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Received for review March 3, 1987. Revised manuscript received August 27, 1987. Accepted September 29, 1987.

Isomerization and Losses of *trans*- β -Carotene in Sweet Potatoes as Affected by Processing Treatments

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Carotene content was altered during the processing of sweet potatoes. The change was dependent upon the treatment employed: blanching (4.0-11.9% increase), lye peeling and pureeing (10.4% increase), steam injection (8.0% loss), canning (19.7% loss), dehydration (20.5% loss), microwaving (22.7% loss), and baking (31.4% loss). Increases in carotene content were attributed to an enhanced extraction efficiency of heat-treated samples. Heat processing induced the formation of predominately the 13-*cis*- β -carotene isomer, and the quantity formed was related to the severity and length of the heat treatment.

Carotenoids represent the most widespread group of naturally occurring pigments in nature (Simpson and Chichester, 1981). The carotenoids in food are primarily of plant origin and β -carotene, with few exceptions, predominates (Panalaks and Murray, 1970). β -Carotene serves as an important nutritional component in foods, as a major precursor of vitamin A, and provides pleasant yellow-orange colors to foods (Francis, 1969; Simpson and Chichester, 1981; Klaui and Bauernfeind, 1981). Recent interest concerning dietary β -carotene has been prompted by studies implicating the ingestion of foods containing carotenes with decreased incidence of certain cancers (Peto et al., 1981; Menkes et al., 1986).

all-trans- β -Carotene exhibits the greatest vitamin A activity of the carotenoids. β -Carotene is subject to degradative changes during food processing and cooking (Gregory, 1985). In general, oxidation is a major cause of β -carotene destruction (Simpson, 1985) while thermal processing of foods may lead to β -carotene isomerization (Sweeney and Marsh, 1971).

In 1974, Lee and Ammerman noted that, in conventional processing of carotene-rich foods, much of the provitamin A activity of the carotenes can be lost due to conversion of *all-trans*- β -carotene to stereoisomers having lower provitamin A activity. Because of the lowered biological activity of the *cis* isomers, characterization of the isomeric forms in cooked and processed products would be desirable (National Research Council, 1980). During processing, several researchers have seen a reduction in the percentage of *all-trans*- β -carotene with the concomitant increase in the 13-*cis* and 9-*cis* isomers (Panalaks and Murray, 1970; Sweeney and Marsh, 1971; Lee and Ammerman, 1974; Ogunlesi and Lee, 1979; Chandler and Schwartz, 1987).

Sweet potatoes are an excellent model system for quantitatively observing changes in β -carotene during processing because of the high concentration of β -carotene in this tissue and low concentrations of other interfering carotenes. Some investigators have studied losses of β -carotene during the handling (curing and storage) and

processing of sweet potatoes (Reddy and Sistrunk, 1980; Walter and Giesbrecht, 1982; Picha, 1985). However, isomerization reactions of β -carotene and losses during food processing and preparation have not been addressed in detail.

The objectives of this study are (1) to quantitatively follow the changes in total β -carotene concentration during various processes for sweet potatoes and (2) to quantitate the isomeric composition of β -carotene as a result of blanching, canning, dehydrating, and cooking.

MATERIALS AND METHODS

all-trans- β -Carotene (type 1) was purchased from Sigma Chemical Co. (St. Louis, MO), and 15-*cis*- β -carotene was donated by Hoffmann-LaRoche (Basel, Switzerland). Chemicals and solvents were reagent grade. Solvents used for high-performance liquid chromatography (HPLC) were filtered (0.45 μ m) and degassed. Raw sweet potatoes *Ipomoea batatas* (Jewel variety, US #1, 300-350 g) were obtained during the 1985 growing season from the North Carolina Agricultural Research Experiment Station, harvested during October, cured (30° C, 85% relative humidity) for 7 days, and then stored (Walter and Schadel, 1982) for 9 months until being processed. The Jewel variety roots are yellow-orange in color.

Processing Conditions. Figure 1 represents a schematic of the sweet potato processing procedure.

Puree. Roots (25 kg) were lye peeled (6% NaOH, 5 min, 101 °C), washed, trimmed, and pureed in a Fitzmill comminutor (Fitzpatrick Co., Chicago, IL) fitted with a 0.06-in. mesh screen. Puree was heated to 81 °C (culinary steam injection) to gelatinize the starch and held for 30 min to allow hydrolysis of the starch by the endogenous amylase enzymes. The puree was then heated (100 °C, steam injection) to inactivate the native amylases (Hoover, 1967; Walter et al., 1976).

