



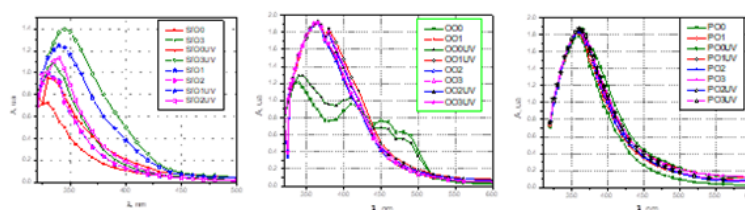
## THERMAL AND PHOTOCHEMICAL DEGRADATION OF COOKED SUNFLOWER, OLIVE AND PALM OILS

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This paper provides information on the number of cooking cycles that could be applied for the most used oils (sunflower, olives and palm) without destroying their qualities. During a cooking period, oils are constantly exposed at high temperatures, so chemical reactions such as oxidation, hydrolysis, isomerisation and polymerization can occur, but the experimental data shows that sunflower, olive and palm oils can be re-used at frying at least three times, the colour, acidity and the peroxide index all being within the range accepted by the literature. Furthermore, experimental data on oils photo-degradation showed that after the third cooking process they decompose into a variety of volatile compounds and monomeric and polymeric products capable of influencing not only the sensory and health quality but also the period that the fried product can be used.



### INTRODUCTION

It is well known that a large amount of food is lost annually due to their degradation over time. This phenomenon damages food from a physical, microbial and chemical point of view.<sup>1</sup> Among these, the biggest interest is the chemical degradation because it involves different types of degradation reactions caused by: light, temperature, oxygen, pH, improper storage, etc. Light is a determining factor in the chemical degradation of food<sup>1,6</sup> because the photodegradation reactions have a negative impact on the appearance and quality of food but also on their chemical composition. Verduin *et al.*,<sup>1</sup> states that food has a complex composition and it is difficult to perform

their complete analysis to establish the degradation, and for this reason it is necessary to know at least the types of photodegradation reactions specific to each class of bioactive compounds that are frequently present in food, such as: carotenoids, chlorophylls, flavonoids and lipids. Thus, in the case of carotenoids, degradation is achieved by autooxidation, thermal degradation, photodegradation and photoisomerization reactions,<sup>7,8</sup> while chlorophylls are degraded by photosensitization and photooxidation reactions.<sup>9–11</sup> A number of literature studies have shown that photooxidation reactions are the basis for the degradation of flavonoids<sup>12,13</sup> and that lipids can be degraded under the influence of light by the means of oxidation reactions, the reaction rate being

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accelerated due to microbial damage and the presence of ultraviolet light.<sup>14,15</sup> Moreover, in case of lipids, degradation products not only have an unpleasant aroma, but are very toxic, leading to serious diseases.<sup>16-19</sup>

Thus, the literature studies confirm that the toxic action of degraded lipids is due to the accumulation of peroxides, aldehydes, ketones, polymers, oxypolymers, aromatic compounds, etc., which appear at high temperatures.<sup>20-22</sup> These lipid degradation products have the effect of inactivating vitamins and breaking down unsaturated fatty acids, leading to pathological phenomena and lipid infiltrations of the liver. Vegetable oils are an indispensable and constant part of the daily diet due to the high content of mono and polyunsaturated fatty acids that are beneficial for health but, at the same time, by exposure to light, they oxidize<sup>18</sup> being considered mainly responsible for lipid degradation.<sup>23</sup>

The degrading effect of light on vegetable oils is responsible for rancidity, a phenomenon due to the oxidation process that can be accelerated by light, ionizing radiation, catalysts, heat and enzymes. The self-oxidation of unsaturated acids in vegetable oils is achieved by a mechanism in which free radicals are formed by hydro-peroxide decomposition or under the action of light. Regarding the latter factor, several literature studies have shown that photooxidative rancidity occurs under the action of both artificial and natural light.<sup>24-28</sup>

