Z Ernährungswiss 34: 308–313 (1995)
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ORIGINAL CONTRIBUTION

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# Relationship between chemical and physical indexes and column and HPSE chromatography methods for evaluating frying oil

## Zusammenhänge zwischen chemischen und physikalischen Kennzahlen und Säulen- und HPSE-chromatographischen Methoden für die Bewertung von Fritieröl

**Summary** Standard chemical and physical indexes, such as color index, acid value, and $K_{270}$ were compared with the polar content and the specific thermoxidative and hydrolytic compounds originated during 75 potato fryings with sunflower oil. The color index, acid value, $K_{270}$ and total polar content showed a significant increase ($p < 0.05$) after the first 30 fryings, followed by a tendency of the last three indexes to reach a near-steady state. Triglyceride polymers and triglyceride dimers showed a significant increase ($p < 0.05$) after 30 fryings, while the oxidized triglycerides were not increased significantly after 30 fryings. The amount of diglycerides and free fatty acids related to hydrolytic alteration did not change significantly throughout the frying operations. The significant correlations between acid value and total polar content, triglyceride dimers, triglycerides polymers and oxidized triglycerides, or between color index and polar content and triglyceride polymers or between $K_{270}$ and triglyceride polymers indicate that these simple and standard methods can be applied and they are as useful as more specific methods of evaluation in the monitoring of frying operations, provided that initial values of all of these indexes are available.

**Zusammenfassung** Chemische und physikalische Standardkennzahlen, wie zum Beispiel der Refraktions- und Farbindex, die Säurezahl und der $K_{270}$-Wert, wurden mit den Gehalten an polaren Verbindungen sowie spezifischen thermooxidativen und hydrolytischen Fettbestandteilen verglichen, die in einem Sonnenblumenöl, das 75 mal zum Fritieren von Kartoffeln benutzt worden war, entstanden sind. Der Farbindex, die Säurezahl, der $K_{270}$-Wert und der Gehalt an polaren Verbindungen stiegen signifikant ($p < 0,05$) nach 30 Fritierungen. Anschließend stabilisierten sich die drei letztgenannten Indices. Der Inhalt an polymeren Verbindungen und Triglyceridendimeren stieg signifikant nach 30 Fritierungen an ($p < 0,05$), während die oxidierten Triglyceride keine signifikante Steigerung aufwiesen. Der Gehalt an Diglyceriden und freien Fettsäuren, als Maß der hydrolytischen Vorgänge, zeigte während des Fritierprozesses keine signifikante Veränderung. Die signifikanten Korrelationen zwischen der Säurezahl und dem Gehalt an polaren Verbindungen, den dimeren und polymeren Triglyceriden und den oxydierten Triglyceriden, zwischen dem Farbindex und dem Gehalt an polaren Verbindungen und polymeren Triglyceriden, oder zwischen dem $K_{270}$-Wert und den polymeren Triglyceriden zeigen, daß diese einfachen Standard-Methoden benutzt werden können und genauso nützlich sind wie mehr spezifische Methoden zur Bewertung von Fritierprozessen, wenn man die Ausgangswerte aller dieser Indices kennt.

**Key words** Deep-fat frying – sunflower oil – column and HPSE chromatographies – color index – acid value – UV absorption

**Schlüsselwörter** Ölbad-Fritierung – Sonnenblumenöl – Säulen- und HPSE-Chromatographie – Farbindex – Säurezahl – Refraktionsindex – UV-Absorption

Received: 27 January 1995
Accepted: 24 July 1995

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## Introduction

Cooking oils used for the preparation of fried foods are exposed to increased temperatures in the presence of air and moisture. Under these conditions a number of chemical reactions takes place, the major of which are hydrolysis, oxidation, thermal decomposition and polymerization (1, 5, 10, 16, 21).

These changes are of considerable interest, both for the performance of the oil as a heat transferring medium and its role as an ingredient of the fat food (6). During the process unavoidable chemical reactions resulting in decomposition products which have a significant effect on the flavor, color and texture of the food, as well as the period of time an oil is usable for frying. In addition, these decomposition products may affect human health (27).

