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Enhancing Quality and Oxidative Stability of Aged Fried Food with γ-Tocopherol

KATHLEEN WARNER,* WILLIAM E. NEFF, AND FRED J. ELLER

Food Quality and Safety Research, USDA, ARS, NCAUR, Northern Regional Research Center, 1815 North University Street, Peoria, Illinois 61604

To determine the effects of γ-tocopherol on the stability of fried food, potato chips were fried in triolein with 0, 100, or 400 ppm γ-tocopherol. Potato chips, sampled at 1, 3, and 6 h of frying time, were aged for 0, 2, and 4 days at 60 °C and then evaluated for odor attributes by sensory analysis and for volatile compounds by purge-and-trap gas chromatography−mass spectrometry. Oil sampled after 1, 3, and 6 h of frying time from the fryer was evaluated for total polar compounds and retention of γ-tocopherol. Oil extracted from the potato chips was also analyzed for residual γ-tocopherol. γ-Tocopherol disappeared rapidly, with only slight amounts of the original 100 ppm level detectable after the triolein was used for frying. γ-Tocopherol significantly inhibited polar compound production in the triolein. Results showed that γ-tocopherol inhibited the oxidation of the fried food even when only very low levels of retained γ-tocopherol were present in the frying oil or potato chips. Nonanal formation was inhibited by γ-tocopherol in aged potato chips. Odor analysis of the aged potato chips showed that samples with 0 ppm γ-tocopherol had a rancid odor after being aged for 4 days. Potato chips with 400 ppm γ-tocopherol had no rancid odors; however, as the level of γ-tocopherol decreased in the triolein and in the potato chips, a weak plastic odor characteristic of oxidized triolein was detected.

KEYWORDS: Frying; fryer oil; γ-tocopherol; nonanal; odor; potato chips; sensory; tocopherol; triolein

INTRODUCTION

Lipid oxidation is a major cause of flavor deterioration in fats, oils, and fat-containing foods; however, it can be inhibited by antioxidants such as tocopherols. Much research has been reported during the past 40 years on the effects of tocopherols during the autoxidation of oils (*1*−*6*), but the results of studies on oil autoxidation cannot necessarily be extrapolated to heated oils or frying oils, because temperature can have a significant effect on the results of oxidation studies using tocopherols. For example, Gottstein and Grosch found that the antioxidant activity order for tocopherols was δ > γ > β > α in lard above 60 °C, but the results were in the order α > γ > β > δ at 20 °C and 40 °C (*7*). In contrast, the antioxidant activity order of tocopherols in a different fat, menhaden oil, stored at 37 °C was γ = δ > α (*8*) which showed the effect of substrate type. Lea and Ward (*9*) suggested that the relative antioxidant activity of tocopherols depends on a variety of parameters, including temperature, composition of fat, form of the fat (liquid, emulsion), and concentration of tocopherols. Erratic effects from high oxidation temperatures were noted by Ragnarsson and Labuza (*10*), who suggested that the mechanism of antioxidation was affected by temperature. Frankel (*11*) has recommended that autoxidation studies on oil be done at temperatures less than 60 °C because higher temperatures gave different results than temperatures <60 °C. In the past several years, researchers studying the effects of tocopherols in heated oils with no frying have shown that the formation of nonvolatile degradation products can be affected by tocopherols (*12*−*14*). Barrera-Arellano et al. (*13*) found that a mixture of α-, β-, γ- and δ-tocopherols inhibited polar compound formation better than δ-tocopherol, which was better than α-tocopherol in triolein heated to 180 °C. In another heated oil study, tocopherols decreased coconut fat oxidation at 160 °C in the order δ > γ > α (*15*). Only a few studies have been conducted on the effects of tocopherols in frying oils in which food was actually fried (*16*, *17*). These studies either compared one tocopherol with other antioxidants or related the differences in the frying oils to differences in the naturally occurring levels of tocopherols. γ-Tocopherol, which is present in almost all vegetable oils, has been shown to inhibit oxidation in heated oils (*3*, *12*) and to decrease in concentration with increasing heating time (*13*). Tocopherol retention in fried food has not been thoroughly investigated and, therefore, is one aspect that should be addressed to fully understand the efficacy of tocopherols in frying applications. Our objective in this study was to determine the effects of γ-tocopherol on the stability of oil during frying, the quality of the fried food, and the shelf life of the fried food. A model system of frying potato chips in triolein with different amounts of γ-tocopherol was used to omit the complications of an oil with many naturally occurring minor oil constituents. This model frying system was used to determine both the

