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COMPARATIVE EFFECTS OF DEHYDRATION PROCESSES

ON PHYSICO-CHEMICAL CHANGES IN FRUITS

by

Chang Yong Lee

A dissertation submitted in partial fulfillment  
of the requirements for the degree

of

DOCTOR OF PHILOSOPHY

in

Food Science and Technology

Approved:

UTAH STATE UNIVERSITY •  
Logan, Utah

1967

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*Chang Yong Lee*  
Chang Yong Lee

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## INTRODUCTION

Drying with the help of sun and wind is one of the oldest methods of food preservation known to man, but artificial drying, or dehydration, has been developed and used extensively only during the last two decades.

The problem in dehydration is that the water content must be decreased sufficiently to maintain the stability of the product by retarding the rates of deteriorative biochemical, microbiological, and enzymatic reactions during subsequent storage. At the same time irreversible changes should not be brought about.

In recent years emphasis has been placed upon dehydro-freezing and freeze dehydration of fruits. In the dehydro-freezing procedure nearly 50 per cent of the original weight is reduced by dehydration and subsequently the product is frozen. This process inactivates enzyme systems in fruit and reduces bulk and weight, hence ease in handling and shipping. Upon thawing dehydro-frozen fruit there is less drip and better moisture control. This process also offers some of the potential advantages of good quality at a substantial saving in cost (Kaufman and Powers, 1957).

In the process of freeze dehydration of foods most of the moisture is removed from frozen foods by sublimation without an appreciable change in the form, color, nutrients, and taste of the product. This process prevents virtually all bacterial and enzymatic actions in food products. The final products become microporous throughout as the ice is vaporized out of it,

hence rehydration can be accomplished rapidly. The freeze dehydrated foods need no refrigeration in handling, transportation, and storage. These advantages give freeze dehydrated foods an enviable position among other dehydrated produce. In spite of these several advantages of freeze dehydrated foods, their current volume is limited by high processing costs. The process is slow and the operation requires a high degree of technical skill. Packaging of the dehydrated product is expensive, because many of these foods are very brittle and friable and thus do not survive rough handling which means that special packages are required. Nevertheless, recent research studies by United States Department of Agriculture economist shows that costs may be reduced in the future as processing plants become larger and more efficient (Bird, 1964, 1965a and 1965b).

Application of several dehydration processes to fruits has been reported by Powers et al. (1956), Harper and Tappel (1957), Jackson et al. (1957), Bean et al. (1957), and Lazar et al. (1961). However, information of the comparative effects of different dehydration process on flavor compounds, color, histology, and rehydration of fruits is meager and so extensive studies are needed.

The investigations reported here were conducted to elucidate the characteristic effects of several dehydration processes on afore mentioned chemical and physical changes in apples, apricots, and peaches.

It is believed that information developed in this study would serve to increase knowledge in the field of dehydration and may consequently aid in the developments of new products and processes.

## REVIEW OF LITERATURE

### Dehydration of Fruit

#### Apples

The most obvious manifestation of deterioration of dehydrated fruits is browning, which may be accompanied by impairment of flavor, texture, and nutritive value. Extensive investigation of the sulfur dioxide treatment used to reduce this effect has been reported. Smock and Neubert (1950) have described the various factors involved in apple dehydration. Talburt et al. (1950), and Walker et al. (1955) developed the dehydro-freezing process for apples and determined the effects of variations in sulfite treatment and dehydration upon quality of the product. Powers et al. (1958) produced dehydro-canned apples without using sulfite and found the storage stability was good through one year at 70<sup>o</sup> F. Lazar et al. (1961) discussed an improved processing method for dehydro-frozen apples by using through-flow drying process which controlled the sulfite content and increased the drying capacity. The important characteristics of dehydro-frozen apples for the bakery products were reported by Guadagni and Harris (1961). Recently, Lazar et al. (1963) developed a dry-blanch-dry (DBD) method that apples are dehydrated without sulfite treatment.

#### Apricots

Apricots are primarily sun-dried. During World War II, however, research on dehydration of apricots was intensified because of an increasing

demand for the products by the Armed Forces, a shortage of labor to produce the sun-dried fruit, and a desire to produce a dried apricot by a more sanitary method than sun-drying (Brekke and Nury, 1964). Mrak et al. (1943) developed dehydrated apricots by application of steam blanching. Powers et al. (1956) produced dehydro-frozen apricots. They employed a two-step sulfur dioxide treatment and produced brightly colored and richly flavored apricots. Schwarz and Nury (1961) studied hot-air dehydration of apricots and evaluated various characteristics of both sun-dried and hot-air dehydrated apricots. Nury, et al. (1960) reported the stability of dehydrated apricots under various atmospheric conditions. Harper and Tappel (1957) discussed briefly the merits of freeze dehydrated apricots.

### Peaches

Mrak and Perry (1948) reported on the dehydration of peaches and observed that blanched peaches are dehydrated rapidly. Bean, et al. (1957) produced low moisture vacuum dehydrated peaches. They reported that the sulfite dipped fruit packed in vacuum showed remarkable quality after storage at 100<sup>0</sup> F for 6 months. Saravacos and Charm (1962) studied the effect of surface active agents on the dehydration of peaches and other fruits. Jackson et al. (1957) and Chichester (1956) discussed the process of freeze dehydration of peaches and the storage stability of the products. Harper and Tappel (1957) also reported on the freeze dehydration of peaches and the relation of prior treatment with sulfur dioxide and the storage stability. Recently, Huang and Draut (1964) studied the effect of moisture on the browning reaction of freeze dehydrated peaches.



### Flavor Compound Changes

Flavor is one of the important attributes of dehydrated fruit products. Volatile compounds, soluble solids, acids, sugars, and tannins influence primarily the flavor and taste of fruit. The chemistry of fruit flavor was reviewed by Kirchner (1949) and McGlumphy (1951). Leonard et al. (1953) and Luh (1961) reported the flavor compounds of canned peaches and apricots. There is no information available concerning flavor compound changes occurring during the dehydration process.

### Gas-Liquid Chromatography

Gas-liquid chromatography has introduced some possibility of quantitative and reproducible analysis of food aroma. Many investigators in the fruit flavor field have used gas-liquid chromatography as an analytical tool in recent years. Buttery and Teranishi (1961) and Teranishi et al. (1962) attempted fruit flavor analysis by gas chromatographic methods utilizing a dual flame ionization detector for direct vapor analysis of fruit flavor. Jennings and Sevenants (1964) isolated major components of peach essence by gas chromatographic methods. Rhoades and Millar (1965) reported the method of flavor analysis for apricots, peaches, and strawberries. Weurman (1961) studied the volatile compounds in raspberries by head space sampling method of gas-liquid chromatography and found that this method was useful for quantitative determination of small amount of volatiles in fruit.

### Color Changes

Changes in the color of fruit during dehydration is an important mechanism causing the deterioration of quality of dehydrated fruit. It is generally known that the browning reaction is the major reason of color change in fruit which may be accompanied by impairment of flavor and nutritive value. Stadtman (1948) discussed nonenzymatic browning of fruit products and gave a general review of the problem of measuring color in dehydrated fruit. Stadtman et al. (1946a) determined the relative degree of browning of apricot by comparison of 50 per cent alcohol extracts with a standardized reference solution.

Joslyn and Ponting (1951) reviewed the literature on enzyme catalyzed oxidative browning of fruit products. They indicated that discoloration does not occur in the intact cells of the fruit susceptible to enzymatic oxidation. When the protoplasm of the cells is altered mechanically by cutting, bruising, or freezing, browning occurs rapidly. Cells damaged by heat will also brown. It may be assumed that the conditions mentioned above will be the major factors causing the color changes during the dehydration processes.

### Histological and Histochemical Changes

Although histological and histochemical changes of fruits during blanching, canning, and freezing have been investigated widely, there is a lack of information on the effects of various dehydration conditions on histology and histochemistry of fruit.

Woodroof (1938) reported freezing effects on the tissues of fruits and measured the size of ice crystals produced by different types of freezing. He reported that quick freezing prevented the formation of large ice crystals in the fruit tissues and subsequently minimized the cellular destruction. Hohl (1948) discussed the effect of freezing and dehydration on the apricot skin. Weier and Stocking (1949) reviewed the effects of heat on the cell and cell walls of fruit. They also pointed out some factors involved in freezing of fruit. Reeve and Leinbach (1953) studied the influence of heat on apple structure and observed that expansion and escape of intercellular gases during heat treatments aided mechanically in cell separation of apples. Sterling (1955) observed that the starch grains in apples are gelatinized by heat and the cell walls are folded and wrinkled during cooking. He also pointed out that fruit cell walls were not broken down by heat in the cooking process.

#### Rehydration Rate

Changes in water-holding capacity of fruit during the dehydration is another important mechanism causing the deterioration of quality of dehydrated fruit. Karel (1963) indicated that the loss of water-holding capacity is a characteristic form of deterioration of dehydrated foods and is usually associated with a decrease in organoleptic acceptability. Kuprianoff (1958) discussed the reversible and irreversible changes caused by withdrawal of water from the food and postulated that the reversibility is greater the more complete reconstitution of the dehydrated product is obtained after

adding water. Goldblith *et al.* (1963) showed that measurement of rehydration ratio is one of the objective evaluation parameters that might be used to determine the quality of freeze dehydrated products.

#### Osmotic Effects of Sucrose in Freeze Dehydration

The absorption of sugar by fruit with added sugar or syrup has long been considered desirable to improve preservation of color and flavor to yield a better tasting product. Many factors appear to affect the penetration of sugars into a canned fruit product. Joslyn and Hohl (1949) studied early the effect of sugars on frozen fruit and found that the sugar solutions inhibit discoloration by their effect in reducing the concentration of dissolved oxygen and reducing the rate of diffusion of the oxygen of the air into the fruit tissue. Joslyn (1949) showed some data on the effect of type of sugar, oxygen solubility and the relation of sugars with the rate of oxidation of ascorbic acid. Perry and Cruess (1953) observed sugar penetration in several frozen fruits by the measurement of soluble solids in fruit and sugar syrups. Guadagni (1949) studied the vacuum impregnation of apple slices with sugar syrups and found that the product of syrup filling was equal or superior to that of steam blanching and sulfiting. Hughes *et al.* (1958) suggested that penetration of the sugars from the syrup into the fruit is probably related to the diffusional properties of the individual sugars. They studied the penetration of maltosaccharides in processed clingstone peach halves with C<sup>14</sup> labeled sugars and found that the rate of sugar penetration is related directly to the concentration. Sterling

and Chichester (1956, 1960) recently observed the penetration of maltosaccharides into canned peaches by using the method of autoradiography and found that radioactivity was localized principally in the cell walls of the peach tissues. All the studies reported above have been conducted on frozen and canned fruit with the syrup. Unfortunately, because of the many components present in canned fruits, and of different processes of dehydration, these findings cannot be directly applied to the problems of sucrose in the osmo-freeze dehydration.

## MATERIALS AND METHODS

### Materials

Apples (Pyrus malus), variety Golden Delicious, apricots (Prunus armeniaca), variety Large Early Montgamet, and peaches (Prunus persica), variety Late Elberta, were harvested from selected trees at the Howell Field Experiment Station, Ogden, in 1964. In 1965, apricots and peaches were harvested from commercial orchards in Price and Ogden, Utah, respectively. The fruits were selected for uniformity of color, size, and maturity. Maturity was determined by visual means, ground color, and firmness. A Magness-Taylor (1925) pressure tester was employed for measurements of fruit firmness.

After harvest the fruits were immediately transported to the Utah State University Food Processing Laboratory and stored at 35-40<sup>0</sup> F until they were processed.

### Dehydration

Apples were normally peeled, cored, halved, and sliced into one-fourth inch thick slices, and immediately placed in a 2 per cent sodium chloride solution to prevent surface browning.

Apricots and peaches were immersed in boiling water for one minute, peeled, pitted, halved, and sliced into six equal sections manually. The sliced

fruits were then dipped in solution containing 0.5 per cent sodium chloride and citric acid in an attempt to control surface browning. Freshly sliced fruits prepared by the above methods were subsequently dehydrated by the following procedures.

#### Conventional hot-air dehydration

Dehydration was carried out by placing fresh sliced fruits in an experimental dehydrator (Figure 1), built at Utah State University Food Processing Laboratory, with a heated-air current at  $175 \pm 5^{\circ}$  F until the moisture content was reduced to between 5 and 10 per cent.

#### Dehydro-freezing

The dehydration process was identical to the above method. However, when the original weight was reduced to about 50 per cent, the product was removed from the dehydrator and placed in cans, sealed, and frozen at  $0^{\circ}$  F.

#### Freeze dehydration

Fresh fruit slices received the following three treatments before freeze dehydration:

Conventional freezing. A single layer of sliced fruits was spread on stainless steel trays and frozen in a cabinet-type freezer at  $0^{\circ}$  F.

Cryogenic freezing in liquid nitrogen. Fresh fruit slices were immersed in liquid nitrogen at about  $-250^{\circ}$  F for 5 minutes and then placed on the dehydration trays.



Figure 1. Experimental hot-air dehydrator.



Osmo-dehydration by sucrose. Fresh apple and peach slices were mixed with an equal amount of sucrose by weight and held at room temperature (75° F) for 15 hours and then removed from the syrup and spread on stainless steel trays and subsequently frozen by the conventional method.

Frozen fruit slices prepared by the above three methods were dehydrated in a freeze dehydration unit (Figure 2, Hull Corporation, Model 651-F5, Hatboro, Pennsylvania) until the vacuum of the chamber reached 80 microns Hg., and the product temperature reached 105° F. The moisture content of the final products at that point was less than 2 per cent. The freeze dehydration conditions are shown in Figure 3. A simplified flow diagram for the preparation of dehydrated fruit is shown in Figure 4.

### Analyses

#### Chemical analyses

Representative samples of apples, apricots, and peaches from each of the dehydration processes as well as fresh fruits as control were analyzed for the following components. Conventional frozen and freeze dehydrated fruits were represented for the chemical analyses among the freeze dehydrated fruits.

pH and acidity. A sample of 50 g of fruit tissue (fresh fruit basis) was homogenized with 50 ml distilled water in Waring blender for 5 minutes. An aliquot of 20 g of the homogenate was placed in a beaker and the pH was read on a Beckman Model H-2 pH meter. The same sample was titrated to a



Figure 2. Freeze dehydration unit.

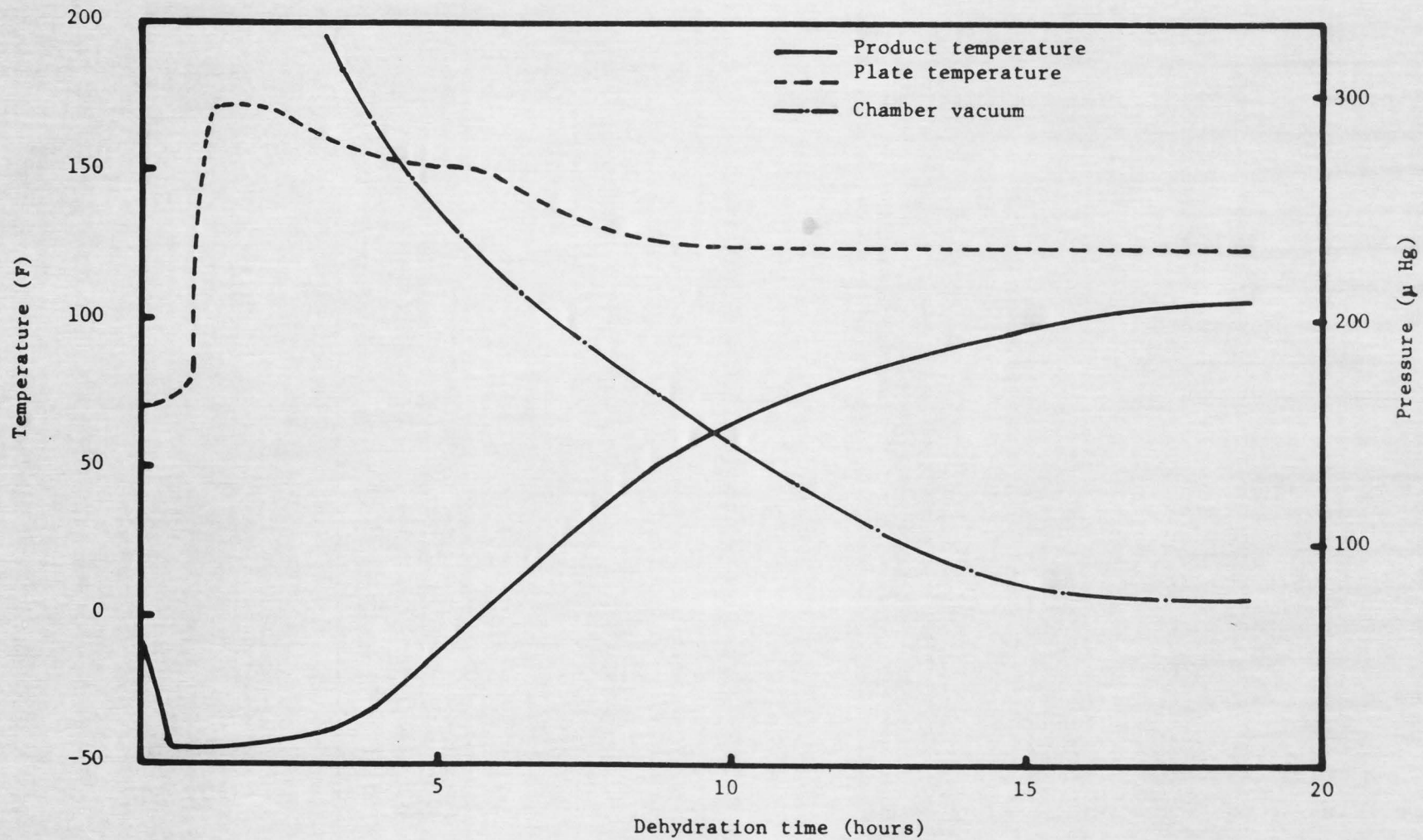


Figure 3. Freeze dehydration curves for sliced fruits.

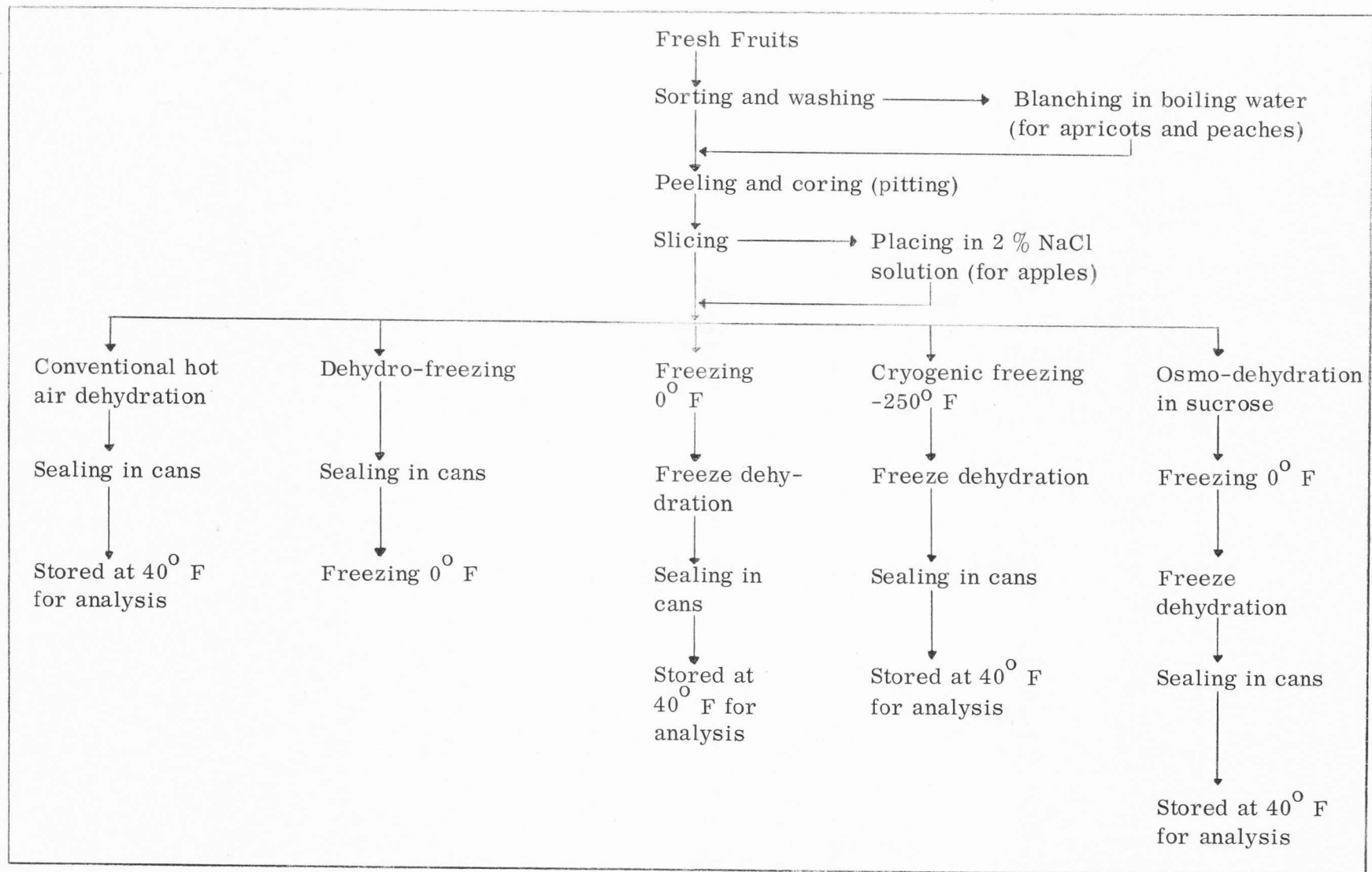


Figure 4. Flow diagram for the preparation of dehydrated fruits.

pH of 8.1 with 0.1 N sodium hydroxide solution and the acidity was calculated as per cent malic acid.

