Regulation of used frying fats and validity of quick tests for discarding the fats

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SUMMARY

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During deep-fat frying, a complex series of reactions takes place resulting in hydrolysis, oxidation and polymerization of the oil. As quality of fried foods is affected by that of the oil, regulations or guidelines have been established in many countries to guarantee high quality foods. In this lecture, present regulations mainly focusing on oil deterioration and setting limits to alteration compounds are reviewed. Among analytical methods to control oil quality, polar compound and polymer determinations stand out.

Studies carried out in different countries indicate the need for improving the quality of frying oil to produce more nutritious fried foods. Practical deep-fat frying can be divided in industrial and fast-food segments, characterized by the use of continuous and discontinuous fryers, respectively. From the analysis of a high number of samples it is deduced that the present situation is very different in both segments and that the main problem is to determine when the fat or oil has to be replaced where there are no laboratory facilities. Hence, simple rapid analytical tests to substitute for official methods are necessary to monitor oil quality in restaurants and fried food outlets. Data of validity of rapid tests will be presented with particular emphasis on the utility of commercialized kits like Oxifrit-Test and Veri-Fry to improve the quality of frying fats and oils.


1. INTRODUCTION

Over the last forty years there has been a great increase in the consumption of frying fats and oils, following the development of a wide range of new products among which frozen prefried foods and fast foods stand out.

During the frying process, the oil or fat is exposed to high temperature in the presence of air and moisture. Under these conditions, a complex series of reactions takes place resulting in the loss of quality of the frying oil and of the fried food. Although general modifications of the main fat constituents -oxidation, polymerization, hydrolysis, etc- are well-known, it is not easy to foresee the rate of fat degradation due to the high number of variables involved in the frying process. Some are linked to the process itself, such as temperature, length of heating, continuous or discontinuous heating, turnover rate, etc.; others to the food subjected to frying, i.e., lipid composition, main and minor constituents, etc.; or else to the oil or fat used e.g., unsaturation degree, initial quality and additives.

Both facts, namely, the important volume of frying fats and oils and the loss of quality during the frying process, have stressed the need for quality control by establishing limits to fat degradation through official or unofficial regulations.

2. REGULATIONS ON FRYING FATS AND OILS

Present regulations have their origin in the recommendations given by the German Society for Fat Research (DGF) to limit the alteration of frying fats for human consumption (Billek et al., 1978). Based on the analyses of a high number of samples, the following criteria were established in 1973:

A used frying fat is deteriorated if,

- without any doubt, odor and taste are not acceptable,
- in case of a doubtful sensory assessment
  - the concentration of petroleum ether insoluble oxidized fatty acids is 1.0%.
  - the concentration of petroleum ether insoluble oxidized fatty acids is 0.7% or higher and the smoke point is lower than 170°C.

It is interesting to observe the primacy of the organoleptic evaluation and the importance of the oxidation compounds in chemical evaluation. The main problem of these initial criteria is the complexity of both analytical methodologies. Determination of oxidized fatty acids (OFA) is a laborious method requiring highly skilled personnel while sensory evaluation is not easy and a expert panel is needed.

The development of polar compound determination (Guhr and Waibel, 1979; Gertz, 1979), later proposed by IUPAC (Waltking and Wessels, 1981) for the control of frying fat quality was an undoubted analytical improvement which contributed to the emergence of the present regulations. Polar compounds (PC) are determined by adsorption
chromatography on silica columns and the value obtained corresponds approximately to the total alteration compounds formed during the frying process. The two main positive characteristics of this evaluation are:

a) It is an objective method clearly related to the quality of the fat. Thus, the higher the level of polar compounds, the lower the quality.

b) It is simple, accurate and reproducible. Moreover, the efficacy of the chromatographic separation can be easily checked.

Given that the amount of PC was found to be very well correlated to the content of OFA, in a new symposium of the DGF in 1979, polar compound determination was recommended as a new criterium. A value of 27% PC corresponding to 0.7% OFA, was initially introduced.

Since then, determination of polar compounds has become the method most generally accepted for quality evaluation of frying fats, as demonstrated by its inclusion in all the present official regulations, summarized in Table I.

