for a product was calculated as:

$$\% \text{ Fat} = \frac{(W_2 - W_1)}{\text{Weight of the sample}} \times 100.$$

#### Rancidity Determinations

The two commonly used methods for measuring rancidity of meat are: (1) Peroxide value (methods of analysis, AOAC, 28.023; 1970) and (2) TBA test (Turner *et al.*, 1954). Out of the two, the TBA test is shown to be much more sensitive to incipient rancidity than the test based on the fat peroxide. Turner *et al.* (1954) demonstrated a poor correlation between peroxide values and the rancidity of meat. This is because of the fact that peroxides are intermediates in oxidation of fats and they can vary considerably as the oxidation

proceeds. The TBA test, on the other hand, takes advantage of color reaction between TBA and aldehydes which are believed to be the flavor compounds responsible for oxidative rancidity. Considering the superiority of the TBA test, the oxidative deterioration of the stored samples was evaluated by the 2-thiobarbituric acid (TBA) test, adapted from Turner et al. (1954) as follows:

A 2.0 grams of dehydrated meat sample was ground and placed with 5 ml. 20% trichloroacetic acid in 2N phosphoric acid, and 10 ml. of 0.01M 2-thiobarbituric acid in a 50 ml. centrifuge tube. The sample mixture was heated in a boiling water bath for 30 minutes, with occasional stirring, then chilled in an ice bath for 10 minutes. After removal of the solidified fat layer with a spatula, the pink colored TBA complex was located in the aqueous layer. 15 ml. of solvent containing a mixture of 2:1 iso-amyl alcohol-pyridine was added and the tubes shaken vigorously for 2 min. and they centrifuged for 15 min. at 2400 r.p.m. to break the emulsion formed during extraction. The clear solvent was extracted into a cuvette and its color measured at 538 nm against a solvent blank. The TBA value is defined as absorption at 538 nm.

#### Phosphates Determinations

The quimociac method for determination of the phosphorus pentoxide was used for determining the polyphosphates in dehydrated meat products. According to this method, accurately weighed meat sample is digested in nitric acid to solubilize the phosphate and to hydrolyze condensed phosphate to the orthophosphate form. The orthophosphate is precipitated as quinolium phosphomolybdate,  $((C_9H_7N)_3 H_3O (PO_4).12 M_o O_3)$  containing 3.207 percent  $P_2O_5$ . Knowing the  $P_2O_5$  content of the specific phosphate salt added to the product, its percent in the meat sample is computed using an appropriate gravimetric factor. The detailed procedure was followed as given by Kramlich et al. (1973) and Brotsky (1974).

#### Color Determinations

The color characteristics of the freeze dehydrated meat products were measured after their rehydration using a Beckman DK-2 Spectrophotometer with reflectance attachment. The total reflectance of the rehydrated samples was recorded in the wavelength range 350 to 800 nm. The standard reflectance material was  $MgO$ , and the instrument was operated using: a time constant 0.2, range 0.5-1.5 and sensitivity of 20. The sample port was covered with M-mylar polyethylene to prevent meat drippings from entering the integrating sphere. For the range of wavelength scanned (350-800 nm), the M-mylar polyethylene film is reported to exhibit no measurable absorption of light (Franke and Solberg, 1973). Each scan was started at 800 nm, and each sample was

adjusted to the same starting point with "100% adjusting control" of the spectrophotometer.

It was recognized that a precise estimation of the color characteristics of the different products, including their storage changes, like non-enzymatic browning, were difficult to accomplish. But to get a physical observation of the color on the surface of the products, as observed by the consumer, reflectance technique was chosen. The reflectance technique is non-destructive, eliminates the need for extraction, and the pigment can be evaluated in its natural environment. As described by Franke and Solberg (1971), percent of  $\Delta R_A$  value at 632 nm was taken to be a measure of total hematin pigment concentration in the rehydrated raw meat samples. Percent  $\Delta R_A$  value at 575 nm was taken to be an index of the non-enzymatic browning in the sample (Borchert and Briskey, 1965).

## DEVELOPMENT OF PROCEDURES TO MINIMIZE FRAGILITY IN DEHYDRATED FOODS

### Exploratory Study

Based on an analysis of the concepts underlying mechanics of breakage in freeze dehydrated foods during their shipment and handling, it was hypothesized that any one or a combination of the following processes or product-treatments may modify their mechanical characteristics and provide stability against fragmentation:

1. Optimization of freezing rate before lyophilization;
2. Formation of a stress barrier (plastic film) coating over the individual dehydrated food item;
- 3a. Modification of the matrix strength by mixing, absorbing or injecting appropriate additives within the product; or,
- b. Provision of an edible or water soluble stress-resistant coating directly and integrally with the food surface using a gum or food colloid.

The following studies were carried out to evaluate the effects of each one of these concepts on the mechanical stability and the overall quality of the freeze dehydrated foods:

#### Freezing Rate

It is well-known that for freezing and freeze-drying of meats, the ice crystal size and location, and the resultant void size and location in the freeze dried product are governed by the rate of freezing. Over twenty-five years back, Koonz and Ramsbottom (1939) demonstrated that the histology of freeze-dried chicken meat is markedly altered by the freezing treatment before drying. When frozen almost instantaneously (using dry ice-acetone at  $-75^{\circ}\text{C}$ ), thin slices of chicken exhibited ice crystals and voids after drying which were minute and evenly distributed within the individual muscle fiber. The muscle tissues frozen somewhat more slowly (at  $-40^{\circ}\text{C}$ ) were found to have fewer ice columns within the fiber, with larger diameter and peripheral distribution. If the freezing temperature was further raised, the water was found to be displaced to the center of the fiber and appeared as a single, large centrally located ice column. When the freezing process was sufficiently prolonged, there was reported to be a particular temperature at which water is lost by the fiber and, in consequence, the freezing takes place external to the fibers. Similar observations on the histology of other freeze dried meat products have been reported by Luyet (1962) and King *et al.* (1968). These changes in the distribution of ice crystals and resulting size and locations of voids as the freezing

rate becomes slower can be interpreted in terms of an increase in the ratio of ice crystal nucleation rate to the ice crystal growth rate for freezing at lower temperatures, affording less opportunity for water-migration to a growing crystallite (King, 1971).

With the above understanding of the nucleation and crystal growth during freezing, it was thought that intercellular movement of the water and the pressure exerted by the large columnar ice crystals on the fiber tissues tend to promote fissuring (especially during the relatively slow freezing characteristically used in advance of lyophilization) and thereby weaken the integrity of the meat products after freeze drying. To study the nature and extent of these effects, experiments were planned using the cryogenic freezing facilities at Airco Industrial Gases, Murray Hill, N.J. For fast freezing, a "Kwik-freeze CO<sub>2</sub> Food Freezer" was employed, in which liquid CO<sub>2</sub> is injected through the spray type nozzle located in front of the recirculating fans. For a slow (or intermediate) freezing rate operation, samples were over-wrapped with aluminum foil to avoid freezer dehydration, and stored overnight in a quiescent-air cold room maintained at -30 to -40°C.

In a "Kwik-freeze CO<sub>2</sub> Food Freezer", the samples are fed at the bottom of a refrigerated compartment through a spiral metal mesh Omni-belt system. The dwell time of the product could be maintained in the range of 2 to 30 minutes. Typical time required to freeze completely the six different products investigated was 8 minutes, when ambient temperature was set at -60°C. The innermost core of the samples had attained a temperature lower than -2°C at the time of removal from freezing chamber. The products were then stored overnight in CO<sub>2</sub> snow and allowed to equilibrate to a uniform temperature throughout.

In general, the fast (or instant) freezing was found to decrease the dehydration rate, which is in agreement with the published literature (King, 1971). It may be pointed out that in the present work, the relative values of dehydration rates were measured using thermocouples by temperature rise curves at the geometric centers of the geometrically similar product samples. The fast frozen samples were observed to take 25 to 40 percent longer drying time.

Table 4 gives a summary of the effects of freezing rates on the quality of freeze dehydrated products. The samples frozen in a lower temperature environment (fast frozen) registered a 15 to 20 percent higher peak shear force as compared to the ones which had undergone slower freezing treatment. This difference in the strength characteristics is a compounded effect of the resistance against breakage and the toughness which the fibrous (or proteinaceous) structure of the meats undergoes. This relatively small degree of difference in strength, associated with a poorer rehydration and texture for the quick frozen samples, lead us to believe that the acceptance of instant (or quick) freezing as a technique to improve mechanical stability of the freeze dried foods is of questionable merit.



Table 4. Summary of the shear strength, rehydration ratio and organoleptic-texture evaluation as related to the different freezing rates prior to dehydration for different products

Product*	Freezing Temp. (°C)	Mean Sample Width, cm	Shear Strength (N) (lbs.)		Rehydration Ratio	Qualitative Comments on Product Texture After Rehydration
			Mean	S.E.		
Beef Patties, raw	-40	6.1	215.3 (48.4)	29.4 (6.6)	2.96	
	-75	6.1	269.9 (59.1)	51.6 (11.6)	2.75	
Beef Patties, cooked	-40	4.1	76.5 (17.2)	14.2 (3.2)	2.15	Fair texture
	-75	4.1	92.9 (20.9)	16.5 (3.7)	2.10	Poor texture, tough
Pork Sausage patties	-40	3.8	44.5 (10.0)	2.2 (0.5)	1.71	"Spongy", disintegrated
	-75	3.8	58.3 (13.1)	5.3 (1.2)	1.65	"Spongy", disintegrated
Shrimp, cooked	-40	1.4	61.0 (13.7)	10.2 (2.3)	3.25	Good texture, disintegrated
	-75	1.4	71.6 (16.1)	4.9 (1.1)	3.10	Tough, poor texture, shrivelled

\*The observations are based on 4 to 8 specimen replicates.

## Plastic Film Overwrap

The surface characteristics of the freeze dried product can govern its resistance against the mechanical damage resulting from such actions as impact, abrasion and vibrations during shipment and handling. The application of an edible food surface coating or of a plastic film package that can grip the food snugly to prevent shattering and fragmentation were considered two alternative solutions to the problem. Overwrapping the freeze dried products with a plastic film appeared to be quite promising, considering the fact that a well designed durable package could resist great levels of mechanical damage which may result during shipment and handling of the product. Also, the necessity of packing the finished product in vacuum or an inert gas within cans could be eliminated.

For overwrapping the samples with plastic film, a Bivac Meat Packaging Machine at DuPont, Wilmington, Delaware was used. With incorporation of vacuum and heat shrink skin packaging, several additional product features were explored, such as putting easy open strips of mylar or polyethylene and putting the samples into trays and overwrapping under vacuum and heat to shrink the film over tray and contents. Saran (0.2 mil) coated iolon film (3 mil/3 mil) was used for these studies, which should result in a cost of approximately 7.2¢ per cycle for recovering about 30 cm x 25 cm of the package area\*. Difficulties were encountered in packaging chicken dice and scallops with this system. Sharp meat fibers tended to pierce the stretched film in one case, and in the other the contents were compressed or crushed. On eight other products, the experiments showed highly promising results.

Drop tests conducted on the overwrapped products against the control samples indicted the former ones to be significantly more resistant to breakage. Sieve analysis on freeze dried fish samples subjected to identical drop-impact treatments are given in Table 5. It may be observed that after 30 drops under similar conditions, a sample comprising three fish squares, each one overwrapped individually in iolon broke into five major fragments, as compared to 20 fragments for a similarly handled control sample (not overwrapped). Since there is no irreversible change incorporated into the samples, the rehydration and organoleptic characteristics of the product are not altered by the film overwrap.

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\* Estimates based on suggested prices by DuPont, Wilmington, Delaware.

Table 5. The effect on fragility of plastic (Iolon) overwrapping on the freeze dried fish blocks evaluated by sieve analysis of the samples gathered after drop testing

Sieve Size No.	Percent Weight Retained on Sieves	
	Control Sample (a)	Iolon Overwrapped Sample (b)
9.3 mm	89.74 (15 frag.)	96.66 (6 frag.)
No. 3	1.01 ( 7 frag.)	-
No. 4	0.40	0.59
No. 8	0.80	0.39
No.10	0.40	0.20
No.20	2.82	0.49
No.50	3.62	0.98
Pan	1.21	0.39

\* Each test was carried out with three whole unit pieces randomly selected and weighed (a) 49.7 g. and (b) 50.95 g., respectively

In principle, the appropriateness of heat-shrink overwrapping of the vacuumized freeze dried products to impart them rigidity and present an abrasion-resistant surface is unquestionable. However, under the directions from US Army Natick Research and Development Command, the above approach was discontinued in favor of alternatives that would modify the inherent characteristics of the freeze dehydrated foods and make them less friable.

### Binders and Surface Additives

A preliminary understanding of the interaction of various food binders, especially starches and gums, with the comminuted meats was gathered from another preceding Natick project completed in our laboratory. However, the scope of the two problems was different, which necessitated a reassessment of the type and concentrations of the various binder systems for comminuted meats. Also, the non-comminuted meats presented altogether different problems, which may not be associated with the meat-binder interaction only, but more so with the mechanics of the interstitial injection (if necessary) and uniform distribution of the binder within the tissue.

The preliminary evaluation of the binders was carried out on products which were relatively simple to formulate, i.e., on comminuted meat patties where the binder can be distributed uniformly by simple mixing. Generally, the freeze dried samples having a mixture of modified starches and wheat gluten showed up to 300 percent increase in shear strength without impairing rehydration and textural characteristics of the products. These observations established that by using suitable binder systems, mechanical strength of the freeze dried products can be considerably improved.

Based on a critical evaluation of the preliminary results from the three procedures outlined above, it was recognized that even though the attainment of an optimum modification in the structure of the products might be the most complex, it was also the most rewarding. However, the importance of a suitable package and optimum freezing rate on the product quality cannot be ignored, and selection of additives-approach for this study does not imply non-usefulness of the former two approaches. But owing to limitations of time and funds, only the most feasible procedure to minimize the fragility, i.e., the improvement of product characteristics through additives, was studied in depth.

### Product Modification Through Binder-Additives

#### Screening of the Additives

The primary objective was to seek out FDA-acceptable additive(s) and their levels of application that would impart decisively higher strength (compared to controls) to the freeze dried meat samples, without impairing their rehydration and organoleptic characteristics. Also,

owing to the inherent differences in the physico-chemical characteristics of the eight food products, the mode of application of these additives, the optimal treatment time, and the interaction of the additives with each other (if more than one is used) needed to be evaluated.

#### Additive-materials

It has been known for over a decade that smaller chunks of chopped raw meat can be coalesced into a solid, integrated body by virtue of the natural meat exudates which are liberated by their mechanical treatment (Meas, 1963). Based on a similar principle, in preparation of meat emulsion for sausage making, the critical protein fraction is exuded under critically controlled temperature conditions (3 to 10°C) by comminution. In either case, the exudate is mostly the myosin fraction of meat proteins, which serves as a natural cementitious material for knitting the adjacent surfaces of meat, which then behave as an integral unit during subsequent handling or processing. The full benefit of the bonding action of the exudate formed on mechanical working is, however, obtained upon heat processing or cooking the meat. The addition of sodium chloride and/or certain phosphates increases the amount and rate of formation of the tacky exudate (Karmas, 1970). Even though the precise mechanism of the action of these salts is not known, it is believed that the phosphates in presence of sodium chloride split the contractile meat protein, actomyosin, into its components actin and myosin, and there occurs a partial conversion of these from the gel into the sol form (Bendall, 1954). Along with the solubilization of proteins, a more uniform dispersion of fat is accomplished. Fat emulsification and stabilization in meats is also attained by such non-meat ingredients as non-fat dry milk solids, gelatin, cereal flours and starches (Pintauro, 1974). The probable effect of most of these additives is to disperse the structural proteins as well as fat pockets more uniformly within the meat products, thereby bridging the micro-discontinuities in the raw meat structure. This dispersed structure is further stabilized upon heat processing or cooking.

Even though all these concepts underlying dispersion of proteins and the resultant structural improvement have been demonstrated in meat samples having their natural moisture, their effect on freeze dried meat remains unresolved. Therefore, the following additives (phosphates, starches, and proteins) were chosen to be investigated:

#### (a) Phosphates:

- (i) Sodium Tripolyphosphate (STPP)
- (ii) Sodium Hexametaphosphate (SHMP)
- (iii) Sodium Acid Pyrophosphate (SAPP)
- (iv) Tetra Sodium Pyrophosphate (TSPP)

- (v) Sodium Aluminum Phosphate (SAP)
- (vi) Sodium Monopyrophosphate (SMPP)
- (vii) Kena (a commercial blend of STPP and SHMP)
- (viii) Freezegard FP-88E (a commercial blend of SHMP, NaCl and Sodium Erythorbate).

Samples of the first six of these food grade phosphates were obtained from Stauffer Chemicals, Westport, Connecticut. The last two, viz, Kena and Freezegard FP-88E are specifically blended for their use in meat and seafood products by Merck Chemicals, Rahway, N.J., and were obtained from that source.

(b) Proteins:

- (i) Gelatin (Knox retail grade)
- (ii) Wheat gluten (Paniplus, Division of I.T.T., Kansas City, Mo.).

(c) Sodium chloride:

(d) Starches:

- (i) National-10
- (ii) National-711
- (iii) National-781742
- (iv) Amaizo-839, in combination with Fro-Dex-24 coarse.

All the four additives listed above are commercially available modified corn starches and were specially recommended by the manufacturer as protein and moisture binders in meat products. The first three are manufactured by National Starch and Chemical Co., Plainfield, N.J. Amaizo-839 and Fro-Dex 24 are the modified waxy maize starch and a low conversion corn syrup (28 DE), respectively, and are produced by American Maize Products Co., Hammond, Indiana. A combination of the Fro-Dex 24 as a dispersant base and Amaizo-839 as binder in low moisture meat products was recommended by this manufacturer.

#### Methods of application of additives

The following techniques were used for incorporating these additives (dry powder or solution form) into the products. Depending on the form of additives, the methods can be grouped as:

(a) For incorporation of phosphates, protein or starch solution:

- (i) spray the solution onto product surface, before or after cooking,
- (ii) soak by immersion,
- (iii) chill after cooking, by immersion,
- (iv) tumble with enough solution to absorb,
- (v) admix with comminuted meats,
- (vi) inject solutions via multiple needle injection system.

(b) For mixing dry powders of the additives:

- (i) dust onto product surfaces (before cooking),  
so that it is dissolved in the surface moisture,
- (ii) admix with comminuted ground meat.

The basic criterion used for selecting the mode of application of additives to any product was the precision with which its uniformity of distribution and concentration within the product could be controlled. In case more than one technique appeared promising, experimental evaluation was carried out to arrive at the most feasible one.

#### Statistical design for screening of the additives

Based on an exploratory work or the published literature, the following variables related to the role of additives were found in determining the physical characteristics (including fragility) of the dehydrated products and their palatability following rehydration:

1. Level of additive(s): General ranges of the different additives, in absence of any other interaction, were either obtained from the manufacturers of the commercial products, or established through exploratory work. These levels of additives were used for screening purposes. More precise evaluation to optimize the various responses was, however, carried out only for the significantly influential additives in subsequent experimentation.
2. Uniformity of distribution of additive.
3. Time of treatment, i.e., the time elapsed after incorporating the additives to the meat product and possible termination of the additive reaction effect. Cooking or freezing of the product were taken as the termination period.
4. Differences in the responses of various meat products to an additive or combinations thereof. For example, for different raw meats the optimum levels and proportions of phosphate and sodium chloride to show synergistic effects are known to be highly variable.
5. Mechanical treatment: The extraction of the tacky meat exudate has been discussed earlier. In comminuted meat products, the effect of mechanical treatment was screened by adding a known amount of natural meat emulsion, obtained by "liquifying" the lean meat in a blender.

Since the comminuted meat products offered a distinct advantage in terms of a precise control over the level and distribution of the additives, the three comminuted meat products (beef patties, raw and cooked; pork sausage patties) were considered more appropriate for the screening experiments. Different formulations of the three comminuted meat products were prepared by varying the combinations of the additives (described earlier), the treatment time, the sodium chloride concentration and the amount of meat emulsion. To minimize the time of experimentation, Fractional Factorial Designs were used. The experimental layout for one of the experiments (a  $2^{6-3}$  factorial design), set up to study the effects of (1) a phosphate (Kena), (2) sodium chloride, (3) a starch (National-10), (4) wheat gluten, (5) meat emulsion, and (6) the treatment time on the strength and rehydration characteristics of the freeze dried patties is given in Table 6. According to all the eight formulations given in Table 6, at least five patties were prepared per formulation for each one of the three products. After freeze drying, three patties from each formulation of a product were subjected to drop tests and one each to rehydration and shear tests. Based on appropriate combinations of all the additives described earlier, formulations similar to those presented in Table 6 were prepared and evaluated similarly. Based on the results of these experiments, the following general conclusions were reached:

1. The addition of phosphates, sodium chloride, wheat gluten and emulsion improved the strength of the patties significantly.
2. The effect of starch on the strength and rehydration of the patties was not distinctly favorable.
3. For pork sausage patties, higher levels of sodium chloride retarded the rehydration owing to a high degree of shrinkage, and it caused a release of fat during the freeze drying operation. The response of the same concentration of sodium chloride on beef patties was not as drastic.
4. The general pattern of responses exhibited by raw as well as cooked beef patties was similar, thereby indicating that the effects of treatments on the same product was not altered significantly as a result of cooking.



Table 6

Screening experiment ( $2^{6-3}$  fractional factorial design) to study the effectiveness of Kena (polyphosphate), sodium chloride, starch, wheat gluten, meat emulsion and treatment time on strength and rehydration of lyophilized meat patties

Formulation	% of ingredients added to raw-ground meat					Held for hrs. ( $x_6$ )	Coded Form <sup>**</sup> of Variable					
	Kena ( $x_1$ )	Salt ( $x_2$ )	National-10 ( $x_3$ )	Gluten ( $x_4$ )	Emulsion ( $x_5$ )		$x_1$	$x_2$	$x_3$	$x_4$	$x_5$	$x_6$
A	0.2	1.0	0	0.15	10.0	4	-1	-1	-1	+1	+1	
B	0.5	1.0	0	0	10.0	1	+1	-1	-1	+1	+1	
C	0.2	2.0	0	0	0	4	-1	+1	-1	-1	+1	
D	0.5	2.0	0	0.15	0	1	+1	+1	-1	+1	-1	
E	0.2	1.0	2.0	0.15	0	1	-1	-1	+1	+1	-1	
F	0.5	1.0	2.0	0	0	4	+1	-1	+1	-1	+1	
G	0.2	2.0	2.0	0	10.0	1	-1	+1	+1	-1	-1	
H	0.5	2.0	2.0	0.15	10.0	4	+1	+1	+1	+1	+1	

\* Treatment time refers to the time (in hours) elapsed between mixing the ingredients and cooking (for cooked samples) or freezing (for raw samples). The samples were held under refrigeration between 2-40C during this period.

