

Article

Oxidation and Flammability Tests for Grape (*Vitis vinifera* L.) Seed Oil

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Abstract: In this work, studies were performed on oxidative stability by determining the transmittance spectra, the components and trichromatic coordinates, and the color differences for grape seed oils (GSO) subjected to a forced oxidation treatment at temperatures of 100 °C and 120 °C, for 4, 8 and 10 h. For this purpose, a constant airflow of 30 L/min was used. GSO was also subjected to flammability tests on a heated cylindrical surface to determine the lowest temperature at which this oil ignites, correlated with the highest temperature at which the oil does not ignite. According to the results, these temperatures are 475 °C and 470 °C, respectively. At these temperatures, the tested oils were darker in color than the reference oil, with the L* parameter having lower values (91.53 and 89.56, respectively). In addition, the correlation coefficients between the evaluated parameters were significant ($p \leq 0.05$).

Keywords: oxidation; flammability; color; transmittance; CIELAB; eco-friendly lubricant

1. Introduction

Grapes (*Vitis Vinifera* L.) have been cultivated since ancient times and are the fruits with the largest world production [1,2]. Various spirits and wine are obtained from grapes, but also a valuable oil from their seeds. In one of the studies [3], it is shown that considering the existence of various types of grapes, the oil yield is variable depending on the origin of the grape seeds. The European Union has 45% of the world's wine-growing areas, 64% of production, 48% of world consumption and 70% of wine exports [4]. Grape seed oil (GSO) is obtained through various methods and techniques that have evolved to increase the extraction yield and to improve the quality of the oil. Moreover, in order to extract GSO, various ecological strategies have been developed, obtaining a superior quality oil by cold pressing the seeds remaining after the process of pressing grapes to obtain wine [5,6]. Some data from the literature [7–10] show that oil is obtained from grape seeds that contain various unsaturated fatty acids, of which linoleic acid and oleic acid predominate. Also, GSO contains tocopherols, tocotrienols, polyphenols, flavonoids, tannins and phytosterols [11–14]. Moreover, studies in the literature show that the percentage of oil extracted from grape seeds depends largely on the grape variety [15,16].

The aspects related to the oxidative stability of GSO have been much addressed in the literature [12,17,18]. Some authors [19] conclude that using antioxidant mixtures obtained from natural plants does not significantly increase the oxidation stability of GSO. However, recent studies [20] emphasize the importance of using aqueous extracts to improve the behavior of this oil to oxidative factors. Thus, the authors of this study show that extraction optimization plays an important role in these studies, emphasizing the need to implement ultrasound-assisted aqueous extraction that leads to an optimization of the extraction yield of phenolic compounds. The use of compounds with good antioxidant activity in the composition of GSO will increase the potential for using this oil in various industrial sectors where high dispersion causes environmental and safety risks. Determination of specific parameters regarding the study of antioxidant activity in terms of GSO and other



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environmentally friendly oils, such as total phenol content (TPC), total tannin content (TTC), total anthocyanin content (TAC), carotenoid composition and α -tocopherol content, DPPH, ABTS, FRAP, etc. is one of the fundamental objectives encountered more and more often in specialized studies in recent years [21–25].

All these studies on GSO promise a real opportunity to use this type of oil as a chainsaw lubricant, especially in the current context of using environmentally friendly ingredients. In the agro-forestry environment, chainsaws are associated with lubrication systems that have total loss. Therefore, the use of mineral lubricants is an important risk factor for the health of chainsaw users [26]. Also, this research encourages the use of GSO as a potential lubricant in the agricultural tractor sector, for which recent studies recommend the use of vegetable oils [27].

Like other vegetable oils, GSO can only be used in areas where the operating conditions of the machines are moderate. Regarding this topic, some researchers [5,6,28–31] have studied GSO as a potential lubricant and ecological fuel. According to Serra et al. [32], this type of oil is recommended as a possible biolubricant that can successfully replace established lubricants, its viability being proven by determining its physical properties and wear rate. The authors of this study concluded that the wear tests indicate that the mineral oil used for comparison as a reference oil performed better than GSO. On the other hand, GSO outperformed the reference oil in terms of certain physical properties such as flash point and pour point.