### Table I
**Limitations established in official regulations on used frying fats and oils**

<table>
<thead>
<tr>
<th>Country</th>
<th>Polar Compounds (%)</th>
<th>Free Fatty Acids (%)</th>
<th>Oxidized Fatty Acids (%)</th>
<th>Smoke Point (°C)</th>
<th>Polymers (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AUSTRIA</td>
<td>27</td>
<td>2.5</td>
<td>1</td>
<td>170</td>
<td>–</td>
</tr>
<tr>
<td>BELGIUM</td>
<td>25</td>
<td>2.5</td>
<td>–</td>
<td>170</td>
<td>10</td>
</tr>
<tr>
<td>FRANCE</td>
<td>25</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>GERMANY</td>
<td>24</td>
<td>2.0</td>
<td>0.7</td>
<td>170</td>
<td>–</td>
</tr>
<tr>
<td>ITALY</td>
<td>25</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>SPAIN</td>
<td>25</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
</tbody>
</table>

As can be observed, PC percentage is limited in all the countries subjected to official regulations although the values differ slightly from one to another. Apart from the initial criteria of OFA and smoke point, free fatty acids and polymer content, the most characteristic groups of compounds originated due to the moisture of the food and to the high temperature of the frying process, respectively, have also been considered in some countries.

Finally, many other countries have adopted specific recommendations or guidelines based on similar criteria (Firestone, 1993) which reflect the increasing interest in the control of used frying fats and oils to improve the quality and nutritive properties of fried foods.

### 3. QUALITY OF USED FRYING FATS AND OILS

An interesting aspect to review next is the quality of frying fats and oils in relation to the existing regulations. Table II shows the results obtained in our laboratory on a significant number of samples belonging to the three segments most characteristic of the frying sector: domestic use, restaurants and fried-food outlets, and industrial frying.

#### Table II
**Polar compounds (PC) in used frying fats from different sources**

<table>
<thead>
<tr>
<th></th>
<th>DISCONTINUOUS FRYERS</th>
<th>CONTINUOUS FRYERS</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>DOMESTIC USE</td>
<td>RESTAURANTS AND FRIED FOOD OUTLETS</td>
</tr>
<tr>
<td>Number of samples</td>
<td>72</td>
<td>190</td>
</tr>
<tr>
<td>Polar compound</td>
<td></td>
<td></td>
</tr>
<tr>
<td>range (wt%)</td>
<td>10.5-42.1</td>
<td>3.1-61.4</td>
</tr>
<tr>
<td>Number of samples</td>
<td></td>
<td></td>
</tr>
<tr>
<td>with PC &gt; 25%</td>
<td>24</td>
<td>69</td>
</tr>
</tbody>
</table>

In general, the industrial segment is characterized by the utilization of continuous fryers (200-800 L) while discontinuous fryers (2-50 L) are used in the others. It is interesting to observe that lower quality was found in segments using discontinuous fryers and mainly in fats collected from restaurants and fried-food outlets.

Samples from continuous fryers were supplied by industries involved in the production of chips, french fries, doughnuts and prefried fish. Normally, a single food was produced in these fryers and high turnover rates were necessary to replace the oil retained in the product during frying. Under these circumstances, polar compound level reaches a constant value below 25% and, only exceptionally, the oil has to be replaced.

Quite in contrast, degradation was much higher in discontinuous fryers. Due to the occurrence of cooling-heating cycles, and of periods in which the oil is heated without presence of food, the turnover rate is, in general, very low and the oil has to be replaced frequently.

These differences would explain, in part, the high percentage of samples with polar compound levels higher than permitted found in the segments using discontinuous fryers. The other complementary reason pertains to the difficulties of controlling quality. Unlike the industrial users, most of these establishments do not have immediate access to laboratory facilities to confirm when a frying oil has to be replaced.

Table III shows detailed comparative results of quality evaluation in different countries based on samples collected by Food Inspection Services in catering, restaurants and fried-food outlets. Limitations for PC and polymer determinations established in...
the regulations have been surpassed in a significant number of samples and this was commonly found in all countries surveyed. In view of these results, it is evident the need for solutions to improve the quality of frying fats, the only way the produce more nutritious foods.

Table III
Polar compound (PC) and polymer contents in used frying fats from restaurants and fast-food outlets

<table>
<thead>
<tr>
<th>REFERENCE</th>
<th>No. of samples analyzed</th>
<th>Polar compounds Range (wt%)</th>
<th>Polymers Range (wt%)</th>
<th>No. of samples with PC &gt; 25%</th>
<th>% of samples with PC &gt; 25%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coon et al. (1986)</td>
<td>100</td>
<td>1-55</td>
<td>–</td>
<td>38</td>
<td>38.0</td>
</tr>
<tr>
<td>Gertz (1986)</td>
<td>125</td>
<td>5.8-57.7</td>
<td>1.7-35.0</td>
<td>44</td>
<td>35.2</td>
</tr>
<tr>
<td>Sebedio et al. (1987)</td>
<td>31</td>
<td>8.2-54.6</td>
<td>0.8-39.5</td>
<td>15</td>
<td>48.4</td>
</tr>
<tr>
<td>Dobarganes and Márquez (1995a)</td>
<td>174</td>
<td>3.1-61.4</td>
<td>0.2-47.6</td>
<td>60</td>
<td>34.5</td>
</tr>
<tr>
<td>Skröcki (1995)</td>
<td>20</td>
<td>7.7-55.8</td>
<td>–</td>
<td>12</td>
<td>60.0</td>
</tr>
</tbody>
</table>