* Send correspondence to: K. Warner, National Center for Agricultural Utilization Research, 1815 North University Street, Peoria, Illinois 61604. Phone: 309-681-6584. Fax: 309-681-6668. E-mail: warnerk@ncaur.usda.gov.

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retention of δ-tocopherol in the frying oil and in the potato chips and the effects of the γ-tocopherol remaining in the aged chips on the quality and stability of the chips.

EXPERIMENTAL PROCEDURES

Materials. Triolein (>99% purity) was purchased from NuChek Prep, Inc. (Elysian, MN). The triacylglycerol was checked for non-triacylglycerol products by peroxide value analysis, polar phase thin-layer chromatography (TLC), polar component analysis, and size-exclusion high-performance liquid chromatography (SEC−HPLC) (*18*). Fatty acid purity was checked by gas chromatography of the trans-methylated triacylglycerol and triacylglycerol purity by reverse-phase HPLC. γ-Tocopherol (99.4% pure) was purchased from Matreya, Inc. (Pleasant Gap, PA). The purity of the γ-tocopherol was evaluated by HPLC (*18*). Both triolein and γ-tocopherol were used without further purification. Idaho Russet potatoes were purchased locally.

Frying Protocol. Thirty-gram samples of triolein with 0, 100, or 400 ppm γ-tocopherol were weighed into 100-mL Pyrex beakers. The beakers were placed in a thermostatically controlled silicone oil bath and heated to 190 °C. Peeled potatoes were cut into pieces approximately 3 cm × 0.5 cm × 1 cm and rinsed several times in cold water. Potato slices were dried and then fried in 3-g batches for approximately 3.5 min/batch every 10 min. Potato chips were sampled for analysis prior to the 1-, 3-, and 6-h frying times and then blanketed with argon and frozen until analysis. Oil samples were taken at time 0 and after 1, 3, and 6 h of frying and then blanketed with argon and stored at −15 °C until analyses could be performed. After the 1 and 3 h oil samples were taken, 3 g of fresh triolein with 0, 100, or 400 ppm γ-tocopherol was added at each time to replenish depleted oil; however, the makeup oil was not intended to restore the oil to the original level of γ-tocopherol. Potato chips were aged for 0, 2, and 4 days at 60 °C in sealed vials. The frying experiment was duplicated.

Extraction of Oil from Potato Chips. Supercritical fluid extractions (SFEs) of potato chips were done with an ISCO model 3560 SFE instrument (ISCO Corporation, Lincoln, NE). Potato chip samples were mixed with ca. 2 g of Leco-Dry (Leco Corporation, St. Joseph, MI) and then placed in an extraction cell containing a glass fiber filter disk (18 mm dia) on the bottom. Additional Leco-Dry was added to nearly fill extraction cell, and a second glass fiber filter was placed on top. Supercritical extractions were done at 10 000 psi and 70 °C with a flow rate of 2 mL/min for 45 min after an initial 1-min static hold. The variable restrictor was held at 70 °C, and extracts were collected in 20-mL precooled (0 °C) and pressurized glass vials. Five milliliters of methylene chloride was added to the collection vial prior to the start of the extraction, and 3 mL of methylene chloride was used to rinse the restrictor after extraction. Solvent was removed under a gentle stream of nitrogen at room temperature, and samples were subsequently stored under nitrogen at −70 °C. SFE-grade CO_2 (Air Products and Chemicals Inc., Allentown, PA) was used.

Total Polar Compound Analysis. Amounts of polar compounds were analyzed in duplicate by the AOCS column chromatography method Cd 20−91(97) (*19*).

