Chapter 87

Physical-chemical characterization of samples of oils used in frying in the city of Castanhal/PA

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ABSTRACT
The production of food through frying is usual due to the practicality of the process and the results obtained, however, the frying medium undergoes changes that reduce its quality until it is no longer usable. Due to the health risks that degraded oils and fats can cause, this work sought to verify the quality status of the oils used in frying in the city of Castanhal/PA. For this, the moisture, acidity, iodine, peroxides and rancidity indexes (Kreis reaction) of seven samples were verified. Of which, four were donated by traders who work with frying, two were owned by the authors and one was commercial soybean oil, used as a control. It was found that the control sample was within the standards. In the other samples, 33.3% had a moisture content above the reference, 100% had a reduction in the iodine index, 66.6% had a peroxide index above the permitted level and 100% tested positive for the Kreis reaction. There were no samples with acidity above the allowed. There was a significant difference at the 5% level in all analyses. It was concluded that the quality of frying oils is below that established by regulatory bodies, making it necessary to periodically follow up with traders in order to adapt the frying process to good manufacturing practices.

Keywords: Analyze, Degradation, Frying, Quality, Oil.

1 INTRODUCTION

The frying technique is widely used in food production because of the practicality of the process and the characteristics provided to the final product. The procedure can be applied to a wide variety of foods and the method most often used is deep frying, in which the material is completely immersed in oil or fat at high temperature and for a short duration. During the process, heat and mass transfer occurs between the food and the frying medium, in addition to several physical and chemical changes in both, such as: water loss, oil absorption, crust formation, color change and flavor development in the food. In the oil or fat used there may be hydrolysis, polymerization, oxidation, etc.

The changes in the food provide the sensory and technological aspects responsible for the acceptance of fried foods. On the other hand, some of these changes are undesirable and can be harmful to the health of consumers, since, during the frying process, degradation of the oil or fat may occur and

subsequently this oil tends to be absorbed by the food, reaching averages of 16% (Teixeira et al, 2021); 9.1% to 28.9% (Albuquerque et al., 2017); 4.1% to 26.1% (Pessanha; Ferreira, 2010); 7.3% to 15.4% (Soares; Rodrigues, 2021), becoming an ingredient of the final product.

Currently, in Brazil, Technical Report No. 11 of October 5, 2004 from the National Health Surveillance Agency - ANVISA, provides about the use of oils and fats used in frying. In this document, standards and limits are established to help small traders and restaurants to maintain the quality of the oil used and, consequently, the food produced. Among the various topics in the Technical Report No. 11, it is recommended that: frying oil should not be reused; it should not work above 180°C or smoke point; continuous frying should be chosen; when it is not being used for cooking, the oil should not be used for cooking. used, the container with the oil must remain closed and that no new oil is added to the one already in use.

It is of utmost importance to control the working conditions of oils and fats during frying, because when the oil is successively reused in the presence of moisture, oxygen and high temperatures, its technological, nutritional and sensory quality is reduced due to the increased level of saturation and production of secondary compounds by hydrolysis and/or oxidation (Jorge, 2009). Among the undesired reactions that occur in the oil during frying, we can mention the formation of acrolein, which according to Nunes (2013, p. 24), is: "a very volatile compound of considerable toxicity, which can cause from irritation to the eyes and mucous membranes, to carcinogenic effects".

With regard to the demand for these foods, the Household Budget Survey (POF), revealed that in Brazil it is common to consume fried foods outside the home, replacing, even traditional meals which in years like 2017 and 2018 represented about 40.6%.

Considering the high consumption of fried food and taking into account the health risks that degraded oils and fats can represent, this study sought to evaluate, through physical-chemical analysis, the quality of oils used in the preparation of these foods in the city of Castanhal - Pará, where there is a growing number of traders who produce and sell these foods in snack bars and squares.

2 MATERIAL AND METHODS

Ten traders who work with frying of various products in the city of Castanhal/Pará were randomly chosen between December 1st and 6th of 2021. Of the total, only four traders agreed to participate in the experiment by donating a sample of the oil used for frying the foods sold, among these, only one person used the pan to fry a single type of food, "potato chips", the others performed the frying of various types of snacks using the same oil. It is worth noting that none of the traders controlled the temperature during frying or stated that they had taken any course in food handling.