Thus, Pignitter *et al.*<sup>24</sup> concluded that cold fluorescent light is the main factor in the oxidative damage of soybean oil stored at home for 8 weeks. Also, the choice of packaging for vegetable oils is important in eliminating degradative processes and ensuring long-term preservation. Although transparent plastic packaging is preferred for vegetable oils in order to quickly quality assess and ensure easy transport to the consumer, a transparent or translucent container made of materials other than glass increases the risk of degradation or oxidation induced by exposure to light and interaction with the packaging material. In this regard, Ahmed *et al.*<sup>29</sup> showed that degradative oxidation processes are accelerated when oils are not stored properly. The authors concluded that the best quality oil is one that is packaged in a glass container and stored in a dark space. However, for long-term preservation of vegetable oils, some authors have suggested the use of preservatives. Thus, Ullah *et al.*,<sup>30</sup> observed that the application of a synthetic antioxidant such

as butylated hydroxyl toluene (BHT) and a natural antioxidant such as red pepper oil, reduce the rate of peroxidation of vegetable oils during their storage in light for 5 weeks, and of these the most efficient was BHT.

The effects of photodegradation of vegetable oils have been the subject of several specialized studies, using UV-VIS spectroscopy, FTIR and HPLC, as tools for highlighting them.<sup>31-33</sup> Thus, Spatari *et al.*,<sup>31</sup> concluded that severe changes in the composition of vegetable oils occur when exposed to light and are detected by UV-VIS spectroscopy, but cannot be detected by FTIR spectrometry. The authors attribute this to the fact that exposure to light causes the degradation of unsaturated fatty acids that cause a change in chromophore characteristics that are detected by UV-VIS analysis, but do not alter the functional groups responsible for typical signals that can be recorded by FTIR analysis. Moreover, the authors also used HPLC in the study of changes in the composition of vegetable oils exposed to light and temperature, and these were consistent with those obtained by UV-VIS.

Along with light, temperature is one of the main factors in food degradation, affecting, in particular, their organoleptic properties and their chemical composition when not properly treated, processed and preserved.<sup>34-37</sup> The literature shows that most changes in flavor and color are due to the Maillard reaction, and the change in texture is the result of both denaturation of proteins and carbohydrates and changes caused by hydration of products.<sup>38</sup>

Also, improper heat treatment can lead to a number of complex changes in the structure of proteins, lipids, carbohydrates or even substances such as enzymes, pesticides, allergens, which have been used in various stages of cultivation and processing of plant materials used as materials. raw materials for obtaining food.<sup>39-44</sup> Vegetable oils degrade rapidly when exposed to high temperatures, the processes occurring are: hydrolysis and accelerated self-oxidation.<sup>45</sup> The most frequently applied methods of preparation of vegetable oils that require high temperatures are: frying, baking, boiling or grilling. Among these, frying requires special attention, because by heat treatment at 180-200°C, vegetable oils change both their properties and nutritional value, with the formation of oxidizing compounds harmful to the human body.<sup>46</sup> Although, the process of frying at high temperatures in vegetable oils becomes more and more popular due to the appearance and taste of food. In scientific literature, there are many studies

on the cooking of vegetable oils and their effects on health.<sup>47-63</sup> According to these studies, oils can be used for cooking, but not at very high temperatures, because the heat changes the flavour and oil quality and the nutritional properties of oil. However, there is no exact number of accepted re-uses for cooking of the same oil. This depends on certain factors such as oil type, frying time, temperature, type of cooked food, etc. Moreover, the oil cannot be re-used if its colour has changed too much (became brownish), if it has changed its viscosity, if it is rancid or has traces of food.<sup>64,65</sup> In terms of choosing the best vegetable oil for cooking, Cui *et al.*,<sup>66</sup> concluded that ideal for thermal heating is to use oil with high combustion point.

