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A series of tests were conducted in which the odors of standard and impaired, precooked, dehydrated sweetpotato flakes were compared with the odors of oxidized carotenoid fractions which had been isolated from sweetpotatoes. In another series of tests the odors of standard and impaired, precooked, dehydrated sweetpotato flakes, white potato flakes, and carrot flakes were compared with each other. The objectives of the study were to determine if beta-carotene or any of the oxidized carotenoid fractions extracted from sweetpotatoes are the precursors of off-odor in precooked, dehydrated sweetpotato flakes and to determine whether or not off-odor development in dehydrated carrot flakes, dehydrated sweetpotato flakes, and dehydrated white potato flakes is caused by the same compounds. Paired tests were used for olfactory comparison of precooked, dehydrated sweetpotato flakes and oxidized carotenoid fractions. Triangle tests were used for olfactory comparison of precooked, dehydrated sweetpotato, white potato, and carrot flakes. Responses of fourteen panel members were recorded on scorecards.

Results of this study indicate that there is a conclusive difference between the odors of fractions 1, 2, 3 purified, and 3 supernatant (phytoene, phytofluene, crystalline beta-carotene, and beta-carotene supernatant, respectively) and good and poor sweetpotato flakes. Results indicate that fraction 8 (*cis*-mutatochrome and unidentified compounds) is conclusively like the impaired sweetpotato flakes in odor. All other carotenoid fractions were found to be similar to both standard

and impaired sweetpotato flakes. However, all fractions except 6 (alpha-5', 6'-epoxide) and 7 (beta-5, 8-epoxide) were judged to be more similar to impaired flakes than to the standard flakes in odor.

The odors of both standard and impaired dehydrated sweetpotato, white potato, and carrot flakes were easily distinguished from each other by panel members. The impaired flakes of sweetpotato, carrot, and white potato were found to be no more alike in odor than the standard flakes of the same foods.

The findings of this investigation indicate that the first three carotenoid fractions, including beta-carotene crystals and beta-carotene supernatant, are not the precursors of off-odor in precooked, dehydrated sweetpotato flakes. Results indicate that off-odor in dehydrated sweetpotato, carrot, and white potato flakes is caused by different compounds, peculiar to each product. There are indications that compounds associated with the carotenoids, especially fraction 8, may be precursors of off-odor in precooked, dehydrated foods.

# AN INVESTIGATION OF THE RELATIONSHIP OF CAROTENOIDS TO OFF-ODOR DEVELOPMENT IN DEHYDRATED FOODS

by

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APPROVAL SHEET

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# CHAPTER I

## INTRODUCTION

Since World War II dehydration has become an increasingly popular method for the preservation of foods. Dehydrated white potato, sweetpotato, and carrot products are among the dehydrated foods which have become very important for use as military rations and as commercial commodities. All three foods have been produced in the form of instant, precooked, dehydrated flakes, as well as in some other forms.

A major problem in producing dehydrated sweetpotato, white potato, and carrot flakes is that of extending the shelf life of the products. Off-odors and off-flavors develop rapidly in each product during storage at ambient temperatures unless certain precautions are taken during processing of the products. Some researchers, noting a high correlation between the development of off-odors and off-flavors and the decline of carotenoid concentration, have concluded that the carotenoids are precursors of the off-odor and off-flavor development. Other researchers have attributed off-odor and off-flavor development to the autoxidation of certain lipids in the dehydrated foods. No specific compounds, however, have been identified as the exact cause of off-odor and off-flavor.

The present study was an attempt to determine by olfactory evaluation whether or not the carotenoids are involved in off-odor and off-

flavor development in dehydrated sweetpotato, white potato, and carrot flakes. If off-odor development is caused by the oxidation of any or all of the carotenoids, then the odor of dehydrated sweetpotato flakes which have deteriorated will be very similar to the odor of those isolated oxidized carotenoids involved in off-odor development. Both sweetpotatoes and carrots contain large amounts of the carotenoids. If the carotenoids are the precursors of off-odor development, the odors of dehydrated carrot and sweetpotato flakes would become less easily distinguishable from each other as the two flakes deteriorate. White potatoes, however, contain only minute amounts of the carotenoids. The odor of dehydrated white potato flakes would become less like the odors of dehydrated carrot and sweetpotato flakes as all three products deteriorate during storage.

# CHAPTER II

## REVIEW OF LITERATURE

### Dehydrated White Potato, Sweetpotato, and Carrot Flakes

Dehydration is a method of food preservation which involves controlled drying of foods. This is usually achieved by artificial thermal means (1). Dehydration has been used for many years to preserve foods, but received its greatest impetus during World War II. Dehydrated foods became necessary during the war years because of the ease of transporting and storing them. Large quantities of dehydrated vegetables and fruits were produced for the armed forces. Consequently, research and development in the processing of dehydrated vegetables and fruits made many extraordinary advances (2, 3, 4). Many dehydrated food products have been improved or developed for civilian use, as well as for military rations. Among the dehydrated vegetables which were introduced during the war and which have become of increasing commercial importance in recent years are dehydrated white potato, sweetpotato, and carrot products.

A form of dehydrated white potato, the potato granule, has been described by Cooley et al. (5) and Tressler (3). The process of producing potato granules originated during World War II, and commercial production was initiated by the British Food Ministry. Production did not begin in America until the postwar years (3), but expanded rapidly to meet military needs. Further impetus was given to dehydration of potatoes by a situation

which developed in the potato industry. In the postwar years the economic position of the potato industry was weakened by a continued high level of production in the face of decreased consumption (6). The need for a new potato product was apparent. Developmental work was begun on a precooked dehydrated potato flake in 1953 at the Eastern Utilization Research and Development Division of Agriculture Research Service at Philadelphia, Pennsylvania (6).

Cording and co-workers (7, 8, 9, 10) described the production of the precooked dehydrated potato flakes. By 1959 potato flakes were being made commercially in Idaho, Maine, Michigan, New York, North Dakota, Oregon and several foreign countries (10). By 1962 the flakes were a well-known commercial article in the United States and in other countries as well (11). Drazga et al. (12) described potato flakelets, a recently developed, denser form of precooked dehydrated potatoes which have some advantages over the flakes.

The development of a dehydrated sweetpotato product has attracted the attention of researchers for many years (13). In the early 1900's dehydrated sweetpotatoes were produced for use as military rations (14). Following the successful development of dehydrated white potato flakes, the Southern Regional Research Laboratory of the United States Department of Agriculture began to develop precooked, dehydrated sweetpotato flakes (6).

Like the white potato industry, the sweetpotato industry also was experiencing an economic decline because of decreased consumption. For

this reason, a new sweetpotato product was needed. Also, a new sweetpotato product was needed to provide an outlet for a substantial volume of the crop which was unmarketable because of grade or size (15, 16, 17).

Commercial production of sweetpotato flakes was initiated by two North Carolina plants in 1962. Plants in Louisiana and other states soon began manufacturing sweetpotato flakes (16). The precooked dehydrated sweetpotato flake process has been described by Deobald (18).

The production of dehydrated carrots was initiated also during World War II. Dehydrated carrots were first produced in the form of dice and strips for use as military rations (4). Production was sharply curtailed at the end of the war, but experimentation has continued in an effort to produce a high quality dehydrated carrot product.

Several researchers (19, 20, 21) have described an explosion-puff method for producing sliced or diced dehydrated vegetables. This method is more applicable than the flake method in producing dehydrated carrots since carrots are more generally served diced or sliced than as mashed carrots (21). The explosion-puff method has also been used for dehydrated potatoes (20).

## Off-odor and Off-flavor in Dehydrated Foods

A major problem in the production of dehydrated white potato, sweetpotato, and carrots is increasing storage stability at ambient temperatures. Tomkins et al. (22) has defined "storage life" as the period during which the color, flavor, and texture of dehydrated

vegetables are rated by a test panel as fair. These researchers reported a marked problem with loss of original color, odor, flavor, and vitamin content of dehydrated foods when stored and transported under the usual conditions available at that time.

Mallette et al. (2) reported similar problems in transporting and storing of dehydrated vegetables and observed a gradual development of an "oxidative" off-flavor in instant mashed potato granules when the product was stored in air. Potato granules containing 7 per cent moisture and stored at 95 to 105°F became inedible after 7 months. They also reported a marked "hay-like" odor which developed in dehydrated sweetpotatoes stored at 95 to 105°F for four weeks and which became more pronounced after 18 weeks.