The collection of approximately 100 ml of oil occurred at the end of the work day, and the samples were stored in amber glass containers with lids, remaining at room temperature until the time of analysis.

In addition to the material collected from traders, two other oil samples were used in laboratory frying experiments, with the difference that the time control of 135 minutes had been previously performed. Both samples were commercial soybean oil and were used for frying breaded chicken drumstick-type snacks. The first sample was coded with the letter A and had been stored in amber glass with a lid and under direct light since May 2021. The second sample, also stored under the same conditions since March 2021, was coded with the letter B.

In order to maintain the anonymity of the vendors, the other samples collected were also coded, being represented by the letters C, D, E and F. In addition to the samples collected, a randomly chosen bottle of soybean oil was purchased from the local market in Castanhal/PA, from which an aliquot was taken without thermal processing to be defined as control sample during the analyses and was coded as G. The frying oil samples collected and analyzed in this assay are shown in Figure 1.

Figure 1 - Frying oil samples analyzed in this experimente

2.1 ANALYTICAL DETERMINATIONS

Analyses of moisture content and volatile matter and acidity indexes, iodine indexes and peroxide indexes were performed, all experiments were done in triplicates, except for the rancidity test through Kreis reaction. The analyses occurred between December 7 and 17, 2021, in the food laboratory of the Pará State University (UEPA), Castanhal Campus.

Moisture and volatile matter content: This was checked following the official Ca 2c-25 method (AOCS, 1997). The results were obtained from equation 1.

Equation 1:

$$\text{Hu}mid\text{ity (\%)} = ((PC + PA) - PF) * 100)/PA$$

Acidity index: we followed the methodology of the Adolfo Lutz Institute (2008), in which neutralization acid-base titration is performed. The results were obtained from the values applied to equation 2.

Equation 2:

$$Acidity\ (\%\ of\ oleic\ acid) = (V * N * Fc * 28.2)/PA$$

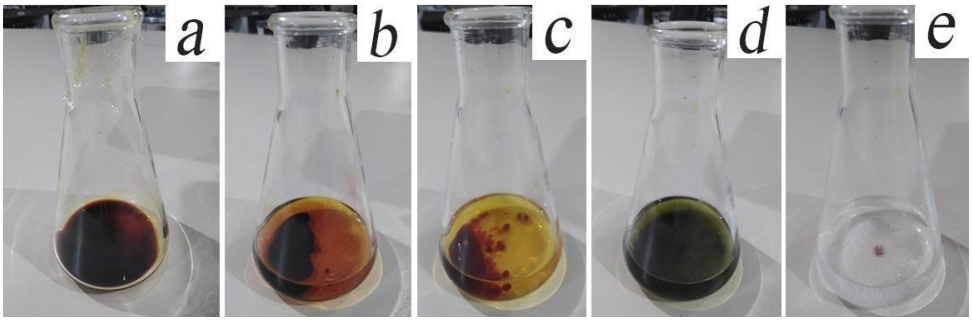
Iodine index: measures the degree of unsaturation of oils by quantifying the mass of iodine absorbed by the oil sample. The result is expressed in gI2/100g (IAL, 2008). The official method for determining the iodine index is Cd 1c-85, performed by iodometry using Wijs solution (AOCS, 2009). However, this method has disadvantages in relation to the reagents required, as is the case of Wijs solution, which according to Aricetti (2010, p.3):

"[...] is a solution of ICl in glacial acetic acid. This solution, besides the high cost, is very toxic and difficult to acquire because it is controlled by the Federal Police."

For this reason, it was decided to adapt the Hubl method proposed by Nunes (2008) and Ataide, Vinagre and Toro (2020). Therefore, for this experiment, a total of 0.125 g of the sample was analytically weighed in a 250 ml conical flask. 5 ml of chloroform and 5 ml of 5% alcoholic iodine solution, prepared at the time of the analysis, was added (Figure 2a). It was stirred gently and allowed to react in the shade of light and oxygen for 30 minutes. After the time, 50 ml of distilled water and 5 ml of 15% potassium iodide solution was added (Figure 2b).