Canning. Puree (5 kg) was filled into 303 × 406 cans and processed (116 °C, 90 min) in a still retort.

Dehydration. Drum-dried product was prepared on a 12 × 19 in. double-drum dryer (160 °C, 25 rpm) with a contact time of approximately 1.8-2 s.

Strips. Hand-peeled roots (5 kg) were cut into strips of variable length (1.9 cm wide × 0.64 cm thick) with use

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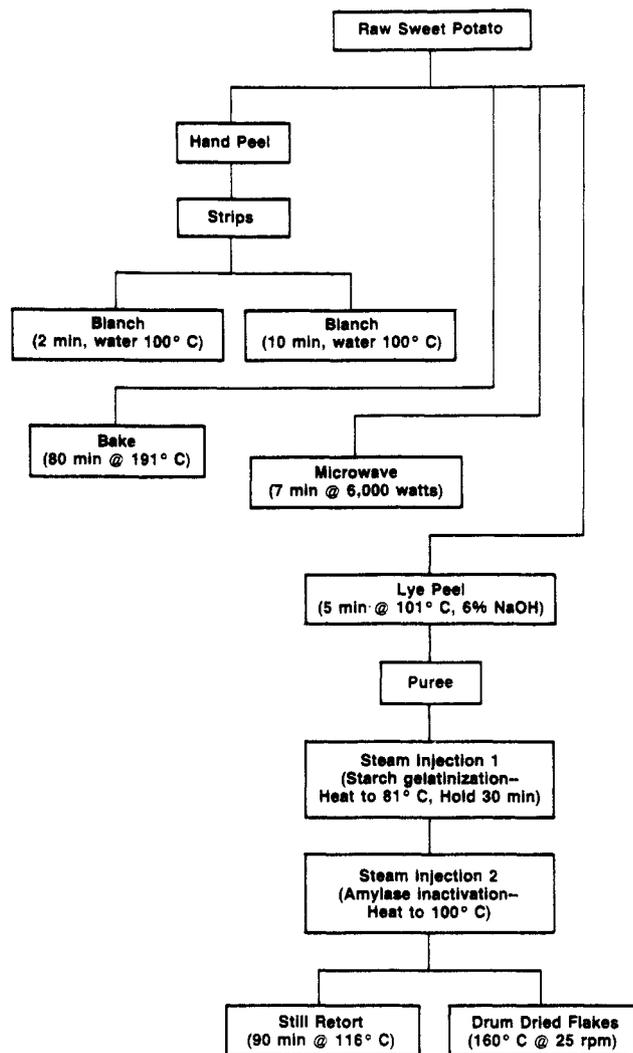


Figure 1. Schematic of sweet potato processing procedure.

of an Urschel dicer (Urschel Corp., Valparaiso, IN) and water blanched (100 °C) for 2 or 10 min.

Baking. Sweet potatoes (2.5 kg) were wrapped in aluminum foil, placed in a prewarmed conventional oven at 191 °C, and baked for 80 min until an internal temperature of 99 °C was reached. After baking, the roots were unwrapped and allowed to cool for 1 h at room temperature.

Microwaving. Roots (2.5 kg) were pierced with a fork, placed in a Litton microwave oven, microwaved at full power (6000 W) for 7 min until an internal temperature of 99 °C was reached, and allowed to cool for 1 h.

Sample Preparation. Representative samples of hand-peeled raw sweet potatoes and strips (750 g each) were cut, and 750 g of water was added and the resultant mixture pureed in a Waring blender (Waring, New Hartford, CN). Baked and microwaved potatoes (2.5 kg each) were peeled and mashed through a conical colander (2-mm pore) to form a homogeneous sample, and 2.5 kg of water was added. To pureed and canned samples, an equal weight of water was also added prior to extraction. To drum-dried flakes (10 g) was added 190 mL of water, and the resultant was allowed to rehydrate.

Solids Determination. Triplicate samples (8 g each) were dried (6 h, 70 °C, 15 psi) in a vacuum oven (National Appliance Co., Portland, OR), and the solids content was determined. Solids content of the blanch water was also measured.