Hendel et al. (4) reported the development of an "oxidative" off-flavor which occurred slowly in mashed potato granules at 75°F. This change was accelerated by lower moisture content and by increasing the oxygen in the atmosphere of the package. The development of an "oxidative" off-flavor in dehydrated potatoes stored in air has also been reported by Tressler (3) and Buttery et al. (23).

Legault et al. (24) reported color fading and the development of a stale or rancid flavor in dehydrated sweetpotatoes stored at 100°F. Deobald et al. (18) reported that precooked, dehydrated sweetpotato flakes, when exposed to air for 24 hours or longer, developed an undesirable "hay-like" flavor and odor. The same off-odor and off-flavor development has been reported by other researchers (14, 25, 26).

As early as 1944 the development of an unpleasant off-flavor and off-odor in dehydrated carrots was reported by Tomkins et al. (22). These researchers described the off-odor as similar to violets and attributed it to the formation of beta-ionone by the oxidation of beta-carotene. Falconer et al. (27) described this same odor in dehydrated carrots stored in the presence of oxygen.

Notable in all reports of the off-odor and off-flavor development in dehydrated food products has been the role of storage temperature, storage time, and exposure of the product to oxygen (2, 3, 16, 22, 23, 24, 25, 26, 27, 28). In order to protect the dehydrated food products in storage, certain processes have been developed which retard the odor and flavor deterioration. Mackinney et al. (29) reported that the necessity of excluding oxygen from packages of dehydrated carrots was known as early as the World War II years. Tomkins et al. (22) reported that storage in the absence of oxygen, using nitrogen or carbon dioxide, retarded the loss of ascorbic acid and carotene and was essential for the preservation of a good quality of dried carrot, though not for dried potato. Legault et al. (24) described a method of in-package desiccation of white potatoes, sweetpotatoes, carrots, and some other vegetables, using nitrogen in the package, to retard off-odor and off-flavor development.

In developmental work of the precooked, instant sweetpotato flakes, it was found that hermetically sealing the flakes in an atmosphere of nitrogen retarded the development of poor flavors during storage (16). Molaison et al. (14) reported that dehydrated sweetpotato dice, canned under nitrogen in an atmosphere containing less than 2 per cent oxygen,

had good keeping qualities at 70°F for as long as 6 years. Deobald et al. (18) reported that good product stability was obtained for more than a year in precooked dehydrated sweetpotato flakes prepared with an antioxidant and in samples of the same batches in which the atmosphere was reduced to 2 per cent oxygen. The flakes so treated could be safely stored at 70°F and 100°F.

Deobald and McLemore (26) found that flakes canned in air and stored at 70°F and 100°F were acceptable in flavor after one month of storage. Flakes stored at 70°F in nitrogen containing 2 per cent or less oxygen retained flavor acceptability with or without an antioxidant for 24 months. However, flakes stored at 100°F were unacceptable after 17 to 20 months. They also found that sweetpotato flakes containing 200 ppm of butylated hydroxyanisole and of butylated hydroxytoluene packaged in air remained acceptable for more than 30 days. If 100 ppm citric acid was used as a synergist, the flakes were acceptable for more than 120 days.

The use of antioxidants and reduction of oxygen increases the storage stability of dehydrated white potato, sweetpotato, and carrot products. Off-odors, however, do develop with continued storage. The exact compounds involved in off-odor development have not been identified, but the problem has been studied by several researchers.

Tressler (3) found that both "oxidative" off-flavor development and oxygen absorption were somewhat retarded by petroleum ether extraction of dehydrated white potatoes before storage. He concluded that the oxidative

change must occur in the fat fraction of the potato.

Buttery et al. (23) studied the relationship of off-flavor development in potato granules to the degree of autoxidation of certain lipids. Off-flavor increased regularly with decreases in the ratio of unsaturated fatty acids to saturated fatty acids. The oxidative off-flavor of dehydrated potatoes, however, was not characteristic of normal fat rancidity. Tests were made to establish the identity of compounds causing off-flavor and off-odor. The compounds found were not the exact ones resulting from oxidation of pure linoleic and pure linolenic acids, but were similar. Buttery et al. (23) concluded that oxygen absorption and the autoxidation of linoleic and linolenic acid were important factors related to off-flavor development in air-packed potato granules.

Weier et al. (30) observed that when carrots are dried the carotene goes into solution in droplets of oil, and that the carotene is degraded concurrently with oxidation of fats. They found a good correlation between off-odor development and carotenoid degradation.

Tomkins et al. (22) and Mackinney and Fratzke (31) found that a loss of 20 per cent of the carotene in dehydrated carrots was accompanied by or paralleled production of off-flavors. Tomkins et al. (22) attributed off-flavor development to the formation of beta-ionone as a result of the oxidation of carotene.

Falconer et al. (27) reported that off-flavor was detected in dehydrated carrots when less than 5 per cent loss of carotene had occurred and found a direct relationship between loss of beta-carotene

and off-flavor development. A similar parallel in deterioration of sensory characteristics with degradation or loss of carotene was reported by Deobald et al. (18) to occur in dehydrated sweetpotato flakes.

Contrary to the findings of several researchers, Purcell (25) found that the amounts of the major carotenoids from samples of deteriorated sweetpotato flakes possessing a pronounced "hay-like" odor and flavor varied no more than plus or minus 10 per cent from the amounts found in samples of raw sweetpotatoes from the same lot. No correlation was noted between carotenoid variation and deterioration of the flakes. Purcell found that only the residue from the composite beta-carotene fractions had an odor resembling that of deteriorated flakes; whereas, pure beta-carotene crystals had a characteristic violet odor. Purcell concluded that compounds associated with the carotenoids could be the primary cause of the undesirable sensory changes in dehydrated sweetpotato flakes.

Deobald and McLemore (26) found that the extent of beta-carotene degradation showed some parallelism with sensory rating of dehydrated sweetpotato flakes when oxygen was not limited in the canning atmosphere of the flakes. However, when antioxidants were used and the oxygen in the canning atmosphere was limited to 6 and 10 per cent, the beta-carotene level did not consistently decrease at the time of off-flavor development.

Deobald et al. (28) found no correlation between beta-carotene disappearance and off-flavor development or oxygen disappearance from the canning atmosphere of precooked, dehydrated sweetpotato flakes. They found that flakes packaged in an atmosphere of nitrogen containing 2 per

cent oxygen developed an off-flavor after several months. The off-flavor was described as "rancid" and was unlike the "hay-like" flavor usually encountered in air-stored samples.

## Sensory Testing Methods

The senses of taste, smell, sight, and feel are often relied upon by food technologists and researchers (32). Selected judges are often used to measure and characterize differences in odor, taste, texture, and other qualities of food (33). One type of testing method used by food test panels is a sensory difference test. In sensory difference testing the investigator is usually interested only in "difference" as such without regard to its nature or direction. Either the panel discriminates or it fails to discriminate between or among samples. The information obtained is the number of judges who indicate the presence or absence of the difference (34). Sensory difference tests are often used in investigations of flavor problems.

Since flavor is odor plus taste, aspects of flavor may be judged by sniffing a food, as well as tasting it (35). Several researchers (33, 35, 37, 38, 39, 40) have studied olfaction and its uses in food research.

Odor is defined as that property of a substance that excites the sense of smell. Nasal stimulation is caused by certain kinds of molecules dispersed in the air. Every slightest change in molecular configuration is attended by odor differences. The sense of smell is exceedingly delicate and the quantity of material required to give a recognizable odor is

infinitesimally small in comparison with the amount needed to produce a recognizable taste. Recovery from odor stimulation is much more rapid than is recovery from taste stimulation. Watts (36) described the sense of smell as the best "all-around trouble shooter" for the chemist in many branches of food technology. In much industrial flavor evaluation, first impressions are gained through the sense of smell. Some researchers have reported better results in studying flavor problems when odor evaluation, rather than taste, was used (33).

A major limitation in using odor analysis in sensory evaluation tests is that odor impressions are now entirely too personal, and a better system of odor analysis and classification is needed (35). There is great need in all sensory evaluation for more objectivity (39).

Several simple test systems have been used in sensory difference testing which fulfill the criteria of objectivity and ease of interpretation. The paired difference test and the triangle test are two of these test systems often used in odor evaluation of foods (39, 40). Several researchers (32, 33, 34, 39, 40, 41) have studied the paired test and the triangle test systems. In the paired test the judge is presented two samples simultaneously or successively. The samples are judged by comparison with each other according to predesignated criteria. Criteria must be understood and reacted to in the same way by all judges. In the triangle test judges are given three samples and are informed that there are two different materials. Judges are asked to indicate either the two identical samples or the odd sample. A control or standard may be used

although it may not be designated as such. The more familiar of the two materials should be used as the control. The triangle method is particularly useful in comparing two samples which are almost alike.