However, the oxidation process influenced by various intervening factors, such as temperature, can reduce the effectiveness of this type of oil used as a biolubricant. In their research, Ramos M. et al. [6] studied the influence of the properties and composition of vegetable oils, such as GSO, on the quality of synthesized biodiesel. All these studies conclude one thing: replacement of mineral oils, with a negative impact on the environment, is only possible through an adequate development of studies on biolubricants. Recent studies [33] show that developing higher-quality lubricants is particularly important in the context of the increasing demand for more ecological lubricating oil with increased performance. Obtaining and studying lubricants obtained from renewable sources is also important in terms of cost reduction.

In this context, great interest has been focused on the use of vegetable biodegradable basestocks for lubricant oils [34]. However, the use of vegetable oils to formulate lubricants has some disadvantages. In his research, Owuna [35] shows that lubricants obtained from vegetable oils, and especially from their crude form, are mostly thermally and oxidatively unstable. On the other hand, vegetable oils contain natural antioxidants that stabilize them against oxidation and extreme temperatures [36,37]. The author of this research concludes that vegetable oils have the potential to be used for the formulation of lubricants. Moreover, they can reduce excessive dependence on the consumption of fossil energy sources. The major problem associated with mineral lubricants is poor biodegradability. In addition, high toxicity has a negative impact on the environment. That is why countless studies [38,39] are oriented towards the identification of potential environmentally friendly lubricants, on vegetable oils, respectively. Thus, research conducted in various parts of the world indicates a growing concern for the use of several types of vegetable oils as lubricants. The authors of these studies emphasized the feasibility of using GSO as a lubricant [32,40], but also of other vegetable oils, such as corn oil [41,42], castor/linseed vegetable oils [43–45], coconut oil [46], sunflower oil [43,45,47,48], rapeseed [45,48], soybean [43,45,48,49] etc.

Therefore, considering that GSO can have enormous potential in its possible use as a lubricant in industrial sectors, the main purpose of this study was to determine, in a new approach, its flammability behavior. Moreover, the study aimed to obtain detailed information about the stability of the GSO color during the oxidation and heating processes and their correlation, especially since we believe this niche has not yet been sufficiently studied and deserves attention.

2. Materials and Methods

The refined oil selected for this study (obtained from grape seed) was purchased from a local store. The oil has been stored in its original packaging to avoid direct contact with light. The tests were carried out under conditions of forced oxidation at temperatures of 100 °C and 120 °C, for 4, 8 and 10 h. A constant airflow of 30 L/min was used for this purpose. The oil samples subjected to analysis (50 cm³) were stored in special test tubes with a 200 cm³ capacity to prevent possible inconveniences due to foaming. A mineral oil bath was used to carry out the studies at 100 °C and 120 °C temperatures. Traditionally, determination of the color of vegetable oils is performed using colorimeters, spectrometers, and other analytical equipment. In this study, color measurements for GSO were made using the CIE L*a*b* system according to CIE (Commission Internationale d’Eclairage/Illumination International Commission) indications. Transmittance spectra were obtained using a UV-Vis spectrophotometer (T60, PG Instruments Limited (Leicestershire, UK)). The measurements were carried out in the range of wavelengths between 380 and 780 nm, the measurement step being 0.2 nm, using a 10 mm glass cuvette. From the spectral transmission curves, the trichromatic coordinates and the trichromatic components were determined (measurements were made using light source D65 and a 10° observation field/perception angle of a human observer). Currently, spectrophotometric methods are widely used in color analysis due to the efficiency and accuracy of the results obtained [50–52]. The tests were performed with up to three replications. The schematic diagram of the experimental setup is shown in Figure 1.