In consequence, there is a need to define the point at which the fat or oil ought to be replaced. This is linked to the content of polar compounds formed during fryings. Today, it is considered that either a fat or an oil for frying purposes must be discarded when its polar content is higher than 25 % (6).

Waltking and Wessels (28) described a method based on the separation by column chromatography on silica-gel of two fractions, one containing the unaltered part of the fat or nonpolar component, which are unaltered triglycerides, and a second one concentrating the altered products or polar components of frying fats.

With the advance of chromatographic techniques such as high performance size-exclusion chromatography (HPSEC), new procedures of evaluation have been developed which offer perspectives on further examination of the polar and polymeric material (7, 11).

Nevertheless, when laboratory facilities are not available, changes occuring in heated oils can be determined by physical and chemical tests, which still constitute a common and useful practice (8, 16). Besides the physical tests, the colorimetric index (related to altered compounds such as unsaturated carbonyl compounds) is now widely accepted. In addition, besides the chemical tests, the determination of the acid value is a standardized method for free fatty acid evaluation. On the other hand, the analytical indexes based on physical and chemical changes are widely used in the fried foodstuff related industries because they are easy to analyze (16). Nevertheless, the relationships between the physical and chemical tests and the analytical methods measuring more specific compounds related to frying fat alteration have not been definitively established.

The aim of this study is to evaluate the alteration of a sunflower oil used in 75 repeated fryings of potatoes by measuring the total polar material, and the specific compounds from this polar fraction, and using analytical methods such as color index, acid value and absorption at 270 nm. In addition, correlations between total polar content and the analytical indexes based on general chemical and physical changes and the number of fryings were studied. Some further correlations between these physical and chemical tests and percentage of total polar content and also between these physical and chemical tests and the most specific compounds related to frying fat alteration such as triglyceride polymers, triglyceride dimers, oxidized triglycerides, diglycerides and free fatty acids were established.

## Materials and methods

### Performance of frying

Refined sunflower oil (Córdoba, Spain) and potatoes were purchased at a local store. The oil was stored light-protected below 15 °C and used as purchased.

Domestic deep-fat fryers with a 3-l aluminum vessel were used for frying. The potatoes were chopped into slices of ca. 2 mm. The proportions of food to frying oil in the repeated fryings were kept at 500 g/3 l. Because so much oil is removed along with the fried potatoes, it was necessary to replenish the fryer bath with unused oil. Throughout the first 20 fryings, the frying bath volume was replenished with fresh oil every four fryings, following a parallel scheme described in a previous work (8). After the 20th frying, the fryer volume was made up with unused oil every five fryings to carry out 10 fryings per day, because the addition of fresh oil every five fryings instead of every four fryings did not change the aim of this study. A total of 75 fryings was carried out. The time required to reach and keep the bath oil at 180 °C, before adding the potatoes was 20 min. The potatoes were then fried for 8 min. After the end of each frying operation, the oil were again heated to 180 °C before starting a new frying. The time required was 10 min. The overall time the oil was heated throughout the whole experiment can be estimated as 25.17 h. The oil loss was about 10 % every five fryings, which implies the addition of 4.5 l of fresh oil throughout the 75 fryings carried out.

### Analytical indexes

Color index was determined according to the method of Wolff (29).

Acid value was performed according to the method outlined in (25).

$K_{270}$ was determined following the procedure outlined in (26).

Aliquots from the unused and used oil from the 20th, 30th, 50th and 75th fryings were taken for analysis.

### Determination of the percentage of the polar fraction

The polar fraction was evaluated by the column chromatographic method of Waltking and Wessels (28), with a modified proportion of petroleum ether/diethyl ether used to fill the column and to elute the nonpolar fraction following the method described in a previous work (10).

Two samples each of unused oil and of used oil from the 20th, 30th, 50th and 75th fryings were analyzed.