γ-Tocopherol Analysis. γ-Tocopherol levels in triolein fryer samples and in triolein extracted from potato chips were determined in triplicate by HPLC with a polar phase column coupled to a fluorescence detector. The HPLC was fitted with a 3-μm particle size ultra silica column (25 × 0.49 cm) (Phenomenex, Torrance, CA). The solvent system was 2% 2-propanol in hexane. Solvent was pumped at 0.5 mL/min. The sample size was 10 μL of a solution containing 50 mg of solute per milliliter of mobile solvent. The fluorescence detector was a model 1046 A programmable unit (Hewlett-Packard, Palo Alto, CA). The excitation wavelength was set at 298 nm, and the emission wavelength was set at 345 nm, with the gain set at 6. A standard curve for 0.6−500 ppm γ-tocopherol was obtained to calculate the content (in parts per million) of γ-tocopherol.

Volatile Compound Analysis by Mass Spectrometry. Volatile compounds were analyzed in triplicate with a purge-and-trap apparatus equipped with a test tube adapter (Tekmar model 3000, Tekmar-Dohrmann Co., Cincinnati, OH) coupled with a model 3400 gas chromatograph (GC) and a Saturn model 3 ion-trap mass spectrometer (Varian, Inc., Walnut Creek, CA). A 50-mg sample was placed in the 1.9 × 7.6 cm test tube and heated at 100 °C for a 9-min preheating period. Volatile compounds were trapped on a 30.5-cm Tenax #1 trap, with a 10-min sample purge time at 170 °C, followed by a 6-min desorption period at 180 °C, with a GC transfer line and valve temperature of 160 °C. Volatile compounds were introduced onto a DB-1701 GC column, with a film thickness of 1 μm, and an area of 30 m × 0.32 mm (J & W Scientific, Folsom, CA). The column was programmed at −20 °C for 2 min and then heated from −20 to 233 °C at 3 °C/min. The helium flow rate through the column was 2 mL/min with 28 mL/min injector split vent flow. The GC injector was set at 240 °C, and the line to the mass spectrometer was set at 230 °C. The ion-trap MS operated in the EI mode with mass scan range of 23−400 *m/z* over 0.8 s. The filament emission current was 25 μA, the axial modulation was 2.1 V, the manifold heater was set at 160 °C, and the filament/multiplier delay was 2.5 min. Compound structural identifications were made both from spectral comparisons with the NIST 92 mass spectrometry library (Varian, Inc., Walnut Creek, CA) and from retention time comparisons with standard compounds.

Sensory Evaluation of Potato Chips. Potato chips were rated in duplicate for odor attributes by a 10-member trained, analytical, descriptive panel experienced in evaluating fried foods for odor and flavor. Odor descriptors on the scoring scales included deep fried, stale, waxy, plastic, and rancid (*20*−*21*). Judges had experience and training in evaluating fresh and aged potato chips with a wide variety of odors at weak, moderate, and strong intensity levels. All evaluations were conducted in a panel room with individual booths, odor control, temperature control, and red lighting.

Statistical Analysis. Data were evaluated by analysis of variance (*22*). Statistical significance was expressed at the $P < 0.05$ level unless otherwise indicated.

RESULTS AND DISCUSSION

Loss of γ-Tocopherol in Fryer Oils. The levels of γ-tocopherol in the fryer oils decreased rapidly and at different rates between the samples with 100 and 400 ppm γ-tocopherol (**Figure 1**). Triolein with 100 ppm γ-tocopherol lost γ-tocopherol to a greater degree during the first hour of frying than did the fryer oil with 400 ppm γ-tocopherol. After 1 h of frying, the sample with 400 ppm γ-tocopherol had 204 ppm γ-tocopherol or 51% of the original level, whereas only 9 ppm (9%) remained in the 100 ppm sample. After 3 h of frying, the tocopherol levels were 88 ppm (22%) and 0.3 ppm (0.3%) for the 400 and 100 ppm samples, respectively. The 400 and 100 ppm fryer oil samples both had low levels of γ-tocopherol (2.4 and 0.2 ppm, respectively) remaining after 6 h of frying. The slopes of these curves are similar to those in the literature on heated triolein with added α- or δ-tocopherol (*13*). Likewise, in a frying study using rapeseed oil with 401 ppm naturally occurring γ-tocopherol, the rate of loss of γ-tocopherol was similar to that found here (*23*).

