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DOI: https://doi.org./10.52091/EVIK-2024/4-2

Arrived: October 2024 / Accepted: December 2024

Comparison of physico-chemical properties of different types of vegetable oils prior to and post deep frying

Keywords: deep frying, oil oxidation and thermal destruction, fatty acid composition, polar compounds, UV-VIS oil spectra, physico-chemical characterization of oils, vegetable oils

1. Abstract

Deep frying is one of the most commonly used methods of food preparation. An essential factor in the frying process is the choice of a suitable oil – it plays a role in the heat transfer and as a product impregnation medium. Repeated use at high temperature leads to a number of reactions of oxidation, polymerization, and thermal destruction, which lead to changes in oils' physical, chemical, nutritional, and sensory properties. Since the changes result from different processes, they cannot be evaluated by a single method. The processes differ in their extent for the different oil types. Therefore, the aim of the present study is to investigate the quality changes in three vegetable oils, namely refined sunflower oil, high oleic acid sunflower oil (HOSO), and rapeseed oil using different physical and physico-chemical methods. General chemical parameters, such as acid value and fatty acid composition were measured by standard methods, namely titrimetry and gas chromatography. The oxidation stability and oxidation degree along with the dynamics of the oxidation processes during frying were assessed by Rancimat method along with UV spectroscopy and electric impedance spectroscopy. The thermal behaviour of the oils and more precisely the amount of polar compounds formed during frying and their effect on the recrystallization phenomena are examined by differential scanning calorimetry.

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2. Introduction

Deep frying is a widely used food preparation technique both in households and restaurants (Choe and Min, 2007). It is also one of the most frequent choices for food cooking due to its simplicity and accessibility. The process of deep frying takes place at high temperatures (160–190 °C) and the oil acts as a heat and mass transfer media that results in a crispy texture and varying flavour for the foodstuff (Kaur et al., 2020; Wiege et al., 2020). Despite its feasibility, deep frying is a complex physicochemical process which is highly influenced by many factors, such as the type and quality of the oil (Yılmaz et al., 2023). As a result of thermal and oxygen exposure, different chemical reactions happen in the oil as hydrolysis, oxidation, polymerization, and isomerization result in thermal and oxidative decomposition of the oils (Dobarganes and Márquez-Ruiz, 2015; Boskou et al. 2006; Turan et al. 2022).

The complexity of these reactions is directly linked to the properties and quality of the used oil and particularly to its fatty acid composition. As a result of different thermo-oxidative reactions, the composition of the oil is changing and the presence of smaller reactive molecules such as free radicals, cyclic fatty acids, or some volatile oxidized compounds may be detected. Their presence leads to alterations in the sensory, functional and nutritional properties of the oils. The extent to which these reactions may occur depends on the amount of unsaturated fatty acids in the oil, the frying time and temperature, and the type of food product that is being fried (Yılmaz et al., 2023; Habarakada, et al. 2021).

It is important to know and to detect the types of byproduct compounds present in oil during thermal processes, due to their potential hazardous effect on the consumers' health (Quek et al. 2022). Back in the 2000's, an international symposium for deep frying was proposed to analyze fats and oils by determining their polar compound content. However, this technique is expensive and time consuming and researchers aim to develop or propose other more effective and accessible methods for quality control of oils before and/or after deep frying (Donner et al. 2000). Many researchers headed toward using change in viscosity and dynamic interfacial tension (Wiege et al. 2020). Changes in these parameters turned out to be indicative of oil deterioration, yet not informative enough about the extent of oil decomposition in different types of oils and their blends, and therefore need to be combined with other techniques in order to have clearer understanding of the occurring decomposition (Cunha et al. 2024).

The main aim of the present study is to establish the potential of different physical and physico-chemical experimental approaches to detect changes in the properties of oils before and after deep frying for up to 9 hours. For this purpose, a comparison between acid values, refractive indices, induction time, UV-VIS spectrophotometry, differential scanning calorimetry, DSC, and Electric impedance spectroscopy will be done. To check the sensibility, these 3 types of oils were chosen: refined sunflower, high-oleic sunflower (HOSO), and rapeseed.