4. RAPID METHODS FOR QUALITY CONTROL OF USED FRYING FATS

Efficiency of quality control is dependent on the existence of objective analytical methods, compatible with the needs of the frying users. In this respect, it is known that, even though polar compound determination is accepted worldwide as the best method for quality evaluation of frying fats, it is only applied in public laboratories connected to Inspection Services and in research studies. The main reason resides in its being an expensive and time-consuming technique requiring technical personnel and laboratory facilities.

Although there are many different analytical techniques to be used for quality evaluation of frying fats (White, 1991), in real practice, food processors with laboratory facilities make use mainly of the percentage of free fatty acids, which is rapid and relatively easy to measure. This analytical method, measuring hydrolytic alteration, has a very poor correlation with polar compounds when samples from different origin are considered but high correlations can be found for a specific industry under well-controlled frying conditions. This is also observed in the case of color evaluation, which is the second analytical method most widely used in continuous frying. Moreover, in some industries, the quality of the finished food is limited by oil free fatty acid content or color, even if the value of 25% PC is far away to be reached.

In discontinuous frying segments, the most generally used methods are based in physical changes which reliability depends on the skill of the fryer operator. The main criteria include color, foaming, smoking, odor and length of fat use (Stevenson et al., 1984). In these establishments, unintentional abuses occur often due to the lack of correlation between this criteria and polar compound percentages.

In search of solutions to these problems, the following rapid procedures have been developed and commercialized to provide a rapid means of testing frying oil abuse.

— Foodoil sensor (FOS)

The Foodoil sensor is a portable electronic instrument, commercialized by Northern Instruments Corp., measuring the change of the dielectric constant in frying fat relative to fresh oil (Graziano, 1979). The instrument is calibrated with fresh oil of the same type as the frying fat and the range of measurement is from 0 to 6. It is suggested that an FOS reading of 4 should be the limit beyond which the frying fat should be discarded.

— OXIFRIT-TEST

Oxifrit-Test is a colorimetric test-kit, commercialized by Merck, which contains redox indicators reacting with the total amount of oxidized compounds in the sample (Meyer, 1979). After mixing two reagents, the oil is added and the developed color of the mixture is compared with a four-color scale with 1 (blue) representing good, 2 (blue-green) still good, 3 (green) replace the fat, and 4 (olive green) abused fat.

— FRITEST

This test, also commercialized by Merck, is a colorimetric test-kit that is sensitive to carbonyl compounds (Von Zeddelmann, 1973). The mixture of sample and reagents is compared to a three-colour scale from yellow to orange. A value of 2 or higher indicates that the fat should be discarded.

— VERI-FRY

This test, commercialized by Libra Laboratory Inc., also contains redox indicators changing from blue to green as polar compounds increase (Litovsky et al., 1991). It is the easiest test to perform as it is only necessary to add the oil to the tube up to a marked sign. The color developed after mixing reagents and oil is compared with a five-color scale. A value of four (green) indicates that the fat should be rejected.

All these rapid tests are cheap, rapid and safe to assess frying fat quality on-site by untrained personnel and only simple or no equipment is required. A significant improvement in the quality of used frying fats would be obtained if food inspectors and fryer operators agreed in a common rapid test and define cut-off levels from which the fats should be replaced.
5. VALIDITY OF RAPID COMMERCIAL TESTS

The last aspect to be defined is the capability of these tests of substituting for official methods where there are no laboratory facilities.

In general, good correlations have been found between all the rapid tests above commented and the standard method for polar compound determination. The highest correlations have been reported in different studies for the measurement of the dielectric constant (Croon et al., 1986; Smith et al., 1986; Augustin et al., 1987). Hence it is very adequate for quality control in restaurants and fried food outlets. The main drawback is, however, that fresh fats and oils differ in dielectric constants and thus the instrument has necessarily to be calibrated against the initial fat. On the other hand, the reading is influenced by the water and lipids coming from the food, which would be particularly important if the fryer is being used for the final preparation of prefried foods because of solubilization of the initial product fat into the fryer.

Oxifrit-test, Fritest and Veri-Fry also correlate well with polar compound determination, coefficients being between 0.7 and 0.8 (Croon et al., 1986; Litovsky et al., 1991). All these colorimetric tests are very easy to handle and take no more than 4-5 minutes. In the case of Oxifrit-test and Veri-Fry, changing color from blue to green, researchers have found the color differentiation easier to read than with the Fritest, changing from yellow to orange.