\*\* High level of the variable is designated +1, the low level is designated -1.

## Formulations Development

### Comminuted meat products

In subsequent experiments, it was attempted to decide upon the optimum concentrations of the ingredients and the modes of their treatments. Theoretically, the behavior of chemical reactions is governed by ascertainable laws, and it should be possible to determine optimum conditions by applying such laws. In practice, however, the underlying mechanisms of the systems are so complex and meagerly understood that an empirical approach was necessary, where each one of the eight products was evaluated individually as influenced by a certain additive system.

(a) Beef Patties, Cooked: Being more representative of the comminuted meat products under consideration, cooked beef patties will be used to describe the procedure and for arriving at the acceptable treatments. Mechanical stability of the samples being one of the prime consideration, it was taken to be its first index of quality. Particle size distributions of the drop-tested samples prepared according to eight different formulations (inclusive of controls) are presented in Table 7. Evaluation of these formulations, comprised of the various proportions of: phosphates, starches, wheat gluten, NaCl and meat emulsion demonstrates that: (a) all the different additive combinations (presented in Table 7) improve the strength of cooked beef patties, (b) starches or starch-polyphosphate combinations are less effective in controlling fragility than appropriate phosphate salts. The effectiveness of the phosphate activity is enhanced by NaC emulsion and wheat gluten. Some of these results (percentages from Col. i, ii, vi, vii, and viii) are presented in Figure 4, which illustrate that:

- (a) the size distribution of the samples after drop testing can be fairly well represented by plotting the data as a log-normal distribution.
- (b) the four additive combinations: (1) sodium chloride;  
(ii) sodium chloride, Kena and wheat gluten (WG);  
(iii) sodium chloride, Kena, and meat emulsion;  
(iv) sodium chloride, Kena, wheat gluten and meat emulsion improve the strength in increasing order.

The effectiveness of each one of these binders or as a composite system can be assessed from Figure 4. The log mean diameter ( $\bar{d}_{gw}$ ) and log standard deviation ( $\bar{S}_{gw}$ ) for the five different samples were estimated from Figure 4, as follows in Table 8.

Table 7

Summary of sieve analyses on the drop tested, dehydrated cooked beef patties with various formulations (the table is based on two or more of the representative tests)

Sieve size, mm (and no.)*	% Weight fraction retained on the sieve from the sample incorporated with (% raw meat basis)						
	Name (Comb.)	15% NaCl iii	15% Emulsion, 2.5% Nat'l-16, 0.2% Nat'l-7II, & 0.5% wheat gluten V	0.5% Kena, 2.0% NaCl, & 0.5% Nat'l-7III V	0.5% Kena, 2.0% NaCl, & 0.15% wheat gluten VI	0.5% Kena, 2.0% NaCl, & 10% Emulsion VII	0.5% Kena, 0.5% Wheat gluten, 2.0% NaCl, & 10% Emulsion VIII
19 mm	0	0	** 9.28 (10)	7.51 (19)	27.90 (6)	34.10 (3)	56.50 (5)
9.5 mm	3.47	11.54	40.02	-	30.60	37.18	21.18
No. 3	7.36	9.11	20.87	12.52	8.08	6.9	5.52
No. 4	8.66	9.51	9.10	12.99	6.02	4.06	2.37
No. 5	6.28	6.07	3.26	13.00	2.41	0.82	0.79
No. 8	34.10	33.40	8.47	13.45	8.43	4.88	3.16
No. 16	25.80	25.30	-	20.97	9.46	5.29	10.45
No. 20	4.55	3.44	4.94	9.39	2.0	3.20	2.42
No. 50	-	1.62	4.60	9.86	2.1	3.29	2.10
Pan	4.90	-	-	0.31	-	-	-

\* See pp 17,19 for sieve sizes corresponding to various sieve numbers.

\*\* The numbers in parenthesis indicate the number of fragments on 19 mm opening screen.

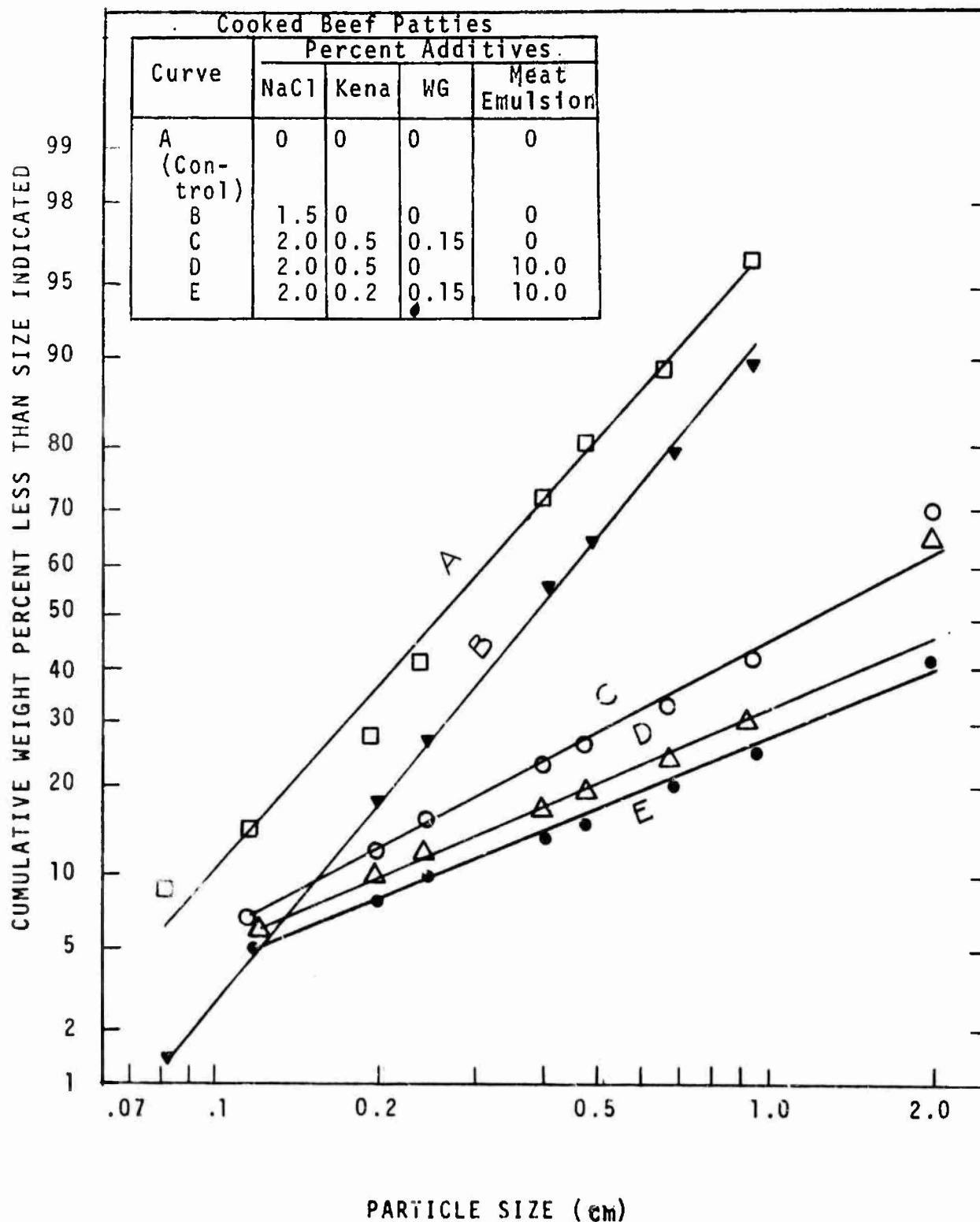


Fig. 4. Log normal probability plots of the freeze dried-cooked beef patties after drop testing. The three curves (C, D and E) illustrate the increase in resistance against breakage by treatment with the appropriate concentration of: salt + wheat gluten; salt + emulsion; and salt + wheat gluten + emulsion, respectively. The geometric mean diameter of the drop tested samples having the treatments E are about 12 times that of the control (A).

Table 8

Summary of the log normal-plots on freeze dried cooked beef patties after drop testing

Additives in Uncooked Ground Meat	Log Mean Diameter $\bar{d}_{gw}$		Log Standard Deviation $S_{gw}$	
	(cm)	(in.)	(cm)	(in.)
A. None (control)	0.25	0.10	5.23	2.06
B. 1.5% NaCl	0.38	0.15	4.83	1.90
C. 2% NaCl + 0.5% Kena + 0.15% WG	1.22	0.48	13.21	5.2
D. 2% NaCl + 0.5% Kena + 10% meat emulsion	2.41	0.95	18.25	7.3
E. 2% NaCl + 0.5% Kena + 0.15% WG + 10% meat emulsion	3.56	1.40	22.35	8.8

It may be clarified that larger  $\bar{d}_{gw}$  and  $S_{gw}$  values, respectively, reflect larger sizes and more non-uniform distribution of particles in a drop tested sample. In other words, a relatively higher value of  $\bar{d}_{gw}$  for samples subjected to the same number and height of drops suggests that it is less susceptible to fragmentation. Accordingly, the samples A to E listed above are in increasing order of resistance against breakage. After subjecting to the similar drop treatments, the mean fragment size of samples C, D and E is between 4.5 to 14 times that of the control sample.

Further tests were run to establish the levels of NaCl, Kena (polyphosphate) and the treatment time for optimum rehydration and strength characteristics. Sensory evaluations using a taste panel were also made on selected samples in the development process. Three different levels of each, Kena (0.2, 0.35, and 0.50%) and sodium chloride (1.2, 1.6, and 2.0%) were chosen which represented 9 different treatments (3 x 3 factorial design). An amount of 0.15% of wheat gluten was added to all these samples and the products were held for 2 hours at 1-3°C before they were cooked. The rehydration and strength characteristics of the freeze dried products are presented in Table 9 which shows a strong interaction between Kena and sodium chloride in terms of both of the response variables. In general, increasing levels of sodium chloride at any one concentration of Kena increases the strength but decreases the rehydration rate of the product. Optimum combinations of Kena and sodium chloride, however, do exist at which the rehydration is not significantly impaired along with remarkable improvement in strength. A formulation consisting of 1.2% sodium chloride, 0.37% Kena and 0.15% wheat gluten offers the most desirable rehydration properties. However, with some sacrifice in rehydration rate, an addition of 0.5% Kena, 0.15% wheat gluten and sodium chloride (1.6 or 2.0%) offers a high degree of improvement in strength.

The effect of treatment time (period elapsed after mixing the ingredients, before cooking the samples), was evaluated by holding the samples; (a) 1.2% sodium chloride, 0.2% Kena, 0.15% wheat gluten; (b) 2% sodium chloride, 0.5% Kena, 0.15% wheat gluten for periods of 1, 2, 4 and 10 hours before cooking. Based on the past experience, the holding periods were chosen such that they represent equal intervals on a logarithmic scale. Data analysis showed no significant difference among the samples representing 1, 2 and 4 hours holding period, thereby establishing a factor of practical importance.

Organoleptic evaluations were carried out on the samples prepared with some of these formulations. Representative results of one of these evaluations are presented in Table 10. Owing to loose and relatively drier texture, poorer appearance, weaker aroma and possible loss of flavoring compounds during cooking and rehydration, the control samples (made by Standard MIL Specifications) were rated significantly ( $\alpha = 0.05$ ) lower than the samples prepared with the better formulations developed above. It is recognized that addition of phosphate salt (Kena) to the meat reduces the cooking losses significantly. Figure 5 shows the effect of various amounts of Kena added to the comminuted meat on the losses during its cooking. It may be observed that an addition of 0.5 percent of Kena to the ground meat brings down the cooking losses to about 18 percent as compared to 33 percent loss for the control samples (0% Kena). Such a large difference in losses during cooking may be rationally associated with the corresponding losses of flavor and aroma from the patties. Also, shrinkage in size resulting from losses during cooking leads to poorer water binding in freeze dehydrated samples on rehydration, which might have been perceived as drier texture by the panel.

Table 9

Summary of the rehydration and drop testing results on cooked beef patties formulated with various concentrations of sodium chloride and Kena\*

Kena Conc. (%)	Sodium chloride concentration (%)									
	0		1.5		1.2		1.6		2.0	
	$\bar{d}_{gw}$	R.R. **	$\bar{d}_{gw}$	R.R.	$\bar{d}_{gw}$	R.R.	$\bar{d}_{gw}$	R.R.	$\bar{d}_{gw}$	R.R.
0	0.25	2.10	0.38	2.03						
	(control)									
0.2					1.14	2.11	0.66	1.86	2.11	1.31
0.35					1.73	2.12	1.40	1.54	1.30	1.22
0.50					1.30	1.98	2.29	1.79	3.56	1.43

$\bar{d}_{gw}$  signifies mean geometric diameter (in cm) of drop shattered samples.

\* All the samples, except the one with no Kena, contained 0.15% wheat gluten and were held for about 2 hours before cooking.

\*\* R.R. (rehydration ratio)  

$$= \frac{\text{weight after rehydration for 10 min. at } 32^{\circ}\text{C}}{\text{weight of the dehydrated material}}$$

Table 10

Sensory evaluation report on cooked - freeze dried beef patties (following rehydration)

Treatment/Additives	Panel* Scores on							
	Appearance		Aroma/Flavor		Texture		Overall	
	Mean	(S.E.)	Mean	(S.E.)	Mean	(S.E.)	Mean	(S.E.)
A. None** (Control Sample)	6.2	(1.66)	4.7	(2.92)	4.5	(2.77)	4.7	(2.91)
B. Mixed with 0.5% Kena, 2% salt and 0.15% wheat gluten, molded into patties, held at 1-20C for one hour and cooked.***	6.6	(1.12)	6.2	(1.47)	6.1	(1.03)	6.2	(1.01)
C. Same formulation as B, but has 10% of meat as emulsion. Process same also.	6.8	(1.24)	5.6	(1.4)	5.5	(1.41)	6.0	(1.24)

\* Panel size = 16 persons

\*\* Sample A scored below acceptability.

\*\*\* In terms of aroma/flavor, texture and overall rating, Sample B was significantly ( $\alpha = 0.05$ ) better than A. The difference between B and C is not significant.



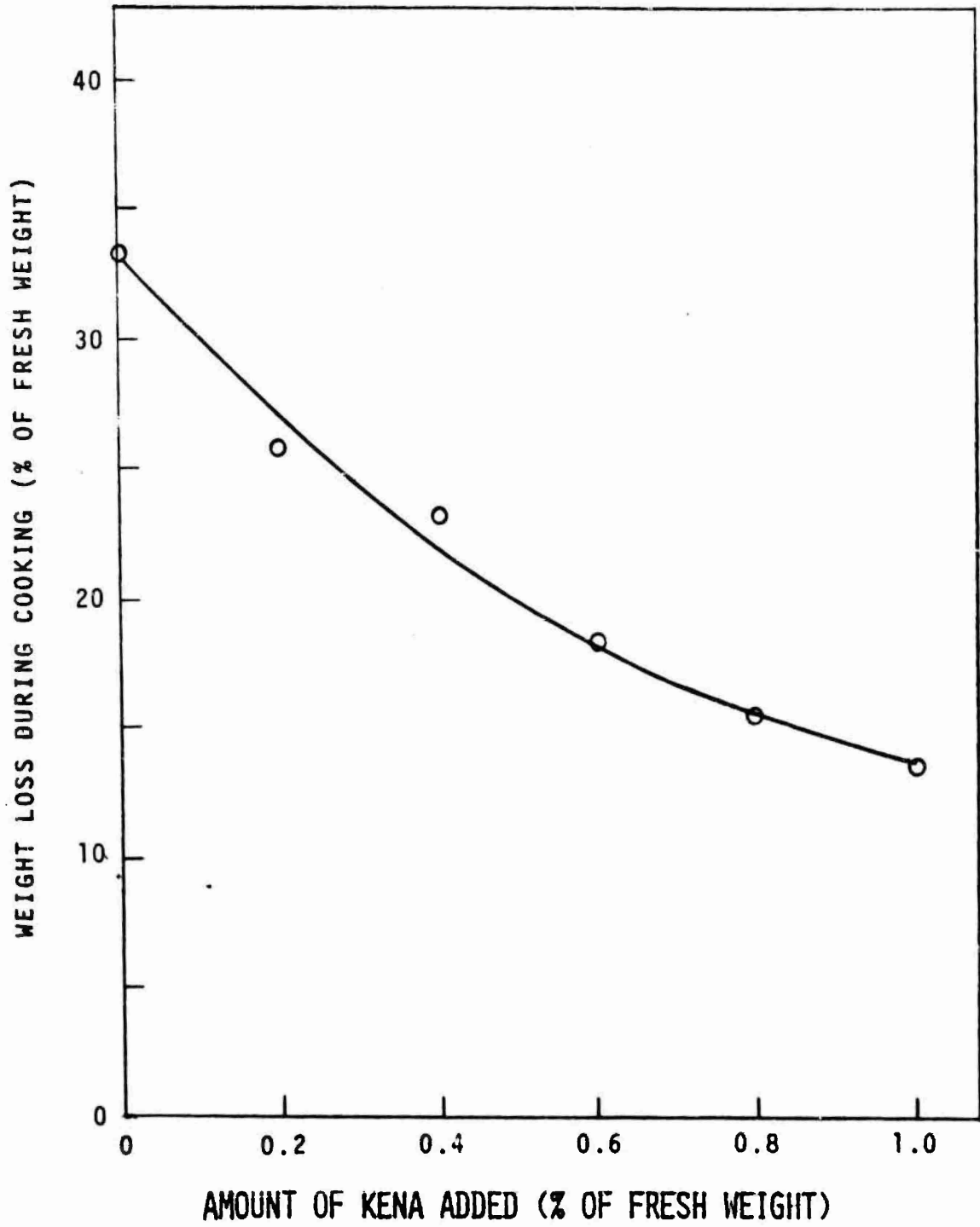


Figure 5. Loss in weight of the beef patties as a function of the amount of Kena

Basically, the same approach as used for cooked beef patties was followed to develop products with optimal characteristics for the other two comminuted meat products (pork sausage patties and raw beef patties). Therefore, for both these latter products, only a summary of the results demonstrating the effect of major treatments (selected ones only) on strength and sensory evaluation will be presented without discussing the procedural details.

(b) Beef Patties, Raw: Size distribution of the weight fraction resulting from drop testing of raw beef patties is given in Table 11. Some of these results are also illustrated in Figure 6. It may be noticed that the modifications incorporated by the various additive-combinations show a similar response as presented in Table 11. However, the control-sample for cooked patties, being considerably more fragile, results in a finer particle size ( $\bar{d}_{gw} = 0.25$  cm or 0.10 in.) as compared to the raw patties ( $\bar{d}_{gw} = 1.02$  cm or 0.40 in.). Panel scores for the treated (raw beef patties) showed an even higher degree of improvement (compared to cooked beef patties) than their respective controls. In terms of appearance, aroma/texture, and overall rating, the treated samples were considered significantly better (see Table 12).

(c) Pork Sausage Patties: Sausage patties were found to be very sensitive to the effect of polyphosphates; more specifically, to the effect of sodium chloride-polyphosphates interaction. At higher concentrations (2% NaCl, 0.5% polyphosphates), a hard gelatinized freeze dried product, practically impermeable to water imbibition, was obtained. A possible explanation of this impermeability might be the excessive extraction of salt soluble proteins (myosin) as binder to the meat, and subsequent shrinkage of collagen (and its irreversibility) during the dehydration process. Therefore, various concentrations of polyphosphates, salt, wheat gluten and their treatment periods were evaluated. Some of the combinations resulting in improved products in terms of strength as well as rehydration are given in Table 13. Table 13 also presents a comparison of the various polyphosphates and the sodium chloride concentrations. Mixing 0.3% STPP (or Kena), 1.25% NaCl, 0.1% gluten and 10% emulsion with the raw pork meat (in addition to various seasonings) and holding the product about two hours before cooking appears to result in optimum characteristics. Panel tests on some of these products are presented in Table 14, which practically follows the same pattern as the previously discussed products.

Therefore, mixing a specified amount of sodium chloride (1.25 to 2.0%, fresh weight basis), polyphosphates (0.3 to 0.5%), wheat gluten (0.10 to 0.15%) and natural meat emulsion (about 10% with the comminuted meat and holding the molded patties for 2 to 4 hours before cooking (or freezing, in case of raw patties) was decided to be a satisfactory procedure to improve mechanical strength as well as organoleptic acceptance of the freeze dried comminuted meat products.

Table 11

Summary of particle size distribution (% weight fraction) of the freeze dried raw beef patties after drop testing

Sieve Size *	Treatment (or additives) prior to freeze drying					
	None (Control Sample)	2% NaCl 0.5% Freez Gard	2% NaCl 0.5% Freez Gard & 0.5% National-711	2% NaCl 0.5% Kena & 0.15% Gluten	2% NaCl 0.5% Kena, 0.15% Gluten & 10% Meat Emulsion	
19 mm	30.88 (9) **	36.33 (7)	0	68.25 (9)	74.67 (8)	
9.5 mm	38.82	18.16	25.79 (40)	-	-	
No. 3	6.02	7.00	11.03	15.45	9.47	
No. 4	4.56	8.29	13.83	0	1.97	
No. 5	12.42	7.42	12.08	3.25	0.73	
No. 8	4.56	8.71	18.46	4.80	4.93	
No. 16	-	7.58	8.46	4.33	4.15	
No. 20	3.53	-	3.43	0	0	
Pan	0	4.97	6.92	3.87	3.36	
Summary Comments	Fragile	Fragile	Fragile	Non-fragile	Non-fragile	

\* See Page 17 & 19 for sieve sizes corresponding to various sieve numbers

\*\* The numbers in parenthesis indicate the number of fragments on the largest opening size sieve.