Sample Extraction. To a 25-g sample was added 60 mL of methanol; the mixture was allowed to stand for 30

min and filtered. The filter cake was extracted by suspending in 70 mL of acetone-hexanes (1:1, v/v) and filtered, and the filtrate was combined with the methanol extract. The filter cake was washed until successive extracts were colorless. The extract was washed (3×) with 100 mL of water. The epilayer was removed and saponified at room temperature for 30 min with 60 mL of KOH-saturated methanol and then washed (3×, 100 mL) free of base with water. The extract was transferred quantitatively to a 100-mL volumetric flask and brought to volume. The entire extraction procedure was performed under reduced lighting conditions. Recovery studies were performed by adding 0.25 and 0.50 mg of *all-trans*- β -carotene to 25 g of raw pureed sweet potato. All samples were extracted in duplicate prior to analysis.

All samples were analyzed by the HPLC procedure outlined by Chandler and Schwartz (1987) with the following modifications. Calcium hydroxide used for column packing was sieved through a 500-mesh screen (Newark Co., Newark, NJ). To 5 g was added 1 mL of water, and the mixture was ground with a mortar and pestle. The mixture was suspended in a solution (40 mL) of acetone-hexanes (0.5:99.5, v/v) and degassed ultrasonically (3×, 10 min) prior to packing. The column was prepared for analyses by washing with acetone-hexanes (1:9, v/v) at a flow rate of 0.4 mL/min for 12 h and then equilibrated to acetone-hexanes (0.5:99.5, v/v). Samples (40 μ L) were injected and eluted isocratically for 18 min at a flow rate of 1 mL/min followed by a step gradient to acetone-hexanes (3:97, v/v). Column eluants were monitored at both 436 and 340 nm and absorbance ratio measurements determined to confirm peak purity and identity (Chandler and Schwartz, 1987). Detector sensitivity was set at 0.05 (436 nm) and 0.02 (340 nm) AUFS. Duplicate injections of each extract from two replicate samples were performed. Statistical differences were determined by the Student's *t*-test (Steel and Torrie, 1980).

Standard Solutions. A stock solution of *all-trans*- β -carotene was prepared from 15 mg of crystalline β -carotene in a 100-mL volumetric flask and brought to volume with hexanes. Appropriate aliquots were sequentially diluted to provide points for a calibration curve. Peak area vs absorbance at 453 nm ($E_{cm}^{1\%} = 2592$; De Ritter and Purcell, 1981) was used to quantitate carotene content.

Isomerization. Isomerization of *all-trans*- β -carotene was catalyzed by iodine according to Zechmeister's (1944) method. Individual isomers were identified by UV-vis spectra obtained on a Linear (Anspec Co., Inc., Ann Arbor, MI) UVIS Model 203 programmable variable-wavelength absorbance detector, in the stop-flow mode, and by comparison to previous literature (Zechmeister, 1944; Stitt et al., 1951; Panalaks and Murray, 1970; Quackenbush, 1987).

RESULTS AND DISCUSSION

Figure 2 illustrates a typical HPLC chromatogram of extracts obtained from pureed, canned, and dehydrated sweet potatoes. Three β -carotene isomers were resolved and identified as 13-*cis*- β -carotene (17 min), *all-trans*- β -carotene (23 min), and 9-*cis*- β -carotene (27.5 min) (Tsukida et al., 1982; Chandler and Schwartz, 1987). Table I lists the observed absorption maxima for each isomer in comparison to literature values. These data provide additional evidence for their identity. Total carotene content and the quantity of individual isomers were calculated from peak area measurements and calibration data. Figure 3 illustrates the calibration curve of *all-trans*- β -carotene concentration vs peak area. Peak area measurements were taken at 436 nm to allow for quantitation of all isomers, using a single calibration curve, since this wavelength is

Table I. Absorption Maxima of β-Carotene Isomers

isomer	obsd abs max (λ), nm			reported abs max (λ), ^a nm		
	A	B	C	A	B	C
15-cis	425	446	474	—	—	—
13-cis	422	445	469	423	444	469
all-trans	431	455	476	432	453	476
9-cis	425	446	473	425	446	473

^a Compiled from Zechmeister (1944), Stitt et al., (1951), Panalaks and Murray (1970), and Quackenbush (1987).