3. Materials and methods

Materials: Refined Sunflower oil (trademarked "Klas"), high oleic sunflower oil (HOSO) and rapeseed oil were obtained from a local Bulgarian market. All other chemicals used in the physical and chemical testing were of analytical grade.

Frying procedure

The frying was performed based on the procedure reported by Holgado (Holgado et al. 2024) with small modifications. 700 ml of the oils were placed in a fryer at 180°C and used to fry potatoes, 2 kg in total divided in portions of 100 g. Each portion required 10 mins time to be ready. After each single portion the oil was left at that temperature for 20 mins. Small samples were taken at specific intervals up to the 9th hour of thermal usage. After cooling to room temperature, the samples were filtered and stored at 4°C in a refrigerator.

Determination of fatty acid composition. Fatty acid composition of the oils was determined by gas chromatography (GC), according to ISO 12966-4:2015 Animal and vegetable fats and oils — Gas chromatography of fatty acid methyl esters — Part 4: Determination by capillary gas chromatography. Fatty acid methyl esters (FAMEs) were prepared by pre-esterification of the samples with 2% sulphuric acid in absolute methanol at 50 °C, as describes ISO 12966-2:2017 Animal and vegetable fats and oils — Gas chromatography of fatty acid methyl esters — Part 2: Preparation of methyl esters of fatty acids. Determination of FAMEs was performed on Agilent 8860 gas chromatograph equipped with a capillary column DB Fast FAME (30 m 0.25 mm 0.25mm film thickness) and a flame ionization detector. The column temperature varied from 70 °C (1 min) to 180 °, at a rate of 6 °C/min, and then from 180 ° to 250 °C at a rate of 5 °C/min; the temperature of the injector was 270 °C and that of the detector was 300 °C, respectively.

Acid value. Acid values were determined titrimetrically, according to ISO 660:2009. Animal and vegetable fats and oils. Determination of acid value and acidity (p. 5).

Oxidative stability. Oxidative stability of the oils was determined by measuring the induction period via conductometric detection of volatile compounds. Rancimat apparatus Methrom 679 was used at 100 °C with an air flow rate of 20 $L.h^{-1}$, as described in ISO 6886:2016. Animal and vegetable fats and oils. Determination of oxidative stability (accelerated oxidation test).

Refractive index measurement. The refractive indices were determined by a standard Abbe refractometer. The measurements were performed at a temperature of 20 °C with an accuracy of $\pm 1\times10^{-4}$. The refractometer was calibrated with water (n_D=1.3330) and isooctane (n_D=1.3915) at 20°C.

Differential scanning calorimetry. The crystallization and melting phenomena of the investigated oils were studied using a differential scanning calorimeter (DSC) 204F1 Phoenix (Netzsch Gerätebau GmbH, Germany). The analysis was carried out during the following temperature regimen: cooling down from 25 °C to −70 °C with a cooling rate of 2 K/min; isothermal step at −70 °C for 10 min; heating from −70 °C up to 100 °C with a heating rate of 5 K/min.

UV-visible (UV-Vis) spectrophotometry. Here, the standard method of the UV spectroscopy was performed on a *Metertech UV/VIS Spectrophotometer SP-8001*.–0.2% w/v–solutions of oil in iso-octan (2,2,4 trimethylpentane) were prepared, mixed well and poured into a quartz cuvette with an optical path-length of 10 mm. Linearity, wavelength, and photometric accuracy in the UV range were verified before the measurements. The spectra in the UV range (220-300 nm) were recorded with a 1-nm resolution and calibrated by means of a pure solvent spectrum. Special attention was paid to the Absorbance (**Abs**) at about 232 and 268 nm, which correspond to the existence of conjugated dienes and trienes, respectively (Malavi et al., 2023, Hashem et al. 2020).