Immediately, the solution was titrated with sodium thiosulfate 0.01 M (standardized with potassium iodide) until a yellow color appeared (Figure 2c), at which point 1 ml of a 1% starch solution was added (Figure 2d), and titration continued until the solution was discolored (Figure 2e). The same procedure was followed for the blank test, without the presence of the sample. The iodine value was calculated using Equation 3.

Figure 2 - Stages of the iodine index titration of the frying oil samples



Equation3:

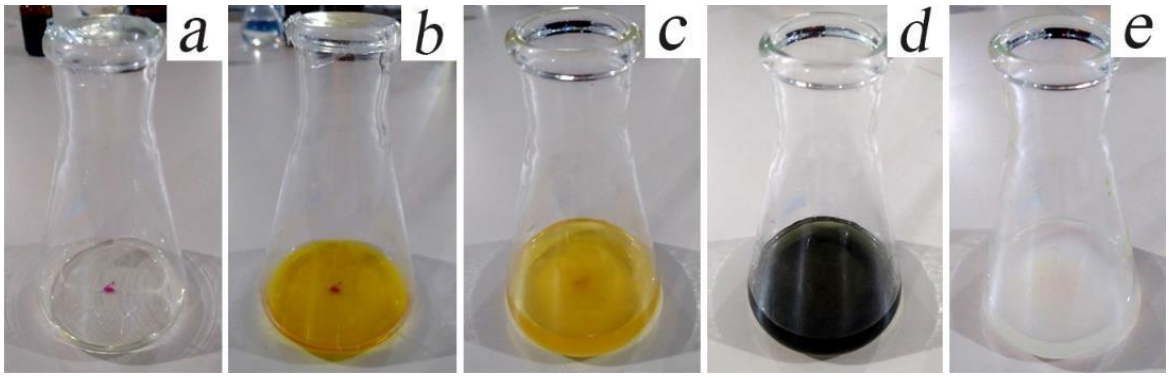
$$\text{Iodine value } (gI2\ /100g) = ((VB - VA) * Fc * 1.27)/PA$$

Peroxide value: we adapted the method proposed by Granato (2017, p.95- 97), in Chemical analysis, functional properties and quality control of food and beverages, changing only the amount of sample and reagents, but maintaining the proportions. To perform the analysis, a mass of 2 g of the sample was weighed analytically in a 250 ml conical flask and 25 ml of the acetic acid-chloroform solution (3:2) was poured over it (Figure 3a) and, after solubilization, 1 ml of the saturated potassium iodide solution (12.8 g of potassium iodide to 10 ml of distilled water) was added (Figure 3b). The solution was stirred briefly and allowed to stand in the shade of light and oxygen for 1 minute. After this time, 50 ml of distilled water was added (Figure 3c), homogenized, and 1 ml of the 1% starch solution was also added (Figure 3d). The solution was titrated with sodium thiosulfate 0.01 M (standardized with potassium iodide) until the dark color disappeared (Figure 4e).

The same procedure was followed for the blank test, without the presence of the sample. The result was expressed in milliequivalents of peroxide per 1000 g of sample (meqO2/kg), the calculation used is shown in Equation 4.

Where: M is the molarity of the sodium thiosulfate solution; VA is the number of milliliters of sodium thiosulfate solution spent in the sample titration; VB is the number of milliliters of sodium thiosulfate solution spent in the blank titration, and PA is the sample mass in grams.

Figure 3 - Stages of the peroxide index titration of the frying oil samples



Equation 4:

$$\text{Peroxide value } (meqO2\ /kg) = (M * (VA - VB * 1000)/PA$$

Kreis reaction: is a rapid qualitative test that indicates the presence of oxidative rancidity products. According to method 333/IV (IAL, 2008), Statistical analysis: the means of the moisture content and volatile matter and the acidity, iodine and peroxide indices were analyzed by variance analysis and Tukey's test in order to investigate the existence of significant differences at the 5% level. For this, we used the software SISVAR, which is a Brazilian program for statistical analysis developed by the Department of Exact Sciences of the Federal University of Lavras - UFLA (FERREIRA, 2008).

3 RESULTS AND DISCUSSION

The results of the physicochemical and statistical analyses of moisture and volatile matter content, acidity index, iodine index, and peroxide index performed on the frying oil samples are shown in Table 1.