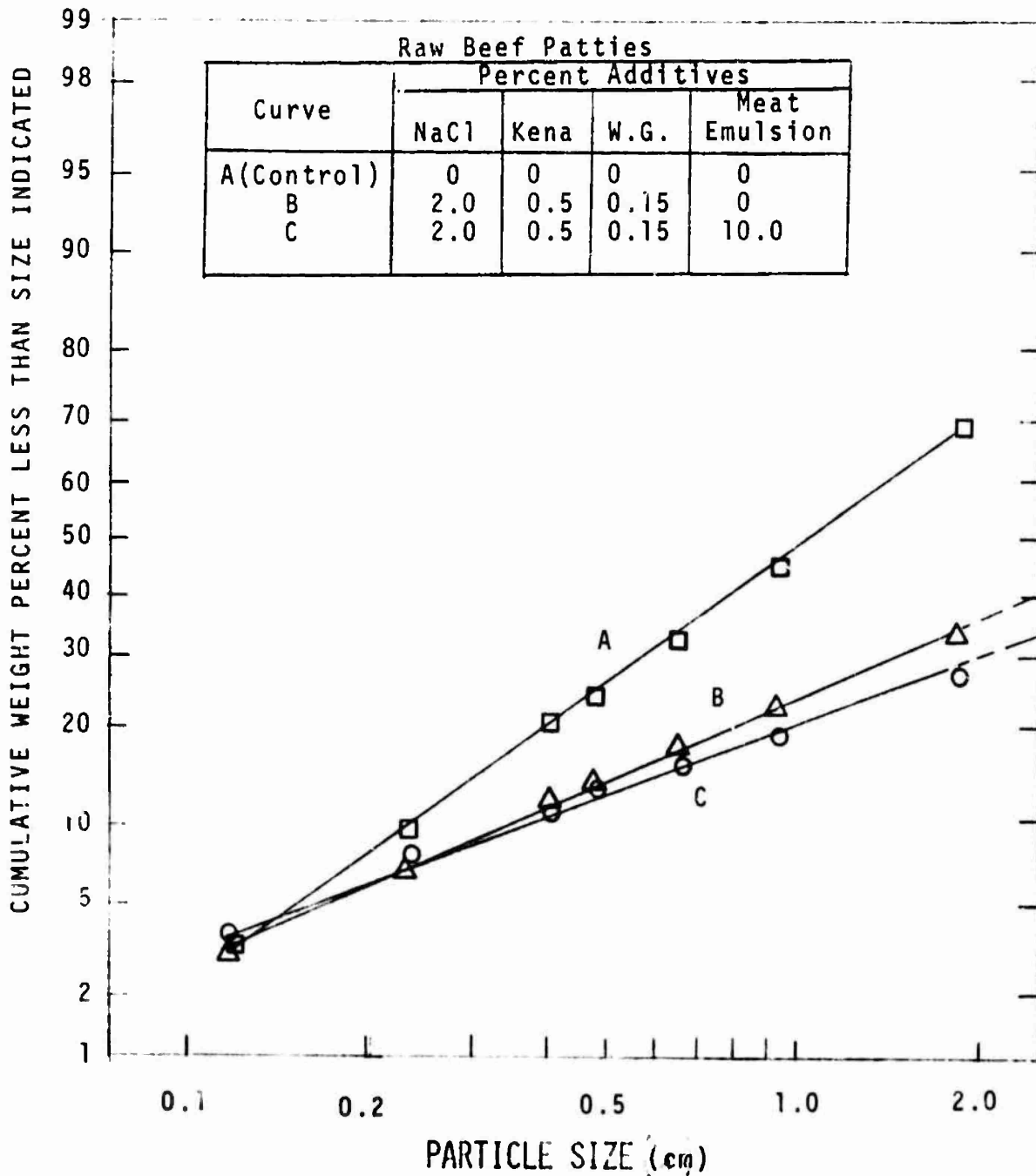


Fig. 6. Log normal probability plots of the freeze dried-raw beef patties. Over 70 percent of the treated sample (C) has size greater than 3/4 in (1.9 cm) against only 30 percent for the control sample. The geometric mean diameter for the treated sample is estimated to be about 4 times that of the control.

Table 12

Sensory evaluation report on raw-freeze dried beef patties (following rehydration and cooking)

Treatment/Additives followed by drying	Panel <sup>1</sup> Scores on							
	Appearance		Aroma/Flavor		Texture		Overall	
	Mean	(S.E.)	Mean	(S.E.)	Mean	(S.E.)	Mean	(S.E.)
A. None <sup>2</sup> (Control Sample)	5.0	(1.75)	5.0	(1.87)	4.3	(2.09)	4.7	(1.97)
B. Mixed with 0.5% Kena, 2% salt and 0.15% wheat gluten, molded into patties, held for one hour at 1-4°C and frozen.	7.0	(1.07)	7.0	(1.07)	5.1	(1.64)	6.4	(1.00)
C. Same formulation as B but has 10% of meat as emulsion. Process also same.	6.6	(1.55)	6.6	(2.02)	5.4	(1.88)	6.0	(1.75)

1. Panel size = 15 persons.
2. Sample A was unacceptable.
3. Both samples B and C are significantly ( $\alpha = 0.05$ ) better than sample A, in terms of their Appearance, Aroma/Flavor, and Overall scores. However, there is no significant difference between samples B and C.

Table 13

Effect of various additives and treatment periods on the strength and rehydration characteristics of freeze dried pork sausage patties (table is based on average of 2 to 5 "replicates")

Treatment (time hrs.) Before Cooking the sample	Parameter 'Evaluated	Additive Concentrations (on fresh meat basis)					0.5% Salt 0.1% W. G. 10% Meat Emul. 0.3% STPP
		MIL-Spec. (Control)	1.25% Salt, 0.3% SHMP	0.1% Wheat Gluten, 0.3% SIPP	10% Meat Emulsion, 0.3% Kena	0.3% STPP	
	$\bar{d}$ gw, cm	0.25	0.38	0.62	0.57	0.56	0.61
	Peak Shear force N (lbs.) R.R.	33.4 (7.5)	48.0 (10.8)	37.7 (13.2)	50.0 (11.2)	50.7 (11.4)	31.1 (7.0)
		1.71	1.50	1.44	1.46	1.54	1.46
		"spongy" "watery"					
2	$\bar{d}$ gw, cm				0.600**	0.79**	0.68
	F, N (lbs.) R.R.				50.0 (11.2)	63.2 (14.2)	39.1 (8.8)
					1.50	1.56	1.50
4	$\bar{d}$ gw, cm				0.57	0.57	0.76
	F, N (lbs.) R.R.				54.3 (12.2)	50.0 (11.2)	46.3 (10.4)
					1.50	1.53	1.39
10	$\bar{d}$ gw, cm				0.76	0.56	0.68
	F, N (lbs.) R.R.				63.2 (14.2)	55.1 (12.4)	43.6 (9.8)
					1.41	1.48	1.40

$\bar{d}$  gw refers to mean geometric diameter (gm) of the drop shattered samples.

R.R. refers to Rehydration Ratio =  $\frac{\text{Weight of the sample rehydrated for 30 sec. at 82 to 99}^\circ\text{C.}}{\text{Weight of the dehydrated sample.}}$

\* Corn meal was gelatinized and, after mixing it with rest of the ingredients, the mixture was kept in chamber maintained at 0°C ambient temperature for about one hour.

\*\* Offers the most desirable characteristics.

Table 14

Sensory evaluation report\* on cooked-freeze dried pork sausage patties (following rehydration)

Treatment/Additives Followed by Drying	Panel Scores On							
	Appearance		Aroma/Flavor		Texture		Overall	
	Mean	(S.E.)	Mean	(S.E.)	Mean	(S.E.)	Mean	(S.E.)
A. Control (conforming to Spec. MIL-P-43383A)	7.0	(1.57)	6.4	(1.60)	7.0	(1.00)	6.5	(1.39)
B. Mixed with 0.3% Kena, 1.5% salt & 0.1% wheat gluten, held for two hours at 1 to 40C	6.1	(1.47)	6.1	(1.28)	4.5	(1.17)	5.8	(1.31)
C. Same formulation as B but has 10% of meat as emulsion. Process same also.	6.5	(1.67)	7.0	(1.21)	6.0	(1.31)	6.5	(1.57)
D. Same as C except for Kena replaced by SHMP	6.6	(1.66)	6.9	(1.57)	7.0	(1.57)	6.5	(1.39)
E.** Same process & formulation as B except for salt conc. decreased to 1.25%	7.2	(2.04)	7.7	(1.83)	6.6	(1.14)	5.7	(1.73)

\*The table represents an average of two tests, each conducted with a panel of 16 persons.

\*\*E was decided to be acceptable treatment.

## Non-comminuted Products

In comminuted meat patties, the ingredients could be mixed in a grinder-mixer, thereby an intimate contact between the meat particles and the additives could be established. For the whole (non-comminuted) meat samples, different treatment procedures were explored mostly relying on the intercellular diffusion of the additives from the surface of a sample to its interior. With an adequate understanding of the time required for distribution of an excipient within the product, and the associated quality problems, the following techniques were evaluated for most of the products:

1. Dusting or tumbling the product with the binder (dry additive mixture) resulting in a fine granular coating on its surface.
2. Soaking the product in a slurry or solution of the additive for the desired time: (a) before cooking; or, (b) after cooking but before freeze dehydration; or, (c) after freeze dehydration. Soaking the dehydrated product in the binder-additive required a secondary drying to bring down the moisture content to less than 2 percent. In any case, owing to concentration gradient, the solutes diffuse partly into the product, leaving a uniform coating over its surface.
3. Spraying the binder solution (of desired concentration) over the surface of the product: (a) after cooking; or (b) after freeze dehydration.
4. Cooking the product in a suitable solution of the additives, resulting in their migration inside the product.

Also, some of the other techniques listed earlier were investigated on certain products. Appropriateness of the different procedures will be dealt with for each product.

Shrimp: In some respects, all the non-communitated products under consideration are unique, but still parallel routes in terms of nature and mode of application of the additives were investigated. Studies carried out on shrimp will generally explain the above generalization.

The effects of the various modes of treating shrimp with gelatin and starch are presented in Table 15. The samples were cooked at a temperature of 74 to 85°C for 8 to 10 minutes in 2% salt (NaCl) solution. It has been suggested that a higher cooking temperature leads to poorer texture in shrimp (Mahon, et al., 1971; Ahmed et al., 1973). It can be observed that the samples cooked in a gelatin or starch solution had poorer rehydration and strength characteristics as



Table 15

Effect of treatments with gelatin and starches on strength and rehydration characteristics of freeze dehydrated shrimp (table is based on mean of 2 to 5 observations)

Treatment Before Freeze Drying	Rehydration Ratio	Strength Characteristics in Dehydrated State			Non-fragmented Sample (% Total Weight) After Drop Testing <sup>2</sup>
		Peak Shear Force <sup>1</sup> (N)	Bioyield Point <sup>1</sup> (lbs.)		
1. None (Control)	2.83	51.6	40.5	9.1	89.0
2. Cooked for 10 min. in 2% solution of:					
(a) Gelatin	2.51	52.0	42.2	9.5	
(b) National-10 Starch	2.61	44.9	35.1	7.9	
(c) National-711 Starch	2.51	57.4	36.9	8.3	
(d) Amaizo-839 Starch (with Frodex 24)	2.37	58.7	44.5	10.0	
3. Dipped (after cooking) in 2% solution of:					
(a) Gelatin	3.44	95.2	60.0	13.5	97.0
(b) National-10 Starch	2.83	78.7	44.5	10.0	
(c) National-711 Starch	3.21	72.5	43.1	9.7	
(d) Amaizo 839 Starch (with Frodex 24)	2.78	68.5	46.7	10.5	95.5

1. Represents mean of 9 observations taken near head, mid-body and tail of three samples for each treatment.

2. Drop tests were not run on all the samples.

3. After cooking the samples were dipped in appropriate solution for 2 minutes.

compared to the controls or to the samples dipped in these additives after cooking. The rehydration ratio of the samples cooked in the additive solution was about 10 percent lower than that of the control, whereas the samples dipped in the additive after cooking exhibited up to about 20 percent higher rehydration ratio than the control. It may be pointed out that the dehydration ratio (ratio of the frozen sample weight before drying to the dry weight) of the sample was 4.3, and, therefore, none of the rehydrated samples attained that level. It may also be observed that soaking the cooked samples in binder solution substantially increased their strength. Of the different starches and gelatin binders, gelatin appears to exhibit the most desirable strength and rehydration characteristics. But, like the effect of dipping into all other solutions presented in Table 15, there is a difference of about sixty percent in the peak shear force and bioyield point for the gelatin treated-dehydrated samples. Such a large difference in these force values indicates a tougher center and possibly an undesirable non-uniform texture.

Organoleptic evaluations carried out on the gelatin-treated samples, which appeared to be the most promising of the treatments presented earlier, are given in Table 16. An extremely low texture rating rendered the gelatin-treated samples unacceptable, with an overall rating of 5.9 against 7.0 for the control. As a result, even though the possibility of a more desirable time-concentration treatment was not ruled out, alternative treatments were investigated.

After an evaluation of the effects of these additives, it was considered necessary to improve the texture of the rehydrated shrimp considerably, in order to obtain acceptable panel ratings. The toughness of fish muscles is attributable to an increased bonding between myofibrillar proteins and a reduction in its extractability (Connell, 1964). Toughening of the muscles of Sacramento blackfish has been attributed to the denaturation of myosin (Chu and Sterling, 1970); protein crystallization and cross-linking of proteins (Mao and Sterling, 1970). Possibly similar mechanisms are responsible for toughening of shrimp during heat processing including freeze dehydration. A surface coating of gelatin or starch contributes to the toughening problem.

Polyphosphates have been used to tenderize shrimp and cod muscles (Love, 1968; Ahmed, *et al.*, 1973). These researchers theorized that the tenderizing effect of polyphosphates was due to weakening of muscle fiber structure and swelling of the fibers to give a protein gel system which increased the water holding capacity of the protein structure. With these concepts supporting the tenderizing effect and the results on the reduction in fragility of the comminuted meats, studying the effects of phosphates on shrimp was a logical step.

Table 16  
 Sensory evaluation report on cooked-freeze dehydrated  
 shrimp (following rehydration): Gelatin evaluation

Treatments	Panel Rating For				Comments
	Appearance	Flavor	Texture	Overall	
A. Control (none)	7.6	6.8	6.6	7.0	Tough, bland
B. Dipped in 2% Gelatin solution after cooking but before freeze drying	7.4	6.1	4.8	5.9	Tough, stringy, fishy

The effects of some of the phosphate treatments on the fragility and rehydration of the freeze dried shrimp are given in Table 17. Although these treatments do not describe the amount and distribution of the phosphate-salt within the shrimp tissue, the favorable responses in terms of breakage as well as rehydration in some cases were encouraging. In general, the extent of mechanical damage was reduced to about half as a result of the phosphate treatment (from about 10 percent for control to about 5 percent for the phosphate treated samples). Among the effects of phosphate treatments on breakage, there was no practical difference, but the rehydration of the samples dusted with the phosphate powder was 10 to 20 percent higher than their respective soaked counterpart. Considering these characteristics of the sample dusted with phosphates, organoleptic evaluations of those treated with Kena, SHMP and STPP were carried out. The results of the preference test are given in Table 18, which shows the superiority of all the Kena and SHMP treatments over the control in terms of Aroma/Flavor, Texture, and overall liking by the panel. Of the two, Kena and SHMP, the former seems to favor better quality results mechanically and in terms of rehydration, in addition to organoleptic preference. Therefore, dusting the samples with Kena was considered to be the desired treatment, but the time of dusting was still to be evaluated precisely. The losses during cooking and the toughness changes in fresh-cooked as well as rehydrated shrimp after freeze drying were evaluated as affected by the controlled duration of time between dusting with phosphate and subsequent rinsing. Relative weights of the samples at each operation, in sequence, for rehydration after freeze drying are given in Figure 7. It can be seen that (a) there is a close parallelism between fresh-cooked weight and weight recovered after rehydration of the freeze dried samples; (b) the maximum weight-yield (as-is, wet basis) for both the fresh-cooked and rehydrated samples correspond to a post-dusting pre-rinsing interval of 10-12 minutes, and is about 13 percent higher than the untreated control (no phosphate treatment); (c) the dry-weight of the control sample is about one percent (fresh weight basis) less than most of the phosphate treated samples. This latter difference might be attributed to the loss of the water soluble compounds including some of those responsible for lower flavor ratings for the control, in organoleptic evaluation.

Toughness of the fresh-cooked and rehydrated shrimp was determined by Shear Test. Force-deformation diagrams were plotted at the centers of three practically equidistant segments of cooked whole, intact shrimp units. The three segments will be referred to as "butt", "mid" and "tip" sections. Five whole shrimp units were randomly selected for each phosphate treatment (including control) and peak shear forces were evaluated at their butt, mid and tip locations. Means of the 15 force values for each treatment are presented in Figure 8. It can be observed that the toughness of the control (no phosphate treatment) increased to over 200 percent after freeze drying (from 8.0 to 12.2 lbs. or 36-76 N force). There is a steep decrease in the shear force of both the fresh-cooked as well as rehydrated samples.

Table 17

Summary of drop tests and rehydration studies on cooked<sup>\*</sup>-freeze dried shrimp with various treatments prior to freeze drying

Treatment(s) Before Freeze Drying	Whole Weight Fraction (%) After Drop Testing <sup>**</sup>	Rehydration Ratio
1. None (Control)	89.8	3.82
2. Cooked, chilled and dipped in 2.5% gelatin for <u>one min.</u>	93.3	3.46
3. Dipped in 10% Kena solution (at 70°F) for one min., before cooking	94.4	3.46
4. Dipped in 10% Kena solution, cooked, chilled and dipped in 2.5% gelatin for one min.	95.5	3.50
5. Dusted with Kena, rinsed with cold water after five min. and cooked	94.2	4.10
6. Dusted with SHMP, rinsed with cold water after five min. and cooked	94.0	3.87

\* Samples were cooked for 10 min. at 85°C in 2% salt water.

\*\* After drop-testing the weight fraction retained at 19 mm opening sieve or pieces comprised of a half or larger shrimp in the underflow

Table 18

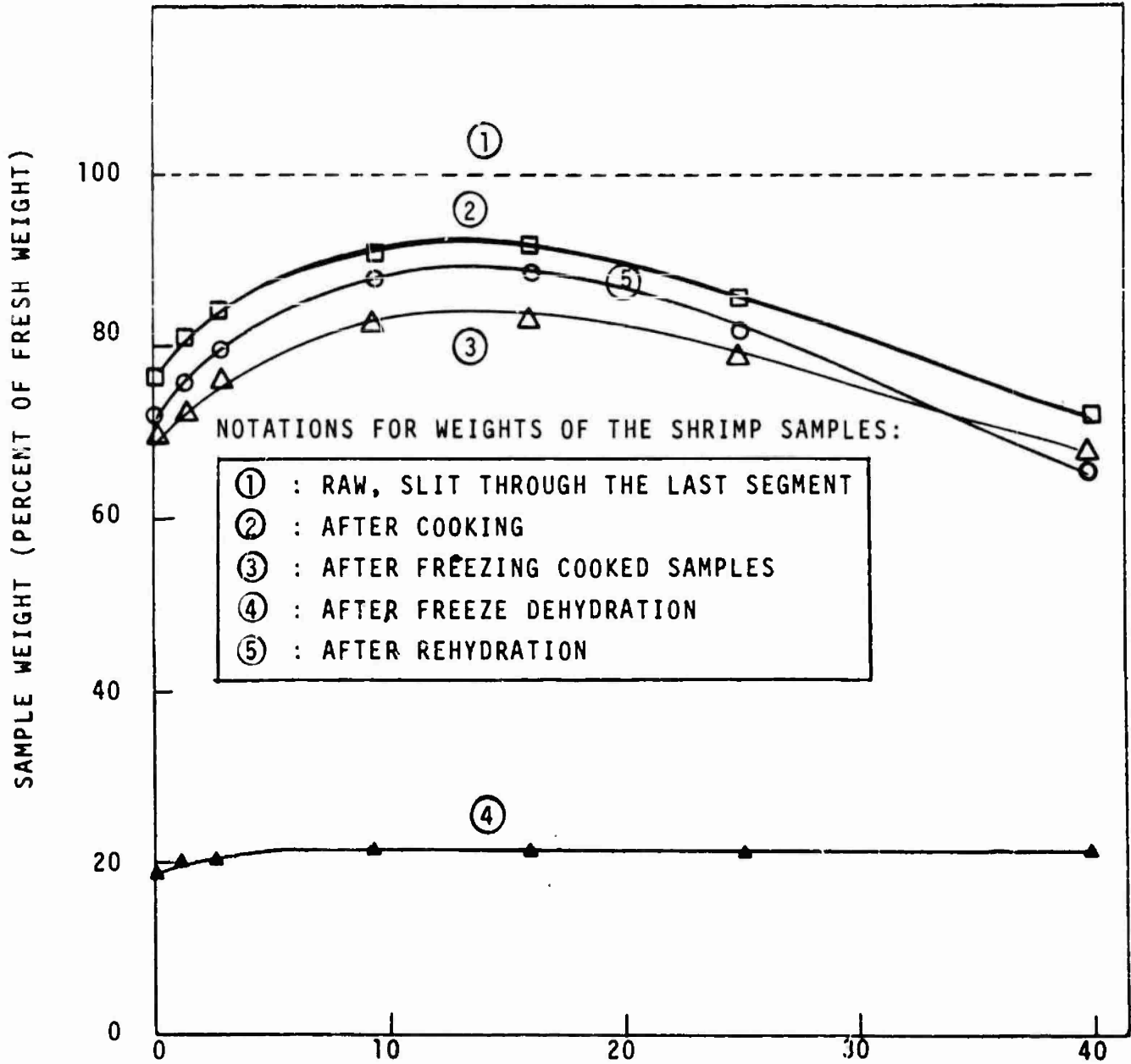
Sensory evaluation report on cooked-freeze dehydrated shrimp (following rehydration): Polyphosphates evaluation (Table is based on three different tests)

	Panel Ratings* For				
	Appearance	Aroma/Flavor	Texture	Overall**	
	Mean Std. Error (S.E.)	Mean (S.E.)	Mean (S.E.)	Mean	(S.E)
A. None (Control Sample)	8.1 (1.0)	6.0 (1.39)	6.7 (2.06)	6.3	(1.68)
B. Dusted with Kena, rinsed with cold water after five minutes and cooked in 2% salt water at 185°F for 10 minutes.	8.0 (1.28)	7.0 (1.78)	8.0 (1.25)	7.57	(1.28)
C. SHMP was used instead of Kena, following the same procedure as B above.	8.1 (1.12)	7.0 (1.84)	7.3 (1.46)	7.3	(1.18)
D. STPP was used instead of Kena, following the same procedure as B above.	7.6 (1.32)	6.3 (1.62)	6.6 (1.98)	6.3	(1.72)

\* Panel size = 15 persons

\*\* Aroma/flavor, texture and overall rating for treatment B, and aroma/flavor and overall ratings for treatment C are significantly ( $\alpha = 0.05$ ) higher than the control.