Soluble solids. A few drops of the filtered homogenate were adopted for the determination of soluble solids with a Zeiss Opton refractometer.

Sugars. A sample of 50 g fruit was homogenized with 50 ml of 80 per cent alcohol solution in a Waring blender for 5 minutes. The homogenate was centrifuged for 5 minutes at 2500 revolutions per minute (International Centrifuge, Model V). The alcohol extract was used for the determination of reducing sugars by the volumetric Schaffer-Somogyi Micro Method as described in the A. O. A. C. (1960). For the determination of total reducing sugars, alcohol extract was inverted with hydrochloric acid and then analyzed by the same method of free reducing sugars.

Volatile compounds. A sample of 150 g fruit was homogenized with 150 ml distilled water in a Waring blender. The homogenate was steam distilled in an all glass apparatus at the rate of 7 ml per minute. About a 150 ml of distillate was analyzed for volatile reducing substance according to the method of Luh (1961) and volatile carbonyl compounds by the method of Lappan and Clark (1951).

Tannins. A 2 ml of alcohol extract was used directly for the analysis of tannins according to A. O. A. C. colorimetric method (1960).

Ascorbic acid. Ascorbic acid was determined by 2,6-dichlorophenol indophenol titration method of A. O. A. C. (1960).

### Gas-liquid chromatographic analysis

Direct head space analysis by gas liquid chromatography was conducted according to the method described by Buttery and Teranishi (1961) with a slight modification. A well mixed 100 g sample was placed in a 250 ml Erlenmeyer flask, sealed with a rubber stopper, and the flask was then placed in a constant temperature water bath at 158<sup>o</sup> F for 30 minutes with occasional shaking. A 5 ml vapor sample was withdrawn by means of a hypodermic syringe and was injected directly into a Micro-Tek Model 2000 gas chromatograph. The instrument was equipped with 30.48 m x 0.64 cm dual columns packed with 80-100 mesh Chromosorb-P coated with Carbowax 20 M (20 per cent). The operation conditions were: column temperature, 212<sup>o</sup> F; injector temperature 350<sup>o</sup> F; detector temperature 392<sup>o</sup> F; carrier gas (He), 70 ml per minute.

### Alcohol extractable color

A 10 gram sample of homogenized fruit was placed in a 300 Erlenmeyer flask containing 200 ml of 50 per cent ethanol and extracted for 20 hours at room temperature with occasional shaking. The colored solution was filtered through Whatman No. 2 filter paper and then the color reading made with the Beckman Model DU spectrophotometer using 1 cm cell. The results were expressed as moisture-free basis optical density according to Nury et al. (1960).

### Histological and histochemical analyses

Representative samples selected from each dehydrated product were employed for histological and histochemical analyses.

Histological techniques. For histological studies, the paraffin-embedding method was applied to each representative sample of fruit. Fresh fruit was cut into small rectangles (0.65 x 0.65 x 1.95 cm) and was killed and fixed in Formalin-Aceta-Alcohol (FAA) solution and dehydrated in a tertiary-butyl alcohol (TBA) series (Johansen, 1940 and Sass, 1961). Dehydrated specimens were transferred to a small vial containing equal parts of TBA and paraffin oil and placed in the oven at 135<sup>o</sup> F for one day. After the TBA evaporated, specimens simultaneously and slowly infiltrated and sank to the bottom of the vial. Specimens were then embedded, blocked, sectioned, and mounted according to Sass (1961).

The preparation of the dehydrated samples for histological examination presented a number of difficult problems. The sample could not be softened and embedded well by standard methods (Johansen, 1940) and gave trouble on microtome sectioning. Conventional hot-air dehydrated and freeze dehydrated fruits were treated according to the following method. Dehydrated fruit samples were cut into small rectangles (0.65 x 0.65 x 2.54 cm) and placed directly into 100 ml vials containing a commercial dehydrating agent ("dehydrant" formula No. S-29, Technicon Chemical Company, Inc., Chancey, New York) and left for one week. During this period the dehydrant was changed <sup>ree</sup> there times, and the dehydrated specimens were then transferred to a commercial cleaning agent ("cleaning agent" formula No. C-650, Technical Chemical Company, Chancey, New York). The specimens were transferred to new cleaning agent twice during five days and then placed in melted paraffin. This was followed

by three changes of paraffin, and finally the specimens were embedded, blocked, sectioned, and mounted.

Fresh and dehydrated fruit samples prepared by the above methods were sectioned with a rotary microtome at 20 microns thickness. The sections were affixed on slides with Haupt's adhesive (Johansen, 1940).

A dilute aqueous solution of Delafield's hematoxylin and fast green was used for the staining of fresh fruit sections according to Johansen (1940). In an attempt to retain the effects of dehydration on the fruit tissue, the sections of dehydrated fruits were stained directly with a dilute fast green in a xylene and alcohol mixture after removal of paraffin from the sections with xylene. The stained sections were washed in alcohol-xylene mixture and xylene, and then mounted in Canadian balsam.

Histological studies on the dehydrated fruit tissue were also conducted after rehydration. Dehydrated samples were rehydrated in distilled water containing about 200 ppm dissolved sulfur dioxide to prevent enzymatic browning and then sectioned with the freezing microtome at about 150 microns. Fresh fruit sections were also prepared after fixing in FAA solution. The sections were dehydrated in an alcohol series (30, 50, 70, and 95 per cent), and stained with orange G, carmine, aniline blue, and fast green. The stained sections were washed twice in 95 per cent alcohol and transferred to absolute alcohol. The sections were then cleaned in xylene and the sections were finally mounted on Canadian balsam.



Histochemical technique. Thick sections of the samples were prepared for histochemical studies by the method of Reeve (1952). For pectin characterization the hydroxylamine-ferric chloride reaction was employed according to McCready and Reeve (1955) and Reeve (1959a and 1959b). The sections were placed in a solution of 0.5 N hydrogen chloride in absolute methyl alcohol and left overnight to esterify the pectins. The esterified sections were thoroughly washed with several changes of methanol and then transferred to a mixture of 5 ml each of solutions of 14 per cent hydroxamine hydrochloride and 14 per cent sodium hydroxide in 60 per cent ethyl alcohol. After five minutes, 5 ml of a mixture of one volume concentrated hydrochloric acid and two volumes of 60 per cent ethyl alcohol were added. Five minutes after this acidification, the sections were moved to 15 ml of 10 per cent ferric chloride in 60 per cent ethyl alcohol containing 0.1 N hydrochloric acid. Three minutes were required for full color development with the ferric chloride. The colored sections were washed in 60 per cent alcohol and then mounted on glycerin.

A weak solution of iodine was applied for the staining of starch granules in apple tissue. Fruit sections were placed in iodine-potassium iodide (IKI) solution (1 g iodine and 1.5 g potassium iodine in 100 ml distilled water) for ten minutes and then washed in water. This was followed by dehydration in alcohol solutions, staining with fast green and subsequently mounting on Canadian balsam as described previously.

A saturated Sudan IV solution in 60 per cent alcohol was prepared for the location of carotene bodies in apricot and peach tissues according to

Weier (1942). Fruit sections were stained with Sudan IV solution for five minutes and then washed quickly in 60 per cent alcohol and mounted immediately either on water or glycerin.

Cell wall measurements were made with a calibrated eye-piece micrometer at X 43 magnification.

Camera lucida and photomicrography. Camera lucida drawings were prepared. Photomicrographs were made using 35 mm Adox film for black-white pictures and 35 mm high speed Ektachrome for color pictures.

Rehydration rate. A 10 gram sample of dehydrated fruit was rehydrated in a water bath operated at 100<sup>0</sup> F to measure the rehydration rate. The rehydration rate was expressed by the ratio of wet weight and dry weight against the rehydration time.

$$\text{Rehydration ratio} = \frac{\text{Wet weight of fruit}}{\text{Dry weight of fruit}}$$

#### Osmo-dehydration of sucrose and autoradiograph

In order to elucidate the effects of sucrose on the dehydration by osmosis and diffusion of sucrose in the pretreatment of osmo-freeze dehydration, the following studies were conducted on apples.

Diffusion pressure deficit of apple. Golden Delicious apples were manually peeled, cored, and sliced into 1 x 1 x 4 cm. The slices were rapidly rinsed in water and blotted to dry with paper towel and then weighed. The sections were immediately placed in 50 ml of 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, and 1.0 Molar sucrose solution at room temperature (75<sup>0</sup> F). Exactly one hour after immersion of a particular slice, the slice was removed, rinsed, blotted, and weighed. The diffusion pressure deficit was calculated

from the ratio of final over original weight against osmotic pressure of the solutions (Meyer et al., 1955).

Changes in weight and soluble solids. Fresh apple slices prepared as the previous method were immediately placed in 50 ml of 0.7, 1.5, 2.0 Molar sucrose solution and in dry sucrose. Weight and soluble solids changes due to sucrose absorption and moisture transfer between solution and fruit were determined at 5, 10, 15, and 20 hour intervals. The soluble solids in the solution and fruit were measured by a refractometer. Each apple slice was homogenized in a blender with 30 ml of H<sub>2</sub>O for 3 minutes in preparation for the determination.

Sucrose diffusion into apple tissue. Diffusion of sucrose into the apple slices was measured by the analysis of soluble solids at different depths of apple slices. Fresh rectangular apple slices (1.5 x 1.5 x 3.0 cm) were placed in 50 ml of 0.7, 1.5, 2.0 Molar sucrose solution and in dry sucrose for 15 hours at room temperature (70° F). The slices were removed from the syrup and frozen quickly in a blast-type freezer at -70° F for 20 minutes and then dehydrated in a freeze dehydration unit. A layer, approximately 1 mm thick, was sectioned from each surface of dehydrated apple slice and powdered for the analysis. Moving inward, second, third, and fourth layers were sectioned (each 1 mm thickness) and prepared in the same manner. In each case 200 mg of the powdered sample was dissolved in 2 ml of distilled water for 15 minutes and then the soluble solids were measured by a Zeiss Opton refractometer.

C<sup>14</sup> labeled sucrose. C<sup>14</sup> labeled sucrose was obtained from New England Nuclear Corporation and Calbiochem. Apple slices were immersed in 50 ml of 0.7, 1.5, and 2.0 Molar sucrose solutions which had 10 µc activity

of C<sup>14</sup> sucrose in each solution. After 20 hour immersion, the slices were removed and frozen quickly in a blast-type freezer at -70<sup>o</sup> F for 20 minutes and then subjected to freeze dehydration. The thin sections (1-2 mm in thickness) were made from the dehydrated apples. The sections were exposed to Kodak No Screen X-Ray film (25.4 x 30.5 cm) for 7-10 days and then the film was developed with Kodak X-Ray developer (Ruck, 1966).

#### Statistical analyses

In order to evaluate dehydration treatments, statistical analyses of the data of flavor compounds were conducted by determination of the least significant difference (LSD) described by Li (1965) and multiple range and multiple F-tests according to Duncan (1955).

## RESULTS AND DISCUSSION

### Dehydrated Fruit

The appearance of dehydrated apple, apricot, and peach slices after dehydration by different processes were changed (Figures 5-12). Conventional hot-air dehydrated fruits were shrunk and hardened. Another obvious difference of conventional hot-air dehydrated fruits from other dehydrated fruits was the color change. Apricots were more susceptible to the browning reaction than other fruits. Dehydro-frozen fruits were slightly shrunk but the color change was minimum compared to the fruits dehydrated by conventional hot-air dehydration. Kuprianoff (1958), in a discussion of bound water in foods, pointed out that the presence of water in the food is very important for properties such as structure or turgidity and even nutritive value and taste. Dehydration may affect these important properties considerably and lead to irreversible changes. Burke and Decareau (1964) mentioned that products which are high in sugars, salts, acids, and flavor components represent more sensitive materials than the products which are low in these components and require greater care in freezing and drying in order to retain these desired organoleptic properties.

There was almost no difference in color and shape between the fresh fruits and the freeze dehydrated fruits. Freeze dehydrated fruits were less shiny compared to the fresh fruits, because the light reflection was affected

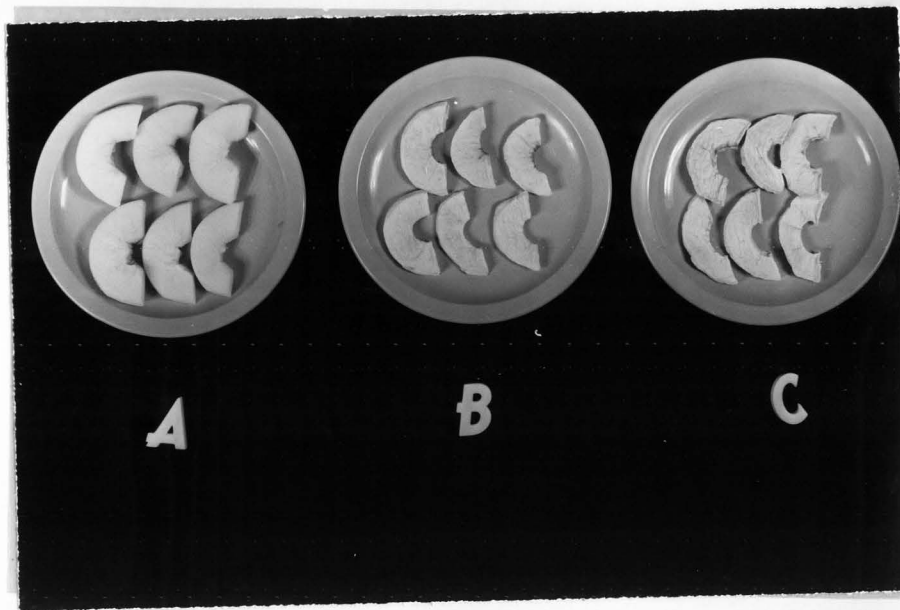


Figure 5. Fresh and dehydrated apple slices.  
A = fresh, B = dehydro-frozen, C = conventional hot-air dehydrated

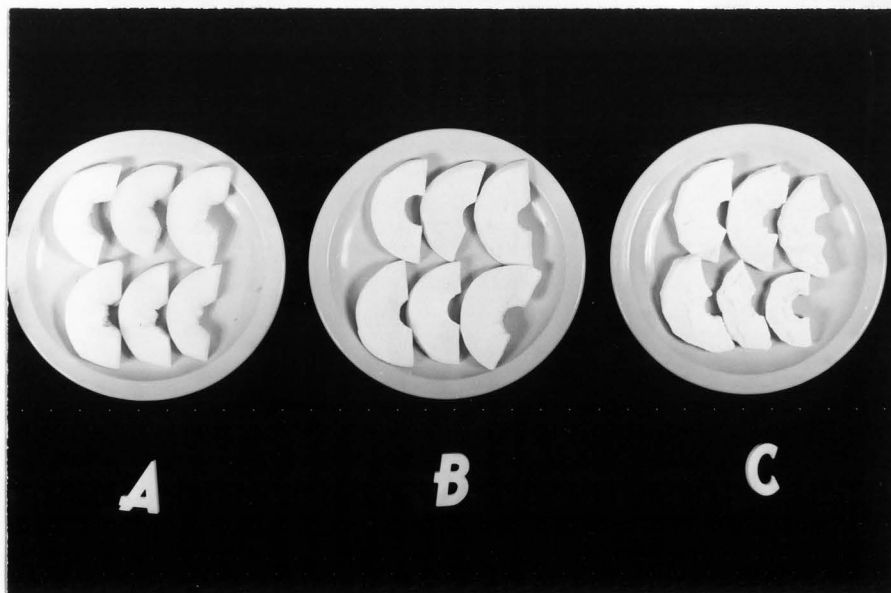


Figure 6. Fresh and dehydrated apple slices.  
A = fresh, B = freeze dehydrated, C = cryogenic frozen and freeze dehydrated



Figure 7. Fresh and dehydrated apricot slices.  
1 = fresh, 2 = dehydro-frozen, 3 = conventional hot-air dehydrated



Figure 8. Fresh and dehydrated apricot slices.  
1 = fresh, 2 = freeze dehydrated

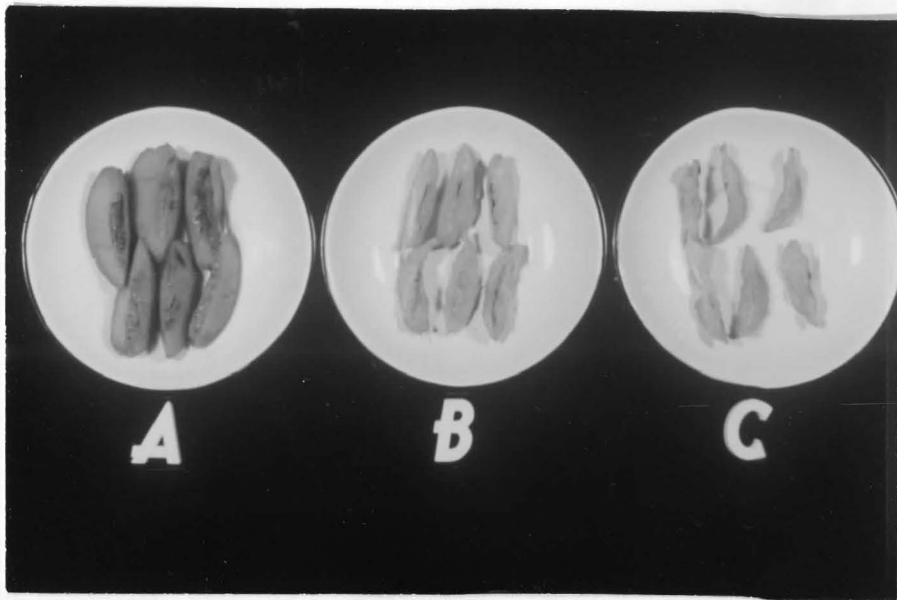


Figure 9. Fresh and dehydrated peach slices.  
A = fresh, B = dehydro-frozen,  
C = conventional hot-air dehydrated



Figure 10. Fresh and dehydrated peach slices.  
A = fresh, B = freeze dehydrated,  
C = cryogenic frozen and freeze dehydrated



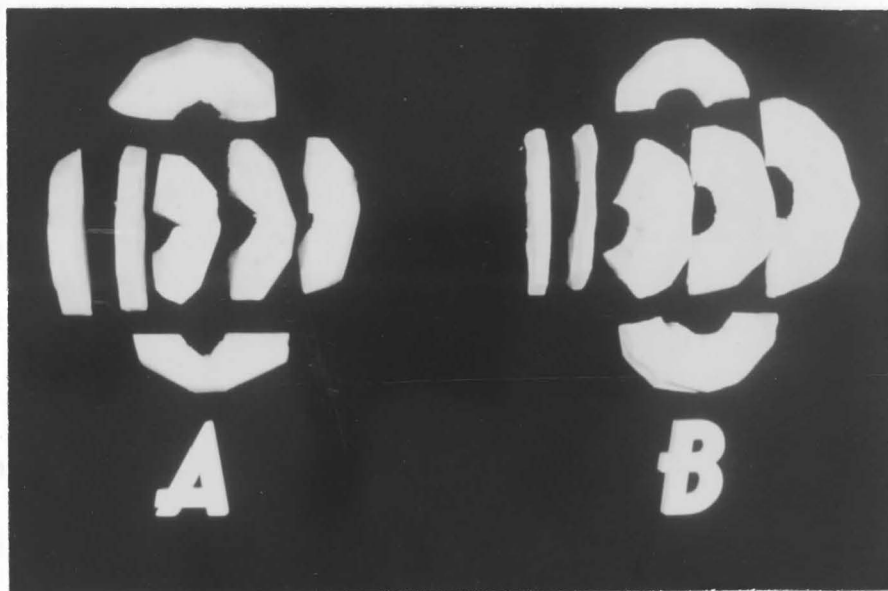


Figure 11. Fresh and dehydrated apple slices.  
A = fresh, B = sucrose treated and freeze dehydrated



Figure 12. Fresh and dehydrated peach slices.  
A = fresh, B = sucrose treated and freeze dehydrated

by moisture in the surface of fruits. Cryogenic freezing influenced the surface color of the freeze dehydrated fruits (Figure 10-C). Woodroof (1938) observed certain objectionable alternations in color when fruits were frozen quickly at very low temperatures. The same results were observed in apricots and peaches. It was assumed that a certain decolorization may be caused by liquid nitrogen itself or cryogenic freezing at extremely low temperature may have caused certain structural changes to the cellular structure of fruits and subsequently produced a different type of light reflection.

