Iron Accumulation in Oil During the Deep-Fat Frying of Meat

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ABSTRACT: Iron accumulation in oil is a potential problem when frying food containing substantial amounts of iron. Selected meat products (skinless chicken breast, beef liver, and lean beef) were ground and fried (*ca*. 2-cm spheres, *ca*. 10 g/sphere) in partially hydrogenated soybean oil (PHSBO). Samples (450 g) of ground meat were fried 3 times/h for 8 h/d for 3 d. Oil samples were collected for analysis for iron (every 8 h) and oil degradation (every 4 h) and replaced with fresh oil. The iron contents of oil samples after 3 d of frying were approximately 0.11, 0.48, and 4.01 mg of iron/kg of PHSBO for the oil used to fry chicken, beef, and liver, respectively. There was a notable darkening in color and an increased tendency to foam for the beef liver oil sample compared with the other samples. After frying, the acid values were 0.9, 1.1, and 1.4 for the oil samples for chicken, beef, and liver, respectively. After frying, the *p*-anisidine values were 11.5, 12.8, and 32.6 for the oil samples for chicken, beef, and liver, respectively; the food oil sensor values were 0.96, 0.96, and 0.83 for the oil samples for chicken, beef, and liver, respectively.

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KEY WORDS: Beef liver, chicken, deep-fat frying, iron, lean beef, soybean oil.

The factors that can affect oil deterioration during frying include those that depend on the process (temperature, time, frying method, vessel type, use of filters/absorbents, oil turnover rate); the type of fat (degree of unsaturation of the oil, additives, and various minor components); and the type of food material (size, form and amount of food, nutrient and other component interchanges that occur between the foodstuff and the frying fat) (1).

Purification to remove undesirable components, including transition metals, is an important step in vegetable oil refining. After refining, chelating agents, such as citric acid, are added specifically to reduce the activity of transition metals in the oil (2,3). Many retail establishments continue the purification process by removing food particulates, oxidation products, and accumulated contaminants using specialized absorbents in combination with filter aids and specialized filtration systems to extend the life of the frying oil.

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Iron is of particular interest since it is present in much greater concentrations in meat than all other transition metals combined. The initial iron content for beef liver is 193 mg of Fe/kg of liver (National Institute of Standards and Technology standard reference material, 1577a); the average for beef is 20.9 mg of Fe/kg of beef meat cuts; and chicken breast contains 4.0 mg of Fe/kg of meat (4,5). If some of that iron is extracted into the oil during frying, the accumulated iron could reduce the frying life of the oil. In beef muscle cuts, approximately 87% of the iron is present as heme iron, whereas only 30% of the iron in chicken breast is present as heme iron.

Iron is a very effective catalyst for oil oxidation and degradation, particularly during vegetable oil storage, since iron accelerates hydroperoxide decomposition, which increases the rate of free radical generation and therefore the rate of oxidation (6). Dobarganes *et al*. (7) suggested that lipid-soluble vitamins and trace metals can leach into the frying oil and inhibit or accelerate oil oxidation depending on their antioxidant or pro-oxidant effect. When the temperature is increased from 85 to 100°C, a significant iron release from heme can occur (4). Preliminary studies under well-controlled conditions have shown that when iron is added to the oil—even partially hydrogenated soybean oil with added antioxidants—and heated to frying temperatures, there is a significant increase in the rate of oxidation and a corresponding decrease in the frying life of the oil (Coscione, A.R., and W.E. Artz, unpublished manuscript).

The catalytic activity of transition metals is a function of the ease with which the metal shifts back and forth between the various valence states. Meat contains relatively large amounts of iron in the form of heme as part of the myoglobin and hemoglobin (8). Heme proteins contain ligands that tend to block positions of potential electron flow, reducing the catalytic activity of the metal. On removal of the heme from the ligand groups, the electron flow and the catalytic activity are enhanced (9).

The high temperatures involved with frying can denature myoglobin and hemoglobin, releasing the iron-containing compounds into the oil. Although both the bound and free forms of iron have catalytic activity, experimental evidence suggests that the free form is more effective (10). Heme iron compounds do have significant pro-oxidant activity (11,12).

Although there is a substantial body of published information on the negative effects of iron on food component and food oil stability during storage and there is also considerable

evidence that heme iron does affect fat/oil stability during storage, little information is available on how much iron can accumulate in oil during frying and how the various forms of iron affect oil stability at frying temperatures. The purpose of this study was to determine whether iron accumulates in the oil. Secondarily, if it does accumulate, the purpose was to determine how much iron accumulates in the oil as a result of frying meat samples. Indices of oxidation were included to meet a limited objective, that is, to demonstrate that substantial iron accumulation could occur in the frying oil prior to the point at which the oil had degraded sufficiently to warrant discarding it. An explanation of the differences in the extent of oxidation based on meat type was beyond the scope of the work.