Among the vegetable oils, the most appreciated by consumers are sunflower, olives and palm. Sunflower oil has a rich content of saturated and unsaturated fatty acids and is characterized by stability, long-term preservation capacity and a high combustion point (232°C) being recommended for cooking.<sup>67</sup> Olive oil is rich in fatty acids and vitamins and is ideal for frying because it does not lose its heating properties over 210°C. Palm oil is rich in antioxidants (vitamin A and E) and is considered as a source of carotenoids. It also has a high combustion point (230°C).<sup>68</sup>

Based on these considerations, the thermal and photochemical degradation of sunflower, olives and palm oils used to cook vegetable products was analysed in this study.

## EXPERIMENTAL

### 1. Choice of materials

For this study, three types of refined oils, namely sunflower, olives and palm, manufactured 2021, were purchased from the supermarket. Vegetable products (potatoes) were used for frying and were purchased from local producers.

### 2. Getting the cooked oil samples

In order to obtain the cooked oil samples, it was thus proceed: after a preliminary preparation, 500 g of peeled potatoes were cut into pieces 1 cm thick and 5 cm long. One

litre of sunflower oil was heated in an iron pan container, at 200°C, after which the potatoes previously prepared were added. During frying, the temperature was kept constant. After about 15 minutes, the product gained a golden colour and was separated from the hot oil.

The separated oil was allowed to cool for 1 hour, after which a further 500 g of fresh vegetable material (prepared as in the previous case) was added again and cooking occurred under the same conditions as the first time. After the second frying, the hot oil was separated from the fried product and left to cool again for 1 hour. After this time, 500 g of fresh vegetable material (prepared as in the previous case) was added again and the frying process was repeated under same parameters. The same method was applied to olive and palm oils, maintaining the same temperature of 200°C.

Samples were harvested for the initial oil and for the fried oil, after each cycle of cooking, so that four samples were finally obtained for each type of oil. Table 1 lists the notations made for each type and oil sample.

### 3. Analysis of uncooked and cooked oil samples

The obtained oil samples are used in subsequent analyses in order to calculate the acidity index, free acidity, peroxide index, saponification index and photodegradation. The chemical reagents required for the analyses were purchased from Merck.

### 4. Determination of acidity index

Concerning the acidity index and the free acidity expressed in oleic acid, these were determined according to the literature method<sup>69</sup>. In this regard, a quantity of sample was weighed into a Berzelius beaker and then heated. A mixture of ethanol-benzene 1:10 was added to the sample and then titrated with a 0.1N NaOH solution with continuous stirring, in the presence of phenolphthalein until the pink colour appeared and persist for 30 seconds. For each type of oil, the titration was done three times, and the final value was the arithmetic mean of the results.

### 5. Determination of saponification index

The saponification index for each type of oil was measured according to the method described in literature.<sup>69</sup> Thus, a quantity of oil was added to a vial and then benzene and a solution of KOH were added. A refluxing refrigerator was added to the vial and the contents were boiled on a water bath for 30 minutes.

After boiling, the hot sample was titrated with a solution of HCl in the presence of phenolphthalein. In parallel, the same operations were made for a control sample. For each type of oil three titrations were performed and the arithmetic mean of the results was the final value.

Table 1

Abreviation for the oil samples

Oil type	uncooked	cooked		
		after 1 hour	after 2 hours	after 3 hours
Sunflower oil	SfO0	SfO1	SfO2	SfO3
Olive oil	OO0	OO1	OO2	OO3
Palm oil	PO0	PO1	PO2	PO3

Table 2

The used notations for photodegraded cooked samples

Oil type	uncooked	Cooked and photodegraded		
		after 1 hour	after 2 hours	after 3 hours
Sunflower oil	SfO0UV	SfO1UV	SfO2UV	SfO3UV
Olive oil	OO0UV	OO1UV	OO2UV	OO3UV
Palm oil	PO0UV	PO1UV	PO2UV	PO3UV