Nevertheless, in quality control of used frying fats it is not so important to achieve significant correlations in the complete range of polar compounds and the selected test than it is to know the capability of the test of defining whether the fat is deteriorated or not. In this regard, there are two possibilities of obtaining false results:

a) False positive results are obtained when the rapid test indicates that the fat should be rejected even though the percentage of polar compounds is lower than the limit established in the regulation.

b) False negative results are obtained when the rapid test indicates that the fat can be further utilized even though polar compound percentage is higher than the limit established in the regulation.

If the primary goal consists on finding an useful rapid test for quality control, both the number and the type of false results should be considered for its selection.

Table IV summarizes the results obtained in our laboratory, corresponding to a study developed in collaboration with the Food Inspection Services in Andalucía (Dobarganes and Márquez-Ruiz, 1995b). Number of samples analyzed and significant results were included in Table III (Dobarganes and Márquez-Ruiz, 1995a). The first column of Table IV presents data of routine colorimetric laboratory test (Perevalov), with the same basis that Oxifrit-test and Veri-Fry. The Perevalov test was first applied to all samples provided our large experience in its interpretation. The other two tests were subsequently applied depending on the availability of oils.

<table>
<thead>
<tr>
<th>No. of samples analyzed</th>
<th>PEREVALOV</th>
<th>VERI-FRY</th>
<th>OXIFRIT</th>
</tr>
</thead>
<tbody>
<tr>
<td>CORRECT RESULTS</td>
<td>174(100)*</td>
<td>157(100)</td>
<td>60(100)</td>
</tr>
<tr>
<td>PC &lt; 25%</td>
<td>107(61.5)</td>
<td>88(56.1)</td>
<td>34(56.7)</td>
</tr>
<tr>
<td>PC &gt; 25%</td>
<td>27(15.5)</td>
<td>28(17.8)</td>
<td>14(23.3)</td>
</tr>
<tr>
<td>FALSE RESULTS</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PC &lt; 25% (False +)</td>
<td>40(23.0)</td>
<td>41(26.1)</td>
<td>12(20.0)</td>
</tr>
<tr>
<td>PC &gt; 25% (False -)</td>
<td>33(19.0)</td>
<td>17(10.6)</td>
<td>9(15.0)</td>
</tr>
</tbody>
</table>

Abbreviation: PC, polar compounds
* Numbers into brackets are percentages on total samples analyzed

Results indicated that, in all cases, a high percentage of correct results was obtained. Also, the three tests gave similar figures of overall false results, although Veri-Fry had the highest percentage of false positive and lowest percentage of false negative results.

Finally, the distribution of false results for different ranges of polar compounds is shown in Table V. In general, the number of false results for high concentrations of polar compounds was very low. It is interesting to note that, even though the major concentration of false results for the two commercialized tests was found in different ranges of polar compounds, very good results were obtained, using any of the three tests, for highly altered samples (PC 40%), considering, additionally, that this group accounted for 10% of the total fats and oils analyzed. Similarly, results were excellent for samples with PC levels below 20%, wherein 55% of the oils analyzed was included.

<table>
<thead>
<tr>
<th>FALSE RESULTS</th>
<th>PEREVALOV</th>
<th>VERI-FRY</th>
<th>OXIFRIT</th>
</tr>
</thead>
<tbody>
<tr>
<td>TOTALS</td>
<td>40(23.0)</td>
<td>41(26.1)</td>
<td>12(20.0)</td>
</tr>
<tr>
<td>PC% &lt; 20</td>
<td>2(1.1)</td>
<td>12(7.6)</td>
<td>2(3.3)</td>
</tr>
<tr>
<td>20 &lt; PC% &lt; 25</td>
<td>5(2.9)</td>
<td>12(7.6)</td>
<td>1(1.7)</td>
</tr>
<tr>
<td>25 &lt; PC% &lt; 30</td>
<td>12(6.9)</td>
<td>5(3.2)</td>
<td>5(8.3)</td>
</tr>
<tr>
<td>30 &lt; PC% &lt; 40</td>
<td>18(10.4)</td>
<td>8(5.1)</td>
<td>4(6.7)</td>
</tr>
<tr>
<td>PC% &gt; 40</td>
<td>3(1.7)</td>
<td>4(2.6)</td>
<td>0(0)</td>
</tr>
</tbody>
</table>

* Numbers into brackets are percentages on total samples analyzed
In summary, it can be concluded that, even if a certain number of false results could not be excluded, the use of any of the two rapid commercialized tests would improve the quality of frying fats. The percentage of altered fats would decrease drastically and, what is more important, fats with polar compound percentages higher than 40% would be virtually absent in real practice.

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