A number of experiments have been conducted to compare sensory methods of measuring differences in food quality. In comparing the paired and the triangle test, Dawson and Dochterman (42) found that one test was no more precise than the other. They did report that the triangle test inspires more confidence because it reduces making correct responses by chance and eliminates judges who cannot identify duplicate samples. Peryam and Swartz (39), however, reported that the triangle test gives greater precision in many instances than other test methods and is easier to conduct. Gridgeman (43) concluded that paired tests and triangle tests are equally powerful and superior to the duo-trio test method, but found that the triangle is somewhat less economical than the paired test. Lockhart (40) suggested that a paired test provides a better method for measuring consumer acceptance and preference of a product, while the triangle method is better for difference testing.

The precision of any difference test depends on the experimental plan, the discrimination of the panel members, and the environmental conditions of testing (33, 44). Many investigations have dealt with the effect of the experimental plan on the precision of results (32, 33, 44, 45, 46). Although the number of samples that can be judged efficiently in one session is limited, the optimum number of samples that can be tested at one session without fatigue depends on the product. The

stronger the odor of a substance, the smaller the number of samples that can be tested at one session before the panelists should rest. Judges will experience psychological fatigue and de-sensitizing of olfactory organs if too many samples are tested at one session.

Harries (47) reported the existence of positional bias in the sensory assessments of food quality by panelists and found a tendency of panelists to treat end samples in a line differently from other samples. This bias can be reduced by care in the physical presentation of samples. Arrangement of samples on the table should be varied so that no one order predominates (51).

Error in an experiment may be reduced by replication of each test unit and by randomized coding of samples (33, 47). Replication reduces error due to variation of judges from day to day. Randomized coding reduces bias that might occur as a result of the way the samples are identified (48).

Peryam (34) reported that in testing a group of samples there is competition between adaptation and memory. If the time interval between samples is lengthened to permit de-adaptation of the sense organ, a greater burden is placed on flavor or odor memory. When samples are presented simultaneously, timing is left up to the judges.

Several researchers (33, 34, 44, 48) in studying sensory evaluation, have found that panel members tend to use all available information in making their judgments. All incidental differences between samples that might be correlated with the factor being tested should be eliminated.

Size, temperature, texture, appearance, and color of samples should be controlled so as not to bias judgments. Color should be masked unless it is a factor being tested. Utensils used should be uniform in color, size, shape, and texture and should be completely odorless.

The selection of panel members is very important in sensory testing of foods. If possible, panel members should be selected on the basis of superior ability to detect sensory differences. They should exhibit intelligence, comprehension, concentration, sustained motivation toward testing, and a keen interest in the problem under study (49).

Variability among panelists and of each panel member from day to day is a serious limitation in sensory testing. Several researchers (34, 38, 49, 50, 51) have studied the problem and have reported a great variability in the smell acuity of individuals. Odor differences always detected by some persons may never be detected by others. There is also the possibility that the smell acuity of an individual may change from time to time. Mitchell (50) reported that subjects did better on sensory tests during the earlier part of the week when they were more rested. Subjects did poorer on taste tests in the very early morning and late afternoon hours.

Blakeslee (52) reported that associations appear to have an influence on one's reaction to the pleasantness and strength of odor. Odors which are strong are often perceived as unpleasant, but what is strong to one may be weak to another. Dawson et al. (49) found that olfactory thresholds were not related to food intake because variations did not occur when the noon meal was omitted.

There is the possibility that variations in smell acuity may be caused by unequal familiarity with materials used, degree of hunger, preference values, diet, smoking, psychological factors, and health (33, 49). Schnieder (53) reported that olfactory acuity was impaired by the presence of a high degree of nasal swelling and obstruction. Acuity was also impaired when the mucosa was pale, dry, and shrunken. A moderate degree of swelling, redness, and wetness of the nasal passage was associated with good smell acuity. Data substantiated the widely held opinion that individuals vary from day to day in smell acuity.

Most researchers agree that to reduce variability among panelists, persons chosen should be in good health, have a good appetite, be free of colds and mouth and sinus infections, and should not be fatigued or under mental strain (32, 33, 49).

Discrimination of panelists may be increased by training and experience (34, 45). A training session should be held in which panel members can receive instruction and practice. Interest in the problem under study can also be aroused in the training session. However, one difficulty which often arises is that of giving enough information to create and sustain interest, but withholding any that might bias answers (33).

In the training period panelists should be presented with a series of samples differing in all the characteristics under study (33). Panel members must have a uniform understanding of the properties to be evaluated, the system of evaluation, and the difference between quality and intensity of sensory stimuli (33). Instructions to the panelists should be clear, concise, and appropriate to the experiment (49).

Testing environment has been found to have an influence on smell acuity and accuracy of judgments. Mitchell (54) reported that for optimum sensitivity, a single panelist at a time should be in the test area. Even without overt interruption, the mere knowledge of the presence of another person provided enough disruption to the subject's concentration to lower sensitivity. The study offers positive evidence of the necessity for concentration and emphasizes the importance of the psychological and the physical conditions of the testing environment. Separate booths for panel members will aid in concentration and prevent exchange of expressions between panelists and biasing of answers (44).

Results of an investigation by Stone (55) did not support the widely held opinion that temperature affects olfactory sensitivity. Results indicated that the temperature of an odorous stimulus is rapidly equilibrated with body temperature.

Most researchers emphasize the need for optimum and uniform test room conditions with no distractions (32, 42, 45, 47). The room should be free of odor and air-conditioned or have other means of proper ventilation and control of temperature. White or neutral gray walls and uniform and adjustable lighting will further minimize distractions. Adjustable lighting is especially important when the color of samples needs to be masked (44).

# CHAPTER III

## EXPERIMENTAL PROCEDURE

The primary objectives of this study were to determine (1) whether or not the odor of oxidized carotenoids could be distinguished from the odor of sweetpotato flakes of standard quality, (2) whether or not the odor of oxidized carotenoids could be distinguished from the odor of sweetpotato flakes of impaired quality, (3) whether the odor of sweetpotato flakes of impaired quality could be distinguished from the odor of carrot flakes of impaired quality as easily as the odors of the standard flakes of both foods could be distinguished from each other, (4) whether the odor of sweetpotato flakes of impaired quality could be distinguished from the odor of white potato flakes of impaired quality as easily as the odors of the standard flakes of both foods could be distinguished from each other, and (5) whether the odor of carrot flakes of impaired quality could be distinguished from the odor of white potato flakes of impaired quality as easily as the odors of the standard flakes of both foods could be distinguished from each other.

### Description of Dehydrated Food Products

The precooked, dehydrated sweetpotato and carrot flakes used in this study were prepared by the Southern Regional Research Laboratory, New Orleans, Louisiana. Sweetpotato flakes were produced from Centennial variety sweetpotatoes, using the newer raw-grind technique. This technique

essentially involves grinding the raw peeled roots, rapidly heating the puree to a starch conversion temperature and holding for a period, cooking the puree at 212°F, and then drying the puree in a double drum dryer.[1]

The dehydrated carrot flakes were produced from Imperator variety carrots. The production process involves peeling and slicing the raw carrots, cooking, then pureeing the cooked carrots, and finally drying the puree in a double drum dryer.[2]

Dehydrated white potato flakes were prepared by a commercial plant. The details of preparation were not available, but the general procedure was similar to that of the carrot flakes.[3]

Two different samples of each type of flake were used in this study, one of standard quality and one of impaired quality. The standard quality flakes of each food had been processed under the best conditions available and canned in a nitrogen atmosphere. Such flakes had no detectable off-odor. The flakes designated as being of impaired quality had been processed and packaged in an atmosphere of air and had developed off-odors. The flakes of impaired quality were also referred to as deteriorated flakes. All flakes, when received, were stored at 0°C until used in this study.

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[1]Letter from H. J. Deobald, Southern Utilization Research and Development Division, Southern Regional Research Laboratory, U. S. Department of Agriculture, New Orleans, Louisiana, March 27, 1967.

[2]Letter from A. E. Purcell, Foods Crops Laboratory, U. S. Department of Agriculture, Southern Utilization Research and Development Division, Raleigh, North Carolina, August 12, 1968.