Table 1 - Results of the analyses performed on the frying oil samples Physicochemical analyses

Samples	Moisture and matter volatile (%)	Acidity (% of oleic acid)	Value of iodine (gI2/g)	Peroxide value (meqO2/kg)
A	0,007^{d}	0,331^{e}	110,7^{b}	47,41^{b}
B	0,012^{d}	0,425^{d}	85,6^{c}	70,11^{a}
C	0,018^{d}	0,687^{b}	83,3^{c}	4,46^{d}
D	0,109^{b}	0,605^{c}	77,5^{c}	5,21^{d}
E	0,008^{d}	0,328^{e}	105,6^{b}	42,47^{b}
F	0,147^{a}	0,839^{a}	105,1^{b}	12,75^{c}
G	0,055^{c}	0,270^{f}	124,9^{a}	1,96^{d}
Reference Values				
ANVISA[1]	-	0,9	-	-
ANVISA[2]	-	0,3	-	10
MAP[3]	0,1	0,3	124 - 139	5

Means followed by the same letter in the column do not differ by the Tukey test at 5% probability. 1: Technical Report n° 11 of 2004;2 : Normative Instruction, n° 87 of March 15th 2021;3 : Normative Instruction n° 49/2006.

The Tukey test showed at 5% that all samples differed significantly from sample G (0.055%), however, only samples D (0.109%) and F (0.147%) showed values of moisture and volatile matter above the limit of 0.1% set by Normative Instruction No. 49/2006 of the Ministry of Agriculture, Livestock and Supply - MAPA. The results varied widely because the oil samples were used for frying different types of products. The highest value was found for sample F, which was used to fry potato sticks. According to research conducted by Jorge and Lunardi (2005), a moisture content of 83.8% was found in potatoes in natura, and after frying, the moisture content fell to an average of 9.13%. That is, the moisture present in the food is evaporated because of the high temperatures and comes into direct contact with the frying medium, consequently, the moisture present in the oil tends to increase according to the duration and type of food used in the process. It is worth noting that moisture is one of the agents responsible for quality changes in oils and fats. According to Luz et al. (2018, n.p): "The presence of moisture in oils and heat favor the hydrolysis reaction of the oil, producing a considerable increase in free acidity generating an unpleasant rancid odor and taste."

As for the index of free fatty acids expressed as oleic acid, a significant difference was found by the Tukey test at the 5% level among all the samples analyzed.

Sample G, commercial soybean oil in natura, showed values of 0.270% and was within the standards set by ANVISA's Normative Instruction No. 87 of March 15, 2021, which stipulates a value of 0.6 mg of KOH/g or 0.3% when expressed as oleic acid for refined vegetable oils in natura.

As already mentioned, ANVISA has acidity limits for oils and fats used in frying, and recommends through Technical Report No. 11 of 2004, that the disposal of this material should be performed before reaching levels of free fatty acids above 0.9% expressed as oleic acid. Thus, the samples analyzed in this experiment, even presenting high levels of acidity, were below the reference values.

Moreover, again the highlight is sample F, which reached an acidity of 0.839%, ratifying the influence of moisture on oil acidification. However, even if all the samples did not exceed the defined limit for acidity, there is a chance that these values may have been higher during the process and, because of this, a kind of false negative occurs. For according to Del Ré and Jorge (2007, p. 1778): "free fatty acids are moderately volatile and an unknown quantity is lost during heating".

Moreover, it should be taken into account that these values refer to samples collected at the end of the working day and their acidity levels would be within limits if their use had come to an end on the same day of collection, but nothing guarantees that vendors have not reused these same oils in new frying. On this issue, one can cite the research of Amaral et al. (2013), in which they monitored 17 pastry shops in the south-central region of the city of Belo Horizonte/MG and found that 41.2% of the vendors used the oil from 4 to 6 days before discarding it, and 17.6% exceeded 7 days of use before discarding it. In view of this, it would be necessary to make a regular monitoring and in partnership with the traders of the city of Castanhal/PA in order to define the way they work and the real condition in which these oils are.

In the results found for the analysis of the iodine index, sample G reached 124.9 gI2/100g, differing significantly from the other samples analyzed, the sample of oil in natura was within the range of 124 and 139 gI2/100g which is stipulated by IN n° 46 (MAPA, 2006). The result also indicates the viability of the alternative method used in this study, since it obtained similar values to those achieved by the official method (Wijs) in the evaluation of vegetable oils in natura. In the other samples that were subjected to frying processes, all of them presented lower iodine levels to a greater or lesser extent, and sample D, with a level of 77.5 gI2/100g, had the lowest value.