Fryer Oil Degradation. Fryer oil stability was determined by total polar compounds for the oils with 0, 100, and 400 ppm γ-tocopherol after 0, 1, 3, and 6 h of frying. Initially, levels of polar compounds were low at 0.5% (**Figure 2**). In preliminary testing, the 6-h frying time was chosen as the end point because the oils had reached approximately 25% total polar compounds, which is close to the standard (>24%) for confirming abused frying oils in Europe (*24*). At each of the sampling times, the fryer oils with either 100 or 400 ppm γ-tocopherol had significantly lower total polar compound percentages than did the oils with no γ-tocopherol, except at the 6-h time, when no significant difference was indicated between the 0 and 100 ppm oils. In most frying oil studies, the polar compound levels of oils increase as the frying time increases (*20*). The slope for the 400 ppm γ-tocopherol oil is not as steep as the slope for the 100 ppm oil, showing an inhibitory effect on polar compound

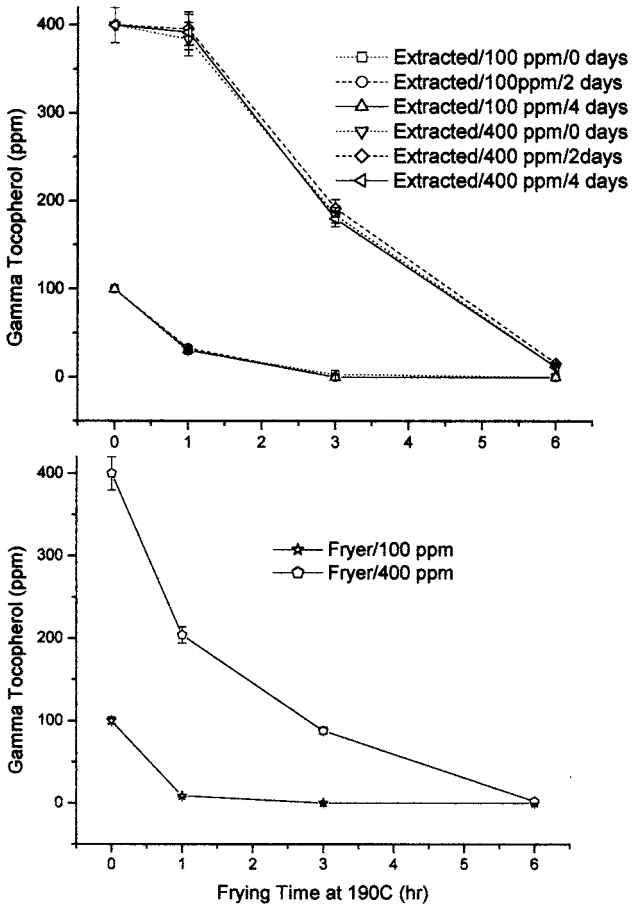

Figure 1. γ-Tocopherol (ppm) remaining in triolein fryer oil after being used to fry potato chips for 1, 3, and 6 h at 190 °C and in oil extracted from potato chips aged 0, 2, or 4 days at 60 °C.