# SHRIMP



HOLDING PERIOD FOLLOWING DUSTING WITH PHOSPHATE (MIN.)

Fig. 7. The relative weights recovered after cooking, freezing, freeze drying and rehydration of the shrimp samples dusted with Kena and held for different lengths of time

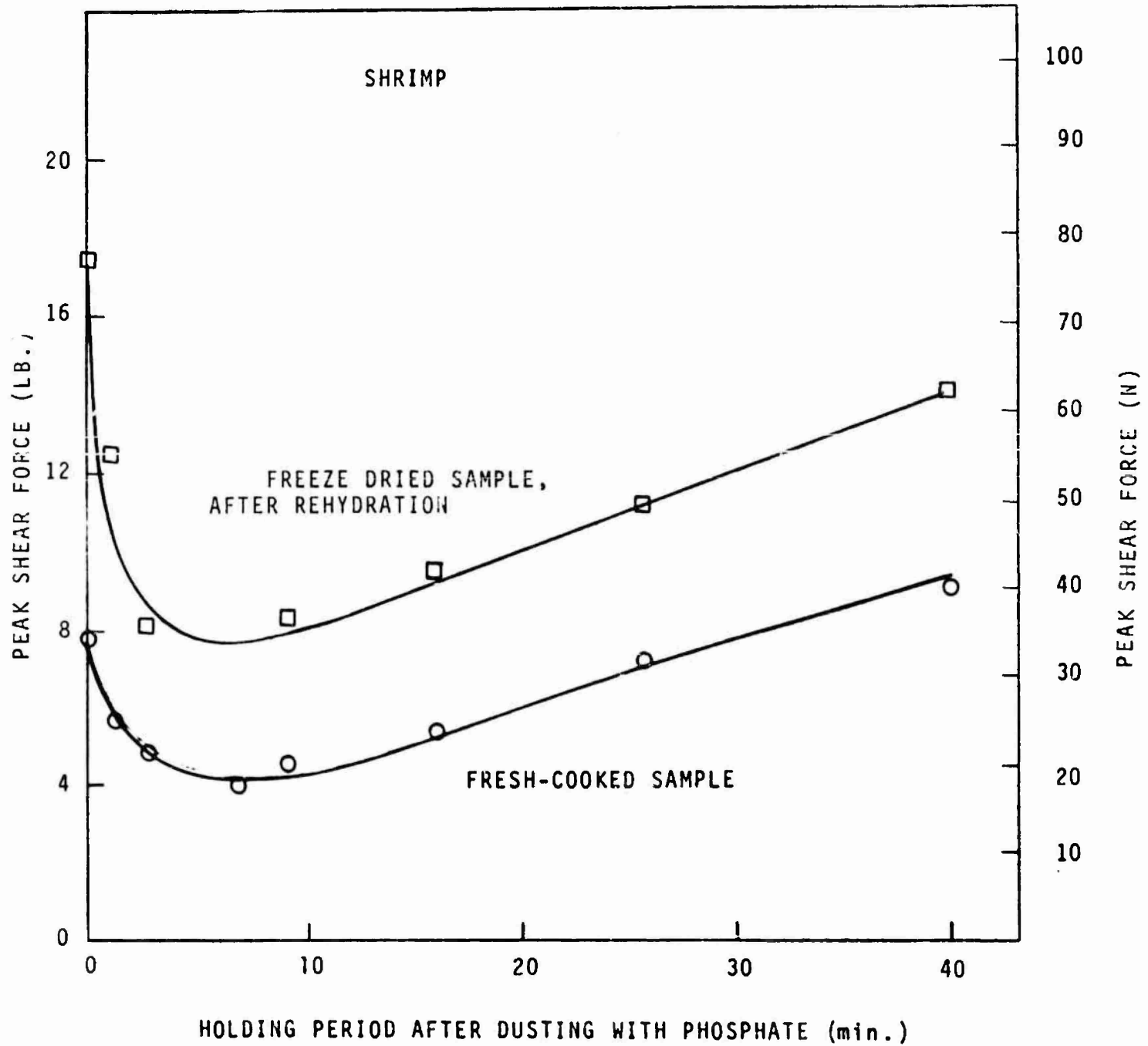


Figure 8: Toughness (in terms of peak shear force) of fresh cooked and rehydrated shrimp presented as a function of the holding period after dusting the samples with Kena



with addition of phosphate. After a post-dusting period of about eight minutes, both of these groups exhibit a zone of least toughness. At the level of phosphate corresponding to this minimum force, the rehydrated samples register a peak shear force of 35 N (7.8 lbs.) which is somewhat less than for the fresh-cooked samples. With longer post-dusting period, the toughness values of both the fresh-cooked as well as rehydrated shrimp increase almost parallel to each other. It is of great interest to observe that dusting shrimp with excess phosphate and holding for 5 to 10 minutes can make the freeze dried product more tender than the fresh-cooked shrimp. This favorable effect might be partly due to reduction in cooking losses by phosphate treatment. With higher cooking losses, probably a shrinkage or collapse of proteinaceous matrix takes place leading to rigidity and toughness of structure. Figure 9 illustrates the relationship between cooking losses and toughness of fresh-cooked shrimp.

The amount of phosphate retained in cooked-freeze dried shrimp was analyzed by the Quimociac method. The amount of STPP uptake as a function of the post-dusting holding time is presented in Figure 10, which demonstrates a logarithmic relationship between two variables. Also, it may be observed that corresponding to a post-dusting holding period of ten minutes, 1.3 percent STPP is found to be retained on the food's dry weight basis. This level of phosphate in dry samples amounts to a 0.25 percent concentration of STPP in fresh moist samples.

Pork Chops, raw: A summary of the effects of dusting and soaking the raw pork chops with various phosphates (poly- and pyro-) on the strength and rehydration characteristics of raw-freeze dried pork chops is given in Table 19. In general, soaking the raw pork chops in a phosphate-NaCl (1:3) solution resulted in "case-hardening" and poorer rehydration as a result thereof. The mechanism of this "case hardening" is well understood, but a similar phenomenon as discussed for pork sausage patties gives rise to the hard gelatinized surface matrix. Dusting (or tumbling) the raw pork chops with phosphates (both poly- and pyro-) generally improved their rehydration property over the controls. Dusting with Kena and holding the samples at refrigerated temperature for one or two hours appeared to be the most favorable choice out of the various dusting treatments studied.

Panel tests on the samples dusted with Kena and SHMP were conducted to study their organoleptic acceptance. Samples with both these treatments were given significantly ( $\alpha = 0.05$ ) higher ratings than the controls, in terms of the attributes (see Table 20). It may be pointed out that even though significantly better than the controls, texture rating for the treated samples was relatively low. A non-uniform phosphate treatment within the sample might be responsible for the "non-uniform" texture, and given the optimum treatment, it is expected that texture and the overall acceptance of the samples will be improved.

Pork Chops, cooked: For cooked-freeze dried pork chops, the effects of various treatments were analogous to their raw-freeze dehydrated counterparts. Dusting the raw chops with Kena (or alternatively, dipping the raw chops in a solution of sodium chloride and Kena), holding

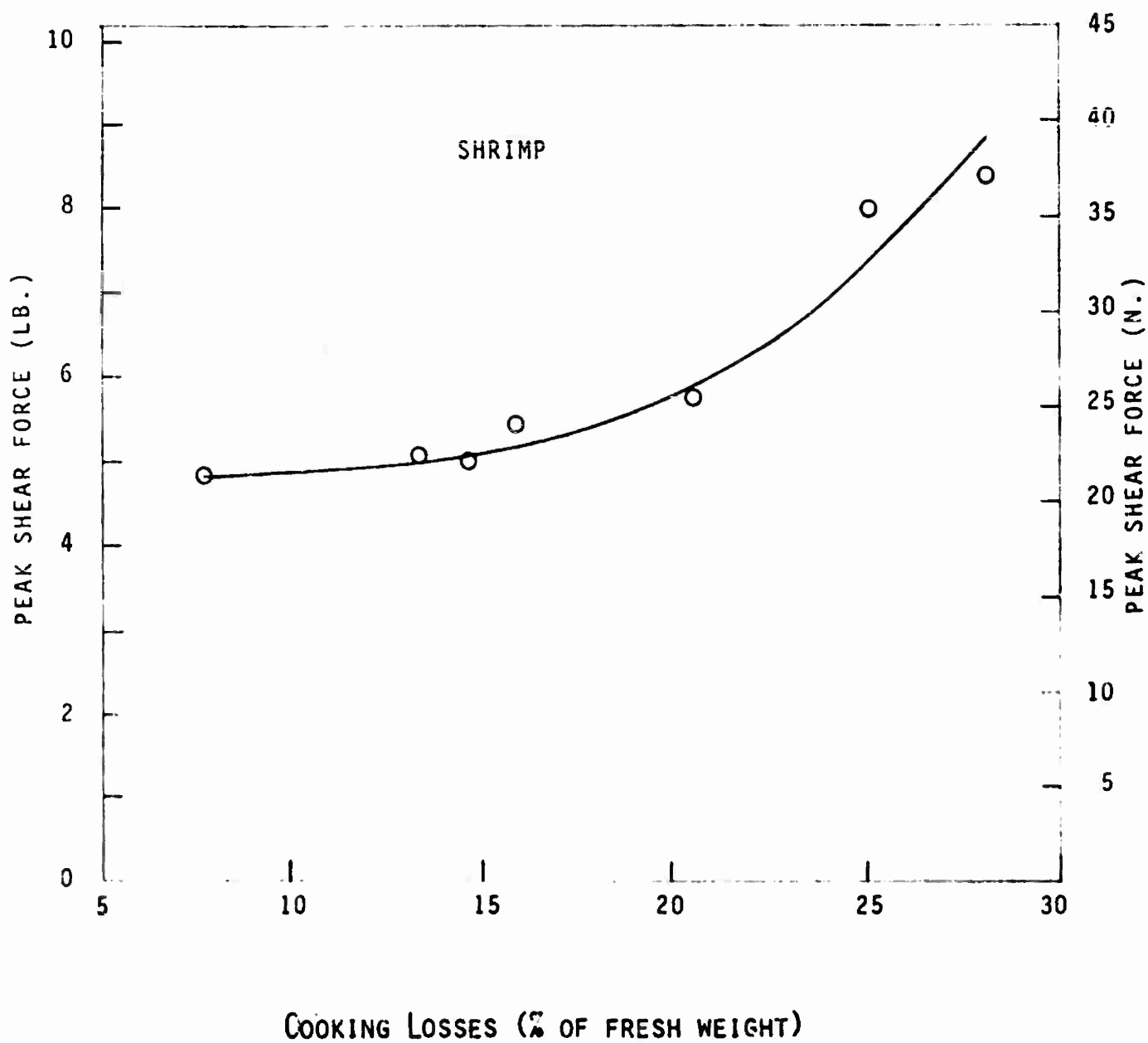


Fig. 9. Changes in the toughness (shear force) of fresh cooked shrimp as a function of the weight loss (mostly moisture) during cooking

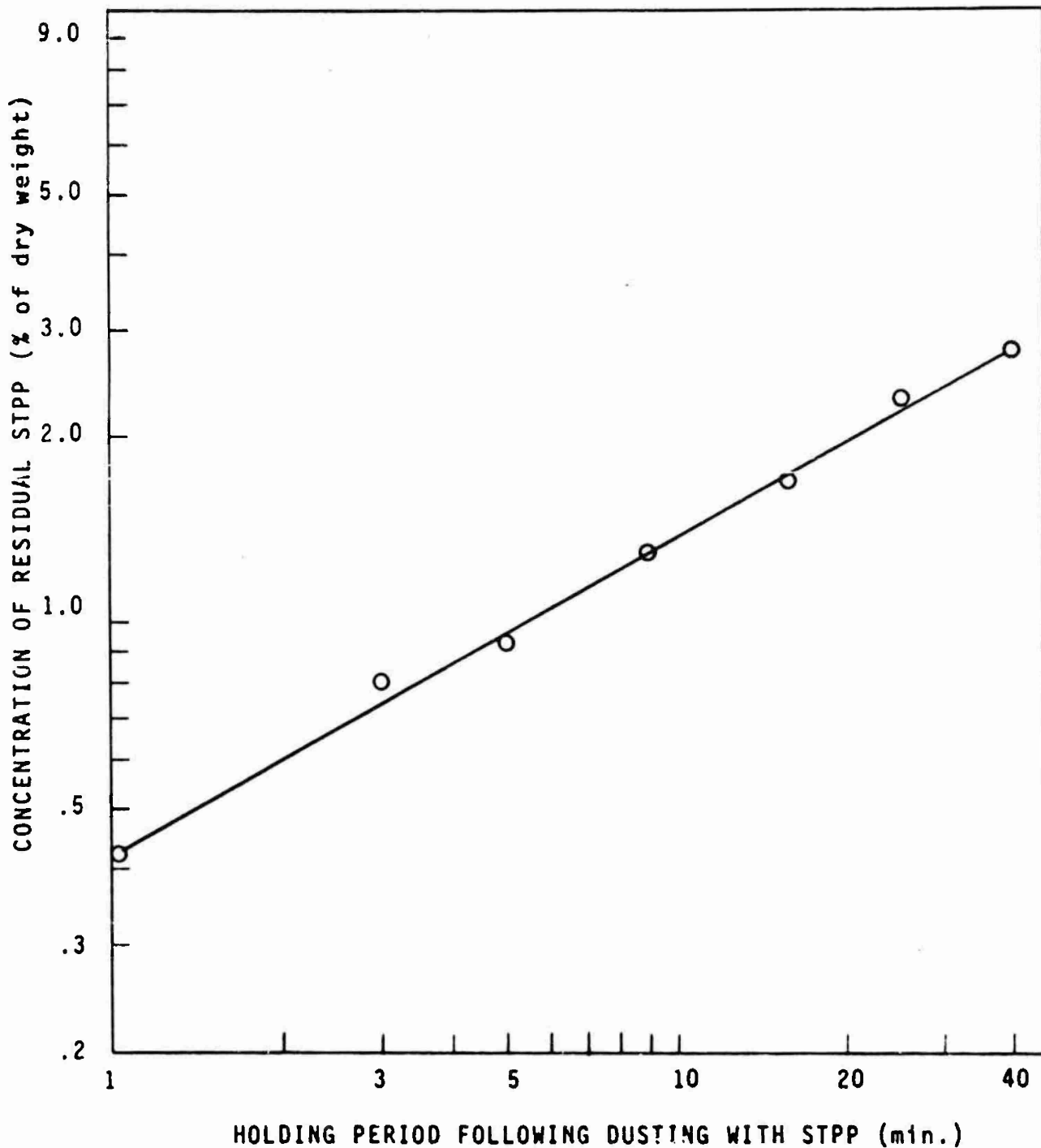


Figure 10. Amount of sodium tripolyphosphate (STPP) retained in the cooked samples as a function of the duration for which peeled, deveined shrimp were coated with excess of STPP powder. (The moisture content of these samples was 81.5 percent on fresh weight basis).

Table 19

Summary of drop tests and rehydration studies on freeze dried raw pork chops with various treatments prior to freeze dehydration (the table is based on an average of three trials)

Treatment	Analysis* After Drop Testing			Rehydration Ratio
	Weight-Fraction Retained on a 19 mm Sieve in Sieve Analysis (a)	No. of Fragments in (a) above per Sample of 3 Units Before Drop Testing (b)		
1. Control	89.2	11		2.11
2. Dusted with one of the following phosphates, held for one hour at 34-38°F and rinsed with cold water before freezing:				
(a) Sodium Acid Pyrophosphate (SAPP)	90.1	10		2.26
(b) Tetrasodium Pyrophosphate (TSPP)	93.8	9		2.41
(c) Sodium Hexametaphosphate (SHMP)	91.9	7		2.79
(d) Sodium Tripolyphosphate (SHMP)	97.8	10		2.32
(e) Kena	95.8	6		3.34
3. Same as 2(e), but held for two hours instead of one hour	97.2	6		2.69
4. Soaked for one hour in (salt + one of the following polyphosphates) (3:1) 20% solution at 1 to 2°C drained and rinsed before freezing:				
(a) TSPP	93.4	6		1.95
(b) SHMP	93.5	6		1.96
(c) Kena	93.4	6		1.32

\* Sieve analysis was carried out after drop testing. For drop testing, the chops were sealed in No. 10 cans and dropped 30 times from a height of one meter.

Table 20

Sensory evaluation report on raw freeze-dried pork chops (following rehydration and cooking)

Treatment	Appearance		Aroma/Flavor		Texture Consistency		Overall	
	Mean	St. Error (S.E.)	Mean	S.E.	Mean	S.E.	Mean	S.E.
None (Control Sample)	4.9	(1.92)	4.2	(2.11)	2.5	(1.19)	3.5	(1.60)
Dusted with Kena, held for one hour at 1-30C rinsed and frozen	6.7	(1.05)	6.3	(1.44)	5.2	(2.41)	5.3	(1.91)
Dusted with SHMP, held for one hour at 1-30C rinsed and frozen	6.5	(1.19)	5.9	(1.82)	4.7	(2.3)	5.1	(1.77)

\* Panel size = 15 persons

\*\* Both the treated samples are significantly ( $\alpha = 0.05$ ) more acceptable than the control sample in terms of appearance, aroma/flavor, texture and overall evaluation.

for about two hours at refrigerated temperature and cooking thereafter imparts the product with desirable characteristics in terms of mechanical stability, rehydration and organoleptic acceptance.

Table 21 shows that incorporation of Kena with or without sodium chloride in pork chops by soaking or dusting results in up to 30 percent reduction in breakage after drop testing as compared to the control. Also, the rehydration of the soaked samples was about 45 percent higher than the control. It may be pointed out (see footnote, Table 21) that for pork chops neither the treated products nor the controls rehydrated after freeze-drying to the pre-drying moisture levels. Also, during cooking, the control samples lost up to 33 percent of their fresh-raw weight, compared to only about 20 percent for Kena + salt treated samples. As a result of these losses, the meat tissue structure had undergone shrinkage and a proportional change in its water holding capacity. The above alteration in the water holding capacity after freeze dehydration is reflected in the rehydration characteristics of the product. A summary of the panel tests on the cooked, dehydrated pork chops after rehydration of the samples is presented in Table 22, which shows: (a) the Kena-treated samples are generally more acceptable to the panel than their untreated counterparts (control); (b) the texture value of the Kena-treated samples is significantly improved over the controls.

Fish Squares: A summary of the drop tests and rehydration characteristics of the dehydrated haddock (Melanogrammus-aleglectinus) fish squares is given in Table 23. The treatments are broadly classified into: (A) those applied before freeze drying, (B) those applied after freeze drying. As a result of the latter of the two treatments, the moisture content of the samples increased and a secondary drying was required. The treatments before freeze drying mostly comprised of adding various phosphates to the fish samples. These treatments did not show any improvement in the fragility of the dehydrated samples. In fact, all the phosphate treatments presented in Table 23 tended to increase the fragmentation of the dehydrated samples over their control. In spite of the theoretical basis and the experimental findings on the products supporting the positive influence of phosphates on binding strength, fish was found to be an exception. Such an anomaly in these results cannot be explained at present.

Effects of surface-coating the fish samples with gelatin and starches were evaluated before as well as after freeze drying. Coating the fresh-moist fish squares with binders resulted in a superficial but impermeable film, which retarded the freeze dehydration. The barrier characteristics of the gelatin film were so strong that it was stretched and ruptured before moisture could migrate during freeze drying. For the samples coated with starches and gelatin after freeze drying, the rehydration and fragility characteristics are given in Table 23. The samples sprayed with gelatin suffered practically no breakage. This favorable effect might be due to the bridging of gelatin over the micro-cracks in freeze dehydrated samples.

Table 21

Summary of drop tests and rehydration studies on freeze dried cooked pork chops with various treatments prior to freeze dehydration

Treatment	Analysis after drop testing*		Rehydration Ratio
	Weight (a) fraction (%) retained on 19 mm sieve after sieve analysis	No. of (b) fragments in (a) above per original sample of 3 units	
1. None (Control)	51.9	8	1.57
2. Dusted with one of the following, held for specified time at 1-30C washed and cooked: (a) Freez Gard, for 2 hours (b) Kena, for 2 hours	30.3 66.3	8 8	1.37 1.47
3. Dusted with Kena, held for 2 hours at 1-30C dipped in gelatin before cooking	70.4	9	1.28
4. Dipped in 20% solution of any one of the following phosphates + NaCl (1:3), held for one hour at 1-30C, washed and cooked: (a) Kena (b) SHMP (c) STPP (d) TSPP	61.9 47.8 10.4 78.0	7 5 1 5	1.82 1.54 1.72 1.56

\*The dehydration ratio for the control samples was 1.94 as compared to 2.30 for those dipped in Kena. This is due to higher degree of fluid losses in cooking, about 30% (dry weight basis for control) compared to 15 percent for Kena or STPP treated.