Since the freeze dehydration method is relatively new in the food dehydration field and its main objective is toward lowering moisture content without appreciable changes in the quality of the product, it may be necessary here to discuss briefly the mechanism of this mode of dehydration.

Harper and Tappel (1957) and Burke and Decareau (1964) reviewed the literatures of freeze dehydration processes. Meryman (as cited by Burke and Decareau, 1964) described the process of freeze dehydration as the solid slabs receiving heat from some external source, the heat being conducted to an ice-dried layer interface, and the water vapor subliming from the interface, moving through the dried layer, and finally escaping to the chamber. Since the material is frozen there is no migration of moisture (this is the principal distinct difference between freeze dehydration and conventional dehydration). It is common to visualize a very sharp break between the dehydrated layer and the ice layer. Food products being dehydrated in bulk will of necessity have an appreciable thickness and the resistance of the

dehydrated material to flow of vapor is important in determining the dehydration rate. Harper and Tappel (1957) pictured the movement of water-vapor from the ice interface to the exterior of the foods. They discussed two mechanisms - hydrodynamic flow and gaseous diffusion - in the freeze dehydration process. If the pressure in the vacuum chamber is lower than the vapor pressure on the ice, there is a gradient in the total pressure, and hydrodynamic flow will occur as a result of pressure gradient. If some residual air is in the vacuum chamber, gaseous diffusion will occur as a result of the concentration gradient.

Heat is transferred to inside the product by thermal conduction from the outer surface of the product. Harper and Tappel (1957) proposed that if dehydration takes place from the surface to which the heat is applied, the heat must be conducted through a layer of dehydrated material. The thermal conductivity of the ice layer is several fold greater than the conductivity of the dehydrated layer (Burke and Decareau, 1964). Because of the low thermal conductivities resulting from the porous structure of dehydrated material, the heat input at the maximum permissible surface temperature will ordinarily be far below that necessary to keep the ice surface close to the melting point. The rate of heat flow is proportional to the product of the thermal conductivity of the dehydrated material and the temperature difference between the outer surface and the ice surface. So the only way of increasing the heat flow rate is to raise the surface temperature.

Bradish *et al.* (1947) discussed two stages of dehydration. At the

first stage, the ice phase recedes within the material and finally disappears. During this stage, the product will be at a low temperature. The second stage of dehydration takes place as the temperature of the product is raised to drive off the water.

### Flavor Compounds

Chemists have long taken an active interest in the flavor problem, because of the general recognition that odor and taste are chemical senses, responsive to the chemical composition of the substances we smell and taste. Food flavor primarily depends upon reactions of olfactory receptors to chemical stimuli. Volatile compounds, soluble solids, acids, sugars and tannins of fruit influence taste and flavor. Farber (1949) reported that the determination of volatile reducing substances may help in evaluating the odor intensity of food, and Leonard *et al.* (1953) observed that the quantities of chemical constituents, such as soluble solids, acids, volatile reducing substances, tannins, and ascorbic acid are correlated with flavor scores in canned peaches. Many of these compounds are susceptible to heat.

### Apples

Data presented in Table 1 illustrate some of the changes of the flavor components of apple slices during the dehydration processes. The acid content of conventional hot-air dehydrated and dehydro-frozen apples decreased while soluble solids, reducing sugars, and total reducing sugars increased compared to the fresh fruits. However, these changes were minimal in freeze

Table 1. Comparison of chemical compounds of dehydrated apple slices<sup>a</sup>

Dehydration method	Acids as malic acid %	Soluble solids %	Reducing sugars %	Total reducing sugars %	Volatile reducing substances $\mu\text{eq}/100\text{ g}$	Volatile carbonyl compounds ppm	Tannins as tannic acid mg/100 g	Ascorbic acid mg/100 g
Fresh (control)	0.57	12.8	6.41	12.33	360	3.2	148	7.2
Conventional hot-air dehydrated	0.49	14.4	7.34	12.98	60	0.8	42	0.5
Dehydro-frozen	0.49	13.7	6.55	12.55	228	2.6	120	3.1
Freeze dehydrated (conventional frozen)	0.55	13.1	6.57	12.68	188	2.3	123	3.6
LSD: 0.05	0.15	0.4	1.10	0.47	54	0.4	15	0.8
0.01	0.22	0.5	1.58	0.68	78	0.6	22	1.1

<sup>a</sup>Averages of four replicates. All values converted to fresh fruit basis.

dehydrated apples. The changes in concentrations of these components were proportional to the heat applied. Heat decomposed acids and hydrolyzed high molecular weight carbohydrates to lower compounds, and subsequently decreased acidity, increased soluble solids and reducing sugars. Significant differences in volatile compounds were observed among dehydrated apples. Conventional hot-air dehydration was significantly different from the other treatments in volatile reducing substances and in volatile carbonyl compounds, whereas there was no significant difference between dehydro-freezing and freeze dehydration. Freeze dehydration appeared to affect volatile compounds of apples more than dehydro-freezing. It may be reasoned that high vacuum applied for a long time during the freeze dehydration process may have removed some low molecular weight volatile compounds.

Tannin compounds decreased as the heat treatment increased. This may be due to high temperature which may have accelerated the oxidation of tannins. Tannins are assumed to play an important part in determining the quality of apples. The relative amounts of sugars, acids, and astringents are probably one of the most important factors in determining the quality of apples and apple products. Sometimes they are referred to as contributing "body" and their presence results in a "heavier" flavor. They are easily hydrolyzed by acids and heat into a variety of products (Russell, 1935). Equally important with the effect of tannins on flavor is their influence on the color of apple products. The chemical reactions of color changes involve oxidation of tannins in the presence of the oxidase enzyme present in the flesh. This

enzymatic oxidation can be greatly retarded by the freeze dehydration process which removes the moisture from fruit to less than 2 per cent; it was observed that when this product was stored at 45<sup>0</sup> F for one and one-half years it did not change color.

Ascorbic acid losses increased with the severity of heat treatment. Freeze dehydrated apples retained a high per cent of ascorbic acid compared to dehydro-frozen and conventional hot-air dehydrated apples. The results of heat effect on ascorbic acid changes concur with the findings of Mrak and Phaff (1947).

### Apricots

There were no significant differences in acidity between fresh and dehydrated apricots with the exception of conventional hot-air dehydrated apricots (Table 2). High temperatures may have decomposed (or evaporated) the low molecular acids. Soluble solids, reducing sugars, and total reducing sugars were high in the conventional hot-air dehydrated apricots. These increases were proportional to the heat applied. Some of these changes may be explained by the hydrolysis of the high molecular to low molecular compounds under the conditions of high temperature and low pH of fruits. On the other hand, these changes were imperceptible in freeze dehydrated apricots.

The chemistry of fruit flavors has been reviewed by Kirchner (1949) but no detailed information on the volatile constituents of dehydrated apricots is available. Farber (1949) reported on the chemical evaluation of odor

Table 2. Comparison of chemical compounds of dehydrated apricot slices<sup>a</sup>

Dehydration method	Acids as malic acid %	Soluble solids %	Reducing sugars %	Total reducing sugars %	Volatile reducing substances $\mu$ eq/100 g	Volatile carbonyl compounds ppm	Tannins as tannic acid mg/100 g	Ascorbic acid mg/100 g
Fresh (control)	1.21	10.0	1.28	3.27	902	9.9	93	7.7
Conventional hot-air dehydrated	0.99	12.4	1.51	3.31	665	3.3	36	3.4
Dehydro-frozen	1.15	11.6	1.34	3.20	798	7.6	57	6.4
Freeze dehydrated (conventional frozen)	1.14	11.0	1.31	3.29	822	9.2	78	6.8
LSD:	0.05	0.08	0.04	0.05	40	0.4	6	0.8
	0.01	0.11	0.06	0.07	57	0.6	8	1.1

<sup>a</sup>Averages of four replicates. All values converted to fresh fruit basis.



intensity by the permanganate titration method. Luh (1961) reported the volatile reducing compounds of canned apricots. Freeze dehydrated apricots were the highest among the dehydrated apricots in volatile reducing substances and in volatile carbonyl compounds, whereas conventional hot-air dehydrated apricots were the lowest. In contrast to the apples, freeze dehydrated apricots retained more volatile compounds than dehydro-frozen apricots and the percentage of volatile compounds retained in dehydrated apricots was higher than those of dehydrated apples. This may be attributed for the differences of volatile components and certain physiological properties between apricot and apple fruits.

Tannin compounds also were affected by heat. Approximately 60 per cent of the tannins decomposed during the conventional hot-air dehydration process. Perhaps this resulted because high temperature accelerated the oxidation of tannins (Russell, 1935) and they may also be involved in color reactions. Losses of ascorbic acid during the dehydration processes showed the same patterns which were evidenced in apples.

### Peaches

There were no significant differences in acids among fresh, dehydro-frozen, and freeze dehydrated peaches (Table 3). Soluble solids, reducing sugars, and total reducing sugars were higher in the dehydrated peaches. Conventional hot-air dehydrated peaches contained the most reducing and total reducing sugars. The increase of these components was proportional to the heat treatment applied and thus caused hydrolysis of non-reducing sugars.

Table 3. Comparison of chemical compounds of dehydrated peach slices<sup>a</sup>

Dehydration method	Acids as malic acid %	Soluble solids %	Reducing sugars %	Total reducing sugars %	Volatile reducing substances $\mu\text{eq}/100\text{ g}$	Volatile carbonyl compounds ppm	Tannins as tannic acid mg/100 g	Ascorbic acid mg/100 g
Fresh (control)	0.56	11.0	1.81	3.85	980	6.6	140	9.0
Conventional hot-air dehydrated	0.53	14.2	3.46	5.57	380	2.9	44	4.4
Dehydro-frozen	0.54	11.5	2.03	3.43	660	4.8	79	6.0
Freeze dehydrated (conventional frozen)	0.55	11.6	2.46	4.17	924	6.1	103	7.7
LSD: 0.05	0.02	0.5	0.19	0.18	45	0.5	7	0.8
0.01	0.03	0.7	0.27	0.25	65	0.7	10	1.1

<sup>a</sup>Averages of four replicates. All values are calculated on fresh fruit basis.

Freeze dehydrated peaches retained about 90 per cent of their original volatiles whereas dehydro-frozen peaches lost about 30 per cent. More than 65 per cent of the tannins decomposed during the conventional hot-air dehydration process, but freeze dehydrated peaches retained more than 70 per cent of original tannins. Ascorbic acid losses increased also as heat treatment increased. Freeze dehydrated peaches retained a high per cent of ascorbic acid.

Leonard et al. (1953) found that canned peaches of higher volatile reducing substances, ascorbic acid, and tannin contents gave higher flavor scores than those with lower contents of these components. Freeze dehydrated peaches were the highest among the dehydrated peaches in these components that mainly contribute to the peach flavor.

In general, the changes of flavor compounds, during the dehydration of peaches, followed the same pattern as those of apples and apricots. However, the changes in volatile compounds of peaches were more similar to those of apricots than apples. With these results it may be assumed that certain primary flavor compounds of apricots are the same types as those of peaches.

#### Gas-Liquid Chromatography

Most fruit flavor is dependent upon volatile compounds. Odor is an important component of the flavor complex of foods and, therefore, is intimately associated with the quality of the product. Advances in gas

chromatographic instrumentation have made possible to separate very complex mixtures and to detect the components of these mixtures at levels which may approach olfactory thresholds (Wick, 1965). Direct gas chromatographic analysis of head space vapors over foodstuffs has been brought to a high level of sensitivity and general applicability.

The chromatograms of head space vapor of the apple, apricot, and peach dehydrated by different dehydration processes are shown in Figures 13 to 15. No attempt was made to identify the individual peaks (volatiles) in this study. However, differences among the treatments were observed by the comparison of relative peaks in the same fruit. All of the dehydration processes reduced the size of the peaks. Several peaks did not appear in the dehydro-frozen and conventional hot-air dehydrated fruits which occurred in the fresh samples. Freeze dehydrated fruits, however, retained most of the numbers of volatile components as did fresh fruits though in smaller quantities.

It may be concluded that freeze dehydration process retains more of the volatile components in fruits compared with dehydro-freezing and conventional hot-air dehydration processes.

Power and Chestnut (1920) found that odorous components of several varieties of apples consisted essentially of amyl ester of formic, acetic, and a considerable portion of acetaldehyde. They also found traces of methanol, ethanol, and possibly some free formic, acetic, and caproic acids. In a series of articles, Power and Chestnut (1922a) claim to have found geraniol

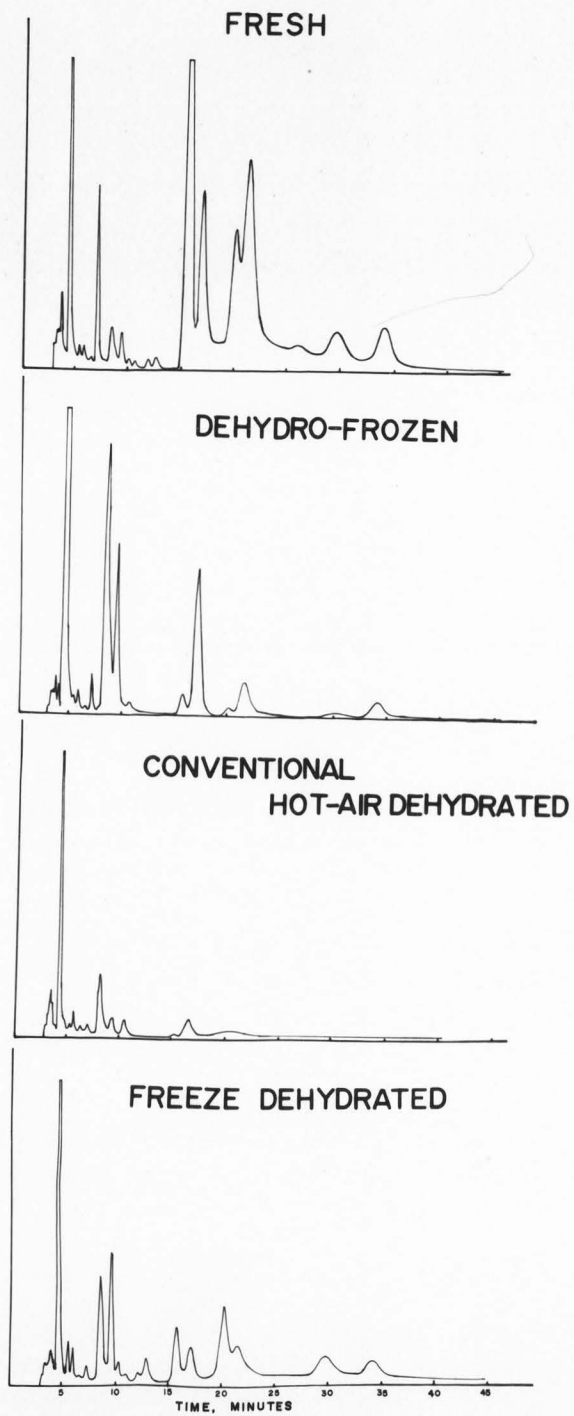


Figure 13. Gas-liquid chromatogram of head-space analysis of fresh and dehydrated apples.

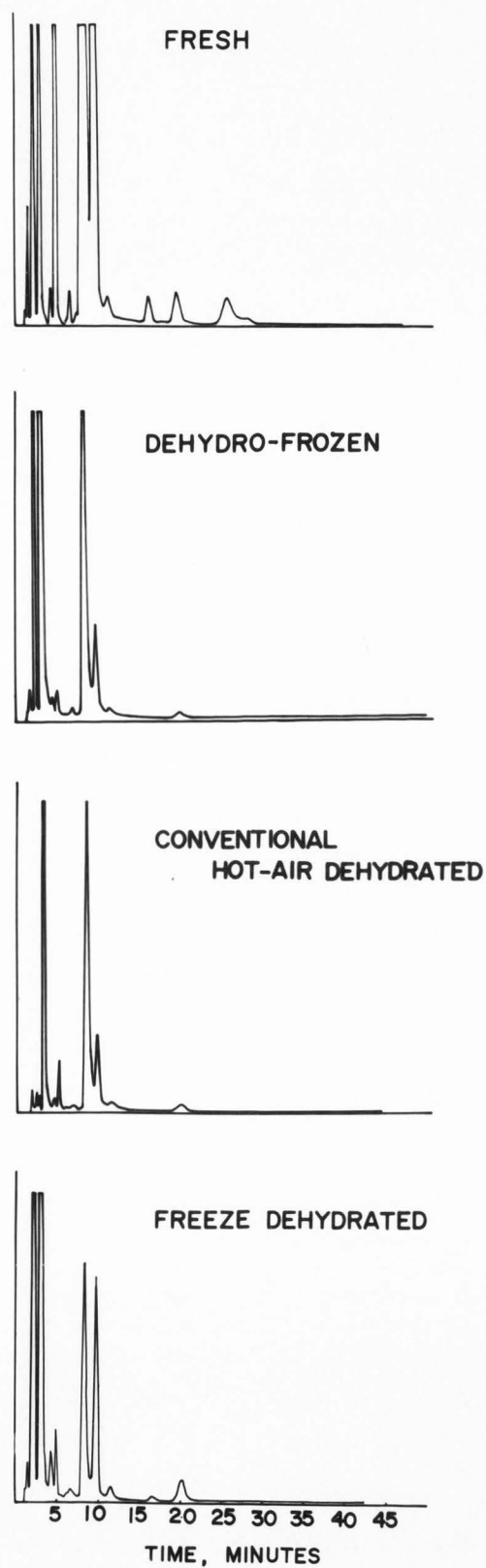


Figure 14. Gas-liquid chromatogram of head-space analysis of fresh and dehydrated apricots.

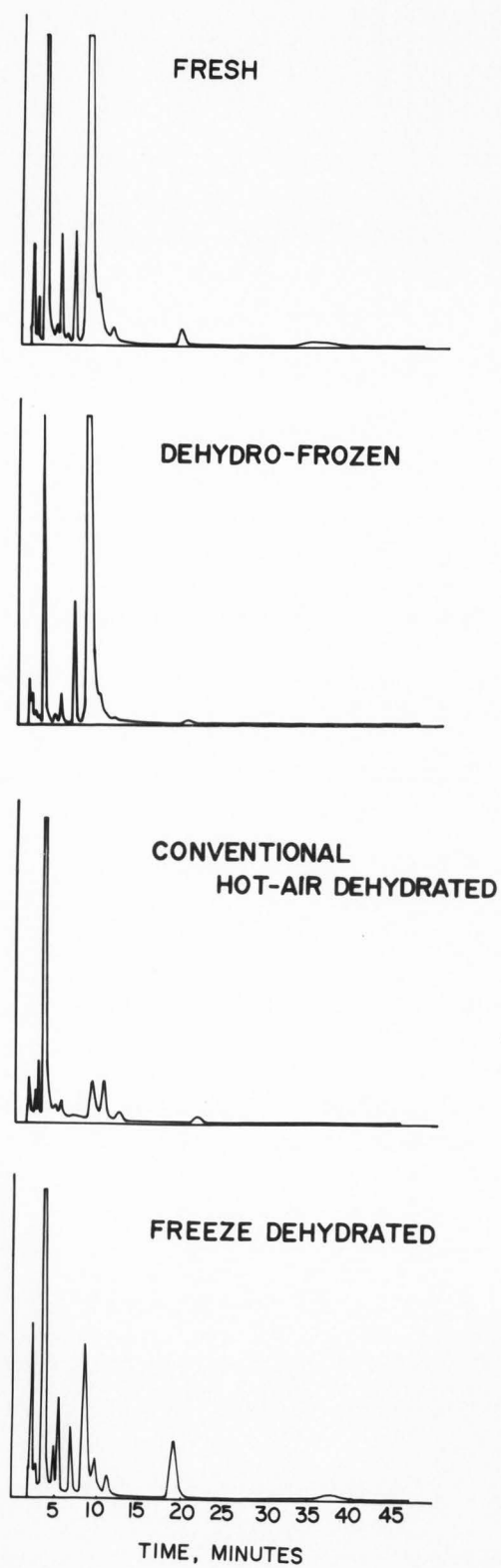


Figure 15. Gas-liquid chromatogram of head-space analysis of fresh and dehydrated peaches.

in ripe apples. Gane (1934) and White (1950) reported ethylene, furfural, three aldehydes, and ten alcohols in McIntosh and Stayman Winesap apples.