Table II. Cis/Trans β-Carotene Isomers in Raw and Processed Sweet Potatoes

sweet potato treatment ^a	% solids	total β-Carotene, ^b μg/g dry wt	13-cis	μg/g dry wt.			% isomers (cis)
				all-trans	9-cis		
raw product	20.8	440.0 ^a	22.5	417.5	—	5.1	
strips (2-min blanch)	21.7	499.2 ^b	39.1	460.1	—	7.8	
strips (10-min blanch)	23.7	457.8 ^a	69.9	387.9	—	15.3	
puree	17.8	485.9 ^b	25.2	460.7	—	5.2	
steam inj (starch gelat)	16.7	495.3 ^b	34.5	460.8	—	7.0	
steam inj (enz inact)	15.6	455.9 ^{ab}	36.9	419.0	—	8.1	
canned	15.8	390.3 ^c	56.7	323.0	10.6	17.2	
dehydration (drum drying)	94.9	349.8 ^{cd}	101.0	248.8	tr	28.9	
microwaved	31.0	340.2 ^d	56.3	283.9	tr	16.5	
baked	31.3	301.8 ^e	69.5	232.3	tr	23.0	

^a See Materials and Methods for processing conditions. ^b Values with different letters are significantly different at $P < 0.05$.

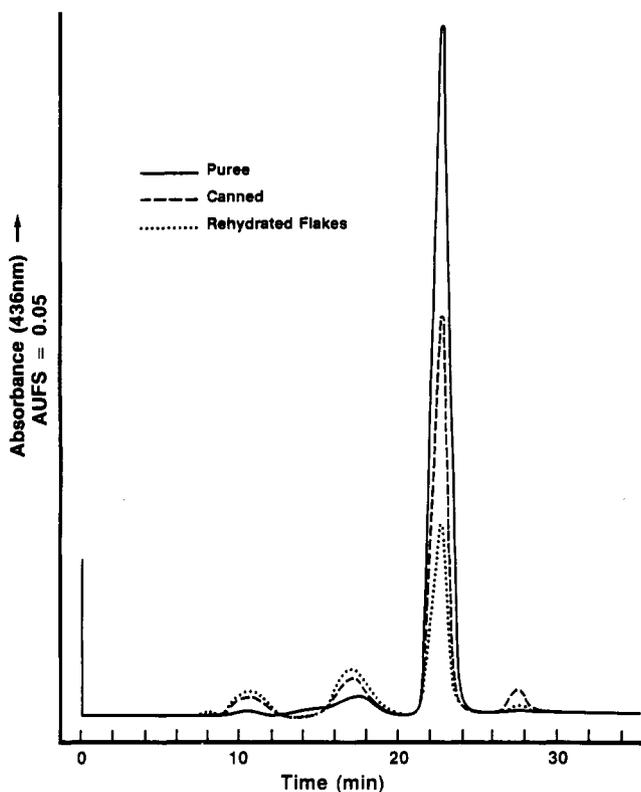


Figure 2. HPLC chromatogram of pureed, canned, and rehydrated flake extracts of sweet potatoes. Isomer positions: 13-cis-β-carotene, 17 min; all-trans-β-carotene, 23 min; 9-cis-β-carotene, 27.5 min.

close to an isobestic point of the three isomers. Note the decrease in peak areas illustrated (Figure 2) for the canned and dehydrated samples relative to the puree. These areas reflect losses in carotene content caused by processing methods and differences in solids content.

Table II lists the quantity of total β-carotene extracted from samples of raw and processed sweet potatoes. Recovery studies were performed, to ensure extraction efficiency, and resulted in 90–95% recoveries. The limits of detection have been reported elsewhere (Chandler and Schwartz, 1987). All values listed were calculated on a dry-weight basis and account for losses of soluble solids.