## 6. Determination of peroxide index

The peroxide index was determined according to the literature method.<sup>69</sup> Thus, an oil sample was introduced into a vial, next chloroform was added and the mixture was homogenized. Then, acetic acid and potassium iodide were added to the vial, which was sealed, shaken, and left to rest in the dark for 5 minutes. Next, distilled water was added to the sample and titrated with a sodium thiosulfate solution, using starch as indicator. At the same time, a control sample was also processed the same way. For each type of oil the titration was done three times, and the arithmetic mean was the final result.

## 7. Photodegradation of cooked samples

50 mL of cooked samples were UV irradiated for 15 minutes using an 18W Hg UV B lamp. The incident radiation intensity was measured as being 2.1 W/cm<sup>2</sup>, and was determined by a Hamamatsu C9536-01 meter with H9958 detector for 310-380 nm, scaled between 1μW/cm<sup>2</sup> and 100mW/cm<sup>2</sup>. The distance between the UV radiation source and the samples was 2 cm. At time intervals, samples (about 4 mL) were taken, and analyzed by UV-Vis spectroscopy (with a SP 870 plus METERTECH spectrophotometer). The samples were continuously agitated (magnetic stirring). The used notations for photodegraded cooked samples are presented in Table 2.

# RESULTS AND DISCUSSIONS

## 1. Analysis of oil samples colour

Figure 1 shows the uncooked and cooked oil samples for each type of oil.

For sunflower oil (Figure 1.a), compared to the unheated sample (UFLN), the fried oil samples (UFL1, UFL2, UFL3) do not show any colour changes, even after the third cycle of cooking. This indicates that the oil is of high quality and the

thermal heating was not above the combustion temperature. Moreover, due to the very careful roasting operation, the oil samples do not show any impurities of the roasted product. The same can be observed with the olive oil samples (UMN, UM1, UM2, UM3) and, respectively, the palm tree oil (UPN, UP1, UP2, UP3). So it can be said that sunflower, olive and palm oils can be reused even after the third time because they do not have significant colour changes and do not have suspensions or sediments at 60°C, as stated in the literature.<sup>13</sup>

## 2. Analysis of acidity index for oil samples

Experimental data on the acidity index and free acidity expressed in oleic acid for all analysed oil samples are presented in Table 3.

For the acidity index, it can be observed that its value increases for each type of oil as it is further cooked, as seen by the increased amount of KOH needed to neutralize existing free fatty acids in one milligram of product. This shows that repeatedly roasting foods in the same oil can result in complete loss of antioxidants and beneficial compounds in the oil composition as well as taste alteration. From Table 3 it can also be observed that the acidity index value is different for all analysed oil samples, which shows that the oil burning point of an oil depends on its type and the purity. Thus, higher combustion point means lower number of free fatty acids and higher purity.

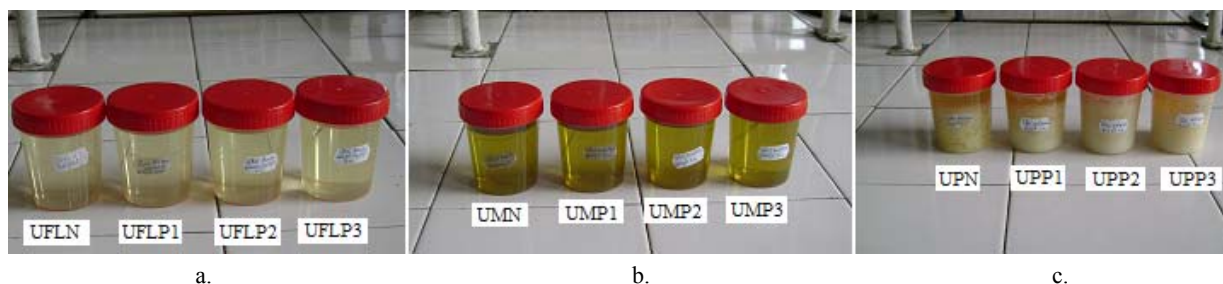


Fig. 1 – Uncooked and cooked oil samples. a. Sunflower oil, b. Olive oil, c. Palm oil.