Power and Chestnut (1921), Kirchner (1949), and McGlumphy (1951) reported that methyl alcohol, formic, acetic, caprylic, and valeric acids, furfural, acetaldehyde, and cadinene were found in peaches. Power and Chestnut (1922b) postulated that linalyl esters of formic, valeric, and caprylic acids were some of the main components of peach flavor. Recently, Jenning and Tang (1966) isolated terpenes and lactones that are relatively higher boiling compounds in apricots. Jenning and Sevenants (1964) identified benzaldehyde, benzyl-alcohol,  $\gamma$ -caprolactone,  $\gamma$ -octalactone,  $\gamma$ -decalactone, and  $\delta$ -decalactone in Red Globe peaches. These are relatively higher boiling point compounds as compared to those identified from apples. As a result of head space analysis which can only detect the more volatile compounds, a smaller number of peaks were observed in apricots and peaches than were found by Jenning and Tang (1966) and Jenning and Sevenant (1964).

### Color

Measurements of ethanol extractable color for dehydrated apples, apricots, and peaches are shown in Figures 16, 17, and 18, respectively. Conventional hot-air dehydrated fruits were the highest in optical density, while freeze dehydrated fruits were the lowest. Dehydro-frozen fruits were between conventional hot-air dehydrated and freeze dehydrated fruits. The differences in



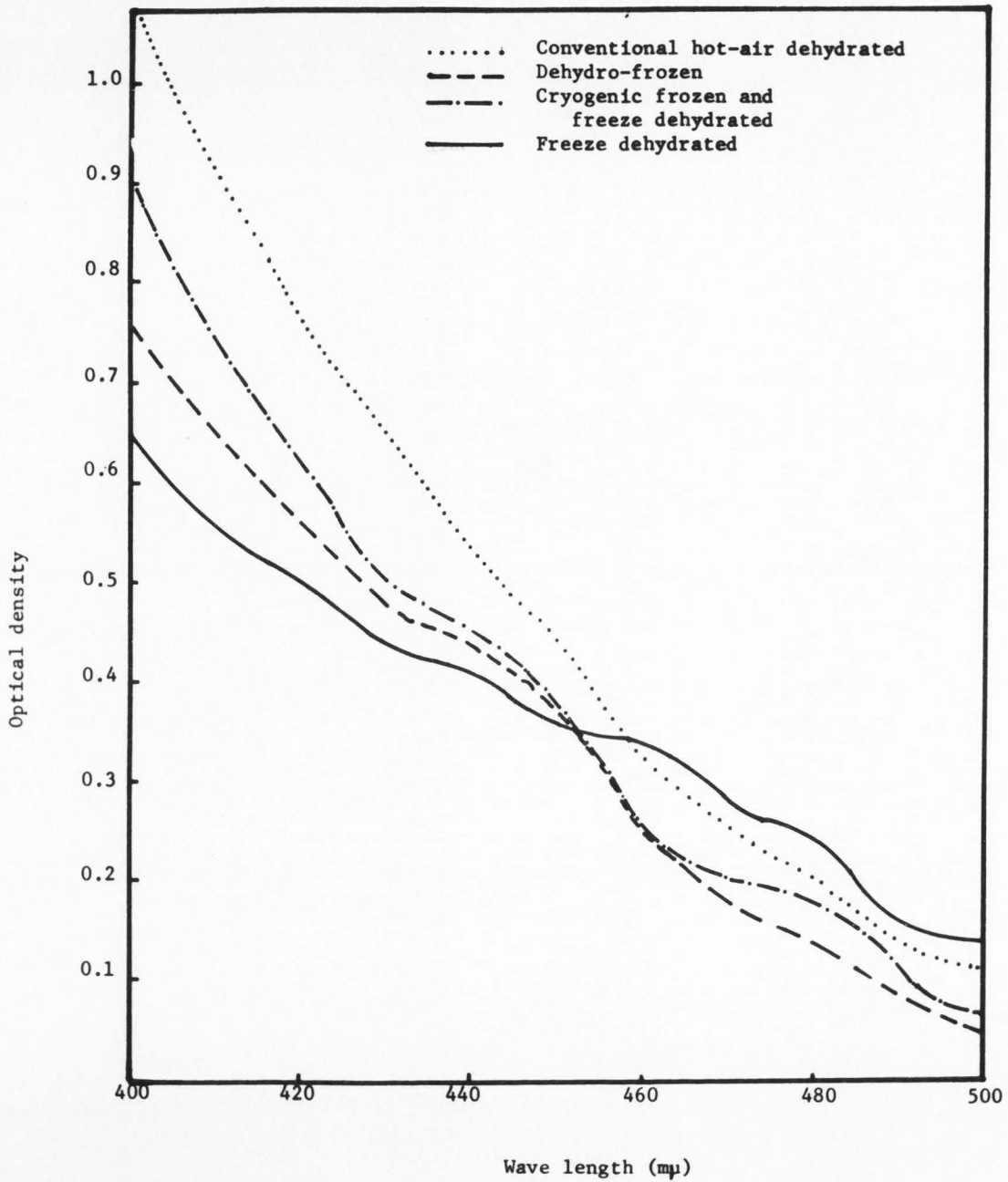


Figure 16. Spectral analysis of ethanol extracts of apples dehydrated by different methods.

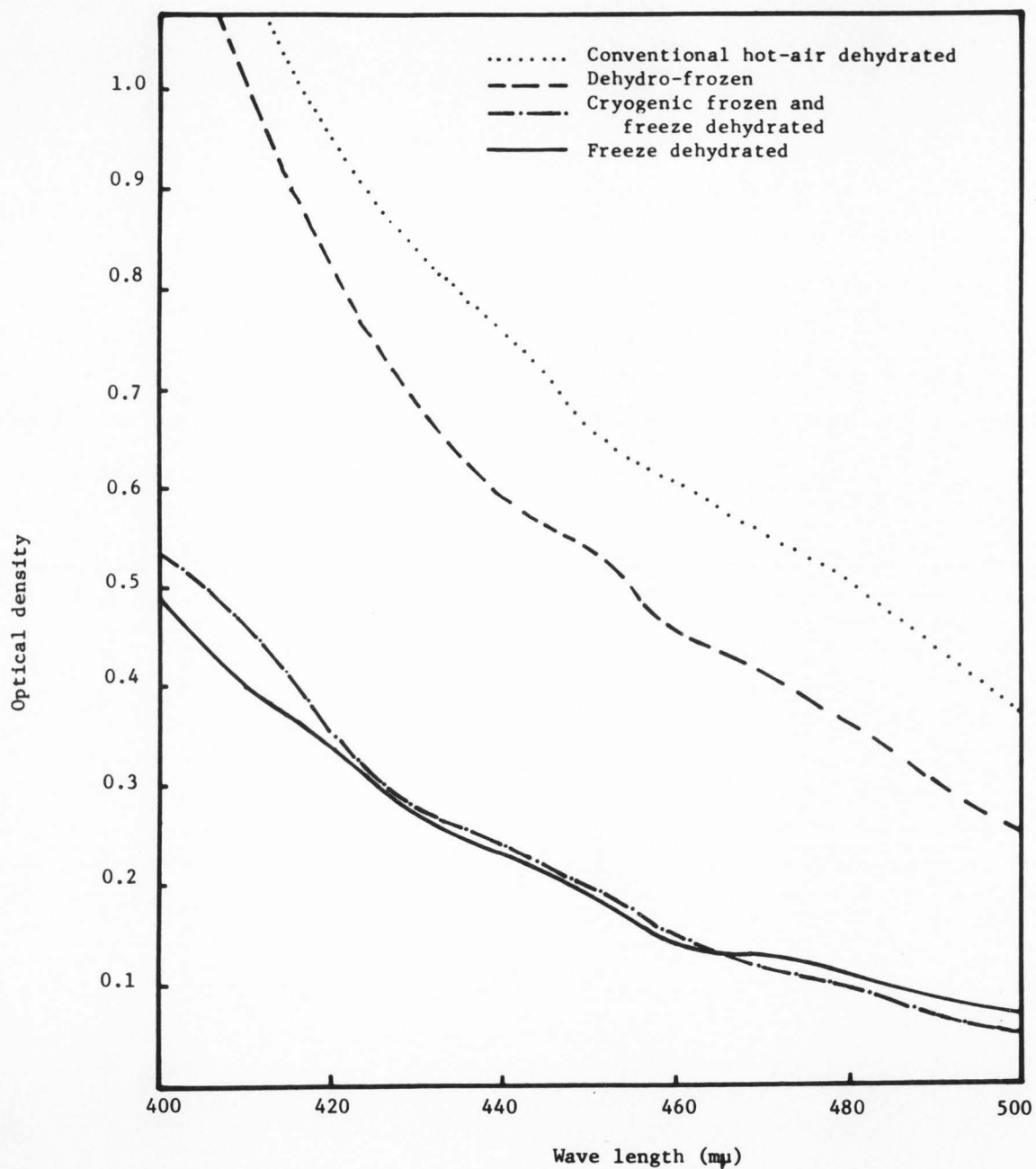


Figure 17. Spectral analysis of ethanol extracts of apricots dehydrated by different methods.

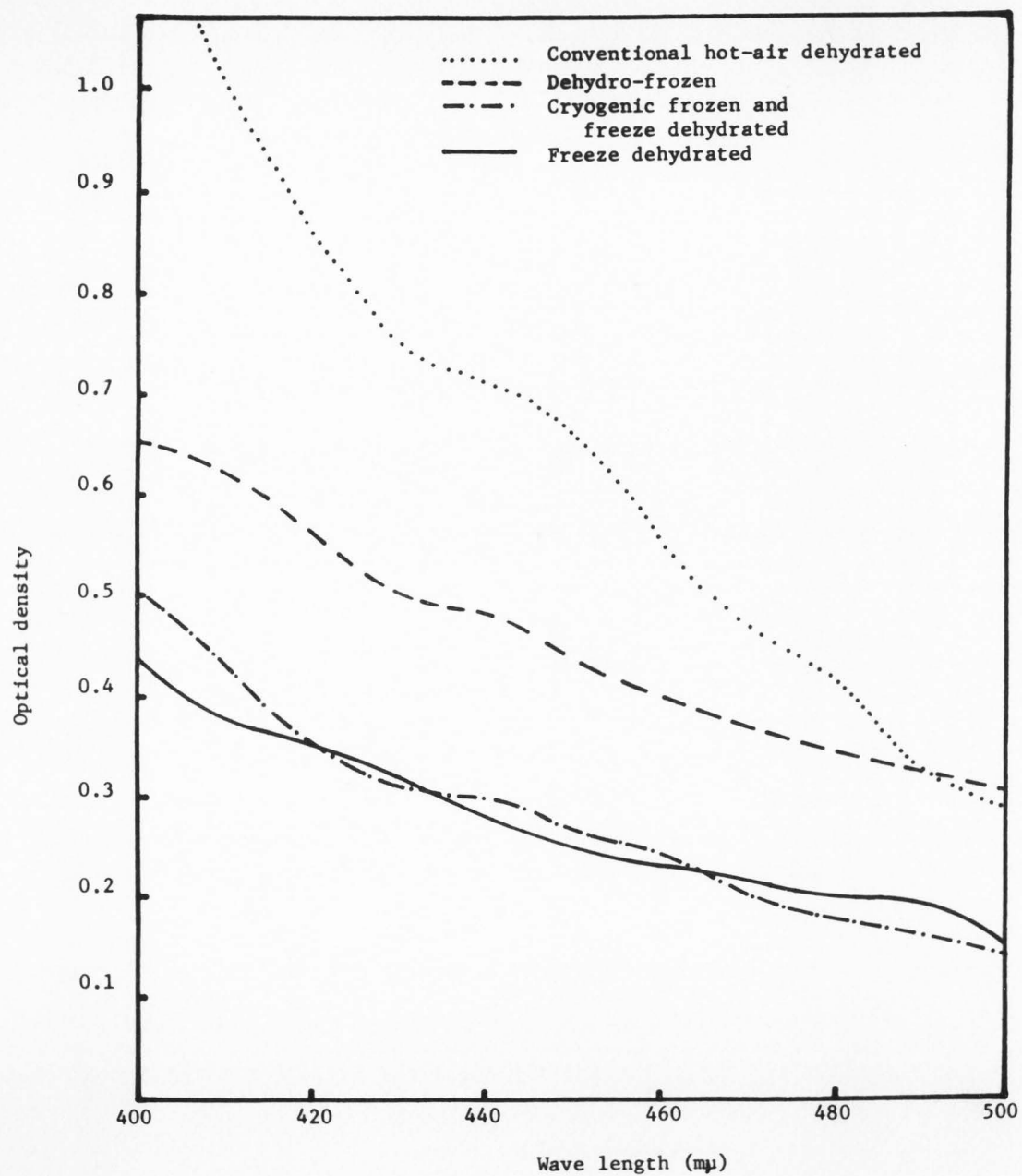


Figure 18. Spectral analysis of ethanol extracts of peaches dehydrated by different methods.

color associated with the three dehydration processes were less significant for apples (Figure 16) than for apricots (Figure 17) and peaches (Figure 18). Apparently the heating during conventional hot-air dehydration and dehydro-freezing accelerated the browning reaction and caused high optical density in apricots and peaches.

Hamburger and Joslyn (1941) determined the absorption spectra of fruit juices at various stages of browning and found that absorption increased rapidly at  $480 \mu\text{m}$  as browning increased. Conventional hot-air dehydrated and dehydro-frozen apricots showed higher optical density in comparison to those of peaches and apples. It may be reasoned that either enzymatic and non-enzymatic browning systems in apricots stimulated more than those of peaches and apples or these systems may be absent in peaches and apples. The samples of all three fruits which were cryogenically frozen in liquid nitrogen and then subjected to freeze dehydration appeared to be discolored as compared to those frozen by regular methods and then freeze dehydrated. However, no significant differences were observed among the optical densities of any of the samples.

Stadtman (1948) stated that the browning reaction in fruit has a relatively high temperature coefficient and the rate of reaction is effected by the time and temperature of dehydration. Stadtman *et al.* (1946b) pointed out the undesirability of using high temperatures to dehydrate apricots to moisture levels below 25 per cent. When fruit of low moisture content is required, the final dehydration should be done at low temperatures ( $77-104^{\circ}\text{F}$ ).

Joslyn and Ponting (1951) pointed out that phenolase activity increases

with increase in temperature until temperatures are high enough to inactivate the enzymes. Ponting and Joslyn (1948) studied the effect of temperature on the rate of oxidation of ascorbic acid by purified apple phenolase in presence of catechol in the range of 41<sup>o</sup> to 149<sup>o</sup> F and found that the rate of oxidation increased with temperature, reaching an optimum at 104<sup>o</sup> F. Fong and Cruess (1929) and El-Tabey and Cruess (1949) reported data on the activity of apricot phenolase and apricot oxidase in tissue at various temperature and pH ranges and found maximum activation at pH 4. They showed that the enzymes in apricots are greatly damaged during drying but are not inactivated. Blanching alone cannot suppress undesirable color changes since non-enzymatic browning reactions can occur while the fruit is being dehydrated. Joslyn and Ponting (1951) explained that all fruits have a sharp pH optimum in enzyme activity. However, apricots appear somewhat unique in being the only fruit tested having a pH of maximum enzyme activity lower than the natural pH; the pH of maximum heat stability of polyphenol oxidase was 3.9 for apricot and 6.2 for apple. This may be one of the reasons that apricots showed higher optical density (increase in color) as compared to those of apples and peaches in this study.

#### Histology and Histochemistry

Edible parts of fruits are largely parenchyma tissues. The parenchyma cells comprise most of the tissue, acting as storage cells for starches and sugars. Cellulose makes up the walls with living protoplasm lying close to

the cell wall with the nucleus contained within. The protoplasm surrounds a vacuole and also contains chloroplasts, leucoplasts, chromoplasts and mitochondria. During processing procedures these materials may be altered in place or may migrate by coagulation, precipitation, or rupture of membranes to other areas of the cell. Weier and Stocking (1949) pointed out that any food processing technique which alters the permeability of the protoplasm, the ability of solutes to be retained within the cell, the elasticity of the cell wall, or the colloidal nature of the cell contents will alter the water retaining power of the cell, and possibly the crispness of the final product. They also stated that the cells of parenchyma tissue are held together by pectic compounds and any processing method which reacts on either the pectic cementing substance or on the cellulose of the cell wall will have an effect on the finished product.

Woodroof (1938), Simpson and Halliday (1941), Reeve and Leinbach (1953), and Sterling (1955) reported the effects of freezing and heat on the fruit and vegetable tissues, but information of the several dehydration effects on the histology and histochemistry of apples, apricots, and peaches is meager.

Histological and histochemical effects of dehydration and freezing on apples, apricots, and peaches are illustrated in Figures 19 to 41. The parenchyma cells were rather intact in the fresh fruits, however, they were shrunk and elongated and a few of them were separated in the conventional hot-air dehydrated fruits (Figures 20, 30, and 37). In several instances it was noticed that cavities were formed by the heat-induced expansion of intercellular gases.

Miladi (1966), on the texture changes in fresh fruit during the processing, stated that the irreversible process occurs when cell reaches 150<sup>0</sup> F. As

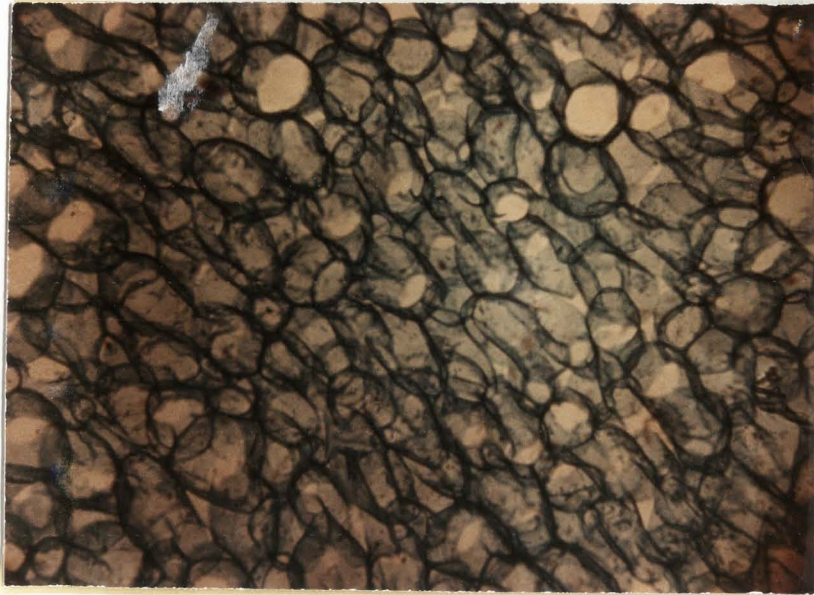


Figure 19. Photomicrograph of parenchyma cells in fresh apple. Note intact cell walls (stained with fast green), X 40.

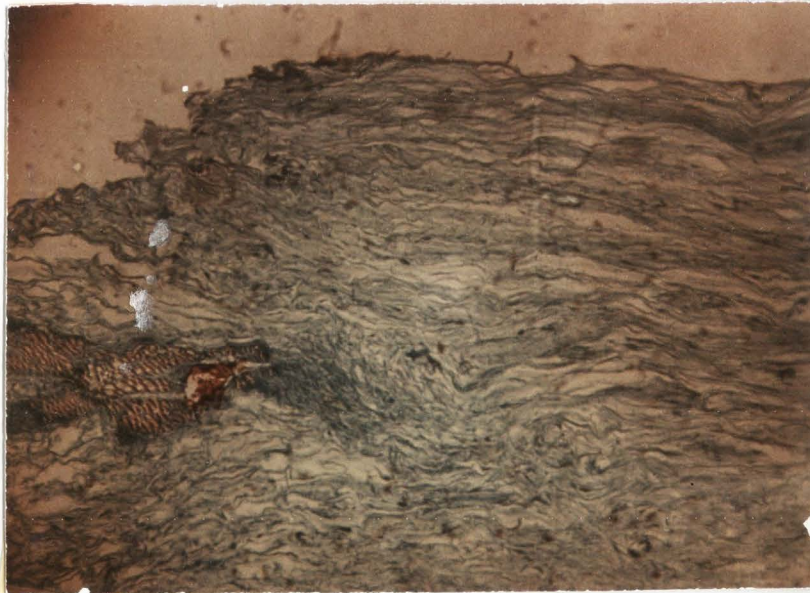


Figure 20. Photomicrograph of parenchyma cells in conventional hot-air dehydrated apple. Note elongated and compressed cell wall and enlarged intercellular air spaces. A vascular bundle shows at left side (stained with safranin-fast green), X 40.