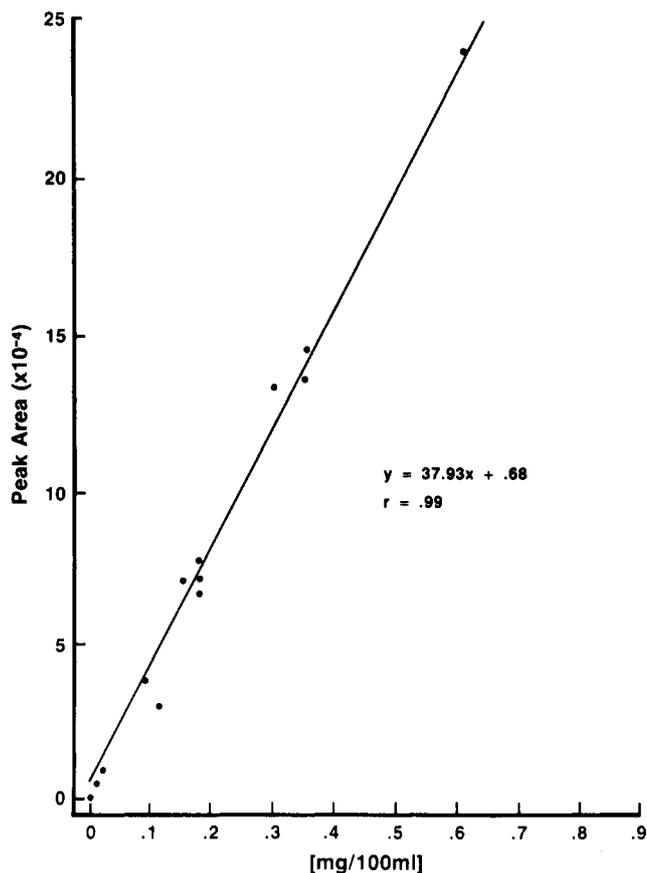


Figure 3. Calibration curve of all-trans-β-carotene peak area vs all-trans-β-carotene concentration.

Strips blanched for 2 min lost 14.7% soluble solids, which increased to 28.2% after 10 min. Changes in soluble solids must be considered when investigating losses of carotenoids in processed products (Ogunlesi and Lee, 1979; Edwards and Lee, 1986). A significant increase ($P < 0.05$) in total β-carotene was noted after a 2-min blanch. We attribute this change to an increase in extractability of carotene in the blanched product. Presumably a change in tissue morphology occurred, thereby allowing greater penetration of organic solvents into the cells and an enhanced release of carotenes. Microscopic examination of raw sweet po-

tatoes showed β -carotene located within the chromoplasts. After heating, the chromoplasts and cell wall are disrupted and pigment droplets are formed (Purcell et al., 1969). After 10 min of blanching, no difference was found between the raw samples and the heated strips. However, a significant decrease was observed relative to the 2-min treatment, which may be caused by losses via oxidative and heat degradation. Oxygen has been found to be a critical factor in β -carotene degradation (Chou and Breene, 1972; Goldman et al., 1983).

The pureed samples, produced from the lye-peeled potatoes, were higher in total carotene content relative to the raw samples ($P < 0.05$). The heat treatment applied to the outer surface of the roots may have caused this increase to occur. Walter and Giesbrecht (1982) also observed an increase in carotene content after lye peeling sweet potatoes. A further increase was noted after subjecting the puree to a steam injection process used to gelatinize the starch. No significant change was found after inactivating the native enzymes by a second steam injection treatment, although a trend ($P < 0.1$) toward lower carotene content was apparent. The samples analyzed after the second steam injection process were more variable in total carotene and accounted for the lack of statistical significance at the 95% level.

Further heat treatment of the puree by canning reduced the carotene content significantly, primarily because of heat-mediated decomposition. The largest decrease (31.4%) in carotene occurred in the baked samples. Microwaved potatoes showed a smaller loss (22.7%). The shorter heat treatment used during microwave processing may be responsible for this result. Previous investigators (Lee and Ammerman, 1974; Reddy and Sistrunk, 1980) have reported similar losses during the processing of sweet potatoes. Drum drying also caused a large loss (20.5%) in carotene content. We attribute this loss to rapid oxidation during drying. Oxidative degradation of β -carotene occurs through a free-radical process (Goldman et al., 1983), and water content has been shown to have a protective effect on carotene stability presumably through its interaction with free radicals (Karel, 1980).

Table II lists the isomer content found in extracts of raw and processed sweet potatoes. Raw samples contained a small quantity of the 13-cis isomer. We have previously reported (Chandler and Schwartz, 1987) no detectable isomers found in fresh raw sweet potatoes. We attribute the formation of this isomer to the long storage period (9 months) employed for potatoes prior to initiating this study. Small quantities of cis isomers have been detected in extracts of sweet potatoes obtained from local markets (Bushway, 1985; Quackenbush, 1987). However, the curing, storage, and handling conditions of the sweet potatoes were not reported.