The separation of the nonpolar and polar fractions was checked by thin-layer chromatography on 0.5-mm-thick 60 F 250 silica gel plates (20 x 20 cm glass). Polar and nonpolar fractions were diluted 50 times (wt/vol) in hexane/diethyl ether (80:20, vol/vol). Samples were applied as 10-µl spots with a 705 Hamilton microsyringe. Plates were developed with hexane/ethyl ether/acetic acid (80:20:1, vol/vol/vol).

### High performance size exclusion chromatography (HPSEC)

The polar fraction previously obtained by column chromatography, as described before, was analyzed by HPSEC, following the method of Dobarganes et al. (11). Isolated polar fractions were analyzed in a Konic 500 A chromatograph (Barcelona, Spain) with a 10-µl sample loop. A Hewlett-Packard 1037 A refractive index detector (Palo Alto, CA) and two 300 mm x 7.5 mm i.d. (5 µm particle size) 0.01 µm and 0.05 µm pL gel (polystyrene-divinylbenzene) columns (Hewlett-Packard), connected in series, were operated at 40 °C. HPLC-grade tetrahydrofuran served as the mobile phase with a flow of 1 ml/min. Sample concentration was 10–15 mg/ml in tetrahydrofuran. All eluents, as well as samples, were precleaned by passing them through a filter (2 µm). The amount of the different alteration compounds was evaluated following the procedure outlined in (10).

### Statistical methods

Results obtained from the different fryings were compared by Newman-Keuls multiple comparison test. Linear correlations were studied using the product moment correlation test (12).

## Results and discussion

The results of the analytical indexes based on general chemical and physical changes obtained from the unused sunflower oil and the corresponding oil after different fryings are given in Table 1.

After 20 fryings the color index of the oil showed a significant increase followed by significant changes until the 75th frying (Table 1) in agreement with the finding of a previous work (22). A high and significant correlation between this index and the number of fryings was also found (Table 2). Frying of food resulted in the darkening of oils, because of oxidation and of the colored pigments from the foods which diffused into the oils (13, 30).

The acid value also increased significantly towards the 75th frying (Table 1). The correlation between the acid value and the number of fryings was high and significant (Table 2). Increases in the acid value are fundamentally due to both hydrolytic reactions and the increase of free fatty acids (FFA), as has been described previously (16). Determination of FFA appears to be the method favored by many operations for quality control evaluation of the frying fats (24). Nevertheless, the level of FFA found in

**Table 1** Analytical indexes based on general chemical and physical changes, total polar content, and thermoxidative and hydrolytic alteration of compounds in the sunflower oil used in 75 successive fryings of potatoes[1)]

| | Frying 0 | Frying 20 | Frying 30 | Frying 50 | Frying 75 |
|---|---|---|---|---|---|
| Color index[a] | 1.95 ± 0.07a | 3.75 ± 0.04b | 5.17 ± 0.31c | 8.07 ± 0.53d | 14.68 ± 0.60e |
| Acid value[a] | 0.05 ± 0.01a | 0.23 ± 0.03b | 0.30 ± 0.01d | 0.35 ± 0.01d | 0.46 ± 0.03e |
| $K_{270}$[a] | 3.63 ± 0.01a | 4.39 ± 0.18b | 7.05 ± 0.19c | 10.98 ± 0.21d | 13.53 ± 0.29e |
| Total polar content[b] | 5.09 ± 0.21a | 15.99 ± 0.40b | 17.99 ± 0.41b | 18.92 ± 0.49b | 19.11 ± 0.40b |
| Triglyceride polymers[b] | 0.10 ± 0.01a | 1.65 ± 0.12b | 2.50 ± 0.20c | 3.15 ± 0.20d | 3.44 ± 0.17d |
| Triglyceride dimers[b] | 0.75 ± 0.12a | 6.25 ± 0.30b | 7.09 ± 0.30bc | 7.37 ± 0.44bc | 7.51 ± 0.34c |
| Oxidized triglycerides[b] | 2.70 ± 0.27a | 6.26 ± 0.30b | 6.49 ±0 .27b | 6.58 ± 0.39b | 6.26 ± 0.30b |
| Diglycerides[b] | 1.11 ± 0.17a | 1.33 ± 0.06a | 1.32 ± 0.10a | 1.39 ± 0.10a | 1.41 ± 0.02a |
| Free fatty acids[b] | 0.43 ± 0.10a | 0.50 ± 0.07a | 0.59 ± 0.08a | 0.43 ± 0.07a | 0.48 ± 0.05a |