Table 22  
Sensory evaluation report\* on cooked freeze dried pork chops  
(following rehydration)

Treatment	Panel Scores on							
	Appearance		Aroma/Flavor		Texture Consistency		Overall	
	Mean	(S.E.)	Mean	(S.E.)	Mean	(S.E.)	Mean	(S.E.)
1. None (Control Sample)	6.2	(1.70)	6.3	(2.13)	5.3	(1.85)	5.6	(1.87)
2. Dusted with Kena, held for one hour at 1-30C, rinsed and cooked before freezing	6.0	(1.00)	6.5	(1.40)	6.1	(2.18)	6.2	(1.54)
3. Dusted with Tetra sodium pyrophosphate, held for one hour at 1-30C, rinsed and cooked before freezing	6.2	(1.58)	6.0	(1.57)	5.6	(1.57)	6.0	(1.61)

\* Panel size = 15 persons



Table 23

Summary of drop tests and rehydration studies on fish blocks with various treatments

Treatment	Analysis of drop tested samples		Rehydration Ratio
	Weight fraction retained on a 19 mm opening sieve	No. of fragments	
A. Treatments before freeze drying:			
(a) None (control)	88.6	6	4.52
(b) Dipped in:			
(i) 12% STPP soln. for 5 min.	82.6	8	4.21
(ii) 12% STPP soln. for 10 min.	86.0	7	4.24
(iii) 12% STPP soln. for 15 min.	81.4	8	4.42
(iv) 12% STPP soln. for 20 min.	82.3	8	4.38
(v) 12% STPP + NaCl (1:1) for 5 min.	81.0	6	4.64
(vi) 12% SHMP + NaCl (1:1) for 5 min.	75.8	3	4.20
(vii) 12% Kena + salt (1:1) for 5 min.	73.8	4	4.12
(c) Dusted with the following and held for one hour at 40°F:			
(i) Kena	71.4	8	4.75
(ii) STPP	79.7	8	5.54
B. Treatments after freeze drying (a secondary drying was needed to bring down the moisture to below 2%)			
(a) None (control)	89.7	15	2.78
(b) Sprayed with 15% gelatin solution	97.0	6	2.78
(c) Dipped in 30% soln. of Amaizo 839 + Frodex 24 DE (1:1) for 15 sec. on each side	82.7	14	2.68
(d) Dipped in 30% soln. of National Starch-781437 for 15 sec. on each side	83.7	22	2.74

\* The fish samples for the treatments before and after freeze drying were obtained from two different sources. Large differences in the rehydration ratio of group A and B may be attributed to the fact that the samples in A were fresh, whereas those in B were obtained as frozen blocks commercially and had undergone considerable moisture loss.

Results of a panel test on the fish samples are given in Table 24. It can be seen that neither of the two samples was acceptable to the panel. Also, there was no significant difference between the two samples. It was understood that the poor Flavor/Aroma in control sample and tough and dryer texture in gelatin coated sample were responsible for the low panel ratings of these samples.

It is possible that a combination of suitable phosphates and gelatin treatment may be developed which would result in more desirable organoleptic properties without sacrificing the mechanical stability attained. Additional studies are, however, needed to establish these anticipated results.

Chicken, Cooked, Diced: The treatments studied for chicken meat can be grouped into: (a) those applied before cooking, and (b) those applied after cooking. Solutions or suspensions of starches and gelatin (up to 50 percent concentration) were sprayed onto the surfaces of cooked, diced frozen meat (i.e., IQF, individually quick frozen). The meat to be sprayed was tumbled constantly to avoid the formation of clusters. Immediately after the treatment, the treated dice were returned to the freeze chamber to prevent thawing. The treatment on the raw meat consisted of soaking the deboned meat in a suitable phosphate or phosphate and sodium chloride solution. For the entire length of the soaking period, the samples were held at refrigerated temperature (about 35°F or 2°C). Subsequently, the meat was frozen, diced and freeze dehydrated. Owing to non-availability of a dicing machine, the meat having undergone phosphate treatment was hand diced, whereas, that used for spray-coating of cooked samples was obtained in already-cooked and diced form from commercial sources. It may, however, be pointed out that to study the effect of any of the treatments, both the control and treated samples were obtained from the same source and processed similarly.

The integrity of the freeze dried samples was evaluated by drop testing. For the samples coated with: (a) National Starch No. 781472, (b) Amaizo 839 with Frodex 24 DE, and (c) gelatin solutions, particle size distributions after 30 drops are given in Table 25. For all the four groups, the initial sample (before drop testing) was controlled to between the range of 6.6 mm and 9.4 mm. After identical drop experience, the higher weight fraction with larger fragment size indicates more resistance of the sample to breakage. It may be seen from Table 25 that over 80 percent of the gelatin treated sample did not undergo any breakage, compared to about 50 percent weights of each of the starch coated and control samples turning into fine fragments when subjected to the same magnitude of stress. With only one percent weight of gelatin on meat, such a reduction in breakage was considered quite impressive.

The samples treated with the three surface coatings along with their control were subjected to panel test. Table 26 shows that all the four samples were unacceptable to the panel. Even though the gelatin treated sample was considered somewhat better than the control, none of the four samples attained an acceptable score of 6.0, in terms of

Table 24

Sensory evaluation report on raw freeze dried fish, following rehydration and cooking

Treatment	Panel Scores on*			
	Appearance	Aroma/Flavor	Texture/Consistency	Overall
A. Control	6.14	5.56	5.28	5.56
B. Sprayed with gelatin after dehydration and given a secondary drying	5.67	6.50	4.67	5.33**

\*Panel size = 16 persons

\*\*Both the samples were unacceptable and the difference between the two was not significant ( $\alpha = 0.05$ ).

Table 25

Sieve analysis of freeze dried diced chicken meat with various treatments after 30 drops from a height of one meter

Sieve no.	Control Sample	Percent Weight Retained From			
		Sample coated with 50% solution of American maize Starch - combination (1.4% dry basis)	Sample coated with 50% solution of flat'1 Starch-781472 (2.1% starch/meat, dry basis)	Sample coated with 5% gelatin solution (1.0% concentration gelatin/chicken, dry weight basis)	
3	53.16	57.04	48.47	81.81	
4	18.77	12.65	17.65	10.52	
8	10.63	12.41	12.71	4.63	
10	3.16	3.10	4.00	0.84	
20	6.31	6.92	9.18	1.47	
40	4.98	4.77	4.94	-	
Pan	2.99	3.10	3.06	0.73	

Table 26

Sensory evaluation report on cooked, diced, surface coated and freeze dried chicken following rehydration

Treatment	Panel Scores on *			
	Appearance	Aroma/Flavor	Texture	Overall Rating
A. None (control)	6.0	4.8	4.3	4.6
B. Sprayed w. 5% solution of gelatin to develop a thin coating on the surface of diced chicken (weight of gelatin=1.0% of the cooked-weight of chicken)	6.3	5.6	4.4	4.7
C. Sprayed with 50% solution of American Maize Amaiizo 839 + Frodex 24 (1:1). (Total starch applied = 1.4% of the cooked weight of chicken)	5.7	4.1	4.7	4.3
D. Sprayed with 50% solution of National Starch 781742 (Total starch applied = 2.7% of the cooked weight of chicken)	6.1	4.4	4.2	4.4

\* Panel size = 16 persons

Conclusion: None of the four samples was considered acceptable

Aroma/Flavor, Texture and Overall rating. It was suggested that the panel members might not be appreciating aesthetically the individual pieces of diced meat, because of their unfamiliar presentation. Therefore, it was decided to rehydrate the diced meat and serve with white sauce, which is recommended to be used with poultry meat. Table 27 gives the panel scores when both the control and gelatin-coated samples were served with white sauce. Such a procedure probably helped in giving the samples somewhat more uniform texture, but neither of the two samples attained a score of 6.0. The poor scores on the samples were mainly attributed to their poor texture (toughness) and lack of flavor. It was, therefore, decided to study the use of phosphates, which had shown significant improvement in the texture and flavor of most of the other products.

Various approaches were studied to incorporate phosphates (mostly sodium tripolyphosphate and Kena in the samples. Deboned samples were: (a) soaked in different concentrations of STPP alone or STPP and NaCl, for a period ranging from 12 to 24 hours, (b) cooked in solution of STPP or STPP and NaCl. Freeze dried samples having undergone these treatments were evaluated for their mechanical stability. Table 28 gives size distribution of the drop tested samples having undergone these treatments. Figure 11 illustrates some of these distributions on a log-normal probability graph. The following observations can be made:

- (a) The size distribution of the fragmented samples can be very closely represented by a log normal distribution;
- (b) The mean sizes of the samples a (soaked in 15% solution of STPP + NaCl for 18 hours), b (soaked in 7.5% solution of STPP for 18 hours), and c (control) as represented in Figure 11 were found to be 1.12, 0.42, and 0.20 centimeters, respectively. As a result of the first of these treatments, over 50 percent of the samples did not undergo any breakage under the mechanical stress which results in fragmentation of over 75 percent in the case of the control.
- (c) The size distribution of the samples cooked in a STPP + NaCl solution is not appreciably different from that of the control. It may, therefore, be interpreted that cooking the sample in phosphate solution does not add to the strength of the freeze dried sample. Either an inadequate time duration or an undesirably high temperature during cooking might be held responsible for ineffectiveness.

After each operation, from cooking to dehydration, relative sample-weight recoveries were measured. Figure 12 illustrates the effect of STPP + NaCl treatments on the sample-weights after cooking, freeze drying and rehydration. It may be noticed that the cooking loss for the control sample was about 37 percent on fresh weighed basis, against less than 20 percent for the phosphate treated samples. The lowest losses

Table 27

Sensory evaluation report on cooked, diced freeze dried chicken (following rehydration), when served with white sauce

Treatment before freeze drying	Panel Scores on *			
	Appearance	Aroma/Flavor	Texture	Overall Rating
A. None (control), served in white sauce	5.0	5.3	5.2	4.8
B. Sprayed with gelatin solution before freeze drying, rehydrated and served in white sauce	5.3	5.4	4.9	5.2

\* Panel size = 23 persons

Conclusion: Both the samples were unacceptable.

Table 28

Sieve analysis of freeze dried chicken meat with various phosphate treatments after 30 drops from a height of one meter

Sieve Size	Percent weight retained from:			
	Control	Sample soaked in 7.5% STPP soln. for 18 hours before cooking	Sample soaked in 15% STPP + NaCl (1:1) soln. for 18 hours before cooking	Sample cooked in 12% STPP + NaCl (1:1) soln.
9.5 mm	24.60	27.91	48.67	16.68
No. 3	11.54	14.82	11.37	13.45
No. 4	9.84	7.50	7.46	7.71
No. 5	4.16	3.56	3.02	3.77
No. 8	9.65	8.66	7.10	9.06
No. 16	12.68	13.47	8.70	13.99
No. 30	8.23	6.35	4.09	10.04
No. 50	7.19	7.12	3.37	8.97
Pan	12.11	10.59	6.22	16.32



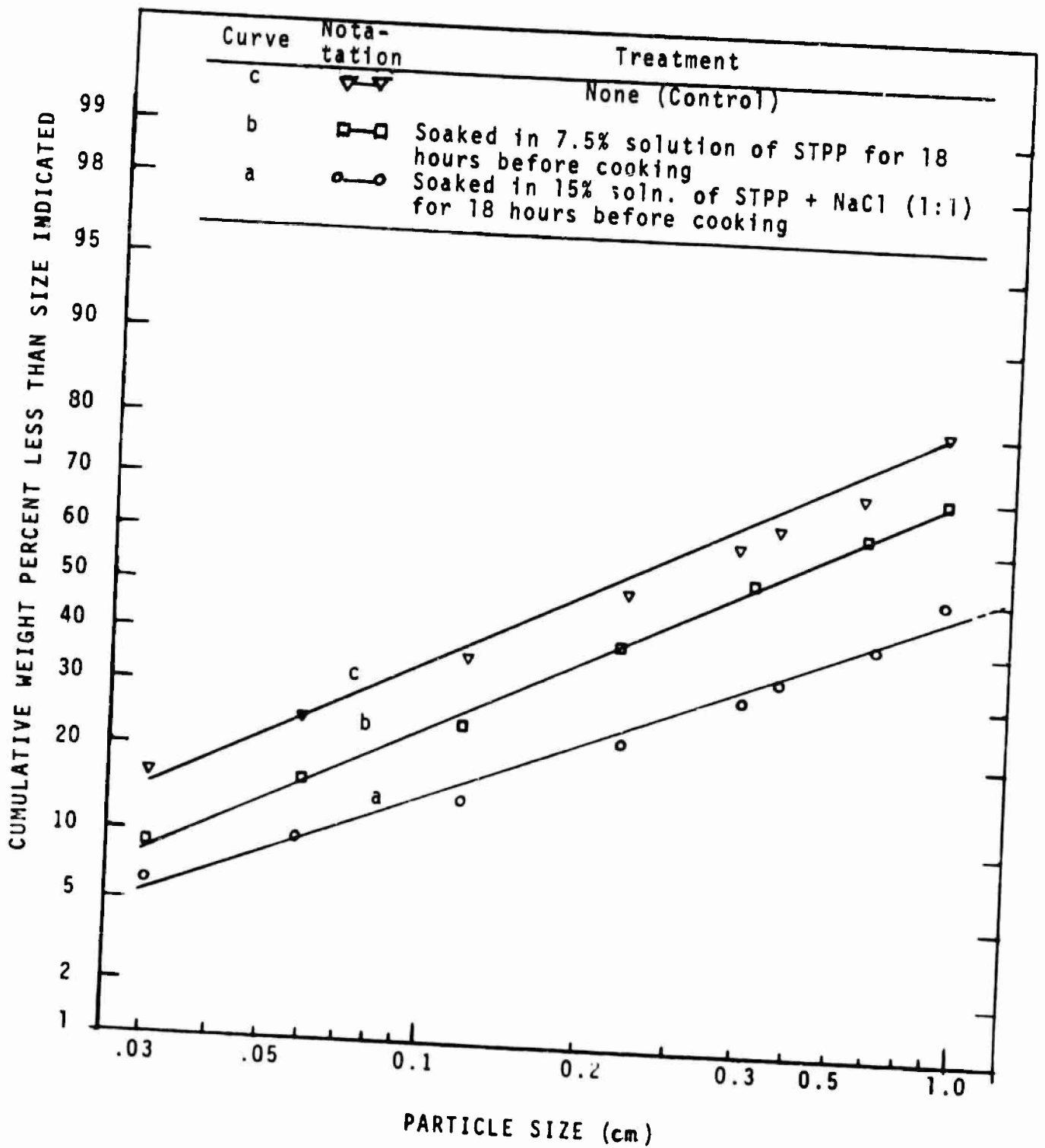


Figure 11. Log probability plot of the freeze dried, cooked-diced chicken after drop testing. The geometric mean diameters for samples a, b and c may be noted as 1.22, 0.41 and 0.20 cm, respectively, indicating the mean size of a to be six times that of c.

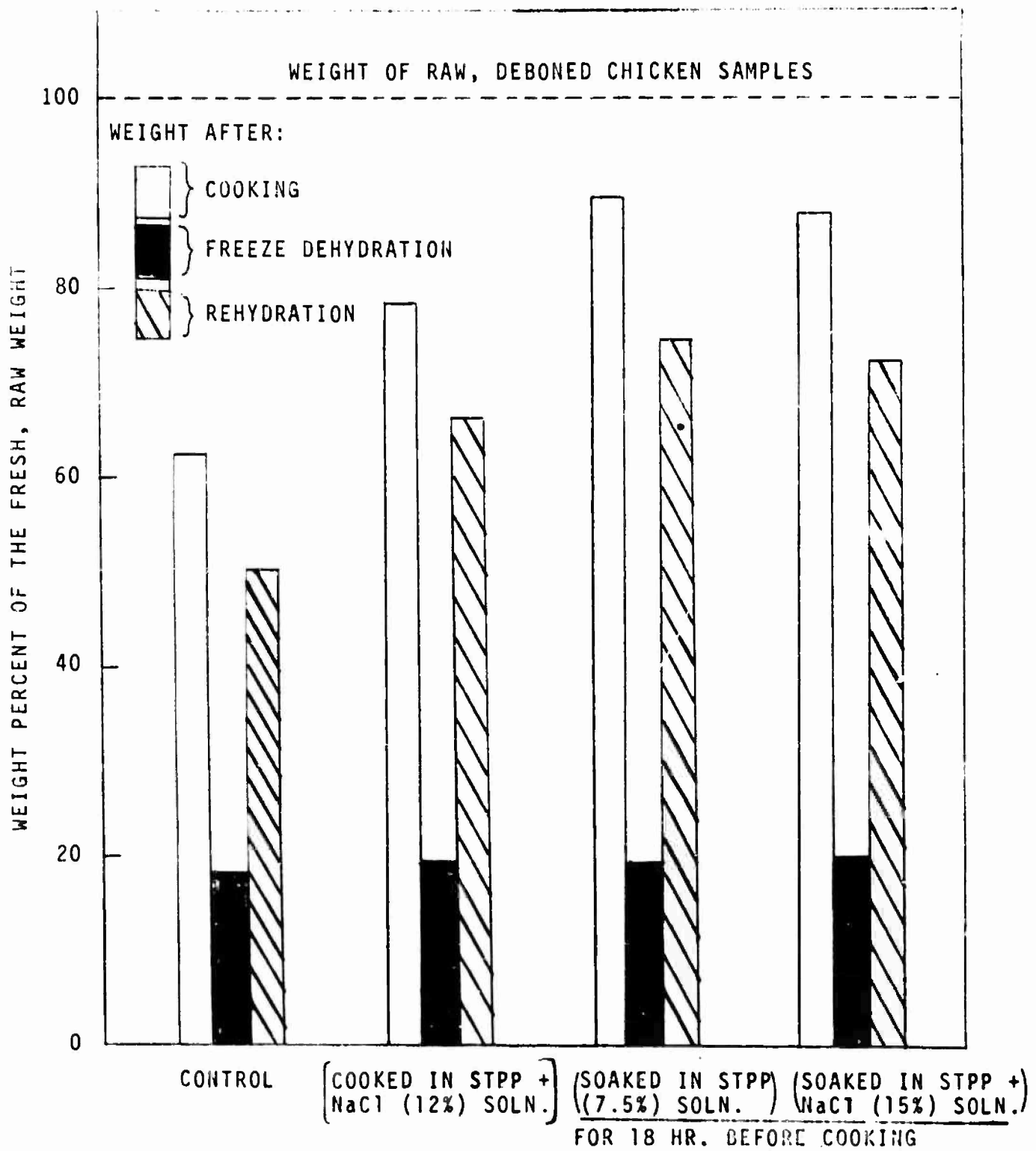


Figure 12. The relative weights recovered after cooking, freeze drying and rehydration of the chicken samples having undergone the indicated salt + phosphate treatments

among these were for the samples soaked in 7.5 percent STPP solution for 18 hours duration and amounted to about 8.5 percent. All the samples rehydrated after freeze drying attained 80 to 85 percent of their cooked weight, resulting in the useable samples with control samples weighing about 50 percent of the fresh weight against about 75 percent recovery for the phosphate treated samples.

The overall effect of the phosphate treatment on the organoleptic acceptance of the samples is presented in Table 29. The samples soaked in 15% NaCl + STPP solution for 18 hours were significantly ( $\alpha = 0.05$ ) more acceptable than the untreated controls, in terms of Aroma/Flavor, Texture and Overall ratings. Also, the panel ratings of the control sample were practically the same as presented in Table 26 and 27. These data indicate the consistency in the panel response, even though the tests were carried out at different times and with the materials from entirely different sources. The significant improvement in Aroma/Flavor, Texture and Overall rating can be explained in terms of the retention of higher water soluble flavor compounds of meat during cooking, retention of bound water and tenderizing effect of the dispersion of proteins in meat.

#### Summary of Treatments

Based on the evaluations of the mechanical stability of the dry samples, and the rehydration, texture, flavor and overall organoleptic characteristics of the rehydrated samples, for seven of the eight products (except fish squares), appropriate phosphate and sodium chloride additive treatments were established to be the most effective. In comminuted meat products, the effectiveness of these additives is augmented by adding wheat gluten and meat emulsion.

As an alternative to the phosphate treatment, a significant improvement in the binding strength of the dry chicken samples was also accomplished by spraying gelatin solution on the surface of the cooked-diced meat. For fish squares, however, phosphate treatment was found ineffective in controlling the breakage in dehydrated samples. A gelatin coating developed by dipping the dehydrated fish squares in gelatin solution, imparted the redried samples with a high degree of resistance against breakage. It may, however, be pointed out that the above treatment did not have any beneficial effect on the other collateral quality factors. A summary of the most desirable formulations for the eight products is given in Table 30. Products prepared according to these formulations were stored under suitable environmental conditions and evaluated at the desired intervals.

Table 29

Sensory evaluation report on phosphate treated, cooked, diced, freeze dried chicken following rehydration

	Panel Scores on *			Overall Rating
	Appearance	Aroma/Flavor	Texture	
A. Control	6.71 ( $\pm 0.81$ ) **	4.67 ( $\pm 0.95$ )	5.00 ( $\pm 0.89$ )	4.88 ( $\pm 0.81$ )
B. Soaked in 15% NaCl + STPP solution (1:1) for 18 hours	6.30 ( $\pm 0.67$ )	6.38 ( $\pm 0.60$ )	6.67 ( $\pm 0.76$ )	6.34 ( $\pm 0.57$ )

\* Panel size = 24 persons

\*\* Figures within parentheses describe the 95% confidence limits on means for each of the characteristics evaluated. The Aroma/Flavor, Texture and Overall ratings for sample B are significantly ( $\alpha = 0.05$ ) higher than the respective ratings for A. The difference between the appearance ratings of the two samples is not significant.

Table 30

Summary of the techniques evaluated to impart the optimum mechanical characteristics to the freeze dehydrated foods

Food Product	Treatment	
	Most Favorable (A)	Alternative Favorable (B)
1. Beef patties, raw	mix w. 0.5% Kena, 2% NaCl, 0.15% wheat gluten, hold 2 hours at 1° to 3°C	same as A, but 10% meat added as emulsion
2. Beef patties, cooked	same as 1A	same as 1B
3. Diced chicken	soak 12 to 18 hrs. in 15% STPP + NaCl (1:1) soln. before cooking	spray w. 2.5% to 5% gelatin solution on cooked meat surfaces
4. Fish squares	dip freeze dried squares in gelatin solution; redry	-
5. Pork sausage patties	mix w. 0.35% Kena, 1.25% NaCl, 0.1% wheat gluten, hold 2 hrs. before cooking	same as A, but 10% of meat added as emulsion
6. Pork chops, raw	dust w. Kena, hold one hour at 1 to 3°C, rinse and freeze	SHMP instead of Kena
7. Pork chops, cooked	same as 6	
8. Shrimp	Dust w. Kena, hold 10 to 15 min., cook in 2% salt water	SHMP instead of Kena

## EFFECT OF STORAGE ON PRODUCT CHARACTERISTICS

All the freeze dried products were packaged in oxygen and moisture impermeable glass containers, with their internal atmosphere having 1.0 percent or less of oxygen. For all the eight products, treated samples (see Table 30) as well as their untreated counterparts used as control samples were stored at 38°C ambient condition. Treated as well as control samples for some of the products were also stored at -6°C. After a period of about six months (22 to 39 weeks for various products), for each product the samples were removed from the storage and evaluated for fragility, rancidity, color and organoleptic characteristics. Gaseous composition (mainly oxygen concentration) of every withdrawn container was analyzed, and moisture content of every sample was determined. The containers having an oxygen content higher than 2 percent (indicative of seal defects) were discarded.