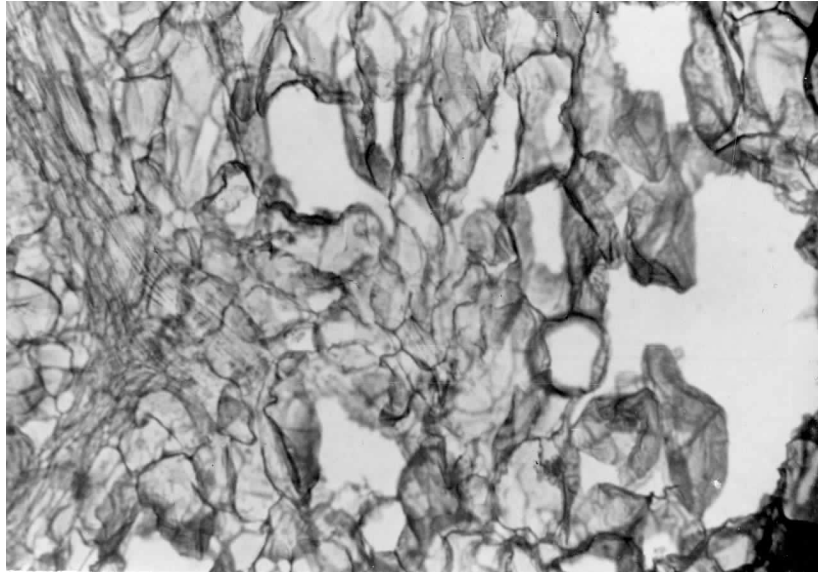


Figure 21. Photomicrograph of parenchyma cells in frozen apple. Note ruptured, folded and crushed cell walls, and large ice crystal spaces. Cells around vascular bundle were not ruptured (stained with fast green), X 40.

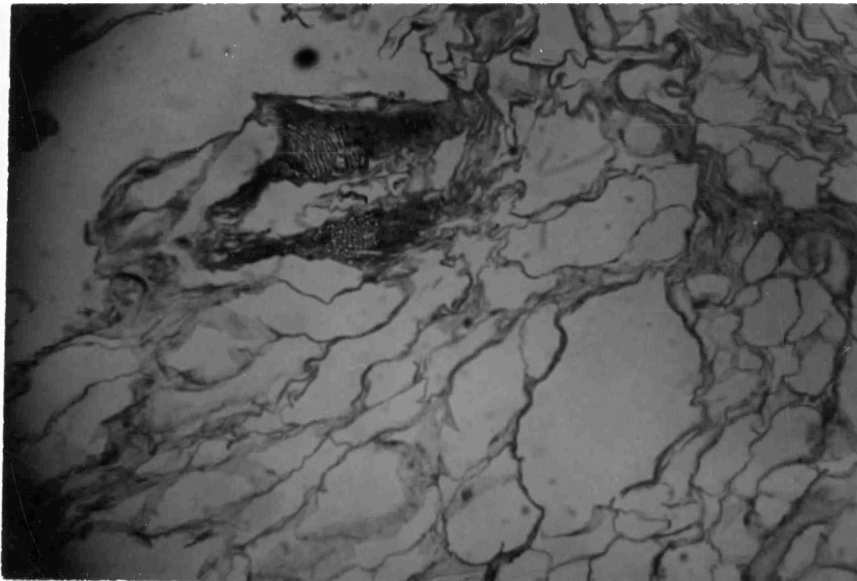
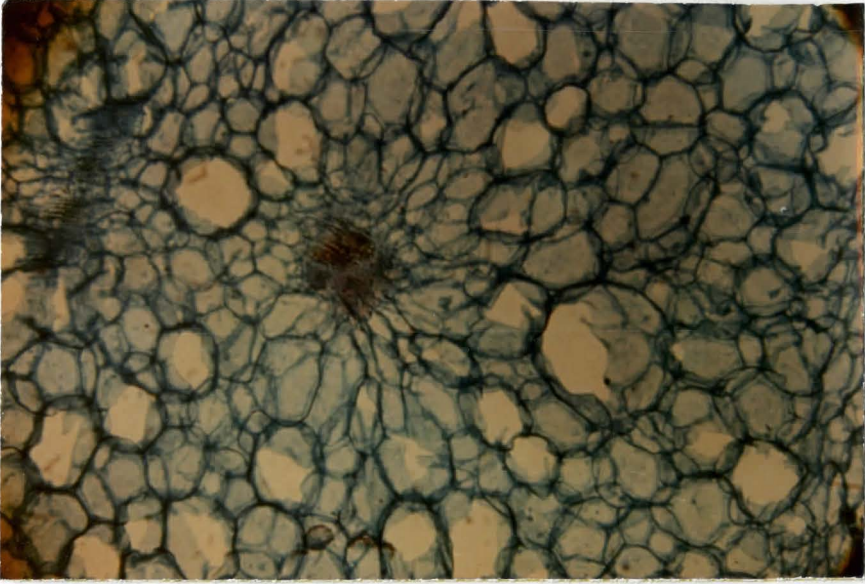


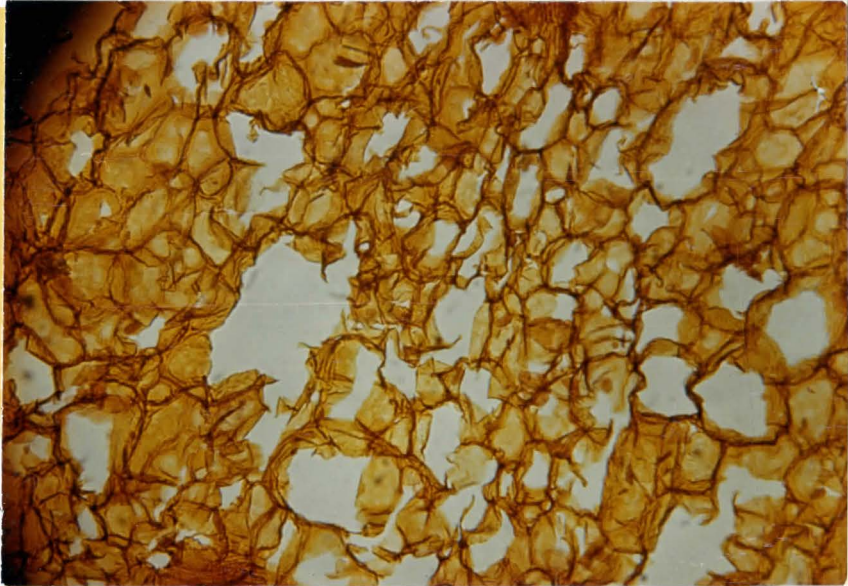
Figure 22. Photomicrograph of parenchyma cells in freeze dehydrated apple. Note ruptured and folded cell walls and large ice crystal spaces (stained with safranin-fast green), X 40.





✓

Figure 23. Photomicrograph of parenchyma cells in cryogenic frozen and freeze dehydrated apple. Note intact cell walls (stained with fast green), X 40.



✓

Figure 24. Photomicrograph of parenchyma cells in freeze dehydrated and rehydrated apple (stained with orange G), X 40.

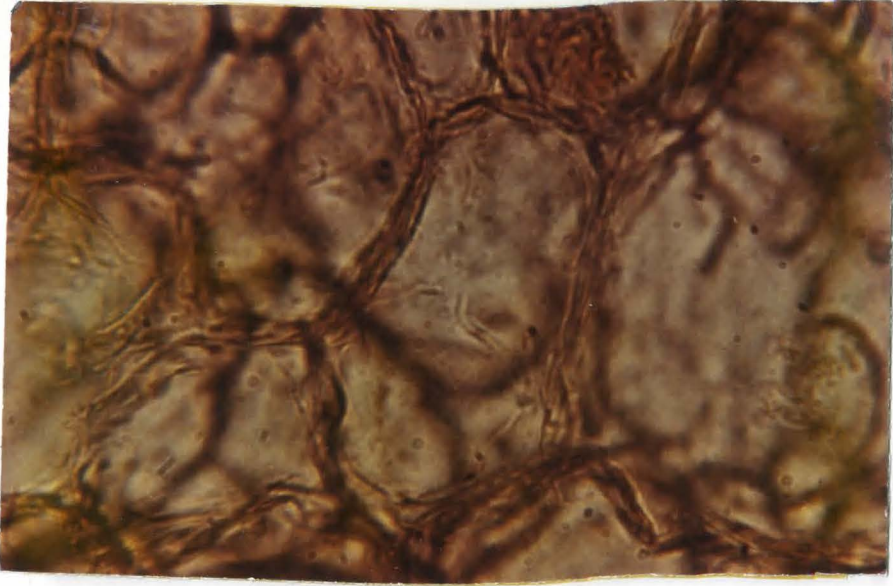


Figure 25. Photomicrograph of cell walls in freeze dehydrated apple. Note an intact cell wall shows that no cell wall components were changed during dehydration process (stained with hydroxyl-amine-ferric chloride), X 150.

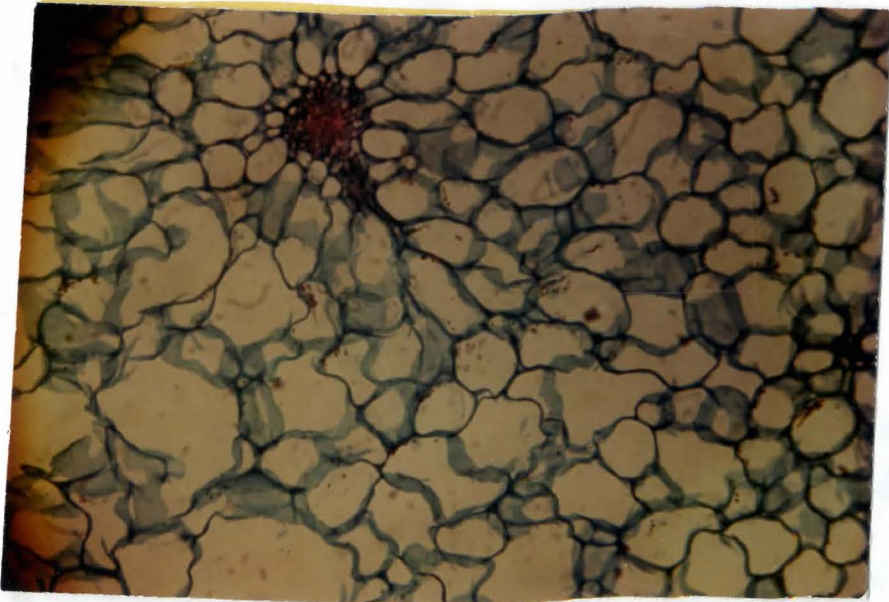


Figure 26. Photomicrograph of parenchyma cells in cryogenic frozen and freeze dehydrated apple. Note starch granules (stained with safranin-fast green and IKI), X 40.

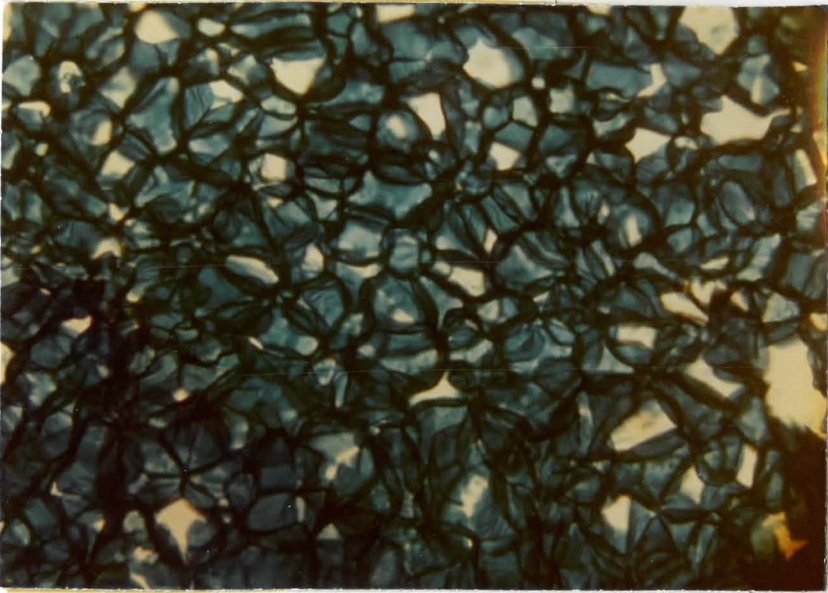


Figure 27. Photomicrograph of parenchyma cells in sucrose treated and frozen apple. Note shrunken cell wall (stained with methylene blue), X 40.



Figure 28. Photomicrograph of parenchyma cells in sucrose treated and freeze dehydrated apple after rehydration. (Stained with anilin blue ), X 40.

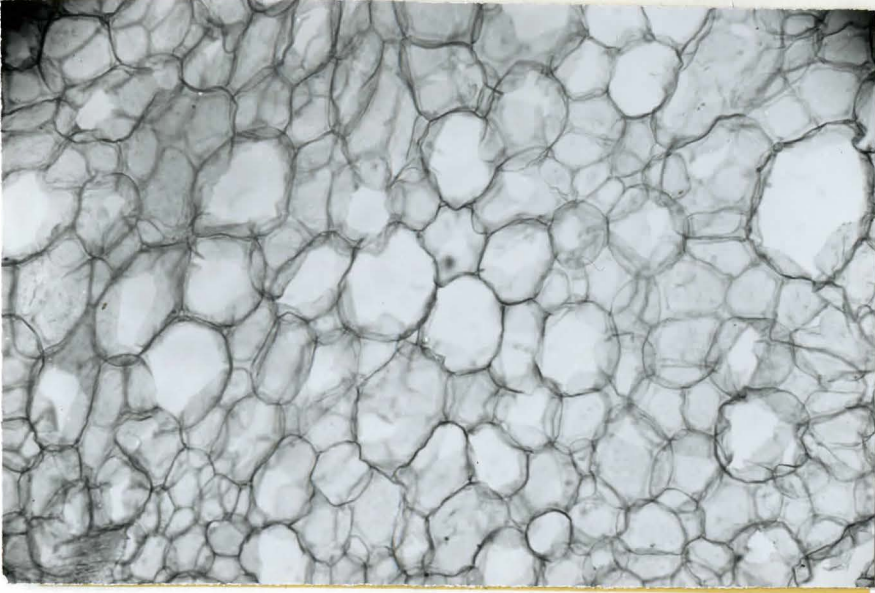


Figure 29. Photomicrograph of parenchyma cells in fresh apricots. Note intact cell walls, X 40.

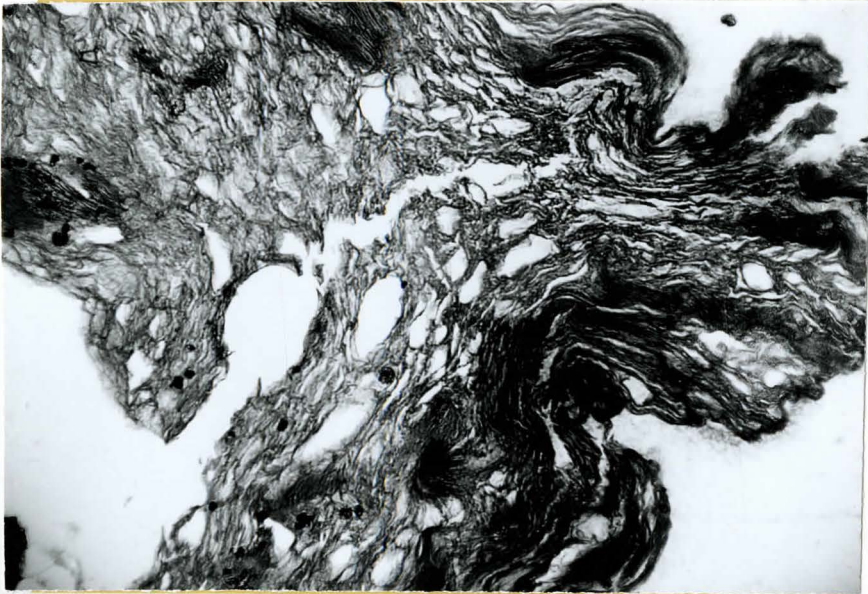


Figure 30. Photomicrograph of parenchyma cells in conventional hot-air dehydrated apricot. Note ruptured, compressed and elongated cell walls and enlarged air spaces, X 40.



Figure 31. Photomicrograph of parenchyma cells in frozen apricot. Note extensively ruptured cell walls, X 40.

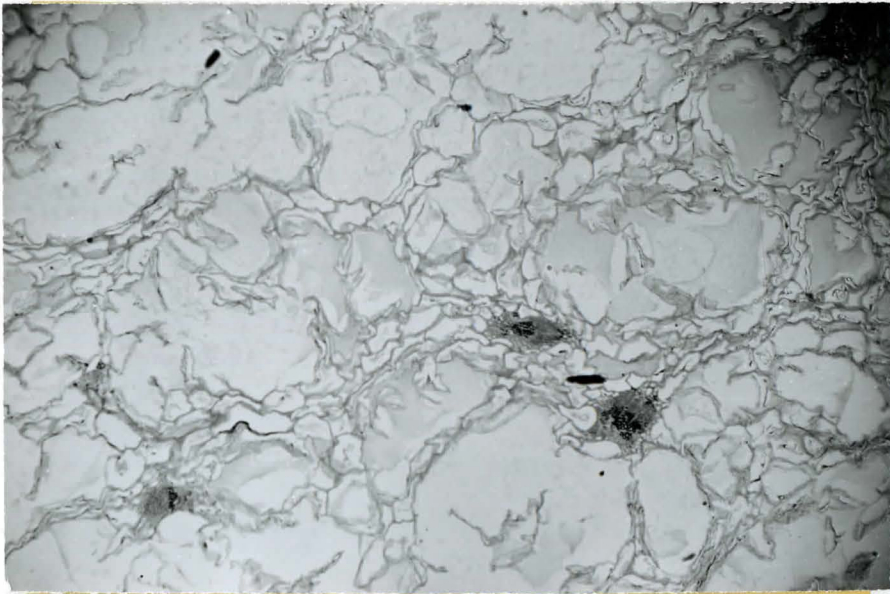


Figure 32. Photomicrograph of parenchyma cells in freeze dehydrated apricot, X 40.

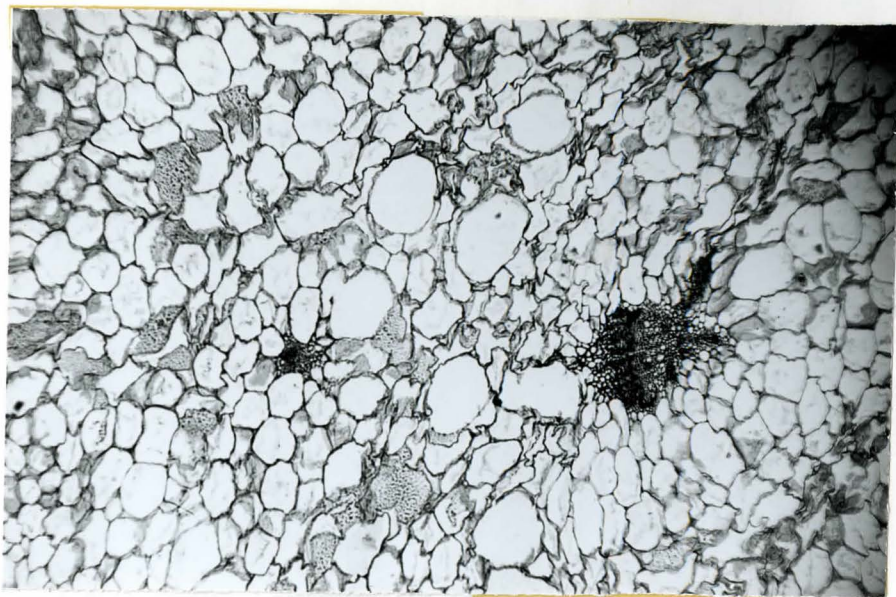


Figure 33. Photomicrograph of parenchyma cells in cryogenic frozen and freeze dehydrated apricot. Note intact cell walls, X 40.

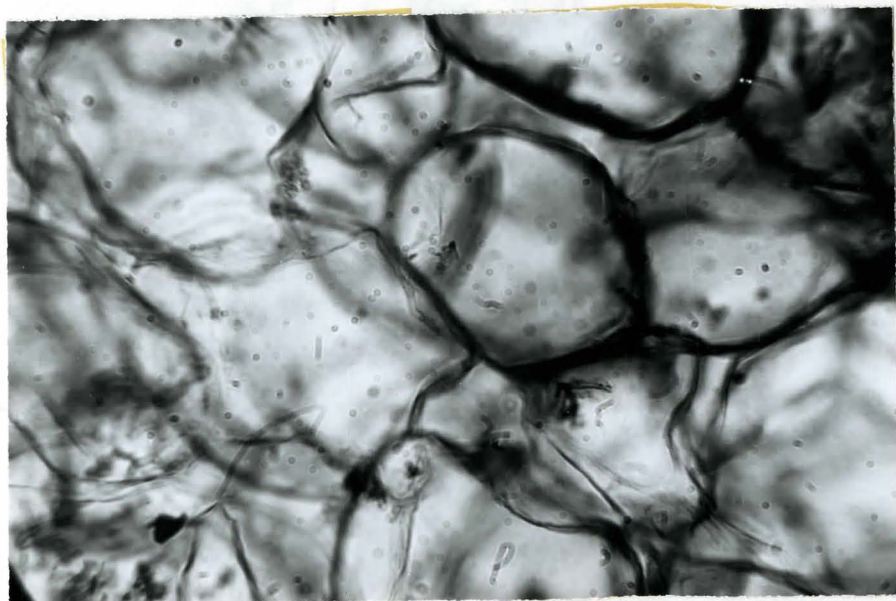


Figure 34. Photomicrograph of parenchyma cells in conventional hot-air dehydrated apricot. Note thinned and broken cell walls, X 150.