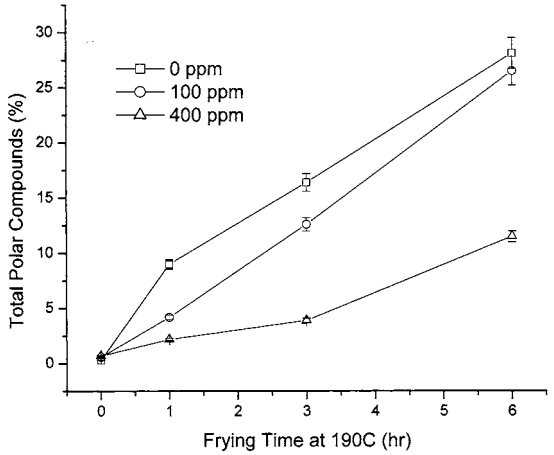

Figure 2. Percent total polar compounds in triolein fryer oil with 0, 100, or 400 ppm γ-tocopherol after being used to fry potato chips for 1, 3, and 6 h at 190 °C.

formation with 400 ppm γ-tocopherol. An exponential effect was noted for the 400 ppm oil, possibly because the oil still had 204 ppm γ-tocopherol after 1 h of frying. After 3 h of frying, an inhibitory effect was still seen with 88 ppm γ-tocopherol retained in the fryer oil, but the slope increased between 3 and 6 h, showing a possible effect of the decreasing tocopherol level. Data for the oil with no tocopherol showed a steeper slope between the 0- and 1-h sampling times, indicating the most rapid rise in polar compounds compared to the other sampling intervals. Data on nonvolatile compounds, including polymer,

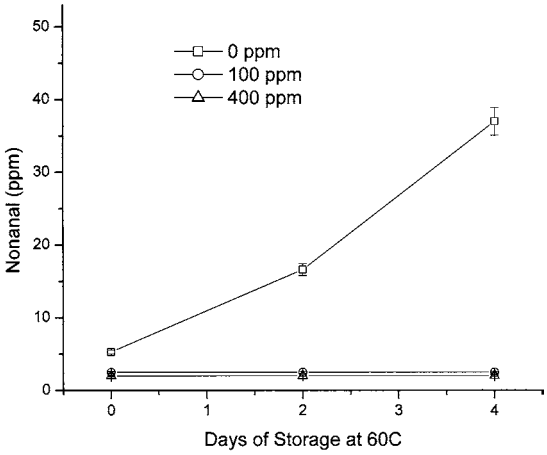

Figure 3. Nonanal in potato chips fried in triolein with 0, 100, or 400 ppm γ-tocopherol at 1 h at 190 °C and aged for 0, 2, or 4 days at 60 °C.

monomeric, diacylglycerol, monoacylglycerol, and free fatty acid components of triolein, showed a significant positive effect in inhibiting the formation of these compounds (unpublished data; manuscript in preparation).

Residual Tocopherol in Potato Chips. Oil extracted from the potato chips was evaluated for γ-tocopherol disappearance after the potato chips were aged 0, 2, and 4 days at 60 °C (**Figure 1**). No significant difference was found in the levels of γ-tocopherol remaining in the potato chips after 0, 2, or 4 days of storage at 60 °C for the sets of samples with either 100 ppm γ-tocopherol or 400 ppm γ-tocopherol. These lack of differences over storage time could be the result of storage conditions that were not severe enough. The percent of γ-tocopherol remaining was different for the extracted oils with 100 and 400 ppm of γ-tocopherol. Approximately 97% of the original amount of γ-tocopherol remained in the 400 ppm γ-tocopherol chips at the 1-h time compared to 31% for the 100 ppm γ-tocopherol chips. At the 3-h sampling time, 46% γ-tocopherol was left in the 400 ppm chips, but the amounts were >1% for the 100 ppm chips. Only 3% γ-tocopherol was left in the 400 ppm potato chips, and no γ-tocopherol was detected in the 100 ppm sample at the 6-h sampling time. The levels of γ-tocopherol in the used fryer oils at 1 and 3 h were lower than the levels in the oils extracted from the unaged potato chips. The reasons for the larger amount of γ-tocopherol measured in the unaged potato chips than in the fryer oil are not clear; however, the method of sampling potato chips and fryer oils is a possible explanation for the differences because potato chip samples were collected over a 30 min period whereas the fryer oil was collected at the end of each frying period of 1, 3, and 6 h.