Electric impedance spectroscopy (EIS): EIS *1910 Inductance analyzer* (Quad tech, USA), was employed to investigate the dielectric characteristics of the oils. For the EIS experiments, a volume of 7 ml oil was placed into a specially designed measuring cell. All tests were performed in triplicate. The vegetable oils show almost perfect capacitive behavior i.e. the phase angle of the electric impedance is -90°, which justify the investigation of the dielectric dispersion, ε' (as the real part of the relative permittivity). The parallel capacitance of the oil, C_p^{oil}, was measured in the frequency range of 100 Hz to 1 MHz (at least 100 experimental points). The values were then used to derive ε_1 with the help of the capacitances of the air (C_p^{air}) and the water (C_p^{water}) so that:

$$\varepsilon' = (C_p^{oil} - C_p^{air} - K_c) / K_c$$

where K_c is a calibration factor $K_c = (C_p^{air} - C_p^{water}) / (\varepsilon^{water} - \varepsilon^{air})$. K_c takes into account the specificity of the cell (stray capacitance etc.) and helps to eliminate any undesirable effects arising from the cell construction.

4. Results and discussion

Fatty acid composition

The results of the fatty acid composition analysis for sunflower oil, high oleic sunflower oil (HOSO), and rapeseed oil are presented in **Table 1**.

Table 1: Fatty acid composition of the investigated oil.

Fatty acid composition, %		Sunflower oil	HOSO	Rapeseed oil
$C_{16:0}$	Palmitic	6.4	5.0	4.4
$C_{18:0}$	Stearic	3.3	2.9	1.4
$C_{18:1}$	Oleic	34.5	82.8	64.7
$C_{18:2}$	Linoleic	54.0	7.3	18.8
Saturated fatty acids, %		11.0	9.1	6.4
Unsaturated fatty acids, %		89.0	90.9	93.6
Monounsaturated fatty acids, %		34.9	83.4	66.2
Polyunsaturated fatty acids, %		54.1	7.5	27.4

Fourteen fatty acids were identified. The major component in the sunflower oil “Klas” is the linoleic acid (54.0%), followed by oleic acid (34.5 %). The fraction of saturated fatty acids was represented by palmitic acid (6.4 %) and stearic acid (3.3 %). The major component in HOSO is oleic acid (82.8 %), followed by linoleic acid (7.3 %), palmic acid (5.0 %), and stearic acid (3.3 %). The oleic acid content of rapeseed oil (64.7 %) is much higher than the sunflower oil; the linoleic acid is 18.8 %. The saturated acid content is close to that of the sunflower oils. The amount of the other fatty acids is lower than 0.5 %.

Acid value and oxidant stability

The acid values of the oils during the frying process (**Table 2**) do not show a regular tendency to increase during frying.

Table 2: Acid value (AV, mgKOH/g) and oxidative stability (IP, h) of the investigated oils during the frying process.

Frying time, h	Sunflower		High oleic sunflower oil (HOSO)		Rapeseed oil	
	AV, mgKOH/g	IP, h	AV, mgKOH/g	IP, h	AV, mgKOH/g	IP, h
0	0.62±0.00	13.80	0.33±0.00	46.4	1.13±0.03	16.9
1	0.56±0.01	11.70	0.36±0.02	44.8	1.01±0.03	16.2
2	0.67±0.05	9.30	0.39±0.03	43.8	1.26±0.10	13.0
3	0.84±0.02	9.20	0.42±0.02	42.8	0.96±0.02	12.6
4	0.91±0.01	9.17	0.30±0.00	39.3	1.18±0.01	14.8
5	0.85±0.01	8.85	0.62±0.03	28.6	1.22±0.05	12.0
6	0.98±0.00	7.67	0.48±0.00	26.2	1.33±0.03	12.4
7	0.96±0.02	7.35	0.53±0.04	21.8	1.35±0.03	9.30
8	1.10±0.10	7.12	0.56±0.00	15.7	1.35±0.05	9.12
9	0.96±0.04	6.30	0.57±0.03	10.43	1.30±0.02	8.48

The results show that the frying process leads to complex hydrolysis and thermal degradation processes. A smooth increase in acid value from 0.56 mg KOH/g at the 1st hour to 1.10 mg KOH/g at the 8th hour is observed for the sunflower oil, with slight fluctuations at the 5th hour. The acid value initially increases during frying, decreases slightly in the middle of the process, and then continues to grow slowly.

A similar trend is observed for the other two oils: the acid value (AV) for HOSO ranges from 0.36 mg KOH/g at the 1st hour to 0.57 mg KOH/g at the 9th hour, and for rapeseed oil, it ranges from 1.01 mg KOH/g at the 1st hour to 1.35 mg KOH/g at the 8th hour, with a slight decrease at the 4th hour (0.30 mg KOH/g) for HOSO and at the 3rd hour (0.96 mg KOH/g) for rapeseed oil.