[1)] Values are mean ± SD. [a]Values (mean of three samples). [b]Values in mg/100 mg oil (means of two samples). Values in the same row bearing different letters are significantly (Newman-Keuls multiple comparison test) different ($p<0.05$).

**Table 2** Pearson correlations between the analytical indexes based on general chemical and physical changes and total polar content and number of fryings in the sunflower oil after being used 75 times to fry potatoes[1]

| | Correlation |
|---|---|
| Color index | 0.9704a |
| Acid value | 0.9675a |
| $K_{270}$ | 0.8452a |
| Total polar content | 0.7999b |

[1] Values bearing the letter a or b are significant ($p<0.01$ or $p<0.05$, respectively).

the frying fat not only reflects those formed during the frying process, but also the level of FFA initially present in the fat before heating (2, 13). In addition, FFA are formed during frying, both by oxidation and by hydrolysis. The rates of these processes vary according to a number of variables including the type of fats being used and initial FFA level. The rates may also vary over time within the same operation. Although the products of hydrolysis have been shown to have little effect on the nutritional quality of the fried food and although changes in this type are generally small in a deep-fat frying operation, the methods used for FFA determination do not permit differentiation between FFA formed by hydrolysis and those formed by oxidation (13). This being the case, use of FFA alone does not permit to indicate when frying fat should be dumped.

After 20 fryings the ultraviolet absorption ($K_{270}$) showed a significant increase followed by significant changes towards the 75th frying (Table 1), in agreement with the findings of other workers (8, 16). A significant correlation between this index and the number of fryings was also found (Table 2). Ultraviolet absorption, which evaluates the formation of conjugated compounds (dienes and trienes) due to the shift of the double bond during frying (23), logically undergoes a much more marked variation in very unsaturated fats which is independent of the total alteration.

The alteration of the sunflower oil employed in 75 repeated deep-fat fryings of potatoes evaluated by measuring the polar content has the great advantage of evaluating compounds specifically related with the degradation (10).

Total polar content increased from 5.09 mg/100 mg oil in the unused oil to 19.11 mg/100 mg oil in the last frying as is shown in Table 1. Billek (4) found that the smell and taste of fats that contain more than 25 % polar material is still acceptable, but when the content tops 30 % the fats were considered unacceptable and deteriorated. In the present work a level of ~19 mg/100 mg oil of polar content was found. This result suggests that the sunflower oil used for frying is still acceptable and could be employed for producing more potato chips. The correlation between the total polar content and the number of fryings was significant (Table 2). Some authors (8, 9) reported an increase of polar fraction along the number of fryings when samples from olive oil used in repeated deep-fat fryings of potatoes were analyzed, and have also indicated that the speed of degradation is proportional to the temperature and the duration of frying.

In addition, the number and variety of products formed in the frying process are great and the nutritional and toxicological consequences of their consumption are largely unknown. Consequently, there is a need to evaluate the hydrolytic and thermoxidative products in the frying fats.

Although many analytical methods have been used for determination of the monomers, dimers and higher polymers of oxidized fats and oils, the techniques of HPSEC may be considered as one of the most promising because it increases the possibility of quantitation of all classes of alteration compounds: polymers and dimers of triglycerides, oxidized triglycerides, diglycerides, and free fatty acids (6, 14).