MATERIALS AND METHODS

A sample of commercial liquid vegetable shortening consisting of partially hydrogenated soybean and cottonseed oils (PHSBO) was obtained from ADM Packaged Oils (Decatur, IL). The oil contained TBHQ (40 ppm), citric acid (20 ppm), and the antifoam agent dimethylpolysiloxane (4 ppm).

General frying procedures. Meat samples (approximately 450 g) of lean ground beef chuck (75.0% moisture, 3.0% fat) (13), ground chicken breast (73.0% moisture, 2.5% fat) (13), or ground beef liver (73.0% moisture, 4.9% fat) (13) were fried for 3.5 min at 20-min intervals for 8 h each day of the experiment, in approximately 5.5 kg of PHSBO (ADM). According to the USDA National Nutrient Database for Standard Reference (13), the protein content for ground lean beef is 21.4%, for beef liver it is 20.4%, and it is 23.1% for skinless-boneless chicken breast.

Each meat sample was heated in a small stainless-steel electric deep-fat fryer (model F175A; Intedge Industries Inc., Whippany, NJ) with an oil capacity of approximately 5.5 L. The surface area of the oil in the fryer was 610 cm^2 (20.2 × 30.2 cm).

Spheres 2 cm in diameter, weighing approximately 10 g each, were prepared from each 450-g portion of ground meat. Preliminary analyses indicated that a frying time of 3.5 min was sufficient to cook each sample completely, so samples were fried for 3.5 min. Each frying experiment was conducted for three 8-h frying periods (one 8-h period/d for 3 d). The average diameter and weight for each sample of fried meat balls were measured and recorded (data not shown). The oil temperature averaged approximately 170°C and ranged from 165 to 180°C. The temperature of the oil during frying was monitored and recorded every 5 min in five locations (at each corner and in the center of the deep-fat fryer) during the entire experiment (data not shown). A single electric deep-fat fryer was used for frying. A 200-g sample of oil was collected after 4 and 8 h of frying each day of heating. Oil samples were place in an amber bottle, allowed to cool, and argon was used to displace the air in the sample bottle. The sample bottles were held in the dark at approximately 5°C. Each time a 200-g sample of oil was removed from the fryer, 200 g of fresh oil was added. Replicates for the iron and oil analyses were taken from the 200-g samples, which were collected every 4 h.

Physicochemical analysis. The iodine value (IV) for the PHSBO was determined in duplicate according to AOCS Official Method Cd 1d-92 (14) prior to heating.

The FA profile of the PHSBO oil sample was determined before frying by GC analysis of the FAME according to AOCS Official Method Ce 2-66 (14). Triheptadecanoin (Nu-Chek-Prep, Inc., Elysian, MN) was used as the internal standard. The capillary GC column used was a DB-1701 (60 m, 0.25 mm i.d., DF = 0.25 mm; J&W Scientific, Agilent Technologies, Folsom, CA). The initial column temperature was 190°C (2 min), and the temperature was then ramped at 0.5°C/min to a final temperature of 225°C (70 min). Helium was used as the carrier gas (1 mL/min). The injector split ratio was 1:100.

Acid value (AV) determinations were done in triplicate based on AOCS Official Method Cd 3d-63 (14).

The *p*-anisidine value (*p*-AV) analysis was based on the formation of a yellow color produced by the reaction of unsaturated aldehydes with *p*-anisidine. Analyses were determined in triplicate using AOCS Official Method Cd 18-90 (14).

The food oil sensor (FOS) provides a rapid instrumental estimate of oil deterioration based on the dielectric constant of the oil (15), since the dielectric constant increases during oxidation. An FOS model NI-21A (Northern Instruments Corp., Lino Lakes, MN) was used to measure the dielectric constant of each oil sample, and the unheated oil was used for calibration. Samples were filtered with edible oil disposable filters (Sysco Corporation, Houston, TX).