[3]Ibid.

## Preparation of Oxidized Carotenoids

Carotenoids were isolated from sweetpotatoes by the method described by Purcell (25). Raw Centennial variety sweetpotatoes were peeled, sliced, and pureed in a Waring blender with two volumes of methanol to coagulate starch and pectin. A Hyflo-Super-Cel filter aid was added at a ratio of 2 grams per 100 grams of tissue, and the mixture was filtered through filter paper in a Buchner funnel. The filtrate was discarded. The dried mat was scraped from the filter paper into a beaker and extracted with 100 ml. of 50:50 acetone-hexane mixture. The material was filtered to dryness and washed with acetone-hexane until the filtrate was essentially colorless. The mat was again extracted by the same procedure. The filtrates were combined in a separatory funnel and allowed to stand until two distinct phases formed. The bottom phase was drained into another separatory funnel and extracted with ether. The ether fraction and the epiphase from the first separatory funnel were combined, and the mixture was washed free of acetone. This mixture was then saponified with one-fourth volume of methanol saturated with potassium hydroxide for 30 minutes. Two phases formed, with the bottom phase containing the saponified material. The saponified phase was drawn off, diluted with five volumes of water, and extracted with ether. The ether phase was combined with the nonsaponified phase and washed free of alkali. The extract was dried by filtering through anhydrous sodium sulfate under a vacuum and concentrated by means of a rotary film evaporator.

Beta-carotene crystals formed as the carotenoid-hexane mixture was concentrated. The mixture was cooled in ice water to facilitate

crystallization of beta-carotene and filtered to remove the crystallized beta-carotene. The filtrate was saved for later steps. The beta-carotene crystals were further purified by dissolving in hexane and recrystallizing several times. The supernatants, containing other carotenoids, were combined and concentrated by evaporation.

The concentrated hexane solution of carotenoids was chromatographed on a column packed with magnesium oxide (Fisher Seasorb) and Hyflo-Super-Cel at a ratio of 1:1 by weight. The column was covered with hexane, and the carotenoid mixture was added to the column and developed with hexane for two hours. When phytofluene and phytoene were distinctly separated from the other carotenes, the column was developed with a 2 per cent acetone, 98 per cent hexane mixture, then with a 5 per cent acetone, 95 per cent hexane mixture until all other pigments were separated.

The column was extruded, and the separate bands were carved out and placed into individual flasks. Thirteen fractions were obtained. A solvent mixture containing hexane, acetone, and methanol (7:2:1, v/v) was added to each flask to elute the pigments from the absorbent. Each fraction was washed with water to remove the acetone and methanol. Fraction number 3, containing beta-carotene not removed by crystallization earlier, was concentrated in a rotary film evaporator. Beta-carotene crystals formed and were removed by filtration. The filtrate, or beta-carotene supernatant, and the beta-carotene crystals were placed into separate vials and designated as 3S and 3P, respectively.

All other fractions were evaporated to dryness in a rotary film evaporator. Each was separately redissolved in 10 ml. of ether and distributed into vials. The ether was evaporated from each vial with hot

air. It was found that the carotenoid fractions obtained did not correspond exactly to those obtained previously in this laboratory from sweetpotatoes of the Goldrush variety (56), and a spectroanalysis was made to identify the fractions.

The special curves of the fractions were obtained with a Cary Model 15 recording spectrophotometer. Identifications of the various fractions were made on the basis of chromatographic behavior, partition coefficients, presence of epoxides, acid induced changes in spectra, and partition coefficients of absorption spectra.

All fractions were labeled according to their position in ascending order on the chromatographic column. They were saturated with oxygen, covered tightly with lids, and stored in a refrigerator until used.

## Preliminary Tests

Preliminary tests were conducted to determine (1) if the score cards that had been developed were effective, (2) if the testing environment was suitable, (3) if test methods chosen were suitable, and (4) how many samples could be smelled by panelists before they experienced olfactory fatigue. It was found that a paired test was suitable for comparing the odors of oxidized carotenoids and standard and deteriorated sweetpotato flakes. A triangle test appeared to be best for comparing the odors of standard and deteriorated samples of the three dehydrated food products.

Although separate booths were not available for use with the test panel, it was found that testing could be carried out effectively when

test units were placed on small tables which faced the walls of the testing room and were some distance apart. Venetian blinds allowed the room to be darkened for masking color differences in samples.

The score card tested for use in the paired test was found to be adequate. However, adjustments had to be made in the score card used for the triangle test. The score card used in preliminary triangle tests asked only that judges choose the odd sample of three. It was found that more questions needed to be asked about the relationship of the odors of the three samples in order that objectives of the test might be achieved.

Panel members agreed that as many as seven units could be tested at each session with the paired test. With the triangle test, six units could be tested at each session before the panel members became fatigued.

## Preparation of Samples

All dehydrated sweetpotato, white potato, and carrot flake samples used in this study were prepared as wet samples because preliminary tests revealed that olfactory evaluation could be made more easily and accurately when samples were wet. Each sample was prepared by mixing one-half cup of dehydrated flakes with one-third cup of lukewarm water in a 10-ounce, nonodorous, plastic glass. Red glasses were used to aid in masking color differences of samples. This precaution was necessary because of the vast color differences between white potato, sweetpotato, and carrot flakes and between the standard and deteriorated samples of each type of flake. It was believed that this color difference, if not masked, would influence the judgments of the panel members. A piece of

aluminum foil was fitted over each glass and a one-inch hole was cut in it to allow the panel members to sniff each sample. This was a further precaution to mask color differences, as well as texture differences.

During testing the oxidized carotenoids were left in the small vials in which they had been placed after isolation from the sweetpotatoes. No attempt was made to mask color differences since the nature of this part of the test did not appear to necessitate such a precaution. The vials were covered with lids which the panel members were asked to remove for sniffing and to replace after they were through testing so that the original potency of the carotenoid fractions could be kept throughout the study. Results of a previous study in this laboratory indicated that excessive deterioration and loss of volatile compounds had occurred in the oxidized carotenoids which were left uncovered for long periods (56). Since there was a good possibility that the deterioration and loss of volatiles affected results of that study, special precautions were taken in the present study to protect the oxidized carotenoids.

For the triangle test dehydrated food flake samples were coded with capital letters. Letters were selected randomly from a box and assigned to samples in the order drawn. Samples of sweetpotato flakes and oxidized carotenoids used in the paired test were not coded because panelists were asked only if the odors of samples were alike or different. The trays on which samples were placed in the paired tests were numbered so that test units could be identified by the researcher.

## Selection and Training of Panel

The test panel was composed of fourteen members ranging from twenty-one to sixty years of age. Three panel members were age 35 or older. All were associated with the School of Home Economics either as students, teachers, or other school personnel. All panel members were chosen on the basis of their willingness to serve on the panel and their interest in the problem.

Seven of the panel members had no experience in test panels. The other seven had been on test panels in experimental food classes or in food or nutrition studies. Two of the panel members participated on the test panels used in previous research in this laboratory on off-flavor and off-odor development in dehydrated sweetpotato flakes (56).

All panel members stated they were in good health. Six panel members reported they never had sinus or other nasal trouble that interfered with their sense of smell. One member stated that she had sinus infection frequently, while the other seven said they seldom had such difficulty. Only one panel member was a smoker.

A training session was conducted to acquaint panel members with the purpose of the study and procedures to follow during testing. A preliminary test revealed that panel members confused a difference in intensity of odor with a difference in quality of odor. Difference in intensity of odor and quality of odor was demonstrated, and it was emphasized that quality, and not intensity, should be the basis for determining odor differences when making judgments. Panel members were shown the proper way to sniff samples and were told to replace covers on samples after sniffing.

Panel members were asked not to talk or make unnecessary facial expressions during testing. The panel member who smoked was asked not to smoke one-half hour before or during testing.

## Design of the Experiment

Daily evaluation sessions were held for seven weeks during April and May, but each panelist was scheduled to evaluate only three days each week. Evaluation times were Monday and Wednesday, 2:00 to 3:00 P.M.; Tuesday, 1:30 to 2:30 P.M.; Thursday, 2:15 to 3:00 P.M.; and Friday, 9:00 to 10:00 and 10:00 to 11:00 A.M. Seven panel members evaluated samples during each time period, but no more than three panel members were in the testing room at the same time. The maximum time needed for each panel member to complete evaluation of all test units was usually 15 to 20 minutes.