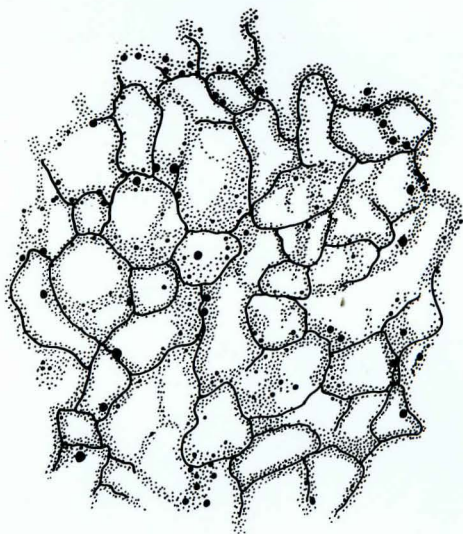


Figure 35. Camera lucida drawing of parenchyma cells in frozen apricot. Note dispersal of carotene pigment and ruptured cell walls, X 60.

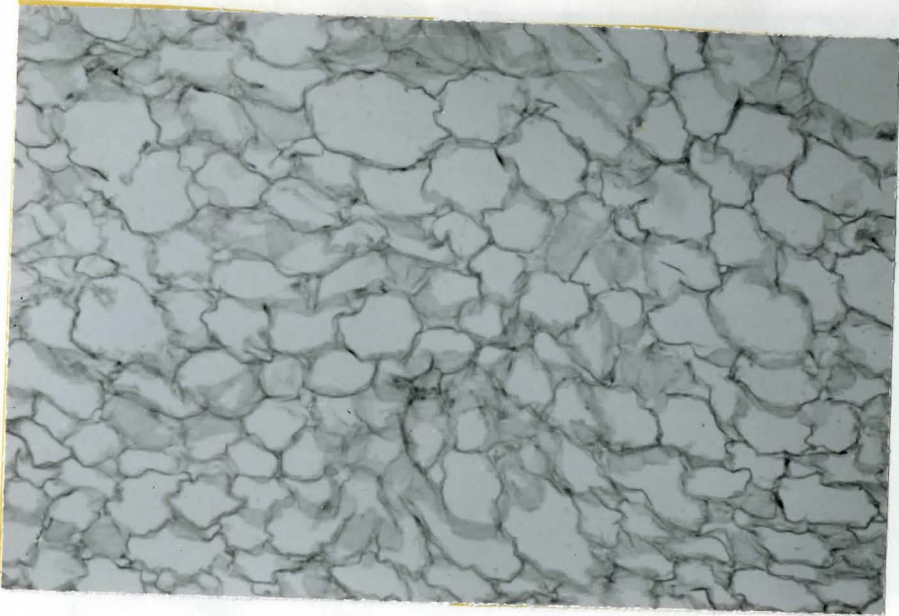


Figure 36. Photomicrograph of parenchyma cells in fresh peach, X 40.

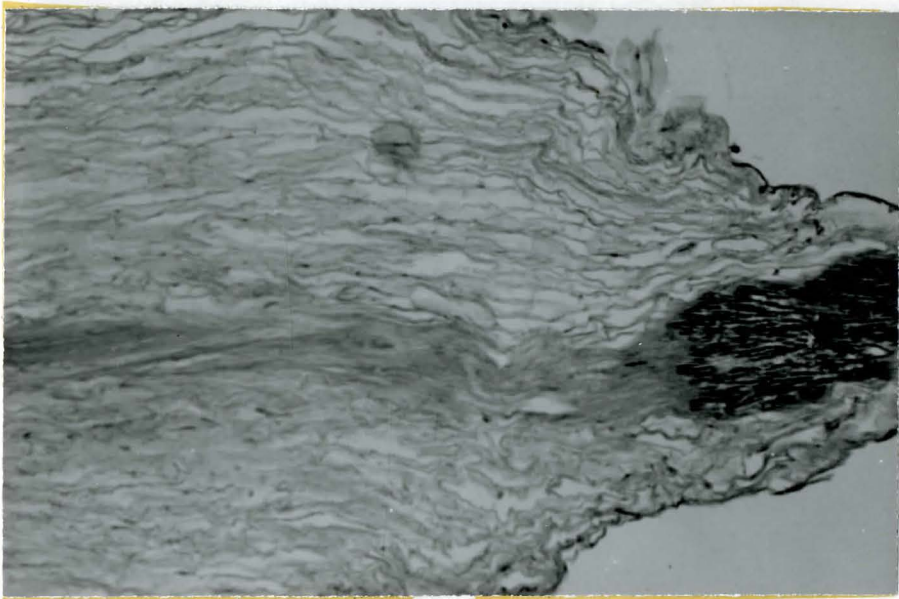


Figure 37. Photomicrograph of parenchyma cells in conventional hot-air dehydrated peach. Note compressed and elongated cell wall. Vascular bundle shows at right side, X 40.



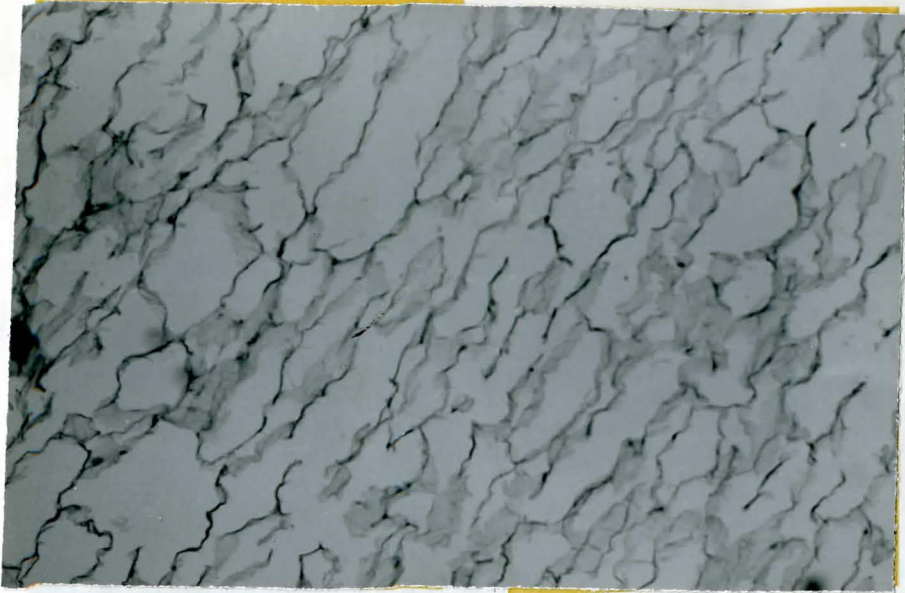


Figure 38. Photomicrograph of parenchyma cells in freeze dehydrated peach. Note ruptured cell walls caused by large ice crystals, X 40.

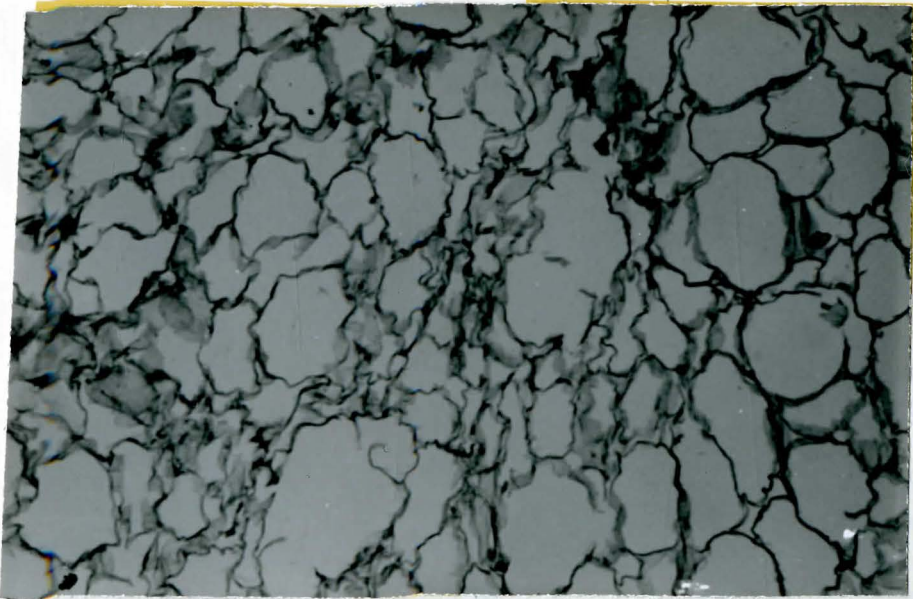


Figure 39. Photomicrograph of parenchyma cells in cryogenic frozen and freeze dehydrated peach. Note the ruptured cell walls at center and lower left corner of picture. Ruptured cell walls were caused by bursting during cryogenic freezing, X 40.



Figure 40. Photomicrograph of parenchyma cells in conventional hot-air dehydrated and rehydrated peach, X 40.

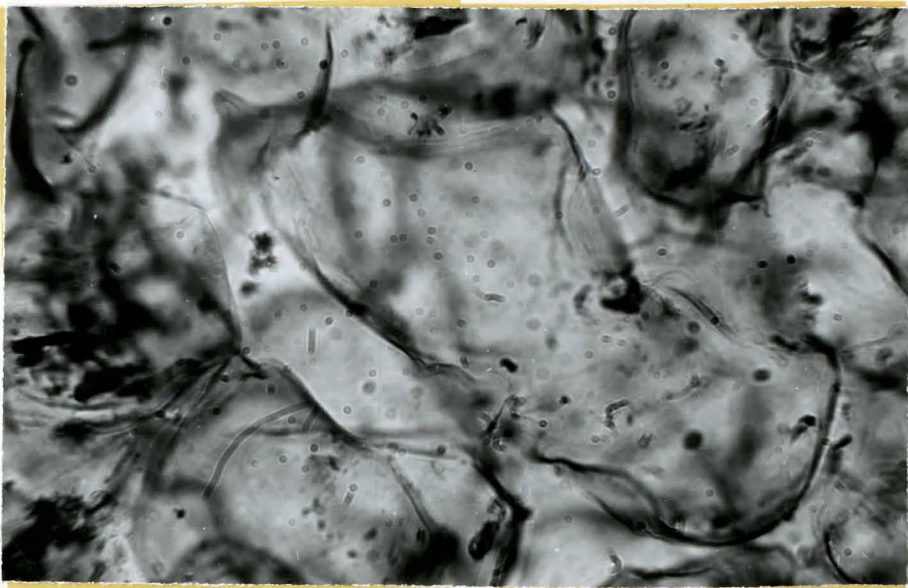


Figure 41. Photomicrograph of parenchyma cell walls in conventional hot-air dehydrated peach after rehydration. Note broken cell wall, X 150.

soon as the selective permeability of the cell is lost, the internal pressure reduced, and the cell sap is extruded into the intercellular spaces. The increased vapor pressure of water at the temperatures encountered during heat processing can result in the filling of intra and inter cellular spaces with gases and the consequent expulsion of fluids ruptures the cell walls and finally consolidation and shrinkage of tissues as the gases decrease in volume during cooling take place.

Sizes of cells, intercellular spaces, and physiological conditions of cell turgor in the fresh tissues are among the factors which influence textural qualities in fresh tissues and which can also affect texture in processed tissue. Weier and Stocking (1949) reported that one of the important changes in the heating of fruit tissues was the expansion and escape of intercellular gases. Reeve and Leinbach (1953) confirmed this in their histological studies of apples. They reported that apples showed considerable internal cracking during steaming as a result of escaping gases.

The expansion and escape of intercellular gases in the fruits during the conventional hot-air dehydration initiated certain cell separations around the cavity. It was also observed that a few cell walls of apricot and peach tissues were ruptured during conventional hot-air dehydration. It appears to be influenced by the result that the trapped moisture inside the tissue vaporized and produced sufficient pressure to rupture the thinner, softer, inner tissue. However, no sign of cell wall breakage was observed in the apples. Sterling (1955) studied the effect of moisture and high temperature

on cell walls in plant tissues and found that the weak cell walls of apples were extensively folded and wrinkled but not broken down by heat and moisture in the cooking process. These results suggest that the histological characteristics of apple tissues are different from those of apricot and peach tissues.

Another pronounced effect of heat was the dissolution of the cell wall components. In most cases the cell walls of the conventional hot-air dehydrated fruits were less thick and less continuous (Figures 34 and 41) than those of the fresh and freeze dehydrated fruits (Figure 25). Simpson and Halliday (1941) and Postlmayr *et al.* (1956) reported chemical and histological changes that occurred during the cooking of carrots, parsnips, and peaches. They observed an increase in pectin and pectic acid and a decrease in protopectin and total pectic substances. Weier and Stocking (1949) stated that the hydrolysis of protopectin was correlated with an increase in softness. In addition, they observed an initiation of cellulose hydrolysis on cooking which subsequently reduced the cell wall thickness and continuity. Chemical analysis of reducing sugars of the conventional hot-air dehydrated fruits supported this result.

Starch granules were observed recurrently in fresh apple tissues (Figure 26). However, they were not found in the fresh and dehydrated apricot and peach tissues. Occasionally a few starch granules were also observed in the freeze dehydrated apples. It may be reasoned that starch granules underwent the gelatinization reaction by heat and were not, therefore, stained with iodine reagent. Weier and Stocking (1949) reported the gelatinization of starch in carrots, potatoes, and parsnips during blanching.

They found that carrot starch had a gelatinization temperature between 104° F and 122° F. The existence of starch granules in the freeze dehydrated apples showed that the low temperature and moisture during the dehydration cycle was not enough to gelatinize the starch granules.

The measurements of cell wall thickness showed also the effect of dehydration on the cell wall components. The average thickness of conventional hot-air dehydrated fruits was thinner than that of freeze dehydrated fruits.

Table 4. Comparison of cell wall thickness<sup>a</sup>

Fruits	Fresh ( $\mu$ )	Convention hot-air dehydration ( $\mu$ )	Freeze dehydration ( $\mu$ )
Apples	1.01	0.58	0.93
Apricots	0.70	0.48	0.61
Peaches	0.93	0.45	0.66

<sup>a</sup>Average of 15 observations

Figures 25, 34, and 41 show photomicrographs of apple, apricot, and peach tissue stained to show pectins after conventional hot-air dehydration. A reduced thickness and continuity of cell walls indicated a probable initiation of pectins and cellulose hydrolysis on dehydration.

Conventional freezing produces large ice crystals and subsequently the thin cell walls are crushed, ruptured, distorted, and separated. Woodroof

(1938) carried out detailed studies on plant tissue in relation to histological changes during freezing procedures and found that rapid freezing reduced the size of ice crystals and the number of ruptured cells in the fruit tissues.

Burke and Decareau (1964) reviewed the literatures in regard to the freezing steps and postulated two important points--the relation between the freezing rate and the structure of the feed materials, and the eutectic temperature--in freeze dehydration. Weier and Stocking (1949) found that, when a rate of freezing is slow, the ice crystals generally form in the intercellular spaces where they may become relatively large. During formation they frequently puncture the thin cell walls and push between the cells reducing the adhesive effect of the middle lamella. Ice crystal formation is dependent on time, temperature, and the material being frozen. If the temperature is high, it takes longer to freeze and the ice crystals tend to be quite large as water moves through the water conducting elements to sites of seeding. Inoculation occurs from external sources through cell membranes that have been punctured by crystals. Kuprianoff (as cited by Burke and Decareau, 1964) proposed that not all freezable water is frozen at about  $-22^{\circ}$  F, but that small amounts of cell juice may remain unfrozen until  $-58^{\circ}$  F to  $-76^{\circ}$  F is reached. He also reported that because of capillary and adsorption forces, even pure water in capillaries may be unfrozen at  $-22^{\circ}$  F or lower. Thus some moisture may remain in liquid state at conventional freezing temperature in this experiment. Woodroof (1938) observed that the size of ice crystals in asparagus could be increased up to 2000 times in volume by slowing up the rate of freezing, as compared to freezing in direct contact with solid carbon

dioxide. These freezing effects are the primary factors of histology of freeze dehydrated products. Burke and Decareau (1964) stated that the freezing step may be as important as--or possibly more important than--the drying step. Figures 21 and 31 showed that fresh frozen fruit tissues are very similar to those of freeze dehydrated fruits. This indicates that most cell and cell wall rupturing in freeze dehydrated fruits occurs during conventional freezing but not during dehydration.

Cytoplasmic materials were dispersed throughout the crushed cell walls. This dispersion separated the carotene pigments in apricots and peaches. As shown in the camera lucida drawing (Figure 35), carotene pigments were dispersed from the chromoplasts. Weier (1944) and Reeve (1943) reported on the lipoidal association in carrots. They stated that vegetable oils may occur free or weakly combined with proteins of the cytoplasm as a lipoprotein complex. This association is readily destroyed by denaturation of the cytoplasm induced by cooking, drying, or freezing, and the lipoidal structure is then freed. The freed oil in dehydrated vegetables may be exposed to air and as a result the carotene pigments may be degraded rapidly by oxidation.

As shown in Figures 23, 33, and 39, cryogenic freezing in liquid nitrogen produced small ice crystals and reduced the number of the ruptured cell walls. The parenchyma cells of freeze dehydrated fruits which were frozen in liquid nitrogen were intact and have similar cellular structures to those of fresh fruits. Hence, it is assumed that the cell damage during the process of freeze dehydration is negligible compared to the other dehydration processes.

The sucrose treatment prior to freeze dehydration had some histological effect on the final products. Figure 27 shows the apple tissues which were dehydrated in sucrose and then frozen by the conventional method. Osmotic action of sucrose removed about 30-40 per cent of the moisture content in the tissue and reduced turgor pressure in the cell, but also rendered the cell wall flexible in certain instances and consequently reduced the cell destruction during the freezing step. Hence, parenchyma cells (Figure 28), after dehydration, retained almost the same structure as those of non-dehydrated apples.

One of the pronounced effects of the sucrose treatment was that sucrose affected the ice crystal formation and produced small ice crystals during the conventional freezing. Luyet and Rapatz (1958) and Nei (1965) studied the mechanisms of ice formation and propagation in some aqueous solutions and described typical forms of crystallization under the various conditions. They observed that high concentrations of sucrose solutions produced evanescent spherulite type ice crystals at lower temperature whereas that of lower concentration produced irregular dendrite type ice crystals at high temperatures.

Another pronounced effect of the sucrose treatment was that sucrose diffused throughout the surface layers of tissues to a certain degree and uniformly covered the surface of the fruit and substantially reduced direct contact with oxygen in the atmosphere. Cox and MacMasters (1942) reported that it is necessary to freeze peaches and other fruits in a sugar solution in order to prevent oxidation and loss of color. Crispness of freeze dehydrated fruits was also enhanced by the sucrose treatment.