For the iron analysis, oil samples were pyrolyzed overnight in a muffle furnace (500°C) following a method adapted from Black (16) and Garrido *et al.* (17). Iron was determined at 259.9 nm with an inductively coupled plasma spectrometer, or ICP (Model ICAP 61; Thermo Elemental, Franklin, MA). Instrument operation, interelement interference correction, background correction, and data collection were controlled using ThermoSPEC/AE 6.20 software (Thermo Elemental). Blanks, calibration check standards, and reference standards were run with each analysis set. Thirty grams of each oil sample was pyrolyzed, and the ashes were dissolved sequentially in two portions of 0.05 M HCl; the first portion was heated to dryness, and the second was used to dissolve the residue and transfer it to a 25-mL volumetric flask. Sufficient deionized water was added to bring the volumetric flask to exactly 25 mL, and the extract was then analyzed by ICP. The iron determination for each sample was done in triplicate.

The polymer content of each oil sample was determined by high-performance size-exclusion chromatography according to the method modified by Artz *et al.* (18).

Comparisons of the degradation parameters of PHSBO as a function of heating time were accomplished using Statistical Analysis System software (SAS Institute, Cary, NC). Data were treated as a factorial design of three sample types

(chicken, beef, and liver) by seven frying times (0, 4, 8, 12, 16, 20, and 24 h of frying), with three frying sample replicates. The data were subjected to ANOVA for main effects and to estimate whether there were significant differences between replicates. For comparisons of treatment means, ANOVA and Duncan's multiple-range test were used, with significance accepted at a level of 95% ($P < 0.05$).

RESULTS AND DISCUSSION

The FA composition of the PHSBO sample prior to frying was as follows: 10.2 ± 0.1% palmitic, 4.8 ± 0.1% stearic, 28.0 ± 0.1% oleic, 53.3 ± 0.1% linoleic, and 3.0 ± 0.0% linolenic. The IV of the oil sample was 103 ± 8. For comparison, the FA compositions of the various meat types are shown in Table 1.

In a preliminary experiment, one 450-g sample of meat was fried each hour for 9 h each day for 7 d, for a total heating time of 63 h. The initial iron concentration of the PHSBO was less than 0.03 mg of Fe/kg of oil, the limit of sensitivity for the method used for iron analysis of the oil samples. After frying was completed, the iron concentrations in the oil samples used to fry the chicken, ground lean beef, and beef liver samples were 0.20, 0.39, and 3.35 mg of Fe/kg of PHSBO, respectively. Since the beef liver and chicken breast samples had not been ground, there was concern that a comparison between the two sample types (ground and not ground) might not be valid, so the experiment was performed again with ground meat samples. All of the figures contain data collected when frying ground meat samples.

The meat samples tested were ground to minimize differences in iron transfer from the meat samples to the oil based on particle size. The iron accumulation that occurred as a function of frying time for each of the three meat sample types during frying is shown in Figure 1.

Because of the difficulty and expense of the iron analysis, and since the primary objective was to determine whether iron can accumulate in oil as a result of frying meat, the iron analysis was performed only once each day of heating. At the end of the frying experiments, the iron concentrations in the oil samples used to fry ground chicken, ground lean beef, and ground beef liver were 0.11, 0.48, and 4.01 mg of Fe/kg of oil, respectively. The iron concentrations were significantly greater for the oil samples used to fry the ground liver (liver oil) than for the fresh oil samples, the oil samples used to fry

TABLE 1
FA Composition (%) of Lean Beef, Beef Liver, and Chicken Breast

FA[a]	Lean raw ground beef	Beef liver	Chicken breast
16:0	25.68 ± 0.10	14.33 ± 0.04	23.08 ± 0.02
16:1	—	—	3.30 ± 0.00
18:0	18.07 ± 0.07	39.59 ± 0.02	10.99 ± 0.01
18:1	41.72 ± 0.20	19.43 ± 0.05	57.66 ± 0.02
18:2	4.04 ± 0.02	27.47 ± 0.02	18.68 ± 0.01
18:3	1.40 ± 0.01	1.47 ± 0.00	1.10 ± 0.00
20:4	0.85 ± 0.01	6.48 ± 0.03	4.39 ± 0.00

[a]Source: USDA National Nutrient Database for Standard Reference, Release 16-1 (2004) (13).