At each session individual trays containing samples and score cards for each test unit were placed on small tables. A chair was provided at each table. After completing a test unit at one table, each panel member placed the score card face down under the tray and moved to another table. Moving from table to table presented no apparent problem since only two or three panel members were present at one time. The arrangement of the samples on trays and trays on the tables were varied at each session. Three replications were made of each test unit.

Paired tests were used in the first series of evaluations in which the odor of oxidized carotenoids were compared with the odor of dehydrated sweetpotato flakes of standard quality and the odor of sweetpotato flakes

of impaired quality. Seven test units were evaluated at each session. Each test unit consisted of a carotenoid sample and a wet sample of either standard or deteriorated sweetpotato flakes. The panel members were asked if the odors of the two samples were alike or different (see Score Card, Appendix I).

In a second series of testing, the triangle test method was used and six test units were evaluated in each session. In this series odors of dehydrated white potato, sweetpotato, and carrot flakes of standard quality were compared with each other. The odors of deteriorated flakes of the same foods were also compared with each other. Each test unit consisted of two identical wet samples of one food flake and one sample of another flake. Standard samples of one flake were compared with a standard sample of another flake, or deteriorated samples of one flake were compared with a deteriorated sample of another flake. Panel members were first asked to identify the odd sample in the triangle. They were then asked if they detected an off-odor and, if so, in which sample or samples. They were told to consider as an off-odor any odor which was unpleasant to them or which they did not associate with the food being tested. They were also asked if they did or did not find it difficult to detect a difference in the odors of samples (see Score Card, Appendix I).

# CHAPTER IV

## RESULTS

Paired tests and triangle tests were given to fourteen panel members to determine if the oxidation of the carotenoids is involved in off-odor development in dehydrated vegetable flakes. Table 1 presents the results of paired tests to determine whether or not panel members could distinguish between the odors of oxidized carotenoid fractions and the odor of standard quality, dehydrated sweetpotato flakes. Three replications were made of the tests, and a summary of all three is presented.

None of the oxidized carotenoid fractions were judged markedly like standard sweetpotato flakes in odor by the panel members. The odors of fractions 1, 2, 3P, and 3S were found like the odor of standard sweetpotato flakes in only 4.76 to 18.60 per cent of the total judgments and different in 81.40 to 94.24 per cent of the judgments. Fractions 4, 5, 6, and 12 were found like the standard sweetpotato flakes in odor in 32.55 to 39.53 per cent of the judgments and different in 60.47 to 67.45 per cent. The odors of fractions 7, 9, 10, 11, and 13 were evaluated like the standard sweetpotato flakes in slightly less than 50 per cent of the total judgments.

Fraction 8 was judged like the standard flakes in odor in 65.85 per cent of the judgments and different in 34.15 per cent. This was the only comparison in which the per cent of judgments finding the odors alike was higher than 50 per cent. Fraction 8 was the only oxidized carotenoid

TABLE 1

ABILITY OF PANEL MEMBERS TO DISTINGUISH ODOR OF STANDARD SWEETPOTATO FLAKES FROM ODOR OF OXIDIZED CAROTENOIDS[a]

| Fraction Number | Designated Oxidized Carotenoids | Percentage of Responses[b] | |
|---|---|---|---|
| | | Alike | Different |
| 1 | Phytoene | 4.76 | 95.24 |
| 2 | Phytofluene | 6.67 | 93.03 |
| 3P | Beta-carotene (pure crystals) | 18.60 | 81.40 |
| 3S | Beta-carotene (supernatant or impure) | 10.00 | 90.00 |
| 4 | Zeta-carotene | 32.55 | 67.45 |
| 5 | Mixture of carotenoid epoxides and sterols | 32.55 | 67.45 |
| 6 | Alpha-5', 6'-epoxide | 37.20 | 62.80 |
| 7 | Beta-5, 8-epoxide (mutatochrome) | 45.23 | 54.77 |
| 8 | Cis-mutatochrome and unidentified compound | 65.85 | 34.15 |
| 9 | Pro gamma-carotene | 47.61 | 52.39 |
| 10 | Gamma-carotene and 2 pro gamma-carotene | 50.00 | 50.00 |
| 11 | Various carotene epoxides and some other unidentified components | 42.86 | 57.14 |
| 12 | Monohydroxy carotenes | 39.53 | 60.47 |
| 13 | Polyhydroxy carotenes | 46.51 | 53.49 |

[a]Per cent of responses of panel members on paired tests.

[b]Based on either 40, 42, or 43 judgments.

fraction which resembled standard sweetpotato flakes in odor according to judgments of panel members.

The results of paired tests to determine if panel members could distinguish between the odors of oxidized carotenoid fractions and the odor of sweetpotato flakes of impaired quality is shown in Table 2. Fractions 1, 2, and 3S were judged like the impaired sweetpotato flakes in odor in only 9.52 to 16.66 per cent of the judgments and different in 83.34 to 90.48 per cent. Fractions 3P, 5, 6, and 7 were judged like the impaired flakes in 26.19 to 38.09 per cent of the judgments and different in 61.91 to 73.81 per cent of the judgments. These results indicate that oxidized carotenoid fractions 1 through 7 were not like the impaired sweetpotato flakes in odor.

Fractions 4, 9, 10, and 12 were found like the impaired sweetpotato flakes in 43.90 to 53.49 per cent of the judgments which indicates that panel members could not determine whether or not these fractions were like impaired sweetpotato flakes in odor. Fractions 11 and 13 were judged like the impaired flakes in 68.29 and 60.47 per cent of the evaluations, respectively, indicating that these fractions may resemble impaired sweetpotato flakes in odor.

Carotenoid fraction 8 was evaluated like impaired sweetpotato flakes in odor in 82.50 per cent of the judgments and different in 17.50 per cent. This result clearly indicates that fraction 8 was like impaired sweetpotato flakes in odor. Fraction 8 was also evaluated like the standard sweetpotato flakes in odor by a large per cent of judgments. However, the percentage of judgments finding fraction 8 like impaired sweetpotato flakes in odor was much higher than the percentage finding

TABLE 2

ABILITY OF PANEL MEMBERS TO DISTINGUISH ODOR OF IMPAIRED SWEETPOTATO FLAKES FROM ODOR OF OXIDIZED CAROTENOIDS[a]

| Fraction Number | Designated Oxidized Carotenoids | Percentage of Responses[b] | |
|---|---|---|---|
| | | Alike | Different |
| 1 | Phytoene | 9.52 | 90.48 |
| 2 | Phytofluene | 11.63 | 88.37 |
| 3P | Beta-carotene (pure crystals) | 26.19 | 73.81 |
| 3S | Beta-carotene (supernatant or impure) | 16.66 | 83.34 |
| 4 | Zeta-carotene | 43.90 | 56.10 |
| 5 | Mixture of carotenoid epoxides and sterols | 38.09 | 61.91 |
| 6 | Alpha-5', 6'-epoxide | 33.33 | 66.67 |
| 7 | Beta-5, 8-epoxide (mutatochrome) | 35.71 | 64.29 |
| 8 | Cis-mutatochrome and unidentified compounds | 82.50 | 17.50 |
| 9 | Pro gamma-carotene | 53.49 | 46.51 |
| 10 | Gamma-carotene and 2 pro gamma-carotene | 51.16 | 48.84 |
| 11 | Various carotene epoxides and some other unidentified components | 68.29 | 31.71 |
| 12 | Monohydroxy carotenes | 50.00 | 50.00 |
| 13 | Polyhydroxy carotenes | 60.47 | 39.53 |

[a]Per cent of responses of panel members on paired tests.

[b]Based on either 40, 42, or 43 judgments.

the fraction like standard sweetpotato flakes. It was predetermined in the study that the percentage of judgments finding the odors of two samples alike must reach the 75 per cent level before it could be concluded that the samples do have the same odor. The comparison of fraction 8 and impaired sweetpotato flakes was the only one in which the odors of the two samples were evaluated alike in more than 75 per cent of the total judgments.

Table 3 presents the results of triangle tests to determine whether the odor of sweetpotato flakes of impaired quality could be distinguished from the odor of carrot flakes of impaired quality as easily as the odors of the standard flakes of both foods could be distinguished from each other. The phrase "as easily as," used here and elsewhere in this paper, is a relative term. It was determined by the percentage of judgments which distinguished the odd sample correctly in a triangle test comparing the odors of impaired flakes of two foods as opposed to the percentage of judgments distinguishing the odd sample correctly in tests comparing standard flakes of the same two foods. Panel members were also asked to indicate on the score card whether or not they found it difficult to distinguish a difference in odors of the samples.