### Rehydration

The effects of various dehydration processes on the rehydration rates of the three fruits are illustrated in Figures 42, 43, and 44. The amounts of water absorbed by dehydrated fruits differed conspicuously in relation to the various dehydration processes. Goldblith et al. (1963) found that the rehydration ratios of shrimp varied according to the conditions maintained prior to and following freeze dehydration. Conventional hot-air dehydration shrunk the cell structure in fruit tissue and subsequently resulted in a hard texture. These undesirable effects caused a low rehydration rate and required more than 30 minutes to be rehydrated completely. Dehydro-frozen fruits in which the original weight was reduced to 50 per cent during dehydration showed the lowest rehydration rates. However, rehydration was completed in less than 15 minutes. It is reasoned that dehydro-frozen fruits contained higher amounts of moisture in comparison to other dehydrated fruits. The freeze dehydration process fostered retention of the original cell structure, which produced porous dehydrated fruit. Subsequently such fruit imbibed water rapidly and rehydration was completed in 10 minutes. Freeze dehydrated fruits which were frozen prior to dehydration at a high temperature ( $0^{\circ}$  F) had a relatively high rehydration ratio as compared to those which were cryogenically frozen at  $-250^{\circ}$  F. The disruption of cellular structure by large ice crystals at the higher temperature (above  $0^{\circ}$  F) may promote a great absorption of water during rehydration as shown by Goldblith et al. (1963). The effects of different freezing conditions were more obvious in apples (Figure 42). In general, the rehydration

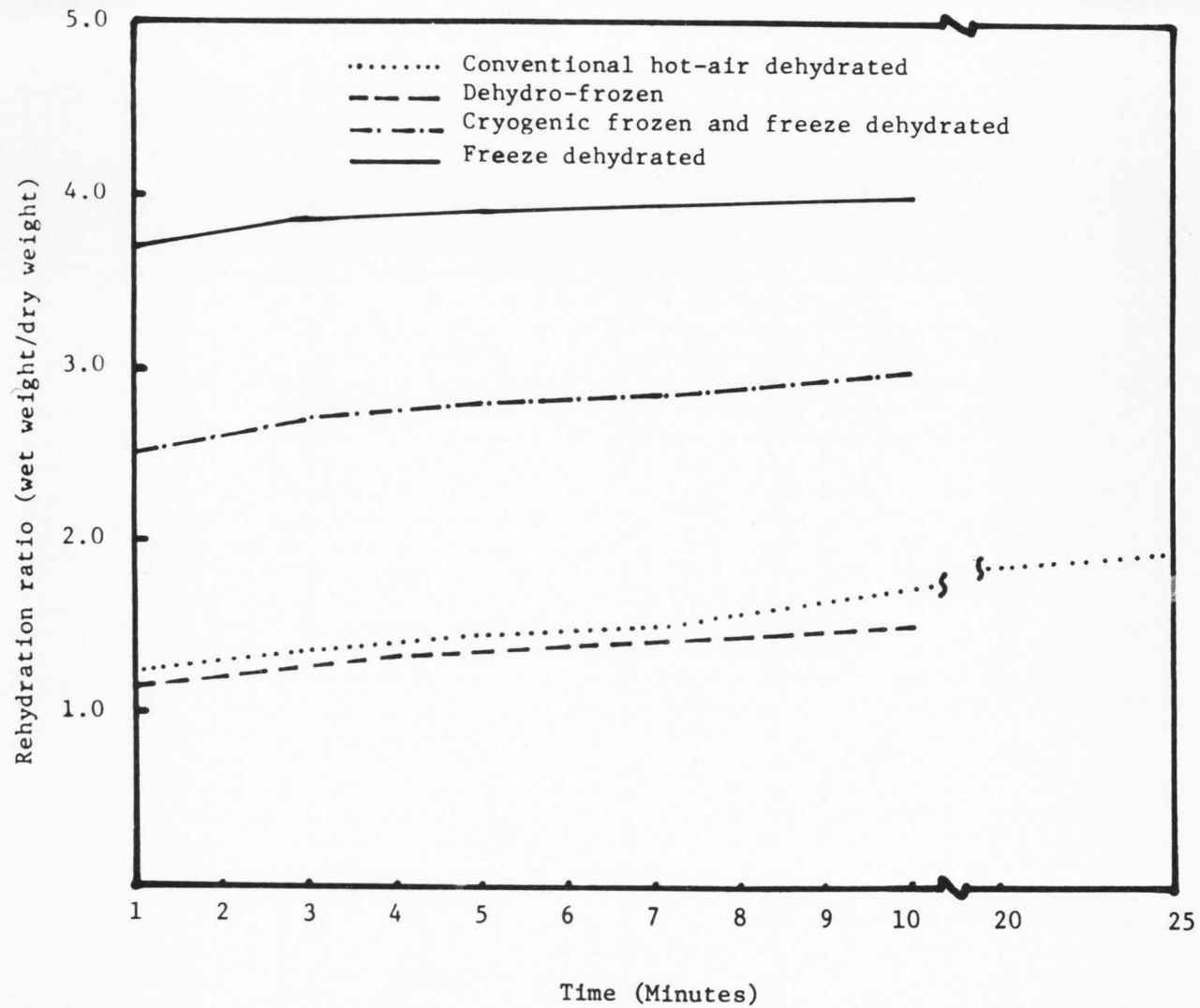


Figure 42. Rehydration rate of apples dehydrated by different methods.

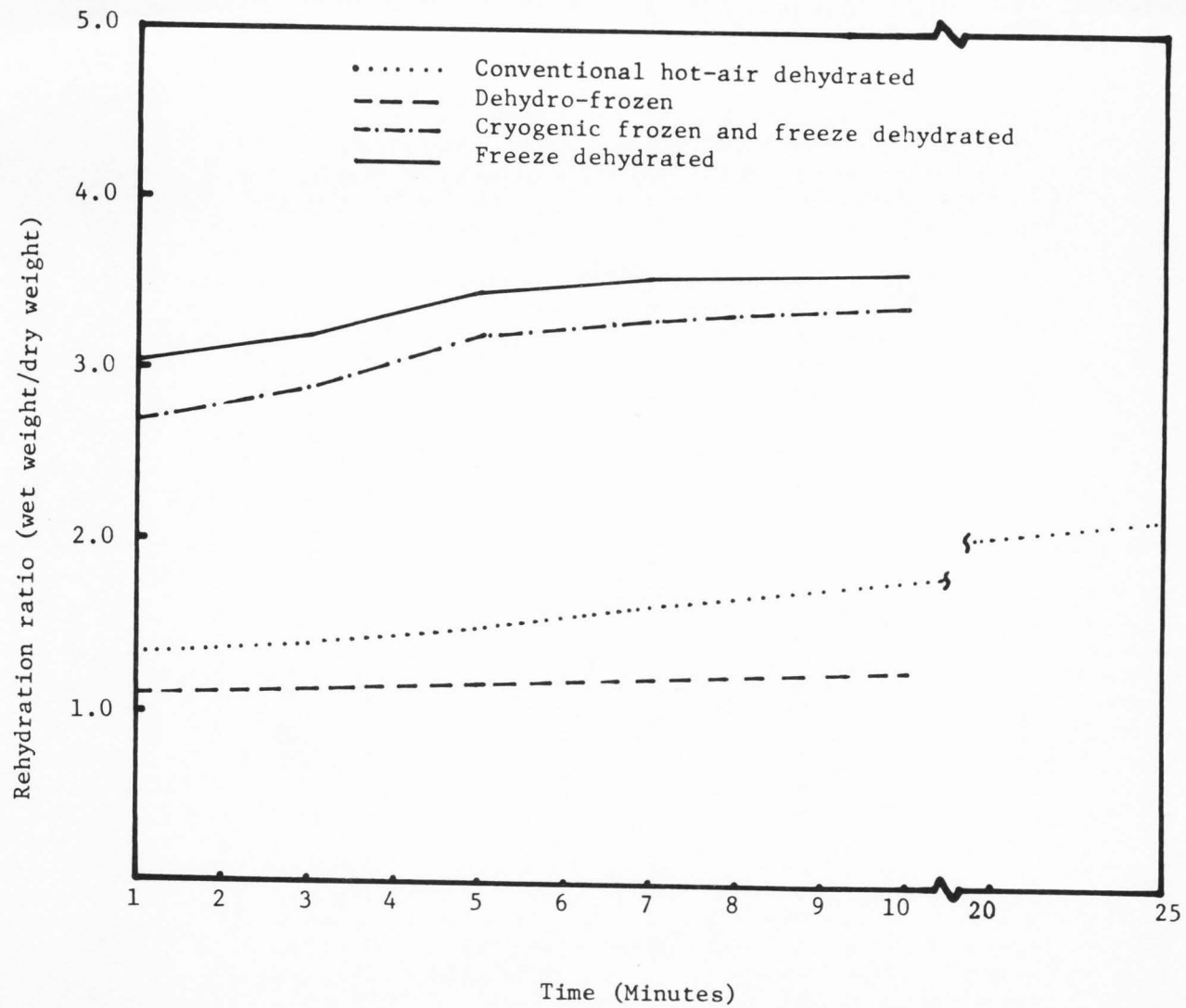


Figure 43. Rehydration rate of apricots dehydrated by different methods.

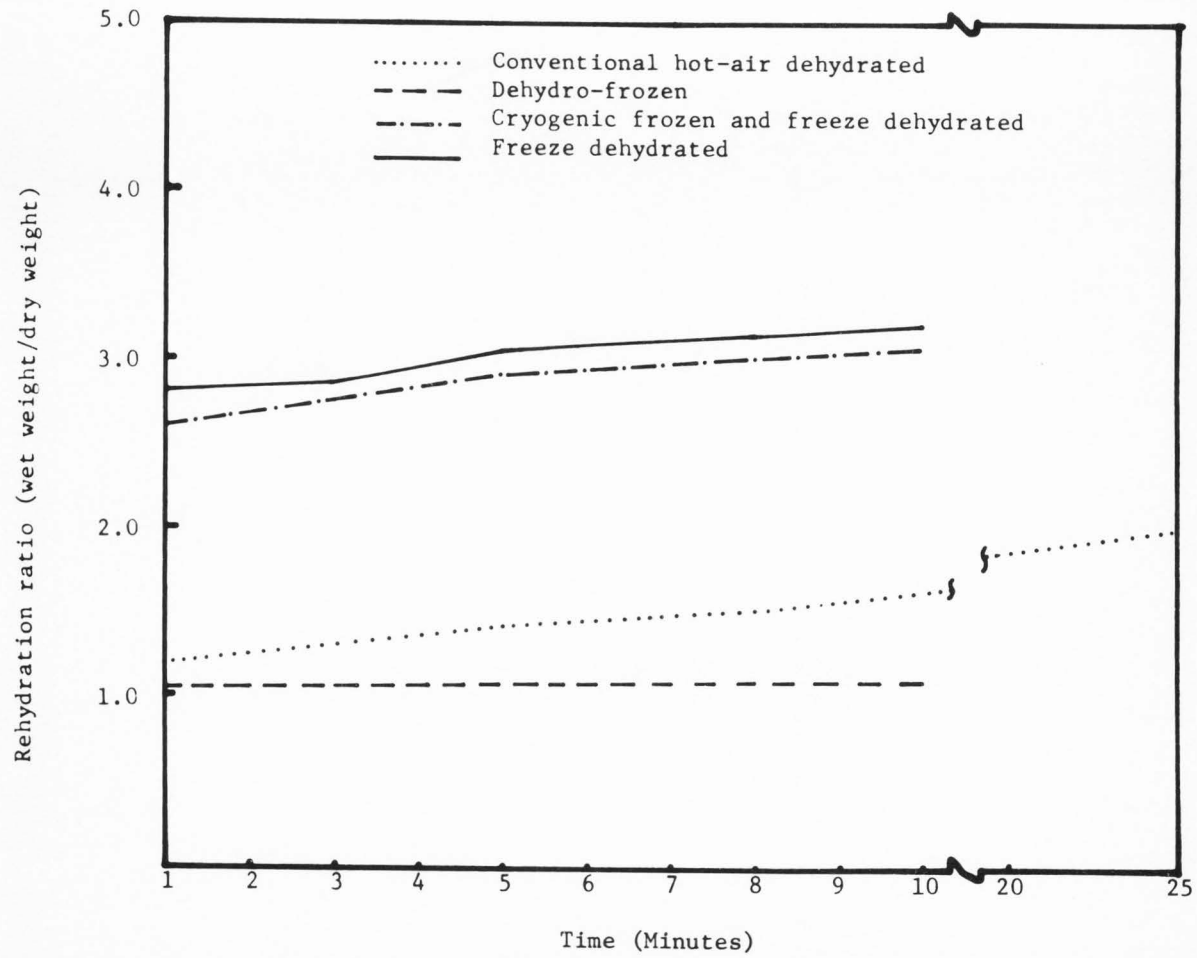


Figure 44. Rehydration rate of peaches dehydrated by different methods.

rates of apricots were more similar to those of peaches than apples. This may be a result of certain structural differences among the fruits.

Even the rehydration rates of freeze dehydrated fruits were high as compared to those of fruits dehydrated by other methods, their textural properties were not similar to those of fresh fruits. It is known that a dehydrated product never regains its original properties because it does not absorb as much water on reconstitution as was present in the original fresh material. Kuprianoff (1958) gave a possible explanation on this problem. He discussed the changes of reconstitution behavior and water-binding capacity during the dehydration. He explained the irreversible changes of low moisture cellulose that the free hydroxy groups of cellulose molecules in its original water-soaked condition are practically all satisfied by water. When the cellulose is dehydrated, these hydroxy groups lose their water and their valence could be satisfied after shrinkage by drawing adjacent cellulose molecules together. Such pairs of hydroxy group may remain in this combination during rehydration causing an irreversible change in the structure.

#### Osmotic Effects of Sucrose

The diffusion pressure deficit of apple slices was around 15.7 atmosphere which corresponds to 0.7 Molar (23.94 per cent) sucrose solution. This provided the information of the minimum concentration of sucrose solution needed for the experiments. The slices which were immersed in 0.7 Molar sucrose solution showed no noticeable change in weight, because the osmotic

pressure of the solution was very close to the diffusion pressure deficit of the fruit. As the sucrose concentration of the impregnation solution was increased, the percentage weight loss of the slice tended to increase (Figure 45). This progressive variation is presumably the main result of osmotic effects, hence, a relatively greater loss of fluid from the slices to the more concentrated sucrose solutions due to osmosis might be expected. The slices placed in dry sucrose showed the maximum weight loss (39 per cent) among the treatments after 20 hours of immersion. It may be assumed in this case that the deliquescing property of sucrose, in addition to the high osmotic effect, might be involved in the change of fruit weight.

Soluble solids in fruit increased as the sucrose content of the solution was increased. This tendency was proportional to the immersion time (Figure 46). The slices immersed in 2.0 Molar sucrose solution gave considerable increase in soluble solids content whereas the slices immersed in 0.7 Molar sucrose solution were the lowest. However, the slices placed in dry sucrose were lower in soluble solids as compared to those of the slices which were immersed in 1.5 and 2.0 Molar sucrose solutions. This was probably due to the absence of solvent ( $H_2O$ ) and/or the saturated concentration which may decrease the molecular movement of sucrose and the subsequent reduction in the rate of the sucrose penetration into the fruit. It is to be noted here that more sucrose from higher concentration (2.0 Molar) penetrated into the fruit and increased the soluble solids in the fruit with the simultaneous decrease of soluble solids in solution. The rate of soluble solids losses in the solution

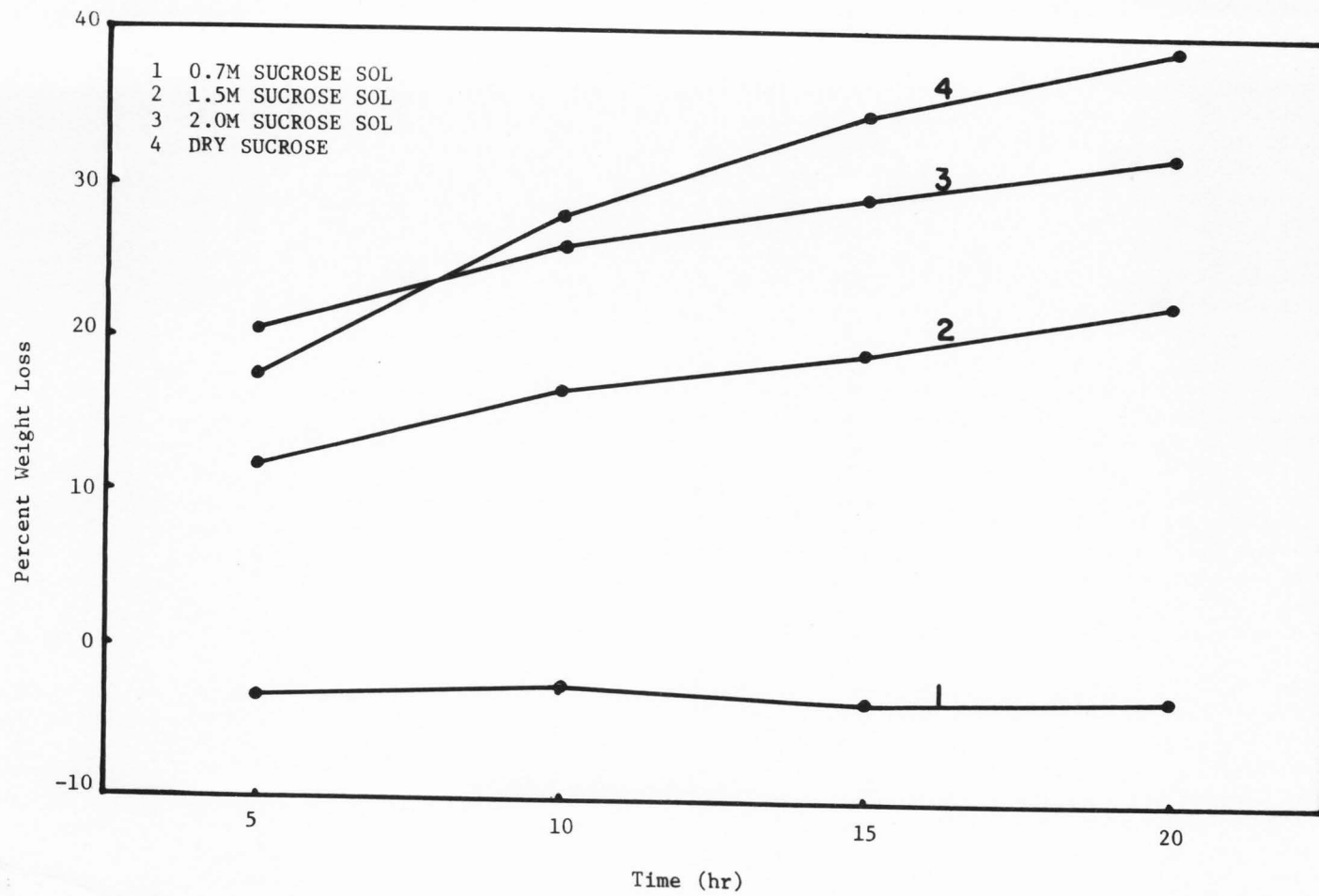


Figure 45. Change in fruit weight during submersion in different concentration of sucrose solution.

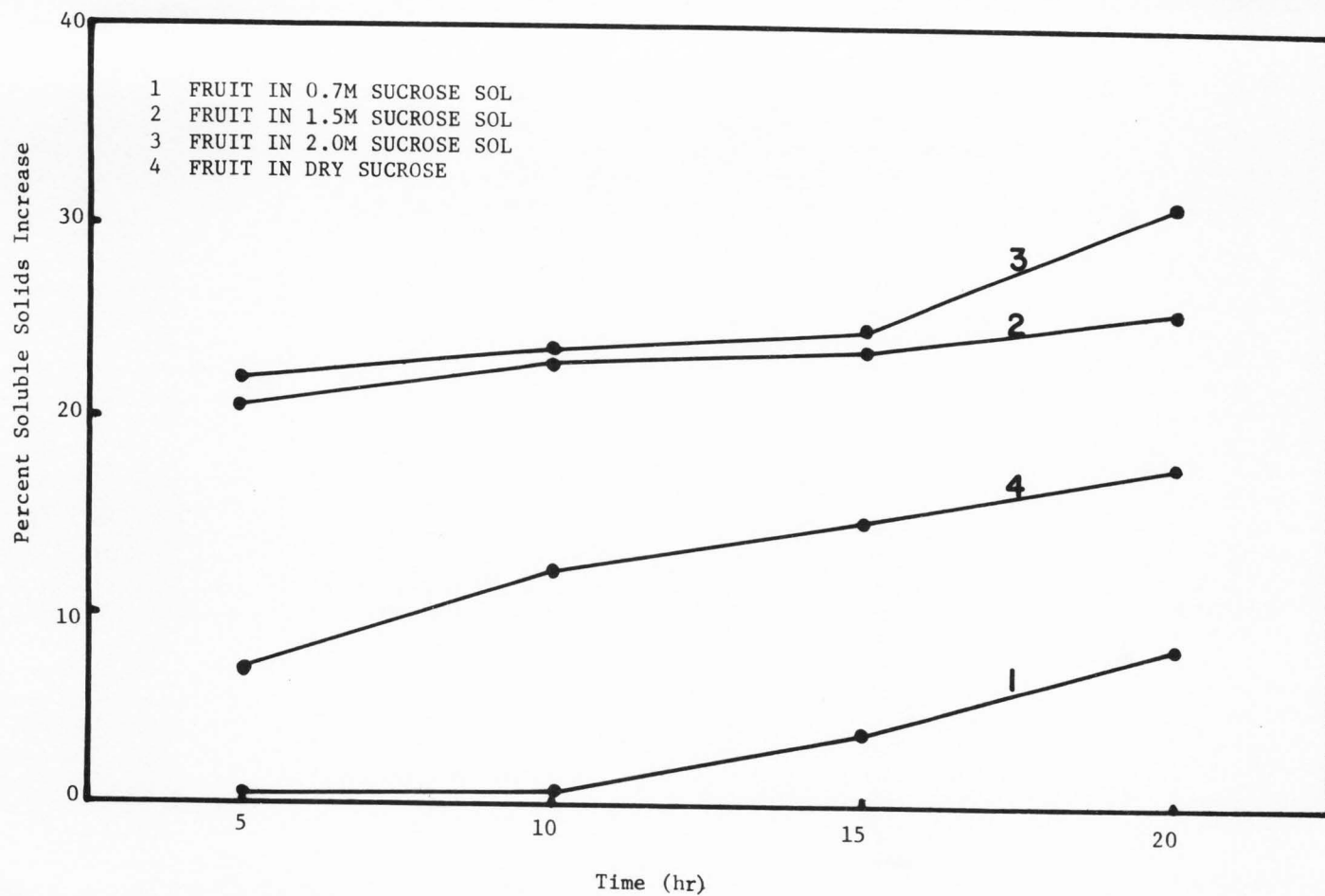


Figure 46. Change in soluble solids content of fruit during submersion in different concentration of sucrose solution.



was almost proportional to that of soluble solids increases in the fruit (Figure 47). The concentration gradient might be distorted to a certain degree due to volume (or weight) change of the fruit, however, this effect was not considered in this study. Hughes *et al.* (1958) found that the high concentration gradient of sugar, from the outside of the peach to its interior, causes an initially rapid movement of sugar into the flesh of the peach despite the rapid movement of fluid from the fruit. On cross-sectioning of the slices, it was observed that water-filled parts usually were translucent throughout, showing complete penetration of the liquid, whereas sucrose-filled parts of the slices were generally opaque except for a narrow band around the surface of the slices. Movement of fluid from apple into the solution probably was due to osmotic pressure differences across the imperfect semipermeable membranes of the apple cells. Hughes *et al.* (1958) explained that the flow of fluid under the osmotic pressure ingredient very likely continued until the activity of the water was equal in the syrup and peach in processed clingstone peaches. The equilibrium condition was not attained during this study. However, it seems reasonable to assume that the diffusion of sucrose and fluid probably continues until the activities of all the gradients are equal within the apple and the solution.