The results of the HPSEC studies from fresh and used sunflower oil samples are presented in Table 1 and the HPSE chromatograms are identified in Fig. 1. These results indicate that the amount of triglyceride dimers, and triglyceride polymers tended to increase rapidly during the first 20 fryings, followed by minor but also significant changes during continued fryings. In addition, oxidized triglycerides also increased during the first 20 fryings but did not increase further with continued fryings. Data suggest that repeated fryings of potatoes in sunflower oil with a fast turnover of fresh oil during the frying operations rapidly increased the level of polar compounds related to thermoxidative alteration in the fryer oil during the first 20 fryings followed by a tendency to reach a near-steady state. According to Bender (3), changes due to the degradation of a fat affected its palatability more than its nutritive value. Most of the identified compounds from heated oils have often been quoted as the usual products of oxidation responsible for the "flavor" deterioration of oils (20). The amount of diglycerides related to hydrolytic alteration did not have the tendency to increase significantly during frying. The amount of free fatty acid remains unaffected. As has been described (11), the quantitation of diglycerides allows the determination of the contribution of hydrolytic alteration, because these compounds remain in the fat. The results clearly indicate that in deep-fat frying of potatoes thermoxidative reactions take place more often than hydrolytic reactions. Several authors reported that after thermal oxidation sunflower oil contained higher amounts of dimers and oligomeric triglycerides than did the unheated oil (14, 18, 19).

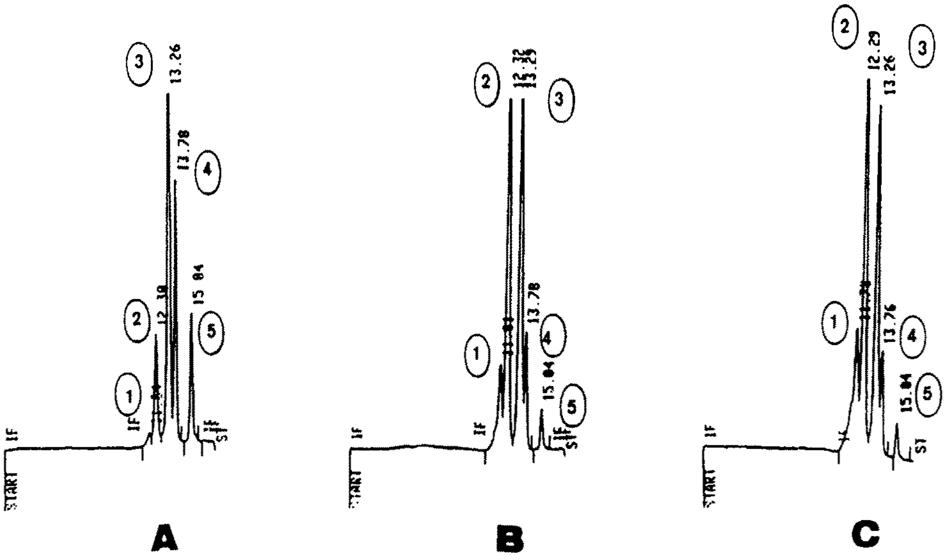

**Fig. 1** High-performance size-exclusion chromatograms of unused (A) and used oil samples: B (20th), C (75th) fryings. Peaks 1, 2, 3, 4, and 5 are triglyceride polymers, triglyceride dimers, oxidized triglycerides, diglycerides and free fatty acids, respectively. Conditions: column, series connected polystyrene-divinylbencene, 300 mm x 7.5 mm i.d. (5 μm particle size) eluent, tetrahydrofuran a 1 ml/min, 10-μl injection volume; refractive index detection.

Table 1 also shows the similarity in the changes produced with continued fryings between total polar content, triglyceride polymers, triglyceride dimers, oxidized triglycerides $K_{270}$, acid value and color index.

Further linear correlations between the chemical and physical indexes and the polar compounds related to thermoxidative and hydrolytic alteration of the sunflower oil used in repeated frying were established.

A significant correlation between color index and polar content or triglyceride polymers was found. Stevenson et al. (24) reported that the color development also parallels the development of polar content. This author described that in a survey done by the Department of Food and Nutrition at the University of Manitoba of 18 food service operations, color was used by the majority as the major indicator for fat dumping. A significant correlation between the acid value and total polar material, triglyceride dimers, triglyceride polymers or oxidized triglycerides was also found.