Volatile Compound Analysis of Potato Chips. With no or low levels of γ-tocopherol left in some of the potato chips, the effect of the remaining tocopherol on the stability and quality of the potato chips was of interest. Measuring the volatile decomposition products in the fresh and aged potato chips provides an important index to the stability of the fried food. Direct decomposition products of oleic hydroperoxides including octanal, nonanal, 2-decenal, and 2-undecenal were measured by purge-and-trap gas chromatography−mass spectrometry (*18*, *25*). Nonanal was produced in the largest quantities; therefore, data from those analyses are presented (**Figures 3**−**5**). All of the monitored volatile compounds followed a pattern similar to that for nonanal. Few differences in the production of nonanal were observed in the unaged potato chips, although the beginnings of a pattern typical of the aged chips began to

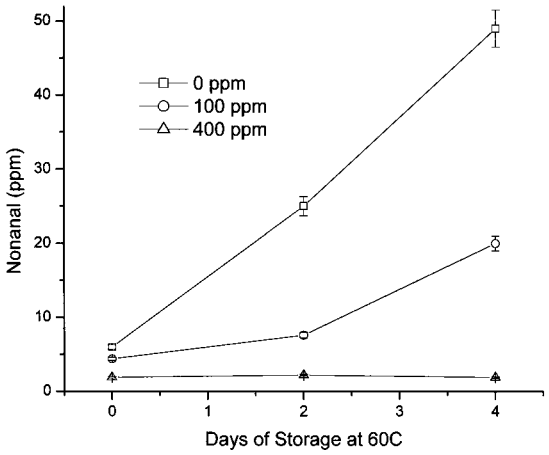

Figure 4. Nonanal in potato chips fried in triolein with 0, 100, or 400 ppm γ-tocopherol at 3 h at 190 °C and aged for 0, 2, or 4 days at 60 °C.

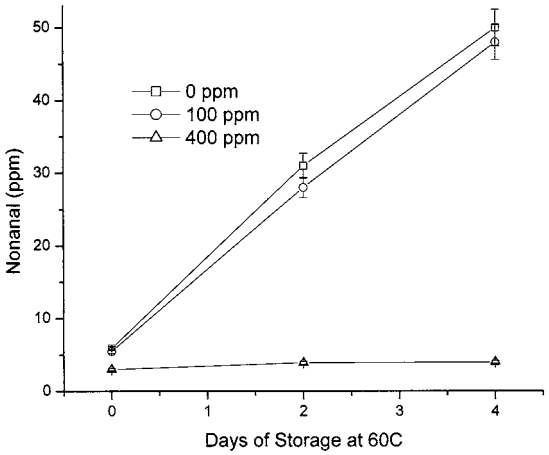

Figure 5. Nonanal in potato chips fried in triolein with 0, 100, or 400 ppm γ-tocopherol at 6 h at 190 °C and aged for 0, 2, or 4 days at 60 °C.

emerge. For example, at the 1-h frying time, there were no significant differences in nonanal levels between the potato chips with 100 and 400 ppm γ-tocopherol, but both of these samples had less nonanal than the control with no γ-tocopherol. At 3 h, the potato chips with 100 ppm γ-tocopherol had significantly more nonanal than the chips with 400 ppm but significantly less than the control. However, by 6 h, there was no significant difference between the 0 and 100 ppm chips, but the potato chips with 400 ppm γ-tocopherol still showed no effect of storage even after 4 days. These changes over frying time for the potato chips with 100 ppm γ-tocopherol followed the same pattern after being aged for 2 and 4 days, indicating that the stability of the potato chip decreased with decreasing levels of γ-tocopherol. The positive effect of γ-tocopherol was evident even though the potato chips fried in the 100 ppm γ-tocopherol oil had less than 2% γ-tocopherol remaining after 2 days of storage. The inhibitory effect could be from the small amount of γ-tocopherol remaining, or it could possibly be an additional effect from tocopherol decomposition products. Hall and Cuppett found positive effects in oils from added rosmariquinone from rosemary (*26*−*28*). Hydroquinones have been found in α-tocopherol decomposition (*29*), and therefore, these types of compounds could possibly also be present in the oils and potato chips tested in this study at levels that could inhibit oxidation. Even though the potato chips fried in the triolein with 400 ppm γ-tocopherol at 6 h had only 12 ppm γ-tocopherol remaining after 4 days of storage, the amount of nonanal in these potato