Generally, the values of the AV increase as free fatty acids are released due to the hydrolysis of the triacylglycerol. This process continues until all susceptible fatty acids have been hydrolysed. The results from the present study indicate a non-linear increase with fluctuations at certain time points. The decrease in AV around the middle of the frying time can be explained by possible reactions between the free fatty acids (FFAs) and the frying food. Other reasons are also that some FFAs are volatile at frying temperatures; they may react with mono- and diacylglycerols, as well as undergo oxidation or polymerization, leading to a temporary drop in AV. Other than that, the distinct fatty acid composition of the oils (high oleic vs. standard sunflower vs. rapeseed) may lead to different rates of hydrolysis and oxidation, explaining variations of the AV in the examined glyceride oils.

The induction period (IP) was also examined during the frying process. An increase in the frying time leads to a decrease in IP value for all samples. HOSO is found to be the most stable vegetable oil among the analyzed oils. The IP of the control oil was initially 46.4 hours, but decreased to 10.43 hours by the end of the process, indicating that the control oil is approximately four times more stable than the oils investigated here after 9 hours. The other oils (sunflower and rapeseed oil) were more unstable against the process of oxidation during the frying process. The IP decreases from 13.80 to 6.30 h for sunflower oil and from 16.9 to 8.48 h for rapeseed oil.

Refractive index

The refractive index values of the oil during the frying process are presented in **Fig. 1**. The obtained values show a significant difference in the mean refractive indices of all investigated oils. The refractive index of the oils increased with frying time. A similar finding was shown by Mudawi et al. (Mudawi et al. 2014). The changes in the refractive index are as follows: from 1.4660 (0 hour) to 1.4668 (9th hour) for HOSO, from 1.4691 to 1.4705 for sunflower oil, and from 1.4705 to 1.4720 for rapeseed oil, respectively. The highest values were observed at the 9th hour, regardless of the oil type. The increase of conjugated fatty acids with time leads to changes in the density and the colour of the oils, so that the refractive index changes as a result of thermal degradation (Godswill et al. 2018). The continuous increase in the refractive index of the oil during repeated frying indicates that deep frying increases their rancidity. Therefore, repeated frying using the same vegetable oil should be discouraged.

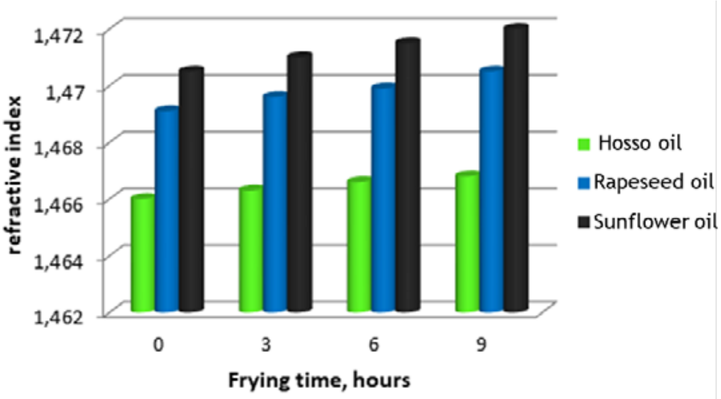


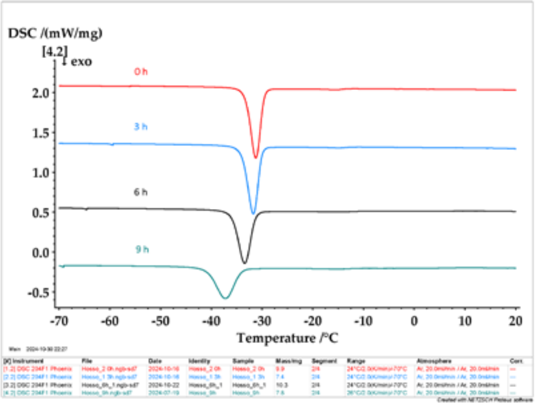
Fig. 1 *The mean values of refractive indices of the oils before and after frying.*