The odd sample was chosen correctly in 84.36 per cent of the judgments when carrot flakes of impaired quality were compared with sweetpotato flakes of impaired quality. In comparing the odors of the standard flakes of carrot and sweetpotato with each other, the odd sample was chosen correctly in 83.75 per cent of the total judgments made. In comparing the impaired quality flakes, only 12.19 per cent of judgments indicated difficulty in distinguishing a difference in odors of samples

TABLE 3

ABILITY OF PANEL MEMBERS TO DISTINGUISH ODOR OF CARROT FLAKES FROM ODOR OF SWEETPOTATO FLAKES[a]

| Comparisons | Percentage of Responses[b] | | |
|---|---|---|---|
| | Distinguished Correctly | Did Not Distinguish Correctly | Stated Difficulty[c] |
| Impaired carrot flakes and impaired sweetpotato flakes | 85.36 | 14.64 | 12.19 |
| Standard carrot flakes and standard sweetpotato flakes | 83.75 | 16.25 | 38.75 |

[a]Per cent of responses of panel members on triangle tests.

[b]Based on 80 or 82 judgments.

[c]Judgments on which panel members stated difficulty in distinguishing a difference between odors of samples.

as opposed to 38.75 per cent of judgments on which panel members stated difficulty in distinguishing between odors of the standard flakes of carrot and sweetpotato.

The results of triangle tests to determine whether the odor of sweetpotato flakes of impaired quality could be distinguished from the odor of white potato flakes as easily as the odor of the standard flakes of both foods could be distinguished from each other is presented in Table 4. In comparing white potato and sweetpotato flakes of impaired quality, panel members made correct judgments in 95 per cent of the total judgments. The odd sample was identified correctly in 87.50 per cent of

TABLE 4

ABILITY OF PANEL MEMBERS TO DISTINGUISH ODOR OF WHITE POTATO FLAKES FROM ODOR OF SWEETPOTATO FLAKES[a]

| Comparisons | Percentage of Responses[b] | | |
|---|---|---|---|
| | Distinguished Correctly | Did Not Distinguish Correctly | Stated Difficulty[c] |
| Impaired white potato flakes and impaired sweetpotato flakes | 95.00 | 5.00 | 11.25 |
| Standard white potato flakes and standard sweetpotato flakes | 87.50 | 12.50 | 23.75 |

[a]Per cent of responses of panel members on triangle tests.

[b]Based on 80 or 82 judgments.

[c]Judgments on which panel members stated difficulty in distinguishing a difference between odors of samples.

judgments when standard white potato flakes were compared with standard sweetpotato flakes. Difficulty in distinguishing a difference between odors of impaired quality flakes was indicated in 11.25 per cent of judgments; whereas, difficulty in distinguishing between the standard flakes was indicated in 23.75 per cent of total judgments. The percentage of judgments in which the odd sample was chosen correctly was greater for the impaired flakes than for the standard flakes. The percentage of judgments indicating difficulty in distinguishing any difference in odors of the flakes was much higher in comparing the standard flakes than in comparing the impaired flakes of white potatoes and sweetpotatoes. These

results suggest that panel members could distinguish slightly more easily between the odors of impaired flakes than they could distinguish between the odors of standard sweetpotato and white potato flakes.

The results of triangle tests to determine whether the odor of carrot flakes and white potato flakes of impaired quality could be distinguished from each other as easily as the standard flakes of the same foods could be distinguished from each other is shown in Table 5. In

TABLE 5

ABILITY OF PANEL MEMBERS TO DISTINGUISH ODOR OF CARROT FLAKES FROM ODOR OF WHITE POTATO FLAKES[a]

| Comparisons | Percentage of Responses[b] | | |
|---|---|---|---|
| | Distinguished Correctly | Did Not Distinguish Correctly | Stated Difficulty[c] |
| Impaired carrot flakes and impaired white potato flakes | 92.50 | 7.50 | 7.50 |
| Standard carrot flakes and standard white potato flakes | 93.75 | 6.25 | 26.83 |

[a]Per cent of responses of panel members on triangle tests.

[b]Based on 80 or 82 judgments.

[c]Judgments on which panel members stated difficulty in distinguishing a difference between odors of samples.

comparing the odors of impaired white potato flakes with the odor of impaired carrot flakes, the odd sample was chosen correctly in 92.50 per cent of the judgments. The odd sample was chosen correctly in 93.75 per cent of the judgments in comparing the odors of the standard flakes with each other. In 7.50 per cent of judgments, difficulty was indicated by panel members in distinguishing a difference between odors of samples of the impaired flakes as opposed to 26.83 per cent of judgments in which difficulty was indicated by panel members in distinguishing a difference between the odors of the standard flakes.

In general, panel members indicated more difficulty in distinguishing a difference between odors of samples when comparing the odor of standard flakes of white potato, sweetpotato, and carrots with each other than in comparing the impaired flakes of the same foods. However, the percentage of judgments in which the odd sample was chosen correctly was not significantly higher when comparing impaired flakes than when comparing the standard flakes.

In the triangle tests comparing carrot flakes, sweetpotato flakes, and white potato flakes, panel members were asked to indicate samples in which they detected off-odors. They were instructed to consider as an off-odor any odor that was unpleasant to them or any odor which they did not associate with the foods used in this study. Table 6 presents the percentages of total judgments in each comparison which indicated off-odors in any of the samples used in that comparison.

When comparing carrot flakes and sweetpotato flakes, off-odor was indicated in the standard carrot flakes in 42.86 per cent of the judgments and in the impaired carrot flakes in 82.14 per cent of the judgments. In

TABLE 6

DETECTION OF OFF-ODORS IN PRECOOKED DEHYDRATED SWEETPOTATO, WHITE POTATO, AND CARROT FLAKES BY PANEL MEMBERS[a]

| Flakes Compared | Percentage of Responses Detecting Off-odor[b] | |
|---|---|---|
| | Standard Flakes | Impaired Flakes |
| Carrot flakes and Sweetpotato flakes | 42.86 | 82.14 |
| | 15.48 | 21.43 |
| Carrot flakes and White potato flakes | 27.38 | 48.81 |
| | 63.10 | 53.57 |
| Sweetpotato flakes and White potato flakes | 27.38 | 20.24 |
| | 53.57 | 73.81 |

[a]Per cent of responses of panel members.

[b]Based on 80 or 82 judgments.

the standard sweetpotato flakes, off-odor was detected in 15.48 per cent of judgments; whereas, off-odor was detected in the impaired flakes in 21.43 per cent of judgments.

Panel members indicated off-odor in the standard carrot flakes in 27.38 per cent of judgments when comparing carrot flakes and white potato flakes, while 48.81 per cent of judgments indicated off-odor in the impaired carrot flakes. Off-odor was detected in the white potato flakes by 63.10 per cent of the judgments and in the impaired white potato flakes by 53.57 per cent of the judgments.

In comparing sweetpotato flakes and white potato flakes, 27.38

per cent of the judgments indicated off-odor in the standard sweetpotato flakes and 20.24 per cent indicated off-odor in the impaired sweetpotato flakes. Off-odor was indicated in the standard white potato flakes by 53.57 per cent of the judgments, but with the impaired white potato flakes, off-odor was indicated by 73.81 per cent of the judgments. Results of this part of the study were irregular and inconsistent. Since no training was given to panel members on what to consider as an off-odor, the "rancid" or "hay-like" odors of deteriorated, dehydrated foods described by other researchers (3, 18, 22, 23, 26, 27) were probably not the only odors which determined panel members' answers. Since it was not known what the panel members considered as off-odors, use of the results of this part of the study was limited.

# CHAPTER V

# DISCUSSION

Results of sensory evaluation clearly indicate that oxidized carotenoid fractions 1, 2, 3P and 3S (phytoene, phytofluene, crystalline beta-carotene, and beta-carotene supernatant, respectively) are different from standard sweetpotato flakes in odor. The odor of the pure crystals of beta-carotene was found to be slightly more similar to the standard flakes than the beta-carotene supernatant.

The other oxidized carotenoid fractions, with the exception of fraction 8, were not evaluated markedly like or different from standard sweetpotato flakes in odor. Fraction 8 (cis-mutatochrome and unidentified compounds) was evaluated like the standard sweetpotato flakes by a large enough percentage of judgments to indicate that there is a similarity in odor.