### Mechanical Stability

At the end of the specified storage period, all the product samples were subjected to drop tests. For some of the products, force deformation characteristics were also determined.

Figure 13 gives a fragment size distribution of cooked beef patties, after being stored for 22 weeks at 38°C. The mean size of the samples A (control), B (mixed with 0.38% Kena, 1.25% NaCl, and 0.15% wheat gluten), and C (mixed with 0.5% Kena, 2% NaCl, 0.15% wheat gluten and 10% meat emulsion) were found to be 0.2 cm, 1.0 cm and 4.0 cm, respectively (see Appendix for the distribution of the drop-tested samples). Within practical experimental limits, these values of the mean diameters are equal to their corresponding samples before storage, as presented in Table 8; viz, 0.25 cm, 1.20 cm and 3.50 cm for A, B, and C, respectively. These figures indicate that the fragmentation characteristics of the cooked freeze dried beef patties prepared according to the above formulations are not altered, and the high degree of resistance against breakage imparted by the treatments (B and C) is retained after an aging period of 22 weeks.

Size distributions of the drop-tested pork sausage patties after 35 weeks of storage and diced chicken samples after 22 weeks of storage are presented in Figures 14 and 15, respectively. For all the eight experimental products, size distribution of the drop-tested samples are presented in the Appendix. It is interesting to note that no appreciable change attributable to storage was observed in the fragmentation characteristics of the treated products or their untreated control samples. The improvement in the mechanical characteristics of the freeze dried products can be, therefore, taken to be unaffected by a storage temperature of 38°C for a duration of up to 39 weeks.

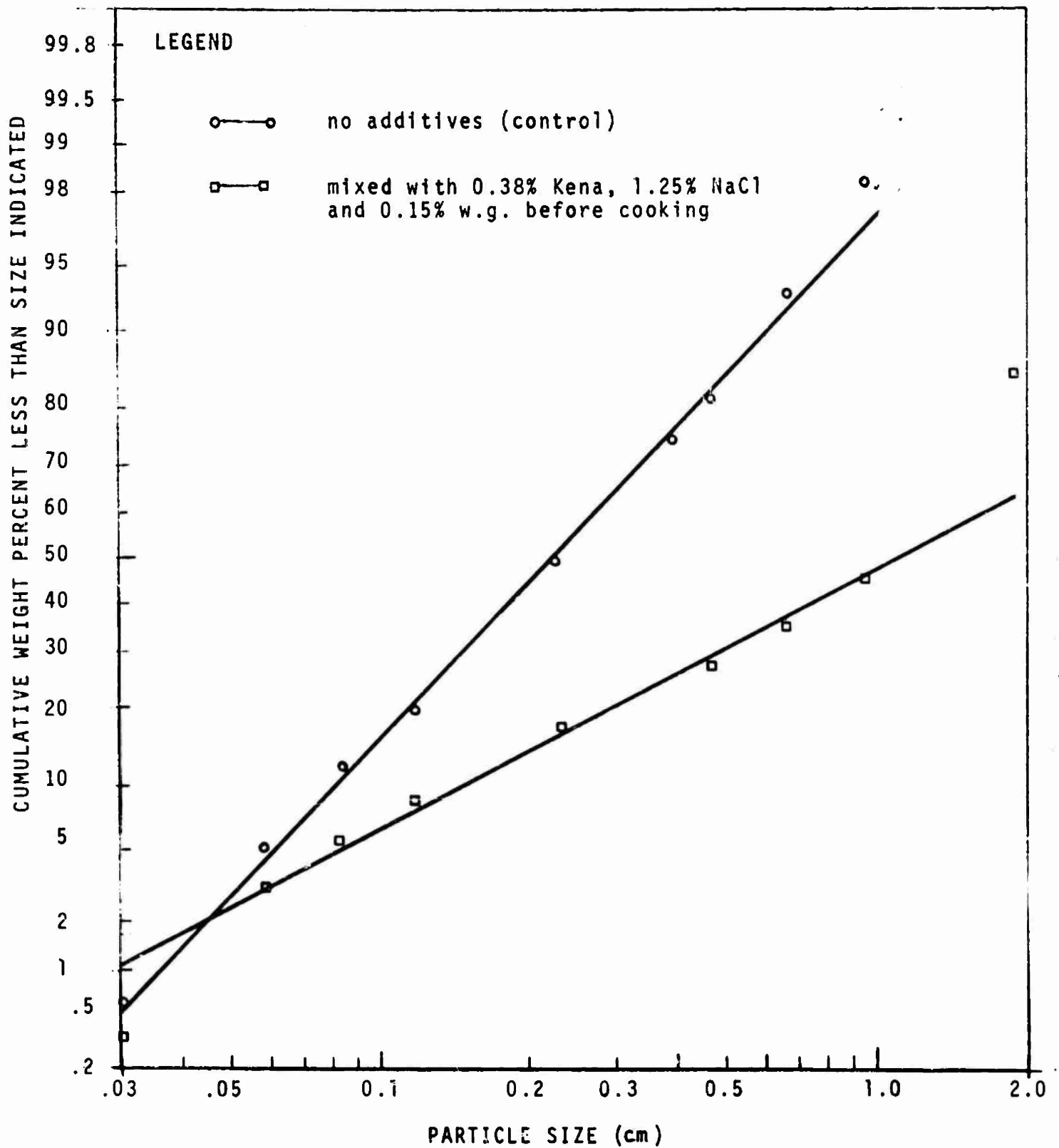


Fig. 13. Log probability plots of the cooked-freeze dried beef patties subjected to drop testing after being stored for 22 weeks at 38°C

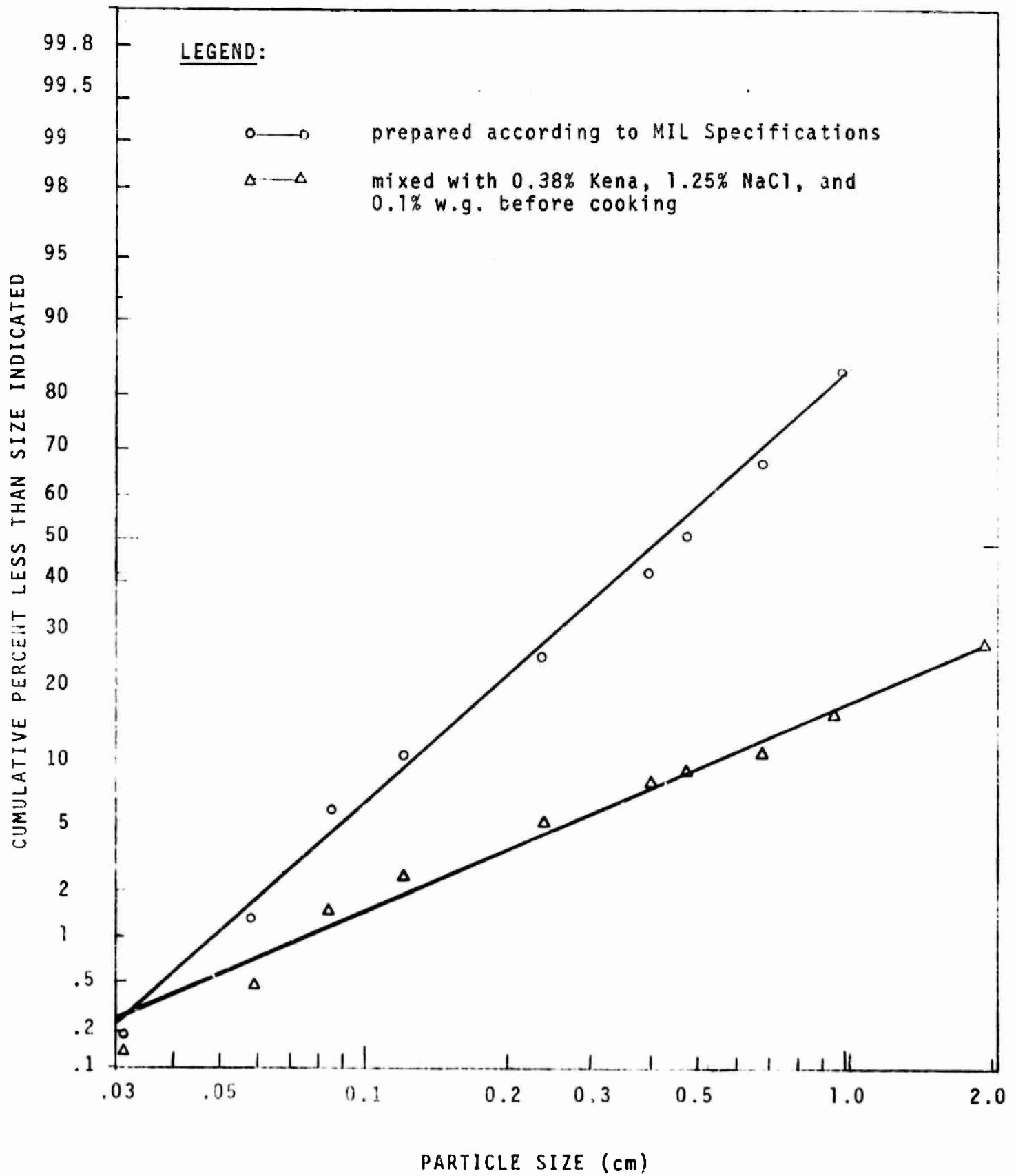


Fig. 14. Log probability plot of the freeze dried pork sausage patties subjected to drop testing after being stored for 35 weeks at 38°C



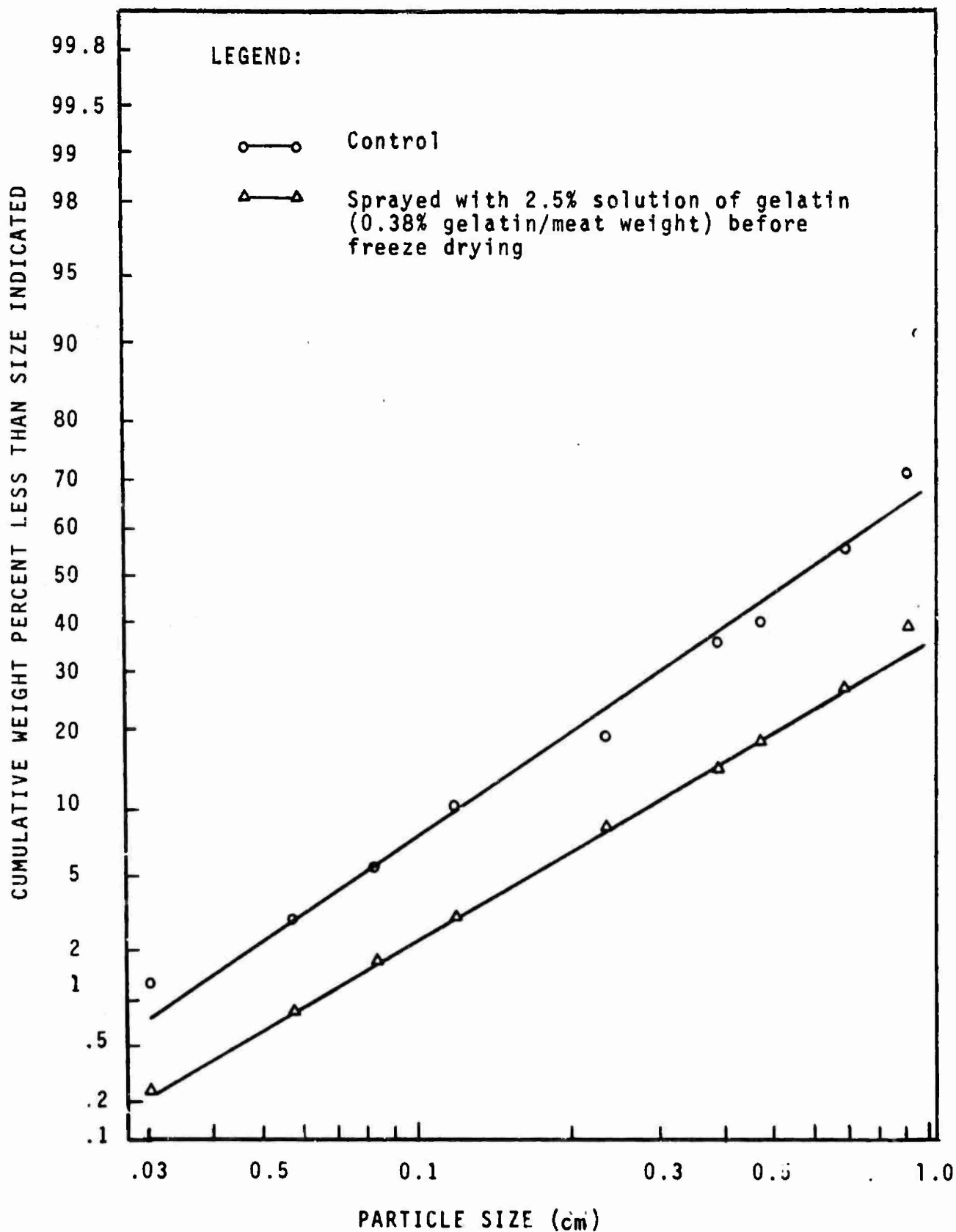


Fig. 15. Log probability plot of the cooked-diced, freeze dried chicken after subjecting to drop test. The samples had been stored at 38°C for a period of 22 weeks prior to evaluation. Over 60% of the treated samples did not undergo any breakage compared to only 30% unbroken for the control sample.

### Rancidity

From every dehydrated product sample, 2.0 grams of a specimen was taken and TBA test was performed on it. TBA values for the dehydrated meat patties and the non-comminuted meat products along with their formulations and storage histories are given in Tables 31 and 32, respectively. Table 31 shows that the TBA values for the treated beef patties were observed to be about 40 percent of their stored controls, whereas that for the sausage patties about 50 percent of the control TBA value was observed. It may also be noticed that both the treated as well as the control samples of the pork sausage patties showed a higher degree of rancidity when stored at  $-6^{\circ}\text{C}$  than at  $38^{\circ}\text{C}$  storage temperature. A TBA score of 1.22 for control against 0.45 for the treated sausage patties stored at  $-6^{\circ}\text{C}$  showed the existence of the antioxidant effect of the additive treatment (possibly phosphates) even at subzero temperature.

Table 32 shows that, like comminuted meat patties, the phosphate treatments corresponded to rancidity ratings of the other meat products at about half of the values for their untreated controls. The rancidity behaviors exhibited by the two products coated with gelatin, namely, chicken and fish squares, were somewhat inconsistent. The TBA value for chicken treated with gelatin was about one third of its control, whereas, the gelatin coated fish squares had about sixty percent higher TBA score than the control sample. The above anomaly of a higher TBA score for treated fish squares may be attributed to the additional exposure to air in the handling required to treat the freeze dried samples and redry them. A TBA score of 0.48, however, cannot be considered objectionable from the consumer standpoint.

### Color Changes after Storage

The curves shown in Figure 16 illustrate the reflectance spectra of freeze dried raw beef patties after rehydration. The information on meat color and browning was gathered from analysis of similar spectra on some of the products.

A relationship between pigment concentration measured by extraction and percent reflectance spectroscopy was presented by Franke and Solberg (1971) as:

$$\text{mg pigment/g meat} = 29.86 (\Delta R_{A632}).$$

In this relationship  $\Delta R_{A632}$  was defined as the height of the 632 nm peak, measured from the  $A_{632}$  750 nm starting point to the top of the peak. Even though such a precise relationship is not hypothesized from the observations on rehydrated meat samples, the relationship might be helpful in getting an idea of the relative concentrations of the pigment in samples with different treatments. Table 33 gives the means of  $\Delta R_{A632}$  from 12 different spectra for each treatment. The pigment

Table 31

TBA values indicating levels of rancidity in the comminuted, freeze dried meat patties after the indicated storage treatment

Product	Additives, on Raw Meat Basis	Storage Variables*		TBA Value (Mean of 4 Samples)
		Temp., °C	Time, Weeks	
Beef patties, raw	(a) None (control)	38	39	0.91
	(b) 0.50% Kena, 2.0% NaCl, and 0.15% wheat gluten	39	39	0.36
	(c) Same as (b)	-6	39	0.33
Beef patties, cooked	(a) None (control)	38	22	0.75
	(b) 0.38% Kena, 1.25% NaCl, and 0.15% wheat gluten	38	22	0.32
Pork sausage patties, cooked	(a) Prepared according to MIL specifications	38	35	0.58
	(b) Mixed with 0.38% Kena, 1.25% NaCl and 0.1% wheat gluten	38	35	0.32
	(c) Same as (a)	-6	35	1.22
	(d) Same as (b)	-6	35	0.45

\* For all the samples, the O<sub>2</sub> content was less than 2 percent of the gaseous volume.

Table 32

TBA values indicating levels of rancidity in the non-comminuted, freeze dried meat products after the indicated storage treatment

Product	Treatment	Storage Variables*		TBA Value (Mean of 4 Samples)
		Temp., °C	Time, Weeks	
Chicken, cooked diced	(a) None (control)	38	22	2.30
	(b) Sprayed with gelatin before freeze dehy- dration	38	22	0.83
	(c) None	38	8	1.23
	(d) Soaked in STPP + NaCl soln. for 18 hours	38	8	0.28
Fish squares, raw	(a) None (control)	38	33	0.29
	(b) Coated with gelatin solution and redried	38	33	0.48
Pork chops, raw	(a) None (control)	38	33	0.43
	(b) Dusted with Kena, held for one hour and rinsed	38	33	0.22
	(c) None	-6	33	0.82
	(d) Same as (b)	-6	33	0.31
Pork chops, cooked	(a) None (control)	38	33	0.46
	(b) Dusted with Kena, held for one hour, rinsed and cooked	38	33	0.28
Shrimp, cooked	(a) None (control)	38	31	0.30
	(b) Dusted with Kena, held for 15 min., rinsed & cooked	38	31	0.17

\* For all the samples, the O<sub>2</sub> content was less than 2 percent of the gaseous volume.

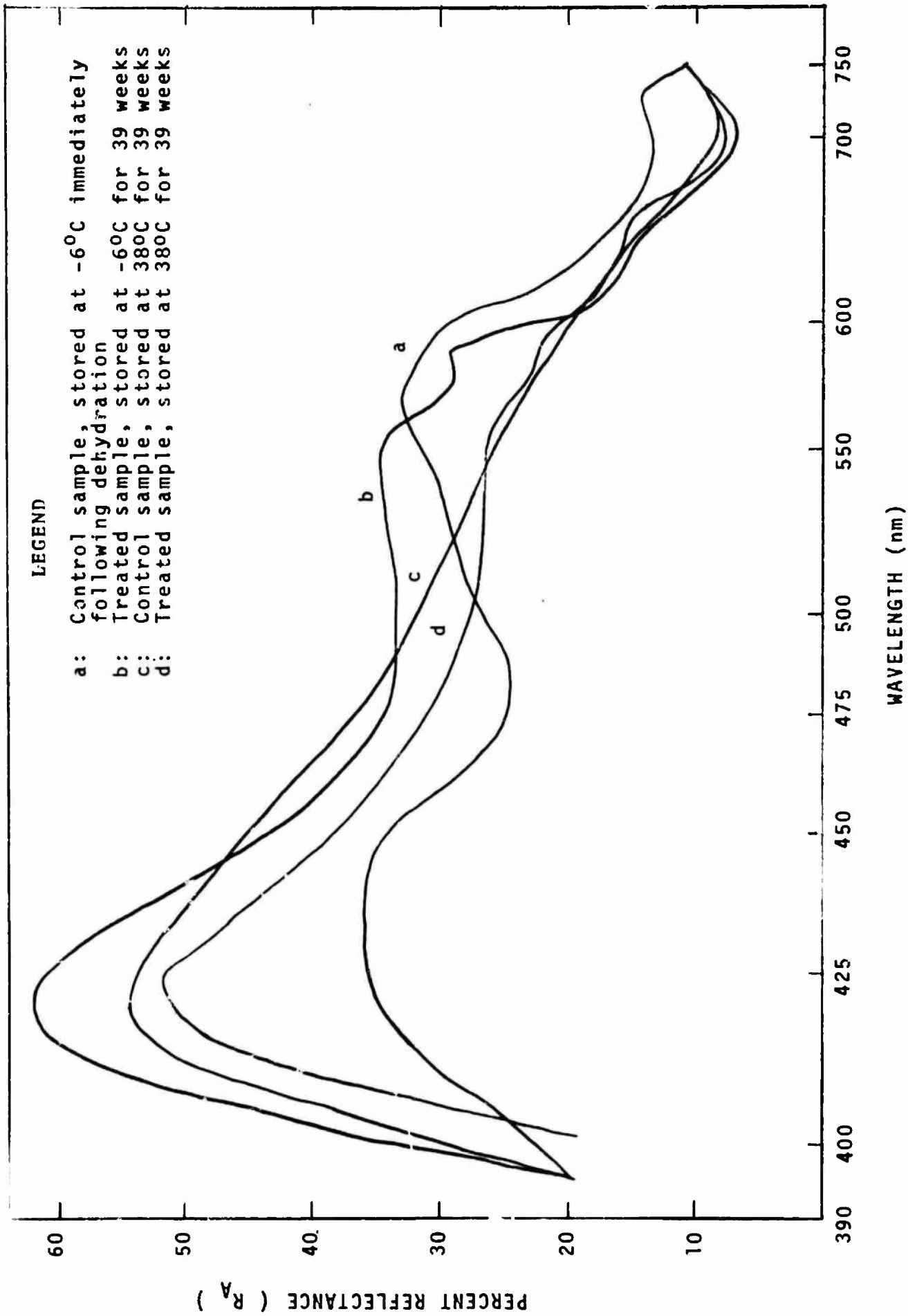


Figure 16. Reflectance spectra of raw freeze dehydrated beef patties after rehydration

concentration in the samples stored for 39 weeks at 38°C was 55 to 60 percent of its concentration immediately following freeze dehydration (i.e., storage caused color diminution). Even though the differences were not large, the  $\Delta R_{A632}$  value for treated sample was about 12 percent higher than the control, i.e., it retained more natural pigment color than the control.