Differential scanning calorimetry

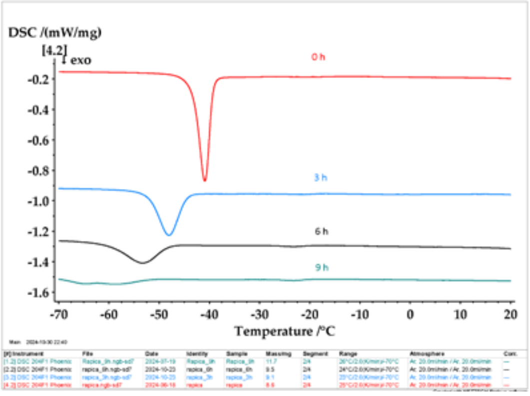
The DSC curves of the studied oil are presented in Fig. 2 (a-c). The cooling thermograms of the oils show a single peak, which corresponds to the exothermic crystallization phenomena. For all types of oil, the temperature and the enthalpy of crystallization decrease linearly with increase in frying time (Table 3). Similar results have been reported by other authors (Al-Khusaibi et al. 2022) and they are attributed to the oxidation process and changes in the chemical composition. In the case of oxidized oils, the chemical composition is affected by the polar compounds formed during frying i.e. hydroperoxides, free fatty acids, mono- and diacylglycerols, glycerol, and oxidized polymers (Chen et al. 2021). In their work Cuvelier et al. (Cuvelier et al. 2012) established a not oil-specific correlation between the total polar compounds (TPC) and the crystallization parameters of vegetable oils, taking into account their polyunsaturated fatty acid content (PUFA), expressed in percentage.

$$TPC\ (\%) = (a - b \cdot \Delta H) + c \cdot PUFA \cdot exp\left(-\frac{PUFA}{d}\right) \tag{1}$$

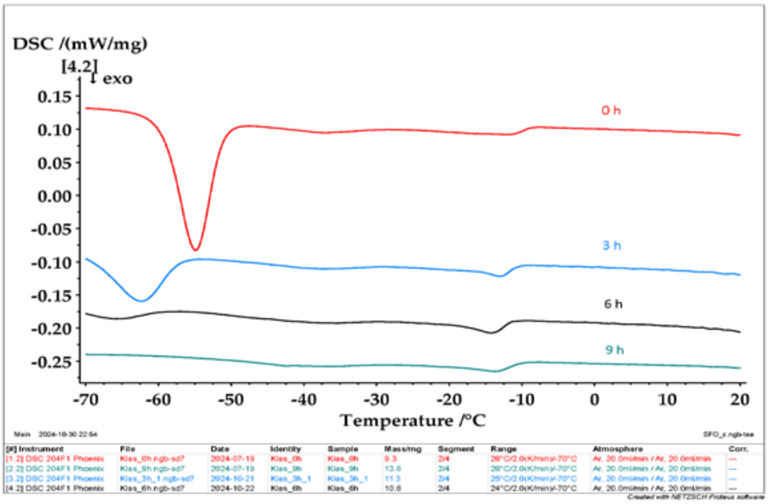
where ΔH is the specific enthalpy of crystallization in J/g. The *a*, *b*, *c,* and *d* parameters have the following values: a = 35.27 ± 1.573, b = 0.7492 ± 0.0402, c = 5.804 ± 0.600 and d = 10.13 ± 0.70.



a)



b)



[#] Instrument	File	Date	Identity	Sample	Mass/mg	Segment	Range	Atmosphere	Corr.
[1.2] DSC 204F1 Phoenix	Klas_0h.ngb-sd7	2024-07-19	Klas_0h	Klas_0h	9.3	2/4	26°C/2.0(K/min)/-70°C	Ar, 20.0ml/min / Ar, 20.0ml/min	---
[2.2] DSC 204F1 Phoenix	Klas_9h.ngb-sd7	2024-07-19	Klas_9h	Klas_9h	13.6	2/4	26°C/2.0(K/min)/-70°C	Ar, 20.0ml/min / Ar, 20.0ml/min	---
[3.2] DSC 204F1 Phoenix	Klas_3h_1.ngb-sd7	2024-10-21	Klas_3h_1	Klas_3h_1	11.3	2/4	25°C/2.0(K/min)/-70°C	Ar, 20.0ml/min / Ar, 20.0ml/min	---
[4.2] DSC 204F1 Phoenix	Klas_6h.ngb-sd7	2024-10-22	Klas_6h	Klas_6h	10.6	2/4	24°C/2.0(K/min)/-70°C	Ar, 20.0ml/min / Ar, 20.0ml/min	---

c)