Table 3

Acidity index and free acidity for analysed samples

No.	Sample	Acidity index, Mg KOH/g sample	Free acidity (oleic acid) %
1.	SfO0	0.605	0.304
2.	SfO1	1.077	0.541
3.	SfO2	1.144	0.575
4.	SfO3	1.211	0.609
5.	OO0	0.471	0.236
6.	OO1	0.942	0.473
7.	OO2	1.077	0.541
8.	OO3	1.144	0.575
9.	PO0	0.605	0.304
10.	PO1	0.740	0.372
11.	PO2	0.807	0.406
12.	PO3	1.346	0.676

Table 4

Experimental data obtained for all analysed oils

No.	Sample	Saponification index, mg KOH/g oil
1.	SfO0	189.351
2.	SfO1	119.221
3.	SfO2	91.169
4.	SfO3	63.117
5.	OO0	186.545
6.	OO1	138.857
7.	OO2	122.026
8.	OO3	91.169
9.	PO0	197.766
10.	PO1	166.909
11.	PO2	147.273
12.	PO3	116.415

Table 5

Peroxide index for oil samples

No.	Sample	Peroxid index, meq/Kg
1.	SfO0	1.35
2.	SfO1	2.2
3.	SfO2	3.5
4.	SfO3	3.9
5.	OO0	1
6.	OO1	1.55
7.	OO2	1.85
8.	OO3	2.7
9.	PO0	1.45
10.	PO1	1.95
11.	PO2	2.6
12.	PO3	3.4

It is known that oils are neutral substances and they have to have a very low acidity, otherwise the high values shows a rancidity of the oil. Banu<sup>24</sup> states that oils can no longer be reused if the acidity, expressed in oleic acid is 1%. From the data presented in Table 2, it can be noticed that each type of uncooked oil has a low acidity, its value increasing after each cooking process. Even so, it can be noticed that after three cycles of roasting, the acidity of each type of oil is less than 1%.

To confirm that with repeated heating the oil changes its chemical composition, the saponification index for each type of oil was analysed. The experimental data obtained for all three types of oils are shown in Table 4.

As showed in Table 4, the saponification index for each oil gradually decrease through repeated frying.

### 3. Analysis of the peroxide index for oil samples

Experimental data on the peroxide index for all analysed oil samples are shown in Table 5.

From the data presented in Table 5, it can be noticed that each type of uncooked oil has a peroxide index below 20 meq/Kg, so the oils can be reused for repeated frying.<sup>70</sup> The value of the peroxide index for each type of oil increases by repeating the heating process. However, it can be noticed that after three cycles of roasting, the specific peroxide index of each type of oil is still less than 20 meq/kg, which can be said that sunflower, olive and palm oils can be re-used for repeated frying at least three times.

### 4. Photodegradation of oil samples

The UV-Vis spectra for all the three uncooked, fried and photodegraded oil samples are shown in Figures 2, 3 and 4.

As showed in Figures 2, 3 and 4, the absorbance occurs at different wavelength for all three types of uncooked oil studied (SfO0, OO0 and PO0) and this can be attributed to the composition of fatty acids, as well as the fact that they can absorb light separately when the wavelength is greater than 220 nm.<sup>20</sup> As for the studied fried oils, the UV-Vis analysis highlights the changes in their qualitative composition. The general increase in absorbance in the case of the three fried oil samples studied may be due to the oxidation products formed during the thermal treatment. Moreover, olive and palm oils showed greater stability than sunflower. This can be attributed to the fact that refined sunflower oil is more thermally sensitive, as it can release a number of chemicals through excessive heating.<sup>71</sup>