Because there is a reduction in the iodine value of the oil used for frying, the double bonds are broken and consequently less iodine is absorbed by the oil that has been heated successively. However, Soares and Rodrigues (2021), when analyzing samples of soybean oil at different times of frying salted snacks, observed an increase in the iodine value throughout the process due to the addition of new oil to that already used, influencing the final result. Besides, it can be seen that the practice of filling the fryer with new oil is very common among those who work with frying, even though it is contraindicated by the

IT n° 11 of ANVISA (2004). About this, Freire, Mancini-Filho and Ferreira (2013, p. 361), mention that: "[...] the addition of new oil to used oil does not allow the iodine index to be used as a parameter to choose when to dispose of the oil". Therefore, it is necessary to use the iodine index in addition to other tests to define the real degree of degradation of the frying oil.

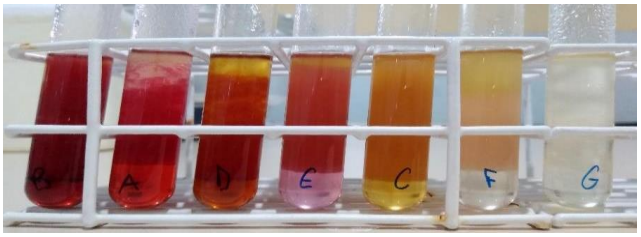
For the analysis of the peroxide index, no maximum and official values were found to define the ideal moment for the disposal of frying oils and fats. For this reason, we defined as limit the value of 10 meqO2/kg provided in IN, no. 87/2021/ANVISA. It should be noted that this limit is specific for vegetable oil without thermal processing. In this experiment, it was found that all the results, except those found in samples C (4.46 meqO2/kg) and D (5.21 meqO2/kg), differed significantly from sample G (1.96 meqO2/kg). Samples C, D, and G were the only samples that were below the limit of 10 meqO2/kg. Among the samples collected from the vendors, sample E (42.47 meqO2/kg) was the highest in peroxides.

Similar values were found by Moraes, Iguti and Correia (2015), when evaluating the oil used in frying potatoes at 140 °C, in which they obtained peroxide index averages of 38.38 meqO2/kg. In contrast, sample B (70.11 meqO2/kg), stored since March 2021, achieved the highest overall result. The peroxide index values indicate the oxidation that occurs when unsaturated fatty acids react with atmospheric oxygen or with the oxygen dissolved in the oil from the evaporation of the water present in fried foods. The chemical oxidation reactions generate products that impart unpleasant odors and tastes to the oil and also to the food.

According to the results obtained, the oxidative rancification process is in progress in samples A, B, E, and F. Samples A and B were no longer in use at the time of the analysis, however, once again there is no confirmation that samples E and F, which had already passed the disposal point, had not been used again by traders.

Finally, through the Kreis reaction, which is a colorimetric method, it was possible to observe a reddish coloration in all samples that had been used for frying, the difference between them was due to the more intense tone of the coloration in oils that had a greater amount of oxidation products, therefore, it was found that samples A, B, C, D, E, and F were positive for oxidative rancidity. Sample G, which represents the oil in natura, did not show any signs of color change, and was thus negative for oxidative rancidity. The results of the analysis are illustrated in Figure 4 in descending order as to color intensity (B, A, D, E, C, F, and G).

Figure 4- Test of rancidity in frying oil samples by means of the Kreis reaction



4 CONCLUSION

In view of the data presented, there is a deficiency in the quality control of the oil used in the frying process carried out by the merchants evaluated, because, according to the results obtained in this experiment, it is confirmed that the quality of the oil is reduced as a result of the lack of production control due to the lack of knowledge about good manufacturing practices.

It is noteworthy that the number of samples evaluated in this study do not faithfully represent the huge number of points that work with fried foods in the city of Castanhal/PA. Thus, it is indicated that further and more comprehensive studies can define the real situation in which these fried foods are being produced and, through these future researches, it is possible to define guidelines together with the traders in order to improve the service offered through the periodic monitoring of the quality of oils and fats used in the frying process.

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