No change in total or isomeric carotene content was observed from freezing sweet potato samples or during frozen storage of extracts. Similar findings were reported by Weckel et al. (1962) upon freezing carrots and by Broich et al. (1983) when freezing, thawing, and refreezing human serum.

Heat processing induced the formation of the 13-cis isomer in all samples except the puree. Although heat was applied during lye peeling of the potatoes, heat penetration occurs only at the surface layers. Cell wall detachment, from lye-peeling treatments, has been reported to occur within 21 mm of the surface (Walter and Schadel, 1982). Furthermore, alkaline conditions have been shown not to cause isomerization reactions (Schwartz and Patroni-Killam, 1985). Dehydrated flakes contained the greatest amount

of cis isomer (28.9%) relative to the other processed products. The quantity of isomer formed was related to the severity and length of the heat treatment. Only canned samples contained appreciable levels of the 9-cis isomer, although detectable amounts were found in flaked, microwaved, and baked potatoes.

Other investigators (Berset et al., 1984; Khachik et al., 1986) have reported the formation of 15-cis- β -carotene during thermal processing. On our chromatographic system, 15-cis- β -carotene was resolved, prior to the 13-cis- β -carotene isomer, in 14.5 min. No detectable quantities of 15-cis- β -carotene were observed in fresh and processed sweet potatoes.

In general, comparing the changes in carotene loss and isomerization relative to processing treatment, we observed a greater susceptibility for isomerization reactions rather than decomposition. For example, a 13.4% change in total β -carotene was found for samples subjected to a 2-min blanch relative to the raw product. However, a 53.0% change in isomer content was measured. After a 10-min blanch, a 4.0% total carotene change was noted with a threefold increase in isomers. These results reflect the lability of *all-trans*- β -carotene to form cis isomers during thermal treatments in contrast to photochemical or other degradative mechanisms. Since appreciable levels of the isomers are formed during various food processes, the amounts of each isomer coupled with their relative biological activities as vitamin A precursors should be used for accurate nutritional content measurements. In addition, maximum nutrient bioavailability can be obtained through processes minimizing isomerization reactions.

Registry No. NaOH, 1310-73-2; *all-trans*- β -carotene, 7235-40-7; 13-cis- β -carotene, 6811-73-0; 9-cis- β -carotene, 13312-52-2.

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Received for review March 16, 1987. Accepted August 26, 1987. Paper No. 10964 of the Journal Series of the North Carolina Agricultural Research Service, Raleigh, NC 27695-7601. The use of trade names in this publication does not imply endorsement by the North Carolina Agricultural Research Service of the products named nor criticism of similar ones not mentioned. This work was partially supported by NIH Biomedical Research Support Grant No. RR07071.

Changes in Carbonyl Compounds in the French Bean as a Function of Cooking and Enzyme Treatment

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The changes in the volatile and total carbonyl compounds in the French bean prior to cooking and after cooking and the subsequent application of endogenous enzymes were investigated. Carbonyl compounds were extracted from treated beans and prepared as DPNH derivatives, which were quantitated both gravimetrically and spectrophotometrically. Gas chromatographic analysis showed that thermal treatment of the beans resulted in increased levels of both volatile and total carbonyl compounds, with a corresponding decrease in longer chain carbonyl compounds. These changes were accentuated by the incubation of the whole cooked beans in the presence of an enzyme extract obtained from germinated beans. Mass spectroscopic analysis indicated the appearance of two dienals (C_{10} and C_{11}) as a result of enzyme treatment. It would appear that enzyme treatment of the cooked bean resulted in changes similar to the cooking of the beans for a prolonged period of time.

The French bean is an important food source because of its relatively high oil and protein content. Cultivated

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in France, and other regions of the world, it is consumed directly or further processed in a manner similar to the better known soybean. Flavor is an important factor in the acceptability of any food product, and the authors have been involved in an ongoing study to understand the chemistry of flavor compounds in the French bean. For example, the French bean, like other oil-bearing seeds, suffers from the development of off-flavors during storage and enzymatic mechanisms are considered to be involved.