Table 33 also gives the values of  $\Delta R_{A575}$  for raw beef patties. Like  $\Delta R_{A632}$ , the  $\Delta R_{A575}$  was defined as the height of the 575 nm peak measured from the 750 nm starting point to the top of the peak. Borchert and Briskey (1965) used reflectance at 575 nm as an index of browning and suggested that the two factors are inversely related. Bowers et al., (1968) suggest the reflectance values to have strong negative correlation with percent ether extract and concentration of reducing sugars. The trend of  $\Delta R_{A575}$  values in Table 33 is similar to that for  $\Delta R_{A632}$ , but in negative direction, indicating that after 39 weeks of storage at 38°C, the treated beef patty samples were somewhat less susceptible to browning than the control.

Observations similar to raw beef were made on some of the other eight products. These observations are presented in Appendix. An extensive study of the color changes in stored samples could not be accomplished with the limited time available for this study.

#### Organoleptic Evaluations

It is evident from the foregoing results on the quality characteristics of the post-storage samples that none of the characteristics evaluated so far was unfavorably affected by the treatments developed in this study. The treated as well as control samples of all the products were, therefore, subjected to sensory evaluations.

Table 34 summarizes the results of sensory evaluations of cooked beef patties following their storage under specified conditions. Samples A (control) and B (prepared with 0.38% Kena, 1.25% NaCl and 0.15% wheat gluten) were stored at 38°C for 22 weeks, whereas, samples C (prepared with 0.50% Kena, 2.0% NaCl, 0.15% wheat gluten and 10% meat emulsion) were stored at -6°C for 39 weeks. The oxygen concentrations in the storage environments of all the samples were maintained at less than 2 percent. In terms of Aroma/Flavor, Texture and Overall evaluation, the treated samples were given significantly higher ( $\alpha = 0.05$ ) ratings than the control samples. These findings are in agreement with the panel evaluations of the cooked beef patties prior to their storage (see Table 10). If the panel ratings in Table 10 are compared with their corresponding response for the post-storage evaluation in Table 34, it is clear that: (a) the quality changes in either the treated or the control samples are not significantly affected ( $\alpha = 0.05$ ) by storage, (b) the overall ratings in terms of Aroma/Flavor, and Texture for control samples decreased by a score of about 1.0 in response to storage at 38°C for 22 weeks, whereas no decrease in panel ratings for the treated samples was observed.

Table 33

Color and browning characteristics of raw beef patties expressed in terms of percent of reflectance, taking RA750 as base. (Each tabulated value is the mean of 12 observations.)

Sample/treatment following freeze dehydration	Percent reflectance	
	$\Delta R_{A632}$	$\Delta R_{A575}$
1. Control, immediately following freeze drying	11.0	21.0
2. Treated, immediately following freeze drying	11.5	21.0
3. Treated samples stored at $-60^{\circ}\text{C}$ for 39 weeks	9.0*	17.5*
4. Treated samples stored at $38^{\circ}\text{C}$ for 39 weeks	6.0	12.6
5. Treated samples stored at $38^{\circ}\text{C}$ for 39 weeks	6.8	14.4

\*Note: Lower storage temperature ~ higher retention of pigment compared to 4 and 5 at higher storage temperature

Table 34  
Sensory evaluation report on cooked freeze dried beef patties following rehydration after being stored as indicated, prior to evaluation

Treatment	Panel* Scores on			
	Appearance Mean (S.E.)	Aroma/Flavor Mean (S.E.)	Texture Mean (S.E.)	Overall Mean (S.E.)
A. Control (prepared according to MIL Spec. and stored at 380C for 22 weeks. Samples had 0.8% moisture and 0.4% O <sub>2</sub> in the storage headspace.).	6.12 (1.86)	3.82 (1.47)	3.59 (2.12)	3.59 (1.97)
B. Mixed with 0.38% Kena, 1.25% NaCl, and 0.15% wheat gluten, held for 2 hours before cooking. (Stored at 380 for 22 weeks. Samples had 1.0% moisture and 1.7% O <sub>2</sub> in the headspace.).	6.59 (1.28)	6.71 (1.83)	6.65 (1.90)	6.56 (1.96)
C. Mixed with 0.50% Kena, 2.0% NaCl, 0.15% wheat gluten and 10% meat emulsion. (Stored at -60C for 39 weeks. Samples had 1.2% moisture and 1.6% O <sub>2</sub> in the headspace.)	5.98 (1.89)	6.88 (1.80)	6.30 (2.11)	6.23 (2.24)

\* Panel size = 18 persons

In terms of Aroma/Flavor, Texture and Overall evaluation, samples B and C were significantly better (  $\alpha = 0.05$  ) than A.



For the other seven products, viz, raw beef patties, diced chicken, fish squares, cooked pork chops, raw pork chops, pork sausage patties and shrimp, the results of the sensory evaluations carried out after the specified storage treatments are presented in Tables 35, 36, 37, 38, 39, 40 and 41, respectively. Except for fish squares, panel tests on the other six products were found to be in agreement with the results on cooked beef patties. Generally speaking, magnitude of improvements in the quality characteristics of the treated-cooked samples was greater than the raw-treated samples of the same products, and the relative improvement in the quality of treated samples over their respective controls was greater for the comminuted meat patties than the non-comminuted meat products.

It was, therefore, concluded that the treatments developed for various products generally imparted improvement in their overall quality characteristics compared to their controls. There was found to be no adverse effect of storage on the retention of these improved quality characteristics.

Table 35

Sensory evaluation report on raw freeze dried beef patties (following rehydration and cooking) after being stored for 39 weeks at indicated temperature prior to the evaluation

Additives and Storage Parameters	Panel Scores on *			
	Appearance Mean (S.E.)	Aroma/Flavor Mean (S.E.)	Texture Mean (S.E.)	Overall Mean (S.E.)
A. None (control). The samples had 0.8% moisture and were stored at 380C, with 1.5% O <sub>2</sub> in headspace.	5.10 (1.48)	3.77 (1.87)	4.43 (1.68)	4.00 (1.52)
B. 0.5% Kena, 2.0% NaCl, 0.15% wheat gluten. The samples had 0.6% moisture and were stored at 380C, with 0.2% O <sub>2</sub> in the headspace.	6.81 (1.40)	5.86 (1.35)	5.48 (2.04)	5.67 (1.50)
C. 0.5% Kena, 2.0% NaCl, 0.15% wheat gluten and 10% moisture and were stored at -60C, with 0.7% O <sub>2</sub> in the headspace.	6.81 (1.07)	6.14 (1.24)	7.00 (1.27)	6.70 (1.16)

\* Panel size = 21 persons

Samples B and C are significantly better ( $\alpha = 0.05$ ) than A in terms of their Appearance, Aroma/Flavor and Overall score. Sample C also has a significantly ( $\alpha = 0.05$ ) superior texture than A as well as B.

Table 36

Sensory evaluation report on cooked, diced, freeze dried chicken following rehydration, after having been stored at 380C for 22 weeks

Treatment	Panel Scores on *				Overall Mean (S.E.)
	Appearance Mean (S.E.)	Aroma/Flavor Mean (S.E.)	Texture Mean (S.E.)		
A. Control	5.75 (1.27)	3.50 (1.57)	4.50 (1.43)		3.80 (1.62)
B. Cooked, diced meat sprayed with gelatin solution	6.65 (1.35)	6.20 (1.96)	5.50 (1.82)		5.90 (2.07)

\* Panel size = 20

In terms of Aroma/Flavor and Overall evaluation, sample B was rated to be significantly better ( $\alpha = 0.05$ ) than A.

Table 37. Sensory evaluation report on freeze dried fish squares following rehydration and cooking

Treatment	Panel* Scores on		
	Appearance	Aroma/Flavor	Texture
A. Control, stored for 33 weeks at 380C	6.00	5.48	5.20
B. Sprayed with gelatin after dehydration and given a secondary drying, subsequently stored for 33 weeks at 380C	5.60	5.90	4.10
			Overall
			5.36
			4.80

\* Panel size = 21 persons

Table 38

Sensory evaluation report on freeze dried raw pork chops, following rehydration and cooking after having been stored at 380C for 33 weeks prior to evaluation

Treatment	Panel Scores on *				Overall Mean (S.E.)
	Appearance Mean (S.E.)	Aroma/Flavor Mean (S.E.)	Texture Mean (S.E.)		
A. Control (prepared according to MIL Spec. Sample had 0.6% moisture and the storage headspace had 0.4% 0.2).	6.56 (1.19)	5.56 (1.19)	4.20 (2.30)		4.50 (1.71)
B. Dusted with Kena, held for one hour at 4-50C, rinsed and frozen. Sample had 0.8% moisture and the storage headspace had 0.2% O <sub>2</sub> ).	6.89 (1.08)	6.39 (1.67)	6.00 (1.71)		6.59 (1.49)

\* Panel size = 19 persons

Sample B was scored significantly better ( $\alpha = 0.05$ ) than A, in terms of Texture and Overall acceptance.

Table 39. Sensory evaluation report on freeze dried cooked pork chops following rehydration

Treatment	Panel* Scores on			
	Appearance	Aroma/Flavor	Texture	Overall
A. Control, stored for 33 weeks at 380C	5.90	5.82	5.12	5.45
B. Dusted with Kena, held for one hour rinsed and cooked. Stored for 33 weeks at 380C	6.01	6.13	5.82	5.94

\* Panel size = 21 persons

Table 40

Sensory evaluation report on freeze dried pork sausage patties following rehydration, after having been stored at indicated temperatures (-60C and 380C) for 35 weeks

Treatment	Storage Temp., °C	Panel* Scores on			Overall
		Appearance	Aroma/Flavor	Texture	
Control (prepared according to MIL Spec.) Mixed with 0.3% Kena, 1.25% salt and 0.1% wheat gluten	(a) 38	6.67	6.12	6.25	6.50
	(b) -6	6.60	5.60	5.60	5.75
	(c) 38	6.38	7.00	5.30	6.10
	(c) -6	6.68	6.10	5.44	5.75

\*Panel size = 16 persons

Table 41

Sensory evaluation report on freeze dried shrimp following rehydration, after having been stored at 380C for 31 weeks prior to evaluation

Treatment	Panel Scores on *			
	Appearance Mean (S.E.)	Aroma/Flavor Mean (S.E.)	Texture Mean (S.E.)	Overall Mean (S.E.)
A. Control (sample moisture content = 1.4%, 0.2 in the head-space = 0.9%)	6.86 (0.79)	5.48 (2.16)	5.90 (1.61)	5.67 (2.11)
B. Dusted with Kena, held for 10-15 min. before cooking (sample moisture content = 1.2%, 0.2 in the head-space = 1.1%)	6.76 (1.09)	6.62 (1.19)	7.24 (1.09)	6.52 (1.40)

\* Panel size = 21 persons

Sample B was scored significantly higher ( $\alpha = 0.05$ ) than A in terms of Aroma/Flavor and Texture.



## DISCUSSION AND CONCLUSIONS

The formulations evolved in this study principally required incorporation of Kena (STPP + SHMP) alone or in combination with wheat gluten and/or NaCl to the fresh meat/seafood products. A surface application of gelatin solution was also found effective in enhancing the desirable quality characteristics of some of the product. In order to arrive at the optimum combinations of the various quality parameters, desirable concentrations of the additives were established for each one of the eight products investigated. Compared to their control samples, the freeze dehydrated products formulated with the additive system developed herein exhibited significantly higher resistance to mechanical breakage, desirable rehydration, better texture and overall organoleptic acceptance, and improved storage stability. Even though the biochemical mechanisms responsible for these effects were not investigated in detail, it was attempted to understand the general nature of these changes. Some of these favorable attributes can, however, be explained in terms of the additives - meat protein interactions.

Several investigators (Bendall, 1954; Ellinger, 1972a; 1972b; Yasui *et al.*, 1964) have contended that such palatability factors as retention of the meat juices during handling and processing, tenderness of cooked meats and fat emulsification are directly affected by the chemical and physical state of meat proteins. For example, tenderness of fresh-cooked meat is reported to be directly related to the presence of meat proteins as actomyosin, or its components, actin and myosin. This, in turn, is affected by the nature and concentration of salts present, the pH of the system, and probably the amount of calcium and magnesium as well as other heavy metal ions present, that would maintain the proteins in an insoluble state (Ellinger, 1972b). Similarly, most of the other physical effects such as mechanical strength, rehydration characteristics can be explained in terms of the protein structure and its water binding capacity.

### Effect of additives on the water binding capacity of meat

About 70% of the water content of fresh meat is located within the three dimensional network of the myofibrils (Price and Schweigert, 1971). If the total space between the filaments decreases, the amount of immobilized water is reduced. Accordingly, based on these principles, relaxation and swelling of the fibers through treatment with ATP,  $Mg^{++}$ , and EDTA has been used to bring about consequent immobilization of water.

The effect of total space between the filaments on bound water of meat can be understood by observing the effect of pH on water retaining capacity (see Figure 17). The minimum water binding appears around pH 5.0, which corresponds approximately to the isoelectric point of fibrillar proteins in the normal ionic environment of meat. Figure 17 also shows the water binding capacity of rehydrated meat (following freeze dehydration). It may be observed that the most deleterious effect

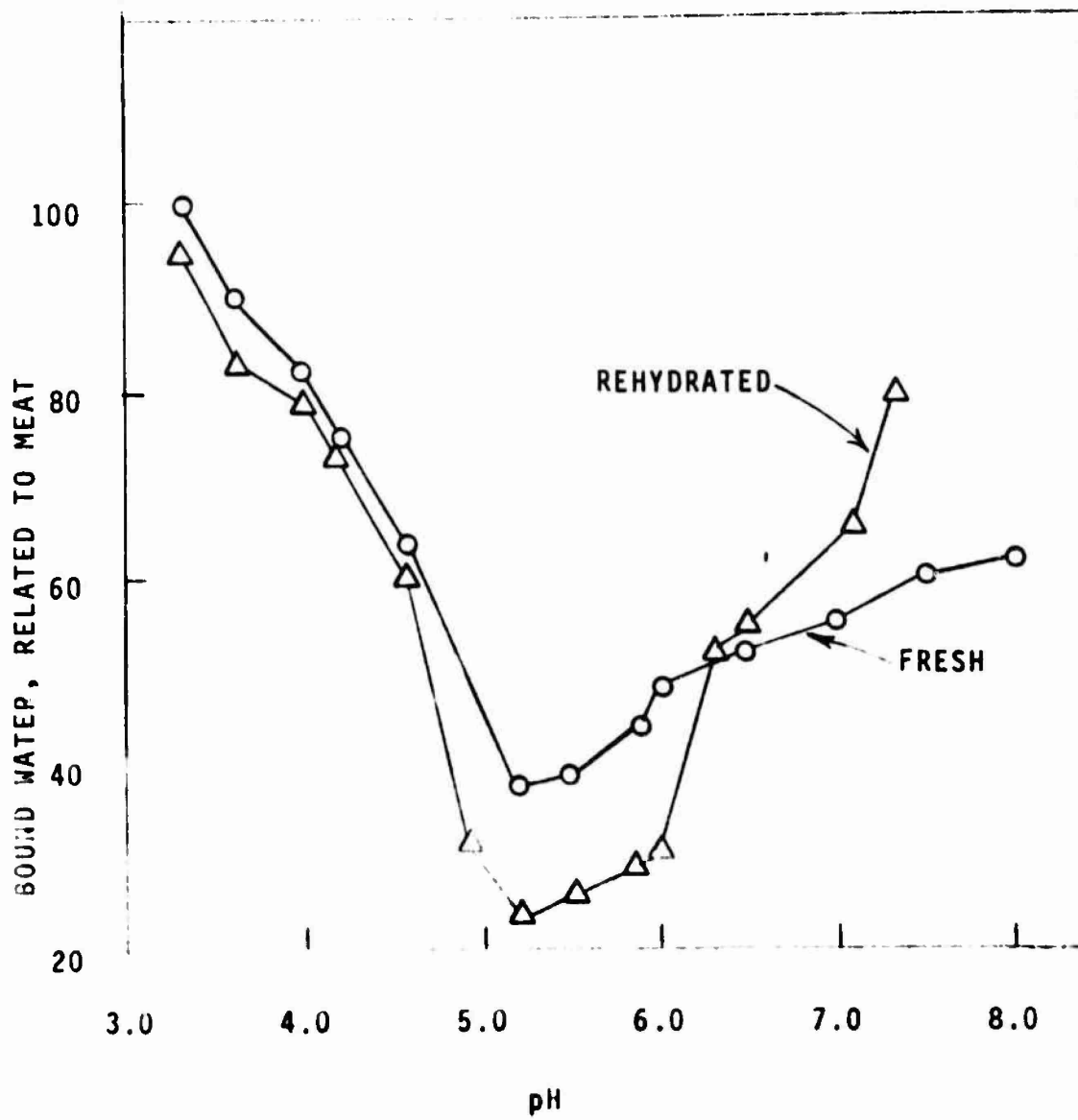


Figure 17. Influence of pH on the water binding capacity of fresh and rehydrated meat (Source: Hamm and Deatherage, 1960)

of freeze drying on water holding capacity is within a pH range of 5 to 6. To explain these changes, Deatherage and Hamm (1960) have hypothesized that removal of water during freeze drying gives rise to a decreased number of protein groups available to hold water after reconstitution. Also, they suggested that drying results in more closed protein structures owing to formation of salt and/or hydrogen bridge type bonds which can be reversed at higher or lower pH.

Addition of salts like sodium chloride on the alkaline side of the isoelectric point has been shown to increase the water retaining capacity and swelling of meat by altering the total and relative numbers of charged groups on the filaments (Bendall, 1954; Swift and Ellis, 1956). This effect in raw meat is explained to be due to displacement of the isoelectric point towards a lower pH and to increased space between the filaments at or above pH 5.0 (Price and Schweigert, 1971).

The effect of polyphosphate treatment on water binding of meat is through solubilizing actomyosin. Tripolyphosphate and pyrophosphate are reported to have a dissociating effect similar to ATP on actomyosin. In the presence of univalent cations as Na<sup>+</sup> or K<sup>+</sup> or divalent cations such as Mg<sup>++</sup>, they are capable of splitting the links between actin and myosin filaments formed at rigor and of enlarging the space between the filaments.

The physical effects of increased water binding of meat products through Kena and sodium chloride were, therefore, understood to be due to the following mechanisms:

- (a) displacement of the isoelectric point of meat proteins to a lower pH,
- (b) increased ionic strength around the filaments,
- (c) dissociation of actomyosin filaments to actin and myosin.

The indirect effects of improvement in water binding capacity were observed in terms of lower cooking losses, reduction in shrinkage (in volume) during cooking, and more desirable rehydration and tenderness of the rehydrated samples.

#### Mechanical integrity of dehydrated products

The ability of phosphates to cause the dissociation of actomyosin into actin and myosin not only has an important effect on the tenderness of all types of meats, but it also provides them with a binding matrix (Bendall, 1954; Ellinger, 1972b). Fukazawa *et al.*, (1961) suggested that the salt soluble proteins, mainly, myosin are responsible for binding of meat particles. The effect of polyphosphates in the presence of halogen ions (Cl<sup>-</sup>, Br<sup>-</sup>, I<sup>-</sup>) consists in the splitting of the contractile protein, actomyosin, into actin and myosin and partial conversion of these from the gel into sol form (Bendall, 1954). The structure of the solubilized or dispersed protein network is further strengthened by

addition of wheat gluten. Microscopic examination of the freeze dehydrated samples after histochemical staining have shown the presence of a continuous proteinaceous matrix holding the individual particles of the meat product. The microdiscontinuities or cracks in the structure of the freeze dehydrated control samples were found to be responsible for their fragmentation. The mechanism underlying reduction in breakage of the treated samples was, therefore, construed to be healing or bridging of these cracks by the dispersed meat proteins.

#### Quality changes during storage

The water binding characteristics, mechanical stability, and texture of neither the treated nor the control samples were altered significantly by a storage period of over six months. The flavor characteristics of the treated and control samples were, however, quite different. Oxidative rancidity determined by means of 2-thiobarbituric acid (TBA) method was found to be significantly lower for the treated samples compared to their respective controls. This favorable effect was probably due to the synergetic activity of the phosphate salts added to the product (Ellinger, 1972b). These changes were perceived by the panel during sensory evaluation, which accordingly, scored the treated samples higher than their respective controls. In terms of their overall evaluations, the effect of storage was more deleterious for the control samples than for those prepared with the additives.

#### Regulatory status of the proposed additives

It is interesting to notice that most of the quality characteristics of the samples formulated with the additive system developed in this study were superior to their controls. The additives recommended for the final formulations include gelatin, sodium chloride, wheat gluten and Kena (STPP + SHMP). Use of gelatin and sodium chloride as food additives is considered safe and there is no limit on their use by regulatory agencies like FDA (Furia, 1972). For individuals having dietary restriction in terms of their intake of sodium, potassium chloride may be substituted in the formulations, without affecting the end results. Use of gluten is approved for use in food products, "provided the protein content of the food does not exceed 13% by weight" (FDA 20). Table 42 lists the "generally recognized as safe" (GRAS) phosphates for use with various food products (FDA), which include both STPP and SHMP (constituents of Kena) as sequestrants. Table 43 lists the specific phosphate additives acceptable for meat processing, by Meat Inspecting Division of USDA, wherein along with the various uses of phosphates recommended limits on their quantity is suggested. Even though the specific application of phosphate "to relate breakage in freeze dried meat products" is not listed, their application under the class of phosphates may be well justified for the present problem. The concentrations of the additives established herein are well within the acceptable limits.

