# Effect of Tocopherols on the Frying Stability of Regular and Modified Canola Oils

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**ABSTRACT:** A study was conducted to compare the relationship between frying stability and levels and degradation rates of tocopherols in regular and three modified canola oils. Oils were heated at 175 ± 2°C for a total of 72 h, with french fries fried intermittently. Frying stability was compared based on the rates of formation of free fatty acids (FFA) and total polar compounds (TPC). Significant differences ($P < 0.05$) were identified between oils using analysis of covariance and *t*-tests for multiple comparisons. No significant differences were observed in the rates of FFA formation among the canola oils during frying. Nevertheless, regular canola (RCO) and high-oleic, low-linolenic acid canola (HOLLCO) oils produced less FFA compared to high-oleic (HOCO) and low-linolenic acid (LLCO) canola oils. However, LLCO and HOCO both had significantly ($P < 0.05$) faster rates of TPC formation compared to HOLLCO or RCO. HOLLCO with the highest level of tocopherols (893 mg/kg) exhibited a slow rate of degradation which accounted for a half-life of 48–60 h of frying. RCO, with a lower level of tocopherols (565 mg/kg), however, had the slowest degradation rate with a half-life of >72 h. In contrast, HOCO and LLCO with 601 and 468 mg/kg tocopherols, respectively, both exhibited a half-life for tocopherols of 3–6 h of frying. An inverse relationship was observed between TPC formation and the reduction of tocopherol. Thus, the greater frying stability of RCO and HOLLCO appears to be affected far more by the rate of tocopherol degradation than by any changes in fatty acid composition.

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**KEY WORDS:** Canola oils, degradation rates, fatty acids, free fatty acid, frying stability, regular and modified, tocopherols, total polar compounds.

A number of studies have reported that modified vegetable oils show some improvement in frying stability over the corresponding regular oils (1–5). However, no significant improvements in frying stability were found by a number of researchers studying modified vegetable oils (3,4,6,7). These findings suggest that fatty acid composition alone may not adequately explain the stability of frying oils. Breeding of oilseeds to improve stability has tended to ignore the minor components, focusing solely on the levels of fatty acids present in the resulting oil. However, the relative frying stability of an oil cannot always be accurately predicted based only on the fatty acid composition. Consequently, minor components must play a significant role in oil stability, particularly if their levels are changed substantially during modification. A recent study (8) showed that the level of linolenic acid was critical to the deep-frying performance of three high-oleic canola oils (HOCO). However, these researchers did not examine any of the minor components in these oils. The objective of this study was to compare the composition and frying stability of three modified canola oils with a regular canola oil (RCO) by monitoring the formation of oil degradation products as well as tocopherol degradation.

## EXPERIMENTAL PROCEDURES

*Oils and french fries*. This study included three modified canola oils—high oleic (HOCO), low linolenic (LLCO), and high-oleic and low-linolenic (HOLLCO)—and an RCO. All the oils were commercially refined and contained only citric acid; no other preservatives were added. French fries were made from Russet-type potatoes, which were peeled and sliced using a Starfrit® potato chipper (Atlantic Productions Inc., Mississauga, Canada). The sliced potatoes were rinsed with cold water prior to being submerged in the oil.

*Frying procedure and oil sampling*. Two replications of 72-h deep-frying trials were conducted using four oils per trial. Oils (2 L) were placed in 2-L capacity domestic deep fryers (Tefal® and SEB® brands, Selongey Cedex, Dijon, France). The oils were heated to 175 ± 2°C and kept at this temperature for 12 h each day for 6 d. Each morning, the oil was replenished with fresh oil to reestablish the initial volume of 2 L. To accelerate the deterioration process, french fries were fried for 6 min each morning and evening. A 1:6 ratio of oil to food was used as this ratio was recommended by Morton and Chidley (9) and used by other researchers (10–12). Oil samples (30 mL) were taken at predetermined intervals throughout frying, flushed with nitrogen, and frozen until analyzed.

*Peroxide value (PV).* PV were determined in duplicate on fresh oils using the AOCS Official Method Cd 8-53 (13).

*Fatty acid analysis.* Fatty acids were methylated prior to analysis by gas chromatography (GC) based on the AOCS Official Method Ce 1-62 (14). The resulting fatty acid methyl esters (FAME) were analyzed on a Hewlett-Packard 5890A gas chromatograph (Palo Alto, CA) using a Supelcowax 10 column (30 m × 0.25 mm i.d.; Supelco, Oakville, Ontario, Canada), a flame-ionization detector, and hydrogen as carrier

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gas (2 mL $min^{-1}$). Initial column temperature was 175°C and then programmed to 235°C at 3°C $min^{-1}$. Injector and detector temperatures were 250 and 260°C, respectively. FAME samples (0.5 μL) were injected with an autosampler. Fatty acids were identified by chromatographic retention time by comparison with authentic standards (Nu-Chek-Prep, Elysian, MN).

*Free fatty acid (FFA) content*. FFA content as a percentage of oleic acid was determined by the Veri-Fry® Pro FFA-75 quick test method (Test Kit Technologies Inc., Metuchen, NJ). A high correlation ($r = 0.94$) was previously reported between this method and the AOCS Official Method Ca 5a-40 (15).

*Total polar compounds (TPC).* TPC were determined using Sep-Pak® Vac 6 cc (1 g) cartridges (Water Chromatography Division, Millipore Corporation, Millford, MA) to separate the polars from the nonpolars. The procedure was performed as described by Petukhov (16), based on the method of Sebedio *et al.* (17). The percentage TPC in the oil was determined by subtracting the weight of the nonpolar fraction from the initial weight of the oil, dividing this number by the initial weight of the oil, and multiplying by 100 based on the AOAC Method 982.27 (18).

*Tocopherols.* Tocopherols were analyzed by the AOCS Official Method Ce 8-89 (19). In brief, oil samples (100 mg) were weighed directly into a threaded high-performance liquid chromatography (HPLC) vial and dissolved in 1.5 mL hexane. Analysis was performed on a Shimadzu LC-10AD liquid chromatograph, with a Shimadzu SIL-10A auto injector and Shimadzu SCL-10A system controller (Shimadzu, Tokyo, Japan). A Hewlett- Packard HP 1046A programmable fluorescence detector was used with excitation λ = 288 nm and emission λ = 331 nm. The column was a normal-phase Prodigy 5 μ silica column (250 × 3.20 mm; Phenomenex®, Torrance, CA). Of each sample, 40 μL was injected with a flow rate of 0.8 mL/min and a run time of 15 min. The mobile phase was 5% methyl-*tert*-butyl-ether in hexane. Levels of tocopherols were quantified using separate calibration curves for α-, β-, γ-, and δ-tocopherol.

*Metal analysis.* The amounts of Cu, Fe, and Ni were determined in the fresh oils following the AOCS Official Method Ca 18b-91 (20). This method involved vaporizing a sample of oil in a graphite furnace, followed by detection of each metal using absorption spectrophotometry. The analyses were performed at the Grain Research Laboratory of the Canadian Grain Commission (Winnipeg, Canada).

**TABLE 1**
**Quality Parameters in Fresh Oils[a]**

| Oil | PV (meq/kg) | FFA[b] (% oleic aid) |
|---|---|---|
| RCO | 1.1 ± 0.02 | 0.05 ± 0.01 |
| HOCO | 0.6 ± 0.01 | 0.04 ± 0.01 |
| HOLLCO | 0.6 ± 0.01 | 0.07 ± 0.01 |
| LLCO | 0.6 ± 0.01 | 0.03 ± 0.01 |

[a]All values are averages of duplicate samples. PV, peroxide value; FFA, free fatty acid; RCO, regular canola oil; HOCO, high-oleic canola oil; HOLLCO, high-oleic, low-linolenic canola oil; LLCO, low-linolenic acid canola oil.
[b]Values obtained by Veri-Fry® Pro FFA-75 quick test (Test Kit Technologies Inc., Metuchen, NJ).

*Statistical analysis.* The rates of TPC and FFA accumulation and tocopherol degradation were compared using analysis of covariance (ANCOVA) with frying time as the covariate variable. The model included the variables of specific oil type, frying time, and the interaction between them. ANCOVA was performed using SAS statistical software (SAS Institute, Cary, NC) and allowed for the comparison of rates (i.e., slopes). To compare rates between oils, *t*-tests for multiple comparisons were used.

## RESULTS AND DISCUSSION

The initial quality of the oils is shown Table 1. The fresh oils were all found to have PV < 1.1 meq/kg and FFA < 0.11%, indicative of good-quality oils (21). Vegetable oils modified to improve stability generally display lower levels of polyunsaturated fatty acids (PUFA), particularly linoleic and linolenic acids (5,22,23). The modified canola oils in this study all showed this pattern compared to RCO (Table 2). RCO fatty acid composition consisted of 57.4% oleic acid, 21.2% linoleic acid, and 10.2% linolenic acid. As expected, all three modified canola oils were lower in linolenic acid (3.0 to 6.7%) and higher in oleic acid (62.7–74.8%) levels. With the exception of LLCO (18:2, 24.6%), the modified oils also displayed lower levels of linoleic acid (9.3 and 12.0%). Although LLCO had a lower level of linolenic acid than RCO, the total PUFA content of LLCO (27.7%) was not much different from RCO (31.5%).

**TABLE 2**
**Fatty Acid Composition of Fresh Oils Expressed as Percentage of Total Fatty Acids[a]**

| Oil | SFA[b] | 18:1[c] | 18:2[d] | 18:3[e] | PUFA[f] |
|---|---|---|---|---|---|
| RCO | 7.5 ± 0.2 | 57.4 ± 0.1 | 21.2 ± 0.1 | 10.2 ± 0.2 | 31.5 ± 0.2 |
| HOCO | 7.0 ± 0.1 | 74.4 ± 0.1 | 9.3 ± 0.1 | 6.7 ± 0.1 | 16.0 ± 0.1 |
| HOLLCO | 8.0 ± 0.1 | 74.8 ± 0.1 | 12.0 ± 0.1 | 3.1 ± 0.1 | 15.1 ± 0.1 |
| LLCO | 7.2 ± 0.1 | 62.7 ± 0.1 | 24.8 ± 0.2 | 3.0 ± 0.1 | 27.7 ± 0.2 |

[a]All values are average of duplicate analysis. See Table 1 for other abbreviations.
[b]Saturated fatty acids.
[c]Oleic acid.
[d]Linoleic acid.
[e]Linolenic acid.
[f]Polyunsaturated fatty acids.

Tocopherols are important minor constituents in oils as they serve as antioxidants and, to varying degrees, slow down oxidative degradation. Consequently, oils with higher levels of tocopherols would be expected to exhibit greater stability. Among the canola oils examined, HOLLCO had the highest level of tocopherols in the fresh oil (Table 3). A level of 893 mg/kg was found in HOLLCO, compared to levels ranging between 468 to 601 mg/kg for the other three canola oils. Tocopherol composition within an oil is also important. It has been shown that different forms of tocopherols exhibit different degrees of antioxidant effectiveness. The order of antioxidant activity (*in vitro*) has been reported to be $\delta > \gamma > \beta > \alpha$ at temperatures between 50 and 100°C (24). However, the antioxidant activity of various combinations of tocopherols has yet to be investigated. The main tocopherol isomer in canola oils was γ-tocopherol which accounted for almost two-thirds of the total tocopherols, with the remainder being α-tocopherol. Canola oils contained only trace amounts of δ-tocopherol. The distribution of tocopherol isomers in canola oil was typical of the values reported previously (25).

A comparison of canola oils revealed no significant differences in the rates of FFA accumulation during frying (Fig. 1). Nevertheless, it was apparent that RCO and HOLLCO produced less FFA compared to HOCO or LLCO. Petukhov (16) compared the frying stability of regular, hydrogenated, LLCO, and HOCO during the frying of potato chips over 40 h. Results indicated that the hydrogenated canola oil had a significantly ($P < 0.05$) faster rate of FFA accumulation compared to LLCO. No significant differences in rates of FFA accumulation were reported among the other oils. Warner and Mounts (4) reported that FFA levels were higher in regular as compared to low-linolenic soybean oil, and significantly ($P < 0.05$) higher in RCO as compared to LLCO heated for 40 h. However, these researchers examined levels rather than rates of FFA accumulation.

Among the canola oils, LLCO and HOCO showed no significant differences in their rates of TPC formation during frying; however both exhibited significantly ($P < 0.05$) faster rates of TPC formation compared to RCO and HOLLCO (Fig. 2). Since TPC are products of oil degradation, the faster rate of formation in these two oils indicated that LLCO and HOCO were significantly less stable during frying than the other two canola oils. The superior frying stability of RCO over LLCO and HOCO was unexpected as both modified oils were lower in PUFA and should have been less susceptible to thermal oxidative breakdown. Warner *et al.* (26) reported that HOCO were more stable than RCO based on the levels of TPC formed during frying. However, their study was conducted for only 18 h and looked at the levels of TPC rather than their rates of formation. Earlier studies by Warner and Mounts (6) also found no significant differences ($P < 0.05$) in TPC between RCO and LLCD during a 40-h frying study. No significant differences were found between RCO and HOLLCO in terms of rates of TPC formation, indicating no apparent differences in their frying stability. These results were also unexpected as HOLLCO contained half as much PUFA compared to RCO (Table 2) and much higher levels of tocopherols (Table 3).

**TABLE 3**
**Tocopherol Content and Composition of Canola Oils[a]**

| Oil | Tocopherols (ppm) | α-Tocopherol (ppm) | γ-Tocopherol (ppm) |
|---|---|---|---|
| RCO | 565 ± 25 | 197 ± 10 | 369 ± 16 |
| HOCO | 601 ± 4 | 180 ± 2 | 421 ± 4 |
| HOLLCO | 893 ± 3 | 290 ± 2 | 603 ± 2 |
| LLCO | 468 ± 34 | 152 ± 13 | 374 ± 44 |

[a]All values are average of duplicate analysis. See Table 1 for abbreviations.

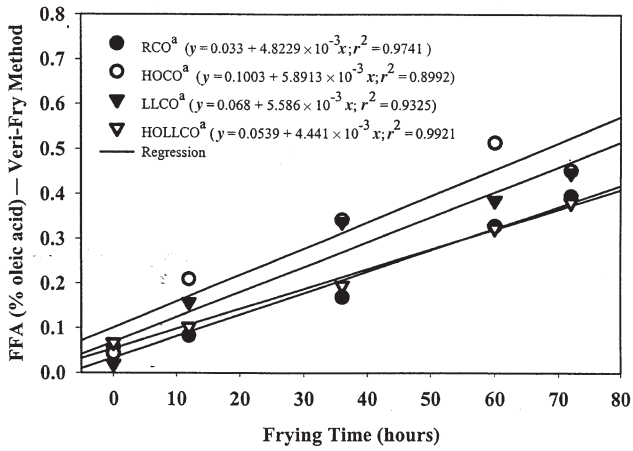


**FIG. 1.** Free fatty acids (FFA) determined by the Veri-Fry Pro FFa-75 Quick Test Method (Test Kit Technologies, Inc., Metuchen, NJ) over frying time for canola oils. Oils with the same letter as a superscript displayed no significant differences in rates of FFA accumulation for $P < 0.05$. RCO, regular canola oil; HOCO, high-oleic canola oil; LLCO, low-linolenic canola oil; HOLLCO, high-oleic, low-linolenic canola oil.

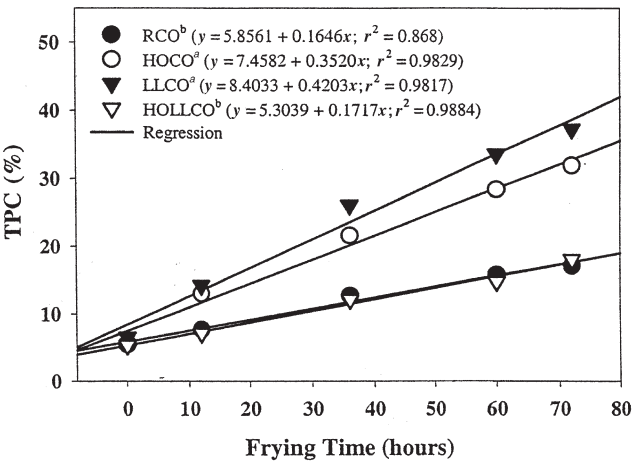


**FIG. 2.** Total polar components (TPC) over frying time for canola oils. Oils with the same letter as a superscript displayed no significant differences in rates of TPC formation at $P < 0.05$. See Figure 1 for other abbreviations.

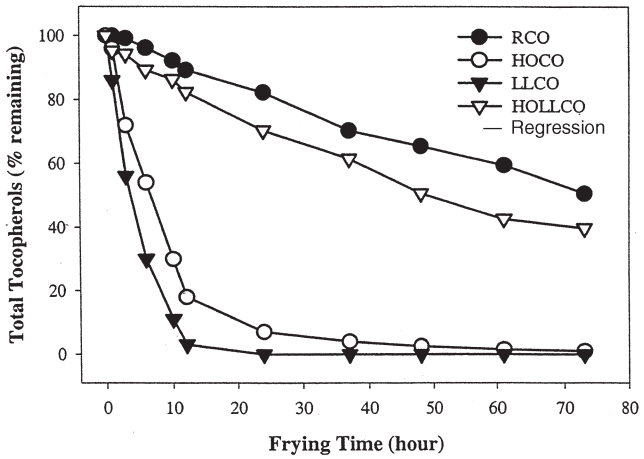


**FIG. 3.** Total tocopherols remaining over frying time for canola oils (% of original amount). See Figure 1 for abbreviations.

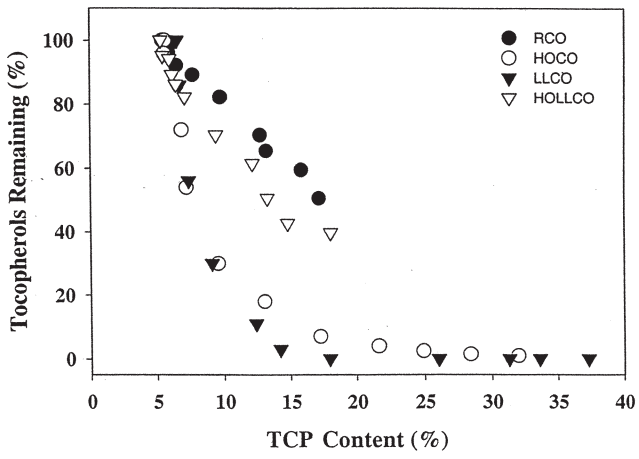


**FIG. 4.** Relationship between formation of total polar components and degradation of tocopherols. For abbreviations see materials. See Figure 1 and 2 for abbreviations.

Examination of tocopherol degradation showed that the rates varied greatly between the oils investigated (Fig. 3). Tocopherol degradation rates are reported as the time required in hours for the concentration of tocopherols in the fresh oils to be reduced by one half or half life. This method was chosen as it represented the half-life of the tocopherols under the conditions of this study. It was also chosen to describe the rate of tocopherol degradation as tocopherol losses followed a linear pattern within this range. Among the canola oils, total tocopherols in HOCO and LLCO degraded at substantially faster rates (Table 4). Within 3–6 h, the initial concentration of tocopherols was reduced by 50% for both oils. The same level of tocopherol degradation occurred between 40 and 60 h for HOLLCO and, by the end of the 72-h frying period, had not been reached for RCO.

With the exception of HOLLCO, γ-tocopherol degraded twice as fast as α-tocopherol in the other canola oils studied. This was in agreement with frying studies by Li (27), who also reported that γ-tocopherol degraded at a faster rate than α-tocopherol using four types of RCO and modified canola oils. The rate of degradation of tocopherol isomers was lowest in RCO with the fastest rate observed for HOCO. Gordon and Kourimska (28) monitored the losses of tocopherols during deep-fat frying of potato chips in low-erucic acid rapeseed oils. They reported that α-tocopherol degraded at a much faster rate than γ-tocopherol.

**Table 4**
**Total and Individual Tocopherol Degradation Rates[a]**

| | Time (h) required to reduce original levels by 50% (rate of degradation ppm/h) | | |
|---|---|---|---|
| Oil | Total tocopherols | Tocopherol α | Tocopherol γ |
| RCO | >72 (3.3) | >72 (1.4) | 60–72 (2.6) |
| HOCO | 3–6 (50.1) | 3–6 (15.0) | 3–6 (35.1) |
| HOLLCO | 48–60 (7.4) | >72 (8.0) | 36–48 (6.3) |
| LLCO | 3-6 (33.0) | 3–6 (12.5) | 3–6 (26.5) |

[a]All values are average of duplicate analysis. See Table 1 for abbreviations.

Since tocopherols act as antioxidants, oils in which they degrade rapidly would be expected to exhibit lower stability. A less stable oil, defined in this study, is one having significantly faster rates of either TPC formation or FFA accumulation. Among the canola oils, HOCO and LLCO exhibited faster rates of tocopherol degradation and were also found to have significantly ($P < 0.05$) faster rates of TPC formation. No significant differences were observed among the canola oils with respect to rates of FFA accumulation. The relationship between rates of tocopherol degradation and TPC formation is shown in Figure 4. Oils with faster rates of tocopherol degradation also tended to have faster rates of TPC formation. Factors contributing to the degradation of tocopherols are incompletely understood. Examination of the trace metals showed negligible levels of Fe, Ni, and Cu in the four canola oils studied. This study points to the danger of focusing solely on tocopherol content when studying the frying degradation of oils. Of more importance is their relative stability during frying. It is apparent, however, that plant breeders can no longer ignore the changes in tocopherols which may accompany the manipulation of fatty acids.

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