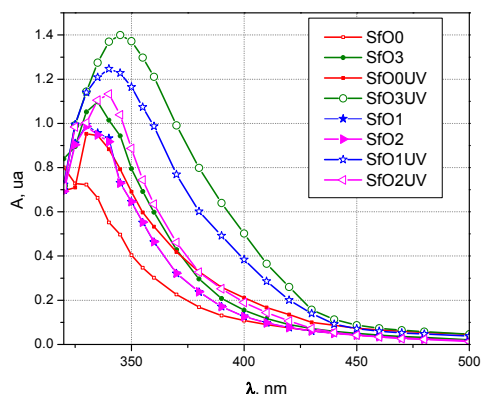


Fig. 2 – The UV-Vis spectra of sunflower oil samples.

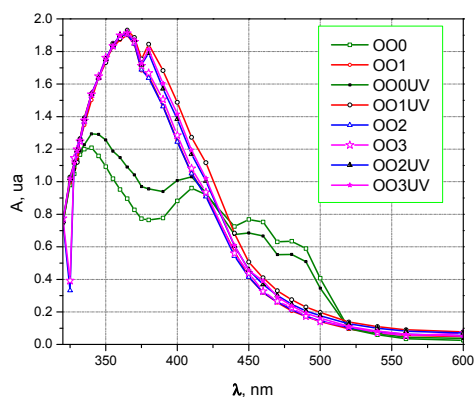


Fig. 3 – The UV-Vis spectra of olive oil samples.

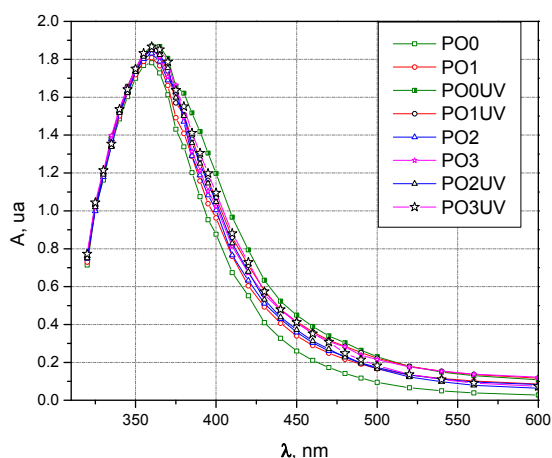


Fig. 4 – The UV-Vis spectra of palm oil samples.

Also UV-Vis analysis show that, for each sample, exposure to UV radiation leads to an increase of unwanted hydrolysis, with the release of fatty acids. Thus, by photodegradation, maximum absorption suffers a hyperchromic and bathochromic shift, justified by the destruction of the ester linkages in the oil with the release of the corresponding fatty acids, effect also observed and reported by Spatari *et al.* (2017).<sup>33</sup> Moreover, for roasted and UV-treated oil samples, it can be seen that the longer the frying time is, the amount of fatty acids increase

## CONCLUSIONS

For all three types of oils analysed, the cooked samples, compared to unheated oil samples show no colour modifications, even after the third cycle, which indicates that the thermal heating was not achieved above the oil combustion temperature. If the roasting process takes place below the combustion temperature, sunflower, olive and palm oils can be re-used even after the third time, because they do not have any significant colour changes and do not have any suspension or sediment at 60°C. Furthermore, after three cycles of roasting, the peroxide index of each type of oil is less than 20 meq/kg, which means that the sunflower, olives and palm oils can be refried at least three times. Experimental data has shown that each type of unheated oil has a low acidity, its value increasing very little after each cooking process, but not more than 1%. By contrast, the saponification index for each type of oil gradually decreases after repeated frying, thus confirming that with repeated heating the oil changes its chemical composition, as demonstrated by the photodegradation of oil samples after three frying cycles.

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