Fig. 2. *DSC thermograms for crystallization phenomena of a) HOSO oil, b) rapeseed oil, and c) sunflower oil “Klas”*

The level of TPC for the sunflower, HOSO, and rapeseed oil are presented in table 3. In general, the level of TPC increased linearly across frying time with the coefficient of determinations of $R^2 > 0.98$. The lowest TPC levels were calculated for high oleic sunflower oil (HOSO) and the highest for the sunflower oil “Klas”, for which at the sixth hour a value of 35.6% was reached. Similar results were reported by Tarmizi et al., who studied the oxidation process of palm olein, soybean oil, rapeseed oil, and sunflower oil during frying (Tarmizi et al. 2019). Taking into consideration that a maximum limit of up to 25% has been suggested in official legislations from different countries (Firestone, 2007), it is suggested that only HOSO oil could be used for 9 hours of frying.

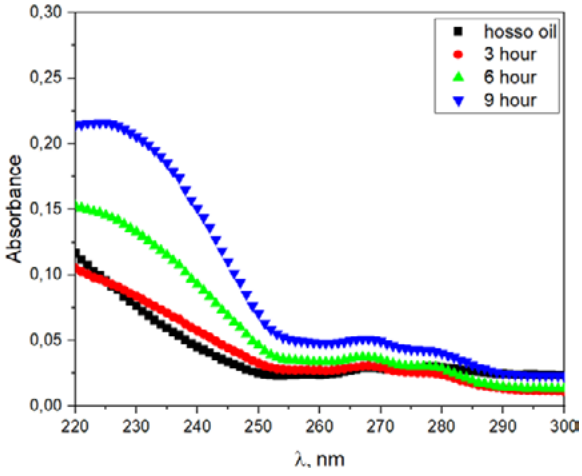
Table 3: The enthalpy of crystallization (ΔH), crystallization temperature (T_c), and total polar compounds (TPC) of the investigated oils.

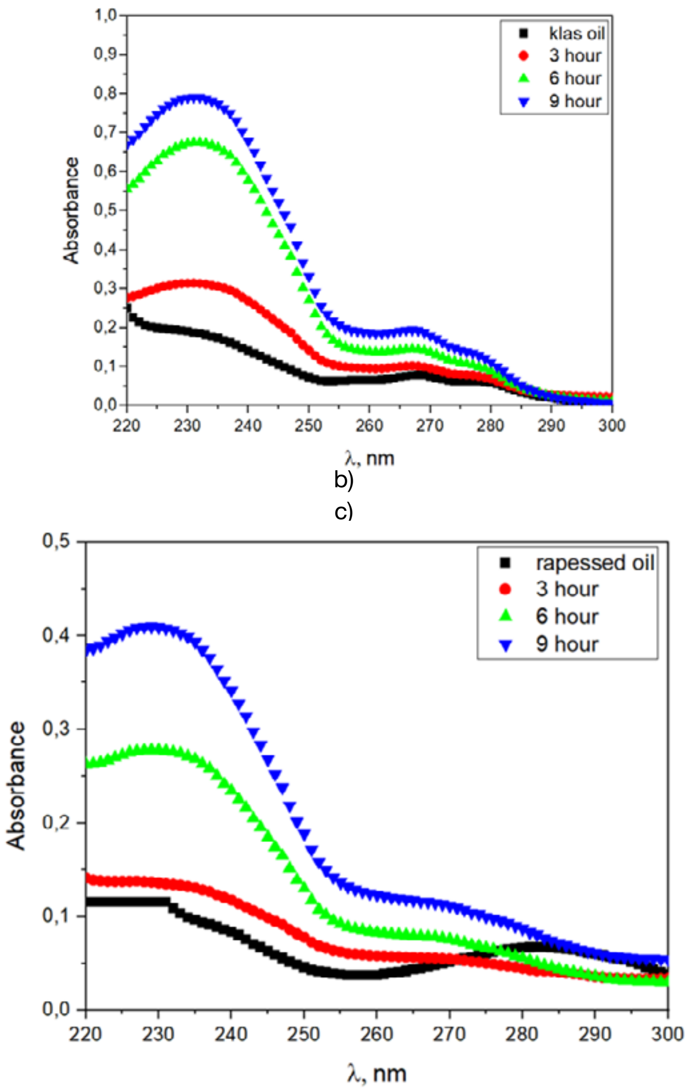
Frying time, h	Sunflower oil			High oleic sunflower oil (HOSO)			Rapeseed oil		
	ΔH, J/g	T_c, °	TPC, %	ΔH, J/g	T_c, °	TPC, %	ΔH, J/g	T_c, °	TPC, %
0	31.1	-54.9	13.4	64.51	-31.2	7.6	55.6	-40.9	4.2
3	12.9	-62.5	27.1	61.60	-31.7	9.8	40.1	-48.0	15.8
6	1.48	-65.3	35.6	58.70	-33.4	12.0	31.2	-53.3	22.5
9	-	-	-	54.18	-37.2	15.4	11.7	-59.0	37.1
R^2	**0.9828**			**0.9866**			**0.9813**		