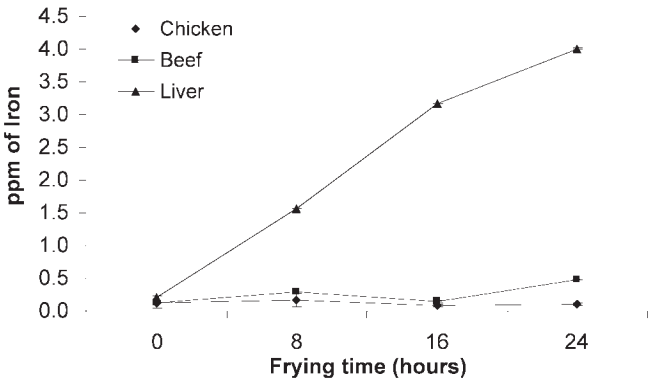

FIG. 1. Iron accumulation in frying oil during the deep-fat frying of ground chicken, ground beef, and ground beef liver. Approximately 450 g of ground meat (spheres of *ca*. 10 g, *ca*. 2 cm in diameter) was fried at an average temperature of approximately 170°C for 3.5 min every 20 min for 8 h/d, for a total of 3 d, in 5.5 kg of partially hydrogenated soybean oil. The error bars indicate SD for the replicates.

the ground chicken, or the oil samples used to fry the ground lean beef. This was probably due to the much greater iron concentration in the liver (4,5) and to the leaching of hemoglobin from the liver into the oil. Normally, beef liver is held in cold water for a short period of time to remove some of the hemoglobin, which was not done for these samples. The material extracted from the liver during frying caused rapid and substantial darkening of the oil, with a substantial accumulation of particulates in the bottom of the frying oil container, possibly due to protein coagulation. No breading was used. The iron content of the chicken oil samples was not significantly different from the beginning of the frying period to the end. For the oil samples in which the liver samples and the lean beef samples were fried, Duncan's multiple-range test showed that the iron content increased significantly with time ($P < 0.05$). The ground beef oil samples and the ground liver oil samples were different from each other at each time interval.

Figures 2–5 contain the physicochemical analysis results for the chicken oil, beef oil, and liver oil samples. Based on the AV, FOS readings, and polymer content values of the three oil sample types (ground chicken breast, ground lean beef, and ground beef liver), there was generally a statistically significant increase in the value of the oxidative indices with an increase in time, which was confirmed by ANOVA. In the case of the *p*-AV, there was no significant increase after 8 h of heating.

The results suggest that the liver oil samples generally had a greater rate of degradation than the other two oil samples (beef oil and chicken oil). A comparison of the means of the degradation parameters for the three different oil samples using Duncan's multiple-range test showed that, with the exception of FOS readings, the oil samples from the three different types of meat were significantly different from each other at a 95% confidence level.

AV results are shown in Figure 2. The AV of the oil samples ranged from 0.88 to 1.4 after the 3 d of heating (8 h/d),

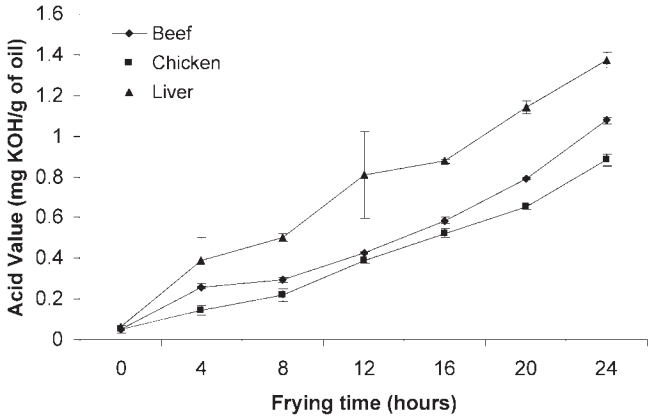

FIG. 2. Acid value results of partially hydrogenated soybean oil during deep-fat frying of ground chicken, ground beef, and ground beef liver. Approximately 450 g of ground meat (spheres of *ca.* 10 g, *ca.* 2 cm in diameter) was fried at approximately 170°C for 3.5 min every 20 min for 8 h/d, for a total of 3 d, in 5.5 kg of partially hydrogenated soybean oil. The error bars indicate SD for the replicates.

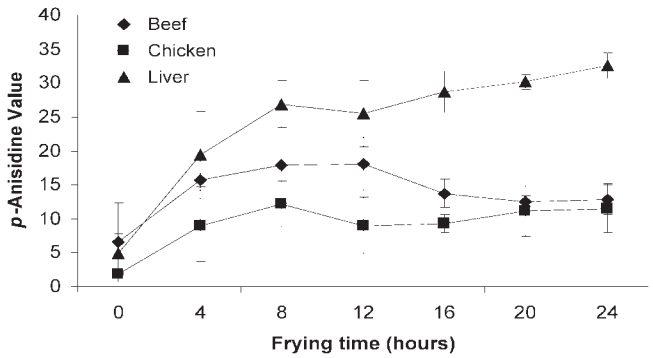

FIG. 3. The *p*-anisidine values of partially hydrogenated soybean oil during deep-fat frying of ground chicken, ground beef, and ground beef liver. Approximately 450 g of ground meat (spheres of *ca.* 10 g, *ca.* 2 cm in diameter) was fried at approximately 170°C for 3.5 min every 20 min for 8 h/d, for a total of 3 d, in 5.5 kg of partially hydrogenated soybean oil. The error bars indicate SD for the replicates.