Carotenoid fractions 1, 2, 3 pure crystals, and 3 supernatant were also found to be distinctively different from impaired sweetpotato flakes in odor. Although none of these fractions were judged like the impaired flakes in odor, the beta-carotene crystals were judged to be more nearly like the impaired flakes than the supernatant of beta-carotene. Results of Purcell (25) and Jones (56) indicated that the supernatant of beta-carotene resembled the impaired flakes in odor more than the beta-carotene crystals did.

Panel members were not able to decide conclusively whether or not the remaining oxidized carotenoid fractions, with the exception of fraction

11, were like the impaired sweetpotato flakes in odor. Fraction 11, composed of various carotene epoxides and some unidentified compounds, was judged to be similar to the impaired sweetpotato flakes, but not definitely like the impaired flakes. Since panel members could not definitely distinguish the odors of these carotenoid fractions from the odor of impaired sweetpotato flakes, it appears that some component of the fractions might be like the impaired flakes in odor. Results of Purcell (25) indicated that compounds associated with the carotenoids might be the primary cause of undesirable sensory changes in dehydrated sweetpotato flakes.

The odor of fraction 8 was found to be conclusively like the impaired sweetpotato flakes in odor. This result indicated that oxidized carotenoid fraction 8 may be a cause of off-odor development in impaired sweetpotato flakes. This fraction is composed of cis-mutatochrome and several unidentified compounds. Off-odor may be caused by any one or all of the components making up fraction 8.

A second series of sensory evaluation to determine if the oxidized carotenoids are the cause of off-odor development in dehydrated foods revealed that the odors of sweetpotato flakes, carrot flakes, and white potato flakes of standard quality could easily be distinguished from each other. The impaired samples of the same flakes were as easily distinguishable from each other as the standard flakes.

In spite of the fact that both sweetpotato and carrot flakes contain large amounts of the same carotenoids, they did not become more alike in odor as they deteriorated. The flakes actually became slightly more easily distinguished from each other as they deteriorated. Although white potato flakes contain only minute amounts of the carotenoids, they

did not become markedly more distinguishable from carrot and sweetpotato flakes as all three flakes deteriorated. These results indicate that compounds other than the carotenoids are the precursors of off-odor development and that these compounds are peculiar to each type of flakes used in this study. Several researchers (3, 4) have attributed off-odor and off-flavor development in dehydrated white potato flakes to the autoxidation of fat fractions in the potato.

The results of this study clearly indicate that the odor of oxidized beta-carotene is different from the odor of standard dehydrated sweetpotato flakes and the odor of impaired sweetpotato flakes. Even though more than 90 per cent of the carotenoid content of sweetpotatoes is beta-carotene, under the conditions of this study, it appears that beta-carotene is not the precursor of off-odor in dehydrated sweetpotato flakes. This finding is in agreement with results of Purcell (25) and with the results of a previous study in this laboratory (56). On the other hand, several researchers (18, 22, 27, 30) have found a correlation between degradation of beta-carotene and off-odor development.

Furthermore, the beta-carotene supernatant was not found to be like impaired sweetpotato flakes in odor, which suggests that this fraction is not a cause of off-odor development. In contrast, Purcell (25) reported that beta-carotene supernatant did have an odor resembling deteriorated sweetpotato flakes.

The only oxidized carotenoid fraction found in this study to be like the impaired sweetpotato flakes in odor was fraction number 8. It was concluded that either the entire fraction or some component of it may be responsible for off-odor development in dehydrated sweetpotato flakes.

Results of the triangle test indicated that sweetpotato flakes and carrot flakes, both containing large amounts of the same carotenoids, did not become more alike in odor as they deteriorated. On the basis of this result, it appears that the carotenoids are not the precursors of off-odor development in dehydrated vegetables flakes. Also, white potato flakes, which contain only minute amounts of the carotenoids, did not become more unlike the carrot and sweetpotato flakes as the three flakes deteriorated in odor.

Under the conditions of this study, it appears that the carotenoids themselves, with the exception of fraction number 8, are probably not the precursors of off-odor development in precooked, dehydrated sweetpotato, carrot, and white potato flakes. Fraction 8, or some compound associated with it, may be responsible for off-odor development in precooked, dehydrated sweetpotato flakes. Also, results indicate that some compound associated with carotenoid fractions 4 through 7 (zeta-carotene, carotenoid epoxides and sterols, alpha-5, 8-epoxide, and beta-5, 8-epoxide) and 9 through 13 (pro gamma-carotene, gamma-carotene and 2 pro gamma-carotene, various carotene epoxides and some other unidentified compounds, monohydroxy carotenes, and polyhydroxy carotenes) might be involved in off-odor development in dehydrated food flakes.

# CHAPTER VI

# SUMMARY AND RECOMMENDATIONS

## Summary

Paired tests were used for olfactory comparison of oxidized carotenoids and precooked, dehydrated sweetpotato flakes. Triangle tests were used for olfactory evaluation of precooked, dehydrated sweetpotato flakes; precooked, dehydrated white potato flakes; and precooked, dehydrated carrot flakes. The purpose of the investigation was to determine if any of the carotenoids tested are the precursors of off-odor in dehydrated foods which contain them.

The paired tests were used to determine by sensory evaluation if the odors of the oxidized carotenoid fractions and the odors of standard and impaired sweetpotato flakes are alike or different. The triangle tests were used to compare the odors of standard, precooked, dehydrated sweetpotato, white potato, and carrot flakes with each other. Also, the odors of impaired sweetpotato, white potato, and carrot flakes were compared with each other in triangle tests. The purpose of this part of the study was to determine whether or not sweetpotato flakes and carrot flakes, both containing large amounts of the carotenoids, become more alike in odor as they deteriorate and to determine whether or not white potato flakes, containing only minute amounts of the carotenoids, become less like the carrot and sweetpotato flakes as they deteriorate. Panel members were asked to choose the odd sample and to indicate whether or not they had difficulty in distinguishing between the odors of samples.

Responses of panel members were recorded on score cards.

Results of the study indicate that there was a conclusive difference between the odors of fractions 1, 2, 3 purified, and 3 supernatant (phytoene, phytofluene, beta-carotene crystals, and beta-carotene supernatant) and good and poor sweetpotato flakes. Results indicate that fraction 8 (cis-mutatochrome and unidentified compounds) was conclusively like the impaired sweetpotato flakes in odor. The other carotenoid fractions were found to be similar to both standard and impaired sweetpotato flakes. However, all fractions except 6 (alpha-5', 6'-epoxide) and 7 (beta-5, 8-epoxide) were judged to be more similar to impaired flakes than to the standard flakes in odor.

The odors of impaired sweetpotato flakes and impaired carrot flakes were no more similar to each other than the odors of the good flakes. Both standard flakes and impaired flakes of carrot and sweetpotato could be easily distinguished from each other by panel members. There was some evidence that carrot and sweetpotato flakes became less alike as they deteriorated in odor. White potato flakes, both standard and impaired, were easily distinguishable from carrot and sweetpotato flakes of the same qualities. The white potato flakes did not become less like the carrot and sweetpotato flakes as all three flakes deteriorated.

Under the conditions of this study, it appears that the first three carotenoid fractions, including beta-carotene crystals and beta-carotene supernatant, are not the precursors of off-odor in precooked, dehydrated sweetpotato flakes. It also appears that off-odor development in precooked, dehydrated sweetpotato, carrot, and white potato flakes is caused by different compounds peculiar to each product. Evidence indicates that

compounds associated with the carotenoids, especially fraction 8, may be precursors of off-odor in precooked, dehydrated foods.

## Recommendations for Additional Investigation

Results of this study showed that keeping samples of carotenoids refrigerated and tightly covered, except while panel members were sniffing, greatly aided in preventing deterioration of carotenoid samples. The procedure also maintained the head space of the vials at a more concentrated level which aided panel members in evaluating. It is recommended that this precaution be taken in any future studies of this nature.

Although the use of red glasses and a darkened room greatly aided in masking color differences of samples, the use of red lights in the testing room might prove more effective. The use of individual testing booths for panel members might aid in increasing reliability of the results in studies of this type.

Sensory tests to determine the description of off-odor which develops in dehydrated carrot flakes and white potato flakes could possibly give insight into what is producing the off-odors. Previous tests were made in this laboratory (56) to obtain a description of the off-odor in precooked, dehydrated sweetpotato flakes.