UV-Vis spectroscopy

The UV absorbance (**Abs**) spectra of the investigated oils with the frying time are shown in **Fig.3** (a-c).

a)





b)

c)

Fig. 3. *Spectra of absorbance during the frying process at 220-300 nm of: a) HOSO oil; b) Sunflower "Klas" oil; c) Rapeseed oil. The numbers in the legend denote the hours of thermal usage.*

The results presented in figure 3 show that:

- UV-Vis spectra for all investigated oils are characterized with two peaks at wavelengths 232 nm and 268 nm. The absorbance at 232 nm corresponds to primary oxidation processes due to the formation of conjugated dienes as a consequence of the peroxidation products of sunflower biodiesel, whereas the absorbance at 268 nm appears as a result of secondary oxidation processes, associated with the formation of conjugated trienes. Analogous results were obtained by other researchers (Malavi et al., 2023; Hashem et al. 2020);
- Values of the absorbance at 232 nm increased from 0.07, 0.19, and 0.11 (0 hour) to 0.22, 0.80, and 0.41 (9 hour) of frying for HOSO, sunflower "klas", and rapeseed oil, respectively. The largest changes in the spectra occur for sunflower oil, followed by rapeseed oil, and the smallest for high oleic oil. These observations correspond to the findings of Šegatin et al. 2020, where the oils were kept at a temperature of 180°C for up to 40 hours. However, our results deviate from the monotonic increases of the band absorbance with time due to the large number of complex chemical processes occurring not only with the air and the moisture in it, but also with the substances of the potatoes;
- The absorbance of the oils increased with the frying time (0-9 hours) of the potato chips.

The time variation of the Abs of the two main bands (at 232 nm and 268 nm) for the investigated oils is shown in **Fig.4(a-c)**. and the alternation in the spectra, more precisely in the absorbance for the two main bands (at 232 nm and 268 nm) for sunflower oil is shown in **Fig.4b**. Both Abs curves show double maxima, divided by a minimum at the 4th hour. The second maximum of the 232-nm curve is more pronounced than that of the 268-nm curve. Such complex behavior is observed in the acidic value (AV) and induction period (IP) value trends, but the extreme values here do not correspond to those of AV and IP. In fact, the tendencies of the

HOSO oil are the same as those of sunflower but the Abs value differences are smaller.