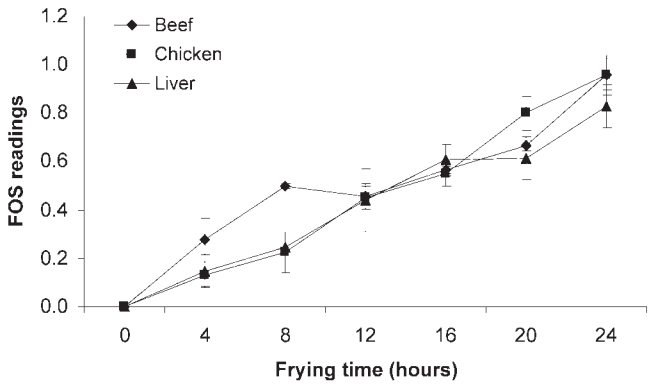

FIG. 4. Food oil sensor (FOS) readings of partially hydrogenated soybean oil during deep-fat frying of ground chicken, ground beef, and ground beef liver. Approximately 450 g of ground meat (spheres of *ca.* 10 g, *ca.* 2 cm in diameter) was fried at approximately 170°C for 3.5 min every 20 min for 8 h/d, for a total of 3 d, in 5.5 kg of partially hydrogenated soybean oil. The error bars indicate SD for the replicates.

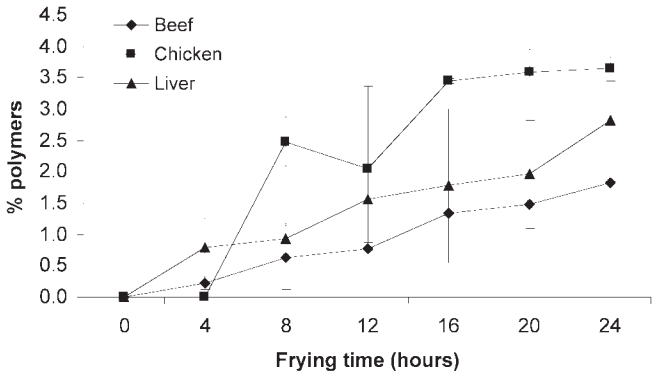

FIG. 5. Polymer content (%) of partially hydrogenated soybean oil during deep-fat frying of ground chicken, ground beef, and ground beef liver. Approximately 450 g of ground meat (spheres of *ca.* 10 g, *ca.* 2 cm in diameter) was fried at approximately 170°C for 3.5 min every 20 min for 8 h/d, for a total of 3 d, in 5.5 kg of partially hydrogenated soybean oil. The error bars indicate SD for the replicates.

or 24 h. Liver samples had a slightly greater moisture content (78%) than did the beef and chicken (75 and 73%, respectively). The additional moisture released into the oil by ground liver may have enhanced the rate of hydrolysis, explaining part of the difference in the FFA content. For the beef and chicken oil samples, the values for each of the time intervals were significantly different from the others.

Figure 3 contains the *p*-AV for the liver oil, chicken oil, and beef oil samples. Duncan's multiple-range test indicated that the *p*-AV of the three oils were significantly different from each other and that the liver oil had significantly greater values as compared with the chicken oil and beef oil samples ($P < 0.05$). Subsequently, after 4 h of heating, the chicken oil and the beef oil samples had no further significant increases in *p*-AV. In contrast, the *p*-AV for the ground liver oil samples continued to increase ($P < 0.05$) with time.

At the end of the frying period, the dielectric constants, as measured by the FOS, did not show significant differences among meat types. The FOS readings from the chicken, beef, and liver oil samples are shown in Figure 4. There was a significant increase in the FOS values for each meat sample type as a function of time.