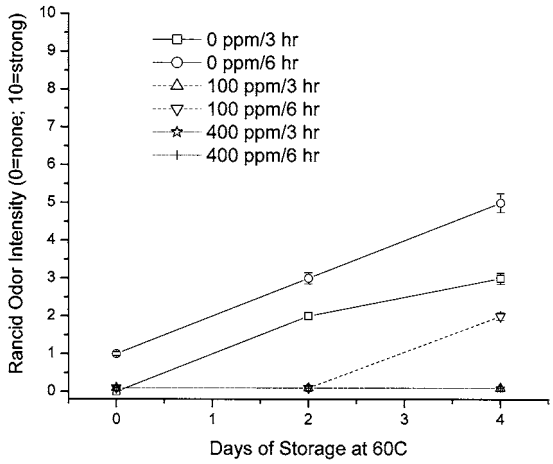

Figure 6. Rancid odor intensities of potato chips fried in triolein with 0, 100, or 400 ppm γ-tocopherol at 3 or 6 h at 190 °C and aged for 0, 2, or 4 days at 60 °C.

chips was no different than the amount in the unaged potato chips fried at the same time.

Odor Analysis of Potato Chips. Volatile degradation products are important markers for oil and fried food deterioration, but sensory analysis of food is the best method for determining the effects of antioxidants on food products. In this study, quantities of potato chips were limited; therefore, the panel evaluated the odor attributes of fresh and aged potato chips rather than the flavor. Preliminary testing in this study showed that the primary odor attributes detected in the potato chips were plastic/waxy; deep fried; stale/cardboard, which is an indication of slight oxidation; and rancid. In odor analyses of the potato chips, all samples fried in the 1-h triolein had the characteristic plastic/waxy odor that is typical of high oleic acid oils (*20*) and heated triolein (*18*). However, in the potato chips fried in the 3-h triolein with either 100 or 400 ppm γ-tocopherol, panelists detected not only a plastic/waxy odor, but also a deep fried odor in the potato chips. This deep fried odor is produced even in triolein because of the formation of 2,4-decadienal by secondary pathways from the oleic acid decomposition products (*21*). Potato chips fried without γ-tocopherol that were sampled at 3 h and aged for 2 or 4 days at 60 °C also had a moderate stale odor intensity. **Figure 6** reports the intensity levels of rancid odor in potato chips fried in triolein used for 3 or 6 h. A very slight rancid odor was detected first in the unaged potato chips that had been fried in the 6-h triolein with no tocopherol. By the time the potato chips had been aged for 2 days at 60 °C, weak rancid odor intensity was reported for the potato chips with no γ-tocopherol that were fried in the 3- and 6-h oil. After 4 days of aging, potato chips fried in oil with no tocopherol had slightly higher rancid odor intensities than they had after 2 days of storage. The potato chips fried in the oil with 100 ppm γ-tocopherol at the 6-h sampling time represented the only sample of potato chips fried with γ-tocopherol to develop a rancid odor. This sample, which had no detectable residual γ-tocopherol (**Figure 1**), also had a high amount of nonanal (**Figure 5**). Rancid odor was not detected in the potato chips fried in triolein with 400 ppm γ-tocopherol. The nonanal and odor analyses were in agreement that even very low levels (~0.1%) of retained γ-tocopherol had a positive effect in inhibiting the oxidation of aged potato chips.

These data on triolein with γ-tocopherol showed positive significant effects in limiting the formation of volatile flavor compounds in aged potato chips. These volatile compounds, such as aldehydes, can have deleterious effects on the flavor

and odor of fried food (*18*). The inhibition of aldehyde formation in this study resulted in a reduction of the rancid odor intensity with increasing γ-tocopherol levels. γ-Tocopherol also had a beneficial effect in extending the fry life of the triolein by inhibiting the formation of polar compounds.

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