Since fraction 8 was found to be conclusively like the impaired sweetpotato flakes in odor, it is desirable that this fraction be further analyzed by chromatography and other means to separate and identify its components. These components might be separately compared with impaired dehydrated sweetpotato flakes in sensory evaluation tests to determine

which of the components are involved in off-odor and off-flavor development in dehydrated foods. Some of the other carotenoid fractions, particularly 9 through 13, which were found to highly resemble the impaired sweetpotato flakes in odor, might be further analyzed in a manner similar to fraction 8.

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# APPENDIX I

## SCORECARDS AND PERSONAL DATA SHEET

SCORE CARDS FOR ODOR DETECTION

Tray No. _____ Name _____

Date _____

PAIRED TEST FOR ODOR DETECTION

1. Circle one word below which describes the relationship of the odor of the two samples.

ALIKE DIFFERENT

---

Name _____

Date _____

TRIANGLE TEST FOR ODOR

1. Two of the samples are identical in odor and one sample is different. Circle the letter of the odd sample.

_____ _____ _____

2. Can you detect an off-odor in any of the samples?

Yes _____ No _____

3. If so, circle the letter or letters of the sample or samples in which you detect off-odor.

_____ _____ _____

4. Check one: _____ I found it difficult to detect any difference in odor.

_____ I did not find it difficult to detect a difference in odor.

PERSONAL DATA

Name: _____

Address: _____

Phone Number: _____

Age: _____ Sex: _____

Occupation: _____

Department or Major: _____

Experience in Test Panels: _____

_____

_____

Health: Good _____ Fair _____ Poor _____

Do you have sinus or other nasal trouble which interferes with your sense of smell?

Never _____ Frequently _____ Seldom _____

Do you smoke? Yes _____ No _____

Any sessions you know of now that you will have to miss? _____

(give dates) _____

# APPENDIX II

# ODOR DETECTION DATA

## DATA TABLES

### TABLE 1

ABILITY OF PANEL MEMBERS TO DISTINGUISH ODOR OF STANDARD SWEETPOTATO FLAKES FROM ODOR OF OXIDIZED CAROTENOIDS[a]

| Position Number | Oxidized Carotenoids | Responses[b] | | | |
|---|---|---|---|---|---|
| | | Alike | | Different | |
| | | Number | Per Cent | Number | Per Cent |
| 1 | Phytoene | 2 | 4.76% | 40 | 95.24% |
| 2 | Phytofluene | 3 | 6.67 | 40 | 93.03 |
| 3 | Beta-carotene (pure crystals) | 8 | 18.60 | 35 | 81.40 |
| 3 | Beta-carotene (supernatant or impure) | 4 | 10.00 | 36 | 90.00 |
| 4 | Zeta-carotene | 14 | 32.55 | 29 | 67.45 |
| 5 | Mixture of carotenoid epoxides and sterols | 14 | 32.55 | 29 | 67.45 |
| 6 | Alpha-5', 6'-epoxide | 16 | 37.20 | 27 | 62.80 |
| 7 | Beta-5, 8-epoxide (mutatochrome) | 19 | 45.23 | 23 | 54.77 |
| 8 | Cis-mutatochrome and unidentified compound | 27 | 65.85 | 14 | 34.15 |
| 9 | Pro gamma-carotene | 20 | 47.61 | 22 | 52.39 |
| 10 | Gamma-carotene and 2 pro gamma-carotene | 21 | 50.00 | 21 | 50.00 |
| 11 | Various carotene epoxides and some unidentified components | 18 | 42.86 | 24 | 57.14 |
| 12 | Monohydroxy carotenes | 17 | 39.53 | 26 | 60.47 |
| 13 | Polyhydroxy carotenes | 20 | 46.51 | 23 | 53.49 |

[a]Per cent of responses of panel members on paired tests.

[b]Based on either 40, 42, or 43 judgments.

TABLE 2

ABILITY OF PANEL MEMBERS TO DISTINGUISH ODOR OF IMPAIRED SWEETPOTATO FLAKES FROM ODOR OF OXIDIZED CAROTENOIDS[a]

| Position Number | Oxidized Carotenoids | Responses[b] | | | |
|---|---|---|---|---|---|
| | | Alike | | Different | |
| | | Number | Per Cent | Number | Per Cent |
| 1 | Phytoene | 4 | 9.52% | 38 | 90.48% |
| 2 | Phytofluene | 5 | 11.63 | 38 | 88.37 |
| 3 | Beta-carotene (pure crystals) | 11 | 26.19 | 31 | 73.81 |
| 3 | Beta-carotene (supernatant or impure) | 7 | 16.66 | 35 | 83.34 |
| 4 | Zeta-carotene | 18 | 43.90 | 23 | 56.10 |
| 5 | Mixture of carotenoid epoxides and sterols | 16 | 38.09 | 26 | 61.91 |
| 6 | Alpha-5', 6'-epoxide | 14 | 33.33 | 28 | 66.67 |
| 7 | Beta-5, 8-epoxide (mutatochrome) | 15 | 35.71 | 27 | 64.29 |
| 8 | Cis-mutatochrome and unidentified compound | 33 | 82.50 | 7 | 17.50 |
| 9 | Pro gamma-carotene | 23 | 53.49 | 20 | 46.51 |
| 10 | Gamma-carotene and 2 pro gamma-carotene | 22 | 51.16 | 21 | 48.84 |
| 11 | Various carotene epoxides and some unidentified components | 28 | 68.29 | 13 | 31.71 |
| 12 | Monohydroxy carotenes | 21 | 50.00 | 21 | 50.00 |
| 13 | Polyhydroxy carotenes | 26 | 60.47 | 17 | 39.53 |

[a]Per cent of responses of panel on paired tests.

[b]Based on either 40, 42, or 43 judgments.

TABLE 3

ABILITY OF PANEL MEMBERS TO DISTINGUISH ODOR OF CARROT FLAKES FROM ODOR OF SWEETPOTATO FLAKES[a]

| Comparisons | Responses[b] | | | | | |
|---|---|---|---|---|---|---|
| | Distinguished Correctly | | Did Not Distinguish Correctly | | Stated Difficulty[c] | |
| | Number | Per Cent | Number | Per Cent | Number | Per Cent |
| Impaired carrot flakes and impaired sweetpotato flakes | 70 | 85.36 | 12 | 14.64 | 10 | 12.19 |
| Standard carrot flakes and standard sweetpotato flakes | 67 | 83.75 | 13 | 16.25 | 31 | 38.75 |

[a]Per cent of responses of panel members on triangle tests.

[b]Based on 80 or 82 judgments.

[c]Judgments on which panel members stated difficulty in distinguishing a difference between odors of samples.

TABLE 4

ABILITY OF PANEL MEMBERS TO DISTINGUISH ODOR OF WHITE POTATO FLAKES FROM ODOR OF SWEETPOTATO FLAKES[a]

| Comparisons | Responses[b] | | | | | |
|---|---|---|---|---|---|---|
| | Distinguished Correctly | | Did Not Distinguish Correctly | | Stated Difficulty[c] | |
| | Number | Per Cent | Number | Per Cent | Number | Per Cent |
| Impaired white potato flakes and impaired sweetpotato flakes | 76 | 95.00 | 4 | 5.00 | 9 | 11.25 |
| Standard white potato flakes and standard sweetpotato flakes | 70 | 87.50 | 10 | 12.50 | 19 | 23.75 |

[a]Per cent of responses of panel members on triangle tests.

[b]Based on 80 or 82 judgments.

[c]Judgments on which panel members stated difficulty in distinguishing a difference between odors of samples.

TABLE 5

ABILITY OF PANEL MEMBERS TO DISTINGUISH ODOR OF CARROT FLAKES FROM ODOR OF WHITE POTATO FLAKES[a]

| Comparisons | Responses[b] | | | | | |
|---|---|---|---|---|---|---|
| | Distinguished Correctly | | Did Not Distinguish Correctly | | Stated Difficulty[c] | |
| | Number | Per Cent | Number | Per Cent | Number | Per Cent |
| Impaired carrot flakes and impaired white potato flakes | 74 | 92.50 | 6 | 7.50 | 6 | 7.50 |
| Standard carrot flakes and standard white potato flakes | 75 | 93.75 | 7 | 6.25 | 22 | 26.83 |

[a]Per cent of responses of panel members on triangle tests.

[b]Based on 80 or 82 judgments.

[c]Judgments on which panel members stated difficulty in distinguishing a difference between odors of samples.