Soluble solids at different depths of apple slices for different concentration of sucrose are shown in Figure 48. High concentration of sucrose was observed at the outermost layer of fruit surface in all treatments. However, there were no appreciable differences in sucrose contents for second, third, and

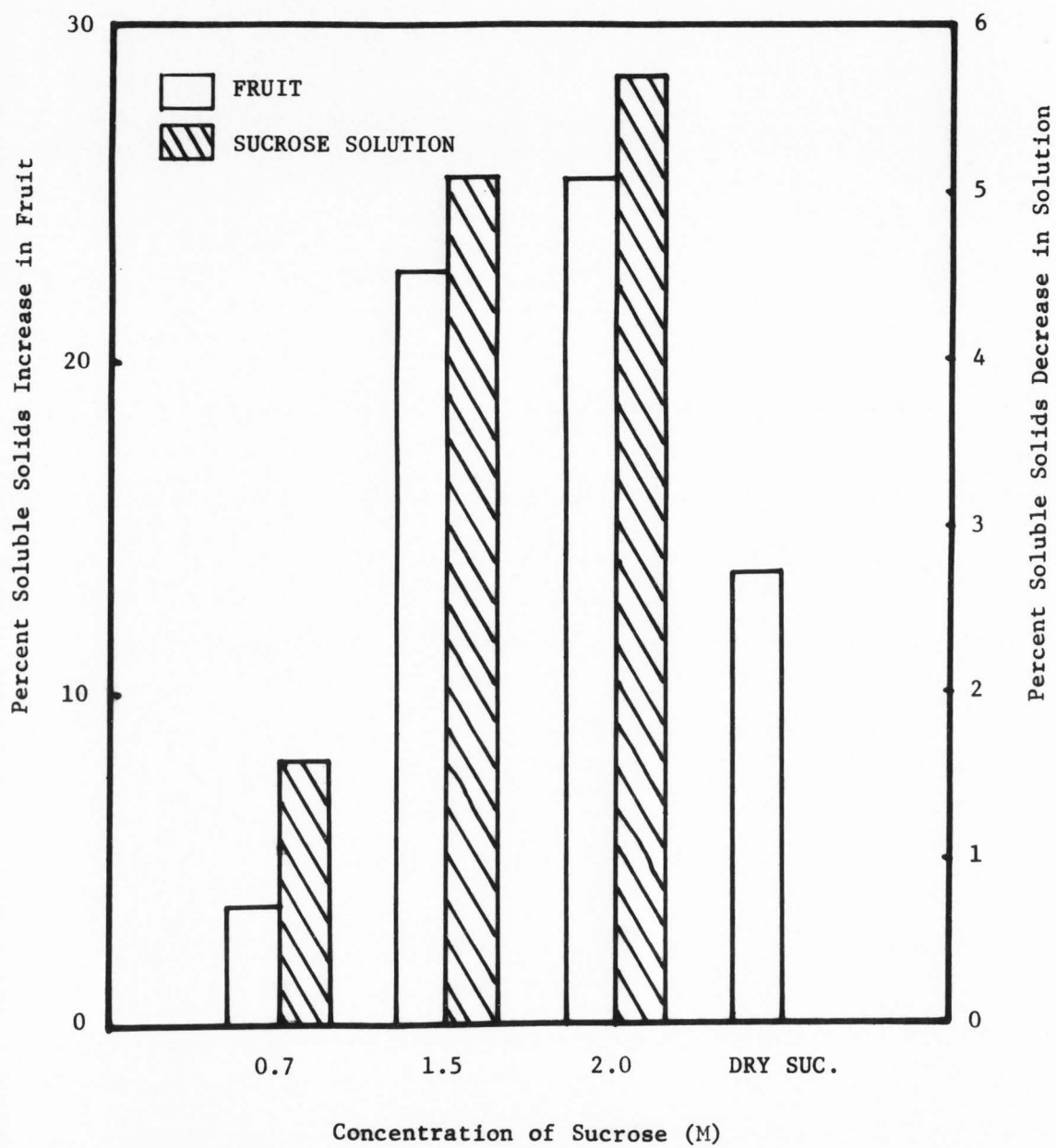


Figure 47. Comparison of soluble solids of fruit and the sucrose solution at different concentration.

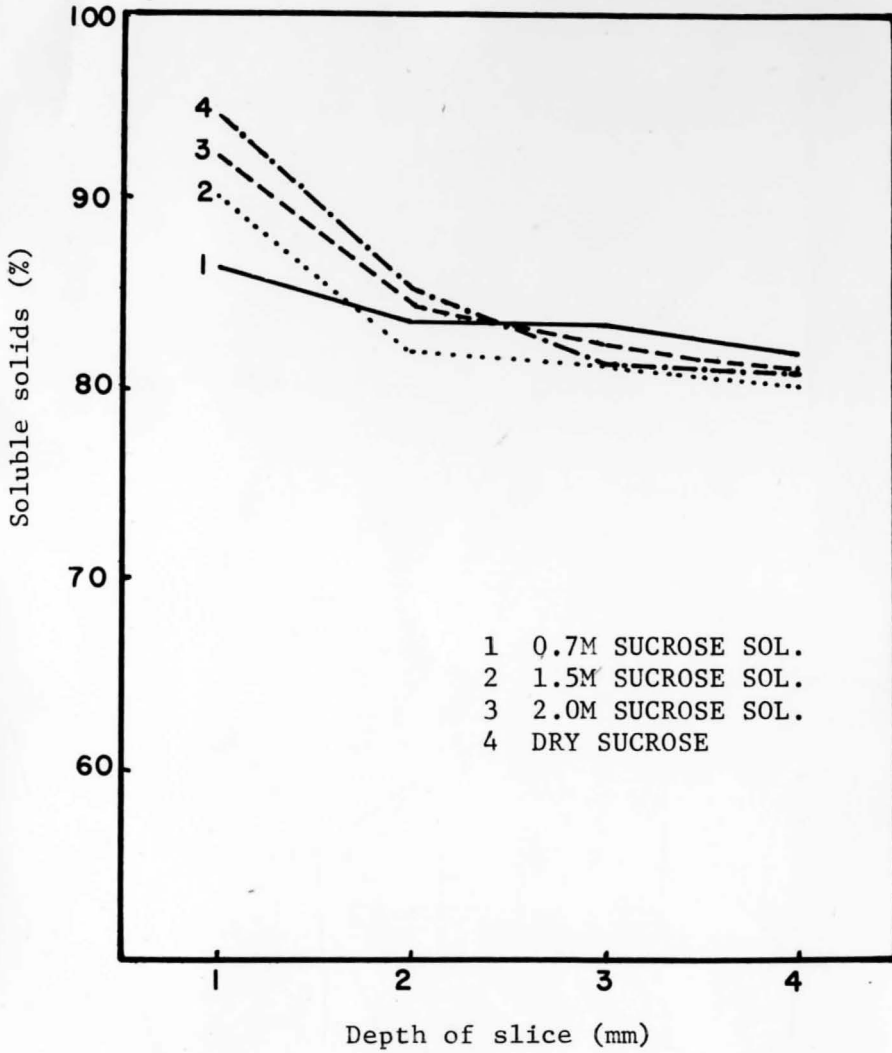


Figure 48. Soluble solids content at different depths of apple slices.

fourth layers, which may show that the time was too short for sucrose diffusion into the center portion of the slices.

Autoradiographs for each of apple slices immersed in different concentration of  $C^{14}$  labeled sucrose solutions are shown in Figures 49-51. The degree of radioactivity on the surface area of slices was proportional to the concentration of sucrose. However, no competent differences in depths of sucrose diffusion into fruit tissue were noticed.

It may be concluded that major portion of the sucrose from the solution was located at the surface area of the apple slices in osmo-freeze dehydration process and increased concentration of sucrose did not follow an increase in rate of diffusion. The discrepancy of the sucrose diffusion into the apple tissue from that of peach as reported by Hughes *et al.* (1958) may be due to different varieties and species of fruits and different processing conditions used in this study.

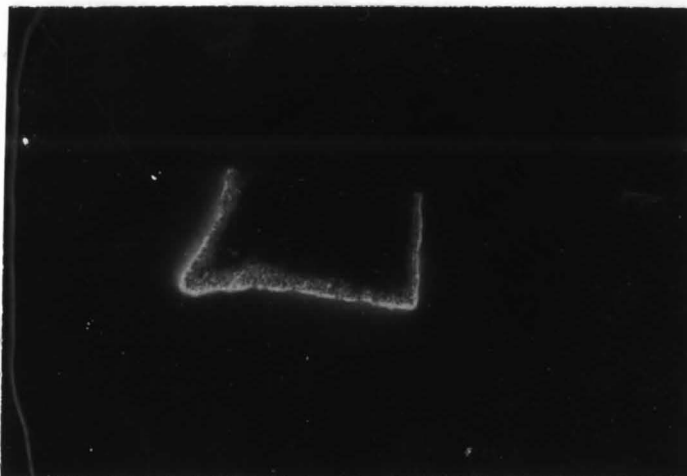


Figure 49. Autoradiograph of osmo-freeze dehydrated apple slice (treated in 0.7 M sucrose solution).

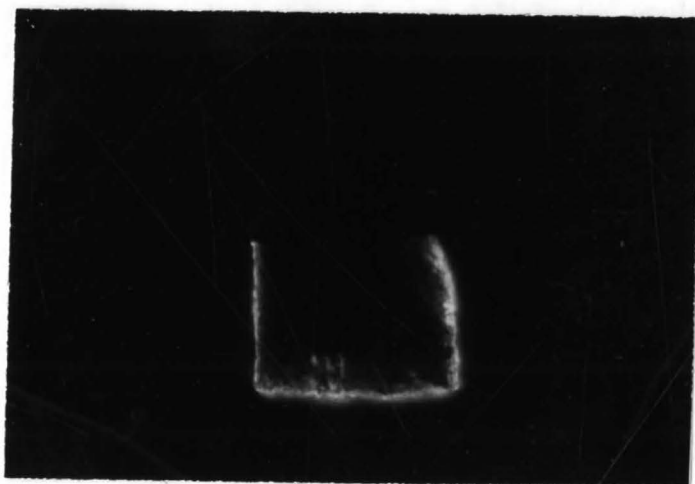


Figure 50. Autoradiograph of osmo-freeze dehydrated apple slice (treated in 1.5 M sucrose solution).

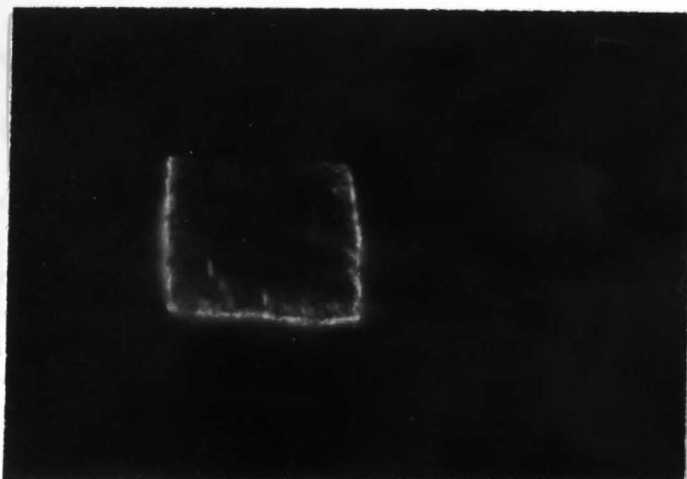


Figure 51. Autoradiograph of osmo-freeze dehydrated apple slice (treated in 2.0 M sucrose solution).

## SUMMARY AND CONCLUSIONS

Comparative studies of the effects of several dehydration processes on the flavor compounds, aroma, histology, rehydration rate, and osmotic changes of fruits were conducted. Fresh apples, apricots, and peaches were dehydrated by conventional hot-air dehydration, dehydro-freezing, and freeze dehydration processes. Several pretreatments were applied to the freeze dehydration process to develop a new fruit product.

Each sample was analyzed for flavor compounds, such as titratable acidity, reducing sugars, volatile reducing substances, volatile carbonyl compounds, tannins, and ascorbic acid. In addition, direct head-space gas-liquid chromatographic methods were employed for the comparison of volatile aroma compounds. Color changes and rehydration rates of dehydrated fruits were also measured.

Histological and histochemical analyses were conducted for representative samples of fruits from each of the dehydration processes.

Effects of sucrose on osmo-freeze dehydrated apples were studied by chemical analyses and autoradiographic methods.

A. Among the different dehydration processes significant differences in chemical compounds which contribute to the fruit flavor were observed. The acid content of conventional hot-air dehydrated fruits decreased while soluble solids and reducing sugars increased. However, these changes were minimal in freeze dehydrated fruits. Some of these changes may be explained by the

hydrolysis of high molecular to low molecular compounds under the condition of high temperature and low pH of fruits. Volatile reducing substances, volatile carbonyl compounds, tannins, and ascorbic acid were more significantly decomposed by hot-air dehydration than the other treatments studied. Freeze dehydration process appeared to have the least effect as compared to other dehydration processes. Direct head-space gas-liquid chromatographic analysis indicated that freeze dehydrated fruit retained volatile aroma components more than the fruit dehydrated by other methods. It may be concluded that freeze dehydration process is superior in maintaining the original fruit flavor and the quality of the products in comparison with the other dehydration processes studied.

B. Conventional hot-air dehydrated fruits underwent more severe browning reactions (high optical density) than those fruits dehydrated by freeze dehydration. These browning reactions subsequently resulted in adverse changes to the products.

C. Conventional hot-air dehydration caused pronounced changes in histology of fruits. The parenchyma cells were compressed, elongated, separated, and a few of them were ruptured. These are the primary reasons of the irreversible changes of dehydrated fruits. Conventional freezing produced extensively broken cells as a result of large ice crystal formation, which seems the primary effects of histological changes in freeze dehydrated fruits. However, cryogenic freezing in liquid nitrogen minimized the cellular damage and produced intact cellular structure in the freeze dehydrated fruits. Hence, it is concluded that freeze dehydration process, with a proper



pretreatment, minimizes the changes in texture of the fruit products.

D. The freeze dehydration process, which fostered retention of the original shape and produced porous dehydrated fruit slices, showed faster rehydration and a higher rehydration rate than fruit slices prepared by other techniques. This finding corroborates the conclusions derived from the histological study of freeze dehydrated fruits.

E. A sucrose treatment prior to freeze dehydration resulted in several advantages to the products. Osmotic action of sucrose that removed moisture from fruits prevented the formation of large ice crystals in the fruit tissues during conventional freezing and subsequently minimized the cellular damage. Certain amounts of sucrose also diffused into the fruit tissues (primarily at the outermost 1 mm layer) and covered surface area as a coating agent and subsequently yielded crispness in texture to the final products which can be consumed "as is" in the dehydrated state.

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APPENDIX

Table 5. Analyses of variance for chemical compounds of fresh and dehydrated apples

Source of variance	Degree of freedom	Mean square							
		Acids	Soluble solids	Reducing sugars	Total reducing sugars	Volatile reducing substances	Volatile carbonyl compounds	Tannins	Ascorbic acid
Replication	3	0.001	0.043	0.603	0.253	1321	0.116	23.6	0.35
Treatment	3	0.006	2.230**	0.870	0.295	61072*	4.070**	8220.6**	30.40**
Error	9	0.009	0.053	0.472	0.088	1162	0.070	94.0	0.25
Total	15								

\*Significant at 5 per cent level

\*\*Significant at 1 per cent level

Table 6. Analyses of variance for chemical compounds of fresh and dehydrated apricots

Source of variance	Degree of freedom	Mean square							
		Acids	Soluble solids	Reducing sugars	Total reducing sugars	Volatile reducing substances	Volatile carbonyl compounds	Tannins	Ascorbic acid
Replication	3	0.00043	0.096	0.0006	0.0014	643.5	0.1137	1.16	0.08
Treatment	3	0.03493**	4.093**	0.0413**	0.0088**	38770.6**	35.0459**	2472.00**	13.90**
Error	9	0.00224	0.043	0.0008	0.0009	617.1	0.0941	12.27	0.22
Total	15								

\*\*Significant at 1 per cent level

Table 7. Analyses of variance for chemical compounds of fresh and dehydrated peaches

Source of variance	Degree of freedom	Mean square							
		Acids	Soluble solids	Reducing sugars	Total reducing sugars	Volatile reducing substances	Volatile carbonyl compounds	Tannins	Ascorbic acids
Replication	3	0.00026	0.026	0.0145	0.0009	318.3	0.0335	4.13	0.28
Treatment	3	0.00056	8.303**	2.1383**	3.5059**	307018.9**	10.9253**	6510.06**	15.91**
Error	9	0.00022	0.100	0.0135	0.0120	809.4	0.0094	17.42	0.25
Total	15								

\*\*Significant at 1 per cent level

Table 8. Result of new multiple range test of chemical compounds of apple

Acidity:				
Treatment	A	B	C	D
Means	0.57	0.49	0.49	0.55
Soluble solids:				
Treatment	A	D	B	C
Means	12.8	13.1	14.4	13.7
Reducing sugars:				
Treatment	A	B	C	D
Means	6.41	7.34	6.55	6.57
Total reducing sugars:				
Treatment	A	C	D	B
Means	12.33	12.55	12.68	12.98
Volatile reducing substances:				
Treatment	A	B	C	D
Means	360	60	228	188
Volatile carbonyl compounds:				
Treatment	A	B	C	D
Means	3.2	0.8	2.6	2.3
Tannins:				
Treatment	A	B	C	D
Means	148	42	120	123
Ascorbic acid:				
Treatment	A	B	C	D
Means	7.2	0.5	3.1	3.6

- A. Fresh  
 B. Conventional hot-air dehydrated  
 C. Dehydro-frozen  
 D. Freeze dehydrated

Table 9. Result of new multiple range test of chemical compounds of apricot

Acidity:				
Treatment	B	A	C	D
Means	0.99	1.12	1.15	1.14
Soluble solids:				
Treatment	A	B	C	D
Means	10.0	12.4	11.6	11.0
Reducing sugars:				
Treatment	B	A	D	C
Means	1.15	1.28	1.31	1.34
Total reducing sugars:				
Treatment	A	B	D	C
Means	3.27	3.31	3.29	3.20
Volatile reducing substances:				
Treatment	A	B	C	D
Means	902	665	798	822
Volatile carbonyl compounds:				
Treatment	A	B	C	D
Means	9.9	3.3	7.6	9.2
Tannins:				
Treatment	A	B	C	D
Means	93	36	57	78
Ascorbic acid:				
Treatment	A	B	C	D
Means	7.7	3.4	6.4	6.8

- A. Fresh  
 B. Conventional hot-air dehydrated  
 C. Dehydro-frozen  
 D. Freeze dehydrated

Table 10. Result of new multiple range test of chemical compounds of peach

Acidity:				
Treatment	A	C	D	B
Means	0.56	0.54	0.55	0.53
Soluble solids:				
Treatment	A	C	D	E
Means	11.0	11.5	11.6	14.2
Reducing sugars:				
Treatment	A	B	C	D
Means	1.81	3.46	2.03	2.46
Total reducing sugars:				
Treatment	A	B	C	D
Means	3.85	5.57	3.43	4.17
Volatile reducing substances:				
Treatment	A	B	C	D
Means	980	380	660	924
Volatile carbonyl compounds:				
Treatment	A	B	C	D
Means	6.6	2.9	4.8	6.1
Tannins:				
Treatment	A	B	C	D
Means	140	44	79	103
Ascorbic acid:				
Treatment	A	B	C	D
Means	9.0	4.4	6.0	7.7

- 
- A. Fresh  
 B. Conventional hot-air dehydrated  
 C. Dehydro-frozen  
 D. Freeze dehydrated