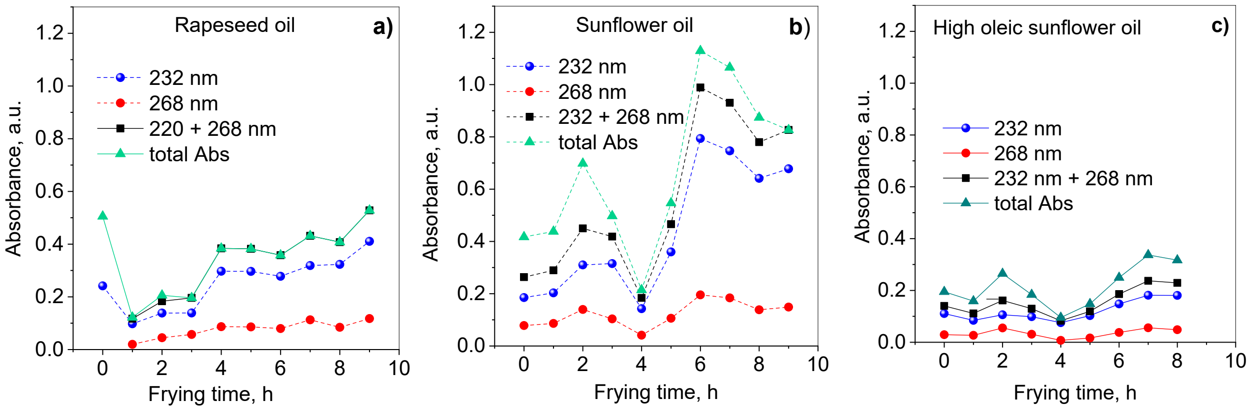


Fig. 4. *Absorbance of the main two peaks at 232 nm and 268 nm for the different oils dependent on time of frying: a) Rapeseed oil; b) Sunflower oil; c) High oleic sunflower oil.*

Electric impedance spectroscopy

In **Fig.5a** a representative plot of frequency dependence on frying time for rapeseed oil is shown. As discussed by many authors (Valantina, 2021; Šegatin et al. 2020), the values are almost independent of the frequency in the range above 150 kHz. The initial low values could be attributed to some pre-electrode processes in the measuring cell. At higher frequencies, the curves appear parallel to each other so for comparison purposes the values at fixed frequency (600 kHz) will be discussed. In **Fig.5b** the variation of with frying time for all three oils is given. The values for the rapeseed oil initially decrease and then gradually increase. For the sunflower oil a double maxima curve is presented. The limits of EIS is demonstrated by the high oleic sunflower oil results, where the values are almost independent of the frying time, which deviates from the UV Abs trend.

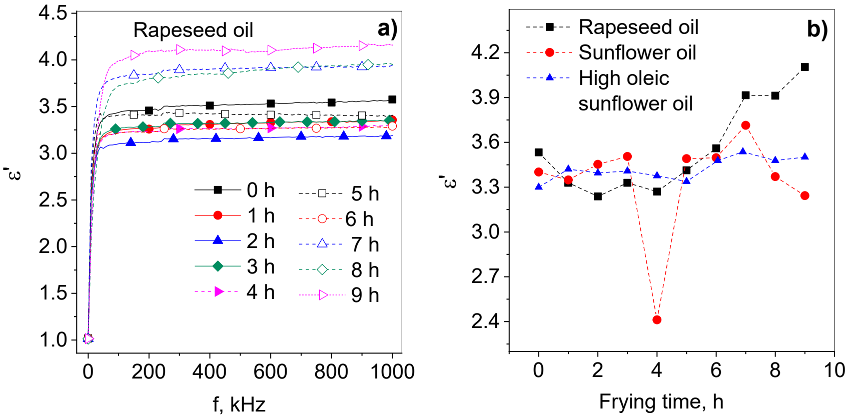


Fig. 5. a) Dielectric dispersion, ε', dependent on frequency for Rapeseed oil for different frying times (the curves are based on 100 experimental points); b) ε' variation with frying time for all used oils. The Standard Deviation was in the limits of 0.005.

5. Conclusion

The aim of the present study was to evaluate the rancidity development during deep frying for 3 types of oils by different physical and chemical methods. The deep frying process affected induction times, making them shorter. With an increase in the frying time, refractive indices of all oil types along with their recrystallization enthalpies, due to the presence of polar compounds, increased. As a result of oxidation processes and formation of conjugated trienes, an alternation in the UV-VIS spectra of all oils in the region of 232 nm and 268 nm was observed. The least changes in all measured parameters were found for the HOSO type of oil.

6. Acknowledgments

Authors are highly grateful to project FT23-FTF-012 “Physical methods for fats characterization” for the financial support of this research.

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