Table 42. Phosphate food additives listed as GRAS by FDA

EMULSIFYING AGENTS

Monosodium phosphate derivatives of mono- and diglycerides

NUTRIENTS AND/OR DIETARY SUPPLEMENTS

Calcium glycerophosphate  
Calcium phosphate (mono-, di-, tribasic)  
Calcium pyrophosphate  
Ferric phosphate  
Ferric pyrophosphate  
Ferric sodium pyrophosphate  
Magnesium phosphate (di-, tribasic)  
Manganese glycerophosphate  
Manganese hypophosphite  
Potassium glycerophosphate  
Sodium phosphate (mono-, di-, tribasic)

SEQUESTRANTS

Calcium hexametaphosphate  
Calcium phosphate (monobasic)  
Dipotassium phosphate  
Disodium phosphate  
Sodium acid phosphate  
Sodium hexametaphosphate  
Sodium metaphosphate  
Sodium phosphate (mono-, di-, tribasic)  
Sodium pyrophosphate  
Sodium pyrophosphate (tetra)  
Sodium tripolyphosphate

MISCELLANEOUS AND/OR GENERAL-PURPOSE FOOD ADDITIVES

Ammonium phosphate (mono- and dibasic)  
Calcium phosphate (mono-, di-, tribasic)  
Phosphoric acid  
Sodium acid pyrophosphate  
Sodium aluminum phosphate  
Sodium phosphate (mono-, di-, tribasic)  
Sodium tripolyphosphate

Source: 121.101(D). Part 121, Code of Federal Regulations, Title 21 (revised as of January 1, 1971), U.S. Government Printing Office, 1971.

Table 43. Phosphate food additives acceptable for use in meat processing

Class of Substance	Phosphate	Purpose	Products	Amount
Cooling and retort water treatment agents	TKPP ) SHMP ) TSPP ) STP )	To prevent staining on exterior of canned goods	Various	Sufficient for purpose
Hot scald agents	SHMP ) STP ) TSP )	To remove hair	Hog carcasses	Sufficient for purpose
Phosphates	DSP ) MSP ) SHMP ) STP ) TSPP ) SAPP )	To decrease amount of cooked-out juices	Cured hams, pork shoulder picnics, loins; canned hams and pork shoulder picnics; chopped ham; bacon	5.0% of phosphate in pickle at 10% pump level; 0.5% of phosphate in product (only clear solution may be injected into product)
Rendering agents	TCP ) TSP )	To aid rendering	Animal fats	Sufficient for purpose
Synergists (used in combination with anti-oxidants)	H <sub>3</sub> PO <sub>4</sub> )	To increase effectiveness of anti-oxidants	Lard and shortening	0.01%

Source: 318.7(4), Code of Federal Regulations, Title 9 (revised as of January 1, 1971), U.S. Government Printing Office, 1971.

### Conclusion

Through appropriate application of GRAS-FDA accepted food additives, freeze dried meat/seafood products have been developed which are significantly resistant to mechanical breakage during their transport and handling. Also, the newly developed formulations impart superior texture, rehydration, flavor and overall organoleptic acceptance. These qualities are found to be retained after storage treatment.

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**APPENDIX**

Table A1. Particle size distribution (% weight fraction) of the raw, freeze dried beef patties, drop tested after being stored at 38°C for 39 weeks

Sieve No./Size	Weight fraction retained from samples prepared with additives as:	
	None (control)	1.25% NaCl, 0.5% Kena and 0.15% wheat gluten
19 mm	45.62	87.61
9.5 mm	22.10	3.84
No. 3	4.30	2.01
No. 4	4.97	0.44
No. 5	11.29	0.50
No. 8	6.12	1.30
No.16	2.83	1.87
No.20	0.63	0.71
No.30	0.80	0.89
No.50	0.87	0.77
Pan	0.10	0.06

Note: Comparison with the fragment size distribution in Table 11 shows similar trends in two samples, before and after storage

Table A2. Particle size distribution (% weight fraction) of the cooked, freeze dried beef patties drop tested after being stored for 22 weeks at 38°C

Sieve No./Size	Weight fraction retained from samples with the additives as:	
	None (control)	1.25% NaCl, 0.38% Kena, and 0.15% wheat gluten
19 mm	0.00	10.44
9.5 mm	0.54	44.13
No. 3	4.35	12.00
No. 4	12.33	7.38
No. 5	7.05	2.17
No. 8	37.57	7.25
No.16	20.19	7.52
No.20	4.70	2.64
No.30	7.86	3.88
No.50	4.82	3.49
Pan	0.58	0.36

Table A3. Particle size distribution (% weight fraction) of the cooked, diced freeze dried chicken drop tested after being stored at 38°C for 22 weeks

Sieve No./Size	Weight fraction retained from samples with:	
	No treatment	Sprayed with gelatin before drying
9.5 mm	9.00	28.86
No. 3	41.86	41.47
No. 4	18.05	11.47
No. 5	11.32	3.82
No. 8	7.59	5.81
No. 16	5.73	5.64
No. 20	1.66	1.07
No. 30	2.17	0.98
No. 50	1.42	0.73
Pan	1.20	0.14

Table A4. Weight fraction of the unfragmented freeze dried fish squares, drop tested after being stored at 38°C for 33 weeks

Treatment	% of the weight retained as major fragments* after drop testing
A. Control	86.49
B. Coated with gelatin solution after freeze drying and redried	95.58

\* fragments retained on 9.5 mm sieve

Table A5. Summary of the drop tests on freeze dried cooked pork chops after being stored at 38°C for 33 weeks

Treatment	% of the weight fraction retained as major fragments* after drop testing
A. Control	45.2
B. Dusted with Kena, held for one hour, rinsed and cooked	58.2

\* fragments retained on a 9.5 mm sieve



Table A6. Fragment size distribution of (% weight fraction) of the raw, freeze dried pork chops, drop tested after being stored at 38°C for 33 weeks

Sieve No./Size	Weight fraction retained from:	
	Control sample	Sample dusted with Kena, held for one hour and rinsed
19 mm	48.24	64.58
9.5 mm	24.68	28.39
No. 3	14.45	4.33
No. 4	3.20	0.96
No. 5	8.21	0.33
No. 8	0.28	0.33
No. 16	0.62	0.48
Pan	0.30	0.60

Table A7. Particle size distribution (% weight fraction) of the freeze dried pork sausage patties, drop tested after being stored at 38°C for 22 weeks

Sieve No./Size	Weight fraction retained from samples prepared with:	
	MIL Spec.	1.25% NaCl, 0.38% Kena and 0.10% wheat gluten
19mm	0.00	71.55
9.5 mm	14.24	15.68
No. 3	18.93	2.10
No. 4	19.83	0.95
No. 5	8.56	0.42
No. 8	15.87	2.10
No. 16	12.10	3.20
No. 20	4.53	2.01
No. 30	4.71	1.59
No. 50	1.26	0.36
Pan	0.04	0.02

Table A8. Weight fraction of the unfragmented freeze dried shrimp, drop tested after being stored at 38°C for 31 weeks

Treatment	Percent of the whole unbroken* sample after drop testing
A. Control	85.53
B. Dusted with Kena before cooking	91.01

\* Each of the samples weighed about 50g to start, and those retained on 9.5 mm sieve were considered unbroken

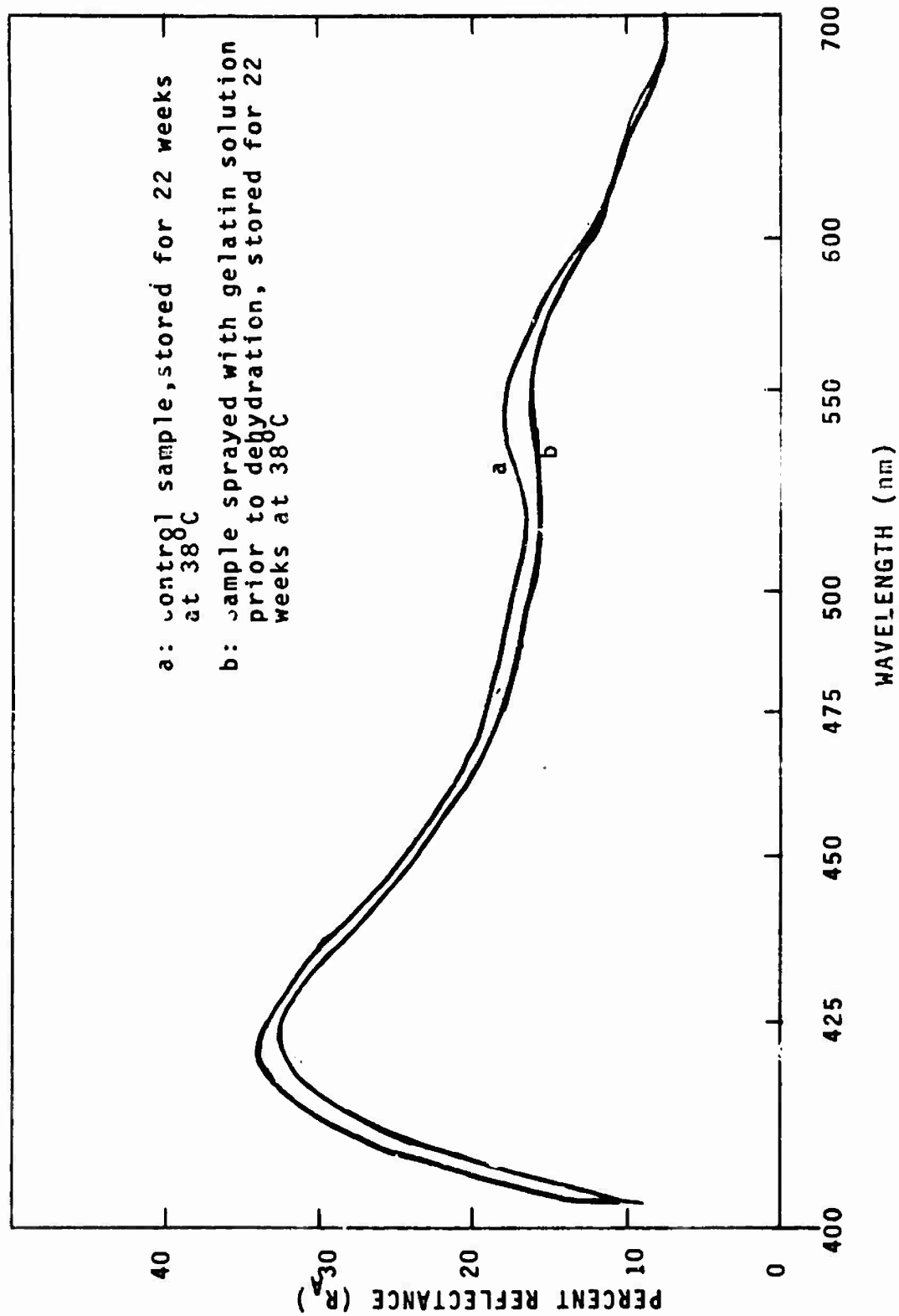


Figure A1. Reflectance spectra of cooked, diced, freeze dehydrated chicken samples following rehydration: Gelatin treatment evaluation

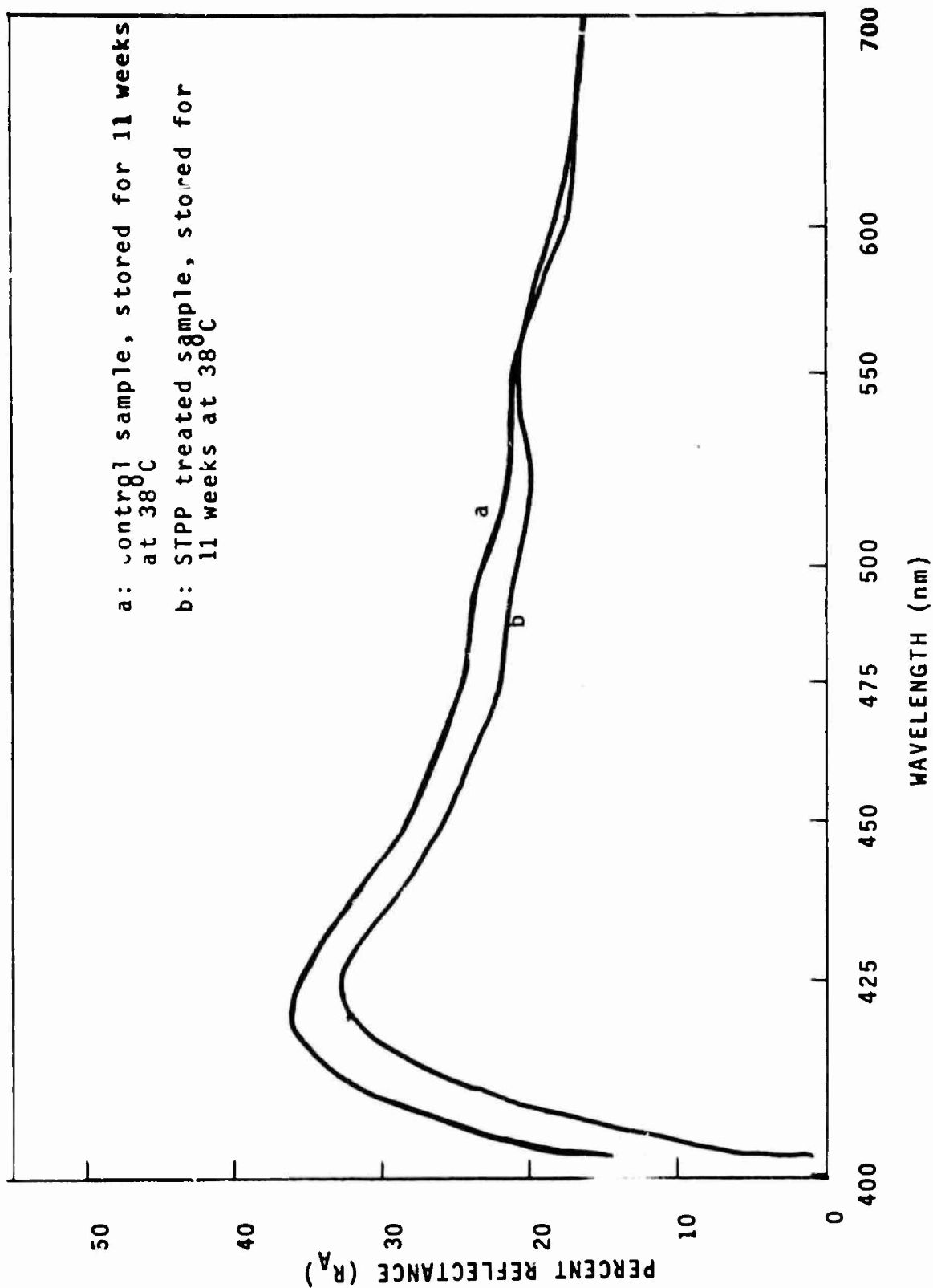


Figure A2. Reflectance spectra of raw freeze dried diced chicken samples after rehydration: phosphate treatment evaluation

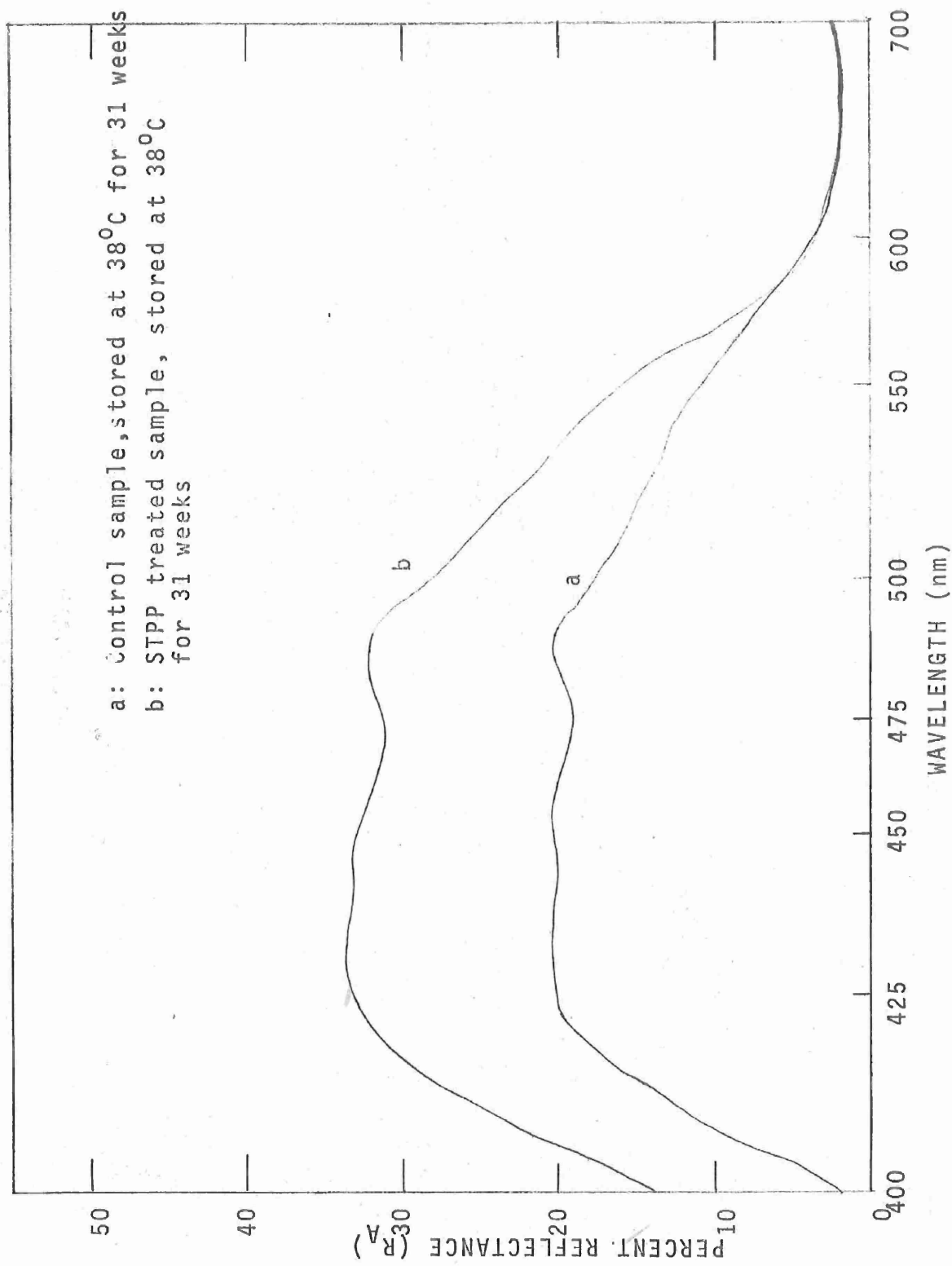


Figure A3. Reflectance spectra of cooked, freeze dehydrated shrimp samples following rehydration

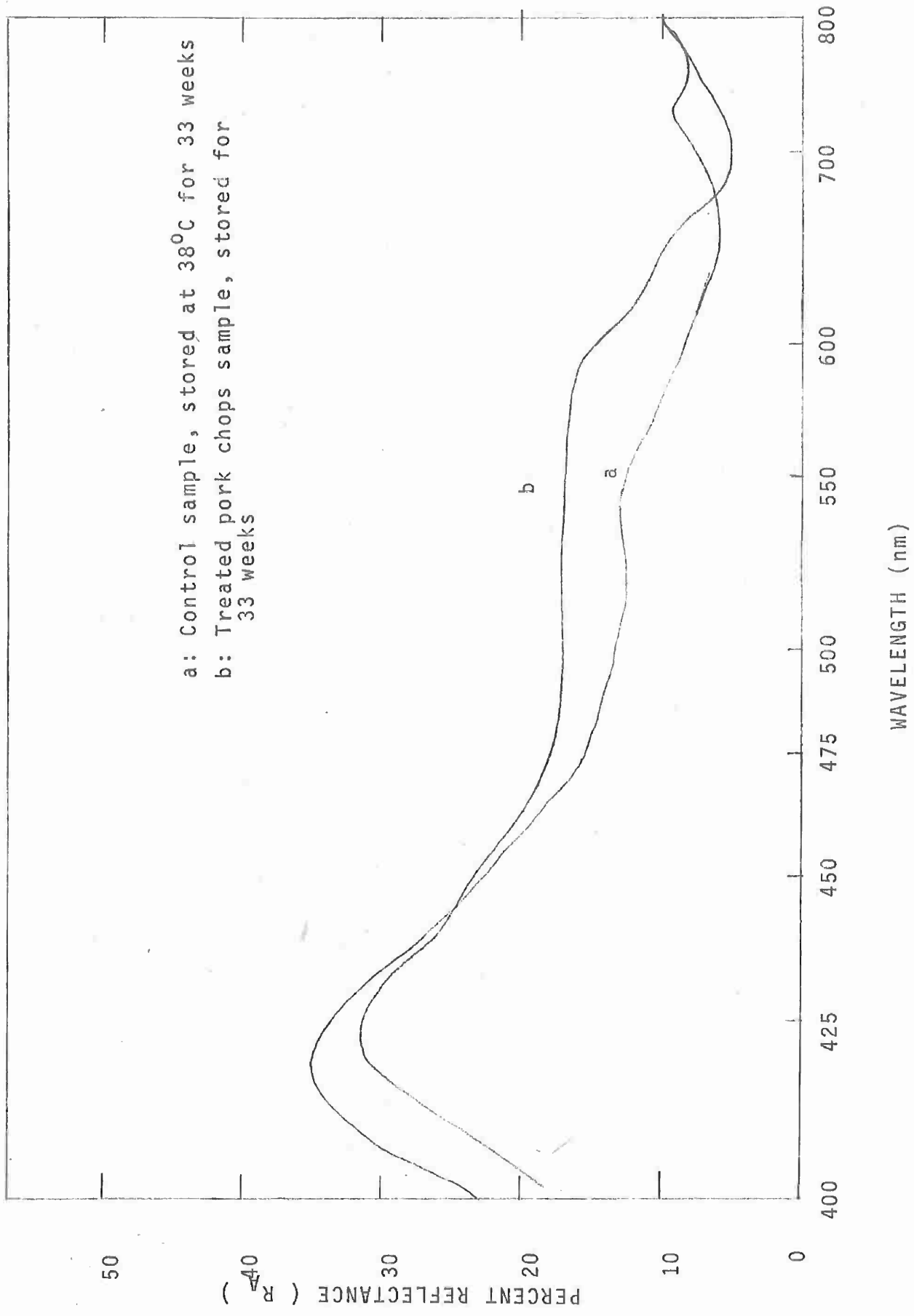


Figure A4. Reflectance spectra of raw freeze dehydrated pork chops after rehydration