Studies also conducted in the Department of Food and Nutrition at the University of Manitoba have shown good correlation between both the acid value and polar determinations for both solid and liquid canola and soybean-based fats in which frozen french fries were cooked (24).

Another study by Hara et al. (17) also showed a good correlation between the contents of polar material and polymers and acid value in agreement with the finding of this work. $K_{270}$ was highly and significantly correlated with triglyceride polymers. Gere (15) found with advancing heating time the amount of decomposition pro-

**Table 3** Pearson correlations between the different indexes based on general chemical and physical changes and polar compounds related to thermoxidative and hydrolytic alteration of sunflower oil used in 75 successive fryings of potatoes

| | Color index | Acid value | $K_{270}$ | Triglyceride polymers | Triglyceride dimers | Oxidized triglycerides | Diglycerides | Free fatty acids |
|---|---|---|---|---|---|---|---|---|
| Color index | | | | | | | | |
| Acid value | 0.9079 | | | | | | | |
| $K_{270}$ | 0.8139 | 0.9183 | | | | | | |
| Triglyceride polymers | 0.8342 | 0.9848 | 0.8975 | | | | | |
| Triglyceride dimers | 0.6407 | 0.9063 | 0.6850 | 0.9325 | | | | |
| Oxidized triglycerides | 0.5182 | 0.8299 | 0.5689 | 0.8688 | 0.9876 | | | |
| Diglyceride | 0.7507 | 0.9450 | 0.7807 | 0.9543 | 0.9733 | 0.9427 | | |
| Free fatty acids | -0.0562 | 0.2109 | -0.1056 | 0.2122 | 0.4151 | 0.4543 | 0.2197 | |
| Polar content | 0.8853 | 0.9088 | 0.600 | 0.9452 | 0.9992 | 0.9823 | 0.9777 | 0.4106 |

$p<0.05$, when $0.553>r>0.684$; $p<0.01$, when $r>0.684$

ducts – i.e., polymer content polar compounds and absorbance at 232 – increases. Most oils samples with higher total polar contents were higher in dienes and trienes (232 mm). These compounds can form polymers and an equilibrium between the rate of formation of conjugated dienes and the rate at which those compounds form polymers may occur during frying (30).

Finally, the linear relationship (Table 3) suggests an adequate correlation between the contents of polymers and polar material with a coefficient of correlation larger than 0.95.

These experimental data suggest that the measurements of polar material and/or polymers, the main deteriorative products in heated oil, provide an adequate means of evaluating frying oils. As the major polar materials are polymerization products, the correlation between the contents of polar material and polymers was fairly good, as shown in Table 3. The polar contents method is of most use in research although it is time-consuming and requires skilled personnel. However, the quick-test methods need little technical expertise and can be done at restaurants and food service institutions. On the other hand, enumeration of the inconveniences of the physical and chemical tests mentioned above is intended only as a warning of their indiscriminative use and in no way implies doubt about their efficiency or use of these tests as criteria for discarding fat, which is in agreement with the results obtained in this study.

## Conclusion

Discontinuous and successive fryings of potatoes in sunflower oil, with addition of fresh oil during the performance of fryings, rapidly increased the amount of total polar material and the amount of polar compounds related to thermoxidative alteration in the frying oil during the first 20 fryings, followed by a tendency of this polar material and some of these polar compounds to reach a near-steady state. In addition, color index, acid value, and ultraviolet absorption also increased with the number of fryings. Correlations suggest that chemical and physical indexes, such as acid value, $K_{270}$, and color index could be applied in the monitoring of frying operations, provided that the values of such indexes in the unused oil are known.

**Acknowledgements** Financial support of this work by the Spanish Comisión Interministerial de Ciencia y Tecnología (Projects ALI 88-0696 and ALI 92-0289-C02-01) is gratefully acknowledged. Thanks are due to Mrs. I. Orvay for preparation of the manuscript.

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