Polymeric TAG are generated during frying as a result of oxidative and thermal reactions. According to Dobarganes and Márquez-Ruiz (19), polymers are the predominant group of nonvolatile degradation products found in used frying fats. The analysis of TAG polymers did not follow the same pattern as the other degradation parameters (Fig. 5). At a confidence level of 0.05, there was no significant difference between the samples of ground beef chuck oil and the ground beef liver oil samples. The polymer content in the chicken oil samples was significantly greater than in the other two samples, although the values for all of the oil samples was relatively low. The polymer contents after 24 h of heating were approximately 1.5% for the beef oil samples, approximately 2.8% for the liver samples, and approximately 3.5% for the chicken samples. It is not apparent why the chicken oil had

the greatest polymer content. Sánchez-Muniz *et al*. (20) suggested that during frying, changes in frying oil composition can occur because of exchanges of fat or oil between the food and the frying medium.

The oil in which ground beef liver samples were fried did have slightly higher temperatures, as compared with either the ground beef oil or the ground chicken oil samples, especially during the final hours of heating. This was probably because of the greater extent of oil degradation for the beef liver oil sample. There were no significant differences ($P > 0.05$) in temperature among locations in the fryer for the same oil at any time during frying.

The turnover rate is an important parameter with respect to the concentration of oxidation products in the frying oil (21). Each time an oil sample was removed (every 4 h) during frying, it was replaced with a fresh sample of oil, which may have contributed to the relatively low percentage of polymers formed. Approximately 20% of the oil was replaced during the experiment, which was relatively short in duration—only 24 h. This reduced the accumulation of iron in the oil slightly because of a moderate dilution effect.

A notable darkening in color was observed after addition of the first liver sample, and it continued as more liver was added. There was also an increased tendency for the beef liver oil sample to foam as heating progressed, probably because of the greater FFA values. In general, those changes were much more pronounced in the liver oil samples than in the other two oil sample types. Since the shape, moisture content, particle size, and fat content of the meat samples were relatively similar, the amount of iron transferred to the oil depended primarily on the initial iron concentration. Beef liver contains approximately 193 mg of Fe/kg, beef contains approximately 21 mg of Fe/kg, and chicken breast contains approximately 4 mg of Fe/kg (4,5). Since beef liver contains almost 10 times as much iron as lean beef and nearly 50 times as much iron as chicken breast, the differences in iron contents of the oil samples were not surprising.

Theoretically, each 450-g sample of chicken could potentially add as much as 1.8 mg of iron to the oil, assuming complete release of all of the iron in each ground chicken sample (450 g × 4.0 mg of Fe/kg of meat). This would increase the oil concentration by approximately 0.3 mg of Fe/kg of oil: (450 g × 4.0 mg of Fe/kg of meat)/(5.5 kg of oil). During the 24 h of frying, 72 samples of chicken were fried, which could potentially have increased the iron concentration in the oil to approximately 23.6 mg of Fe/kg of oil. Using similar calculations, the concentration of iron could have reached 123.7 mg of Fe/kg of oil after frying 72 samples of ground lean beef and 1,137 mg of Fe/kg of oil after frying 72 samples of ground beef liver. By simply dividing the actual increase in iron concentration attributable to the meat samples by the concentration increase that could have occurred had all of the iron been released into the oil from each meat sample, one can determine the percentage of iron released. Based on the final concentrations of iron in the three oil samples, the percentage of iron released for all three meat types was essentially the same (the percentage was 0.34% for the ground chicken, 0.36% for the ground lean beef, and 0.35% for the ground liver). This assumes the initial iron concentration in the unused oil was 0.03 mg of Fe/kg of oil. The equation for that calculation is

$$\text{percentage of iron released} = ([C] - [C_O])/[C_{TM}] \times 100\% \quad [1]$$

where $[C]$ is the iron concentration at the end of heating, $[C_O]$ is the initial iron concentration in the unused oil, and $[C_{TM}]$ is the theoretical maximum concentration if all of the iron in all of the meat samples is completely released into the oil. For chicken, the calculation is

$$0.34\% = \frac{(0.11 \text{ mg of Fe/kg of oil} - 0.03 \text{ mg of Fe/kg of oil})}{23.6 \text{ mg of Fe/kg of oil}} \times 100\% \quad [2]$$

Most important, the results unequivocally confirm the hypothesis that iron can accumulate in frying oil when meat-based food products are deep-fat fried and that iron can accumulate in sufficient concentrations (0.1 to 4.0 mg of Fe/kg of PHSBO) to potentially accelerate oxidative reactions.

To accurately assess the effect of the heme iron at temperatures in the range encountered during frying, careful comparisons must be made in model systems in which the conditions are well controlled and defined, and in which the heme iron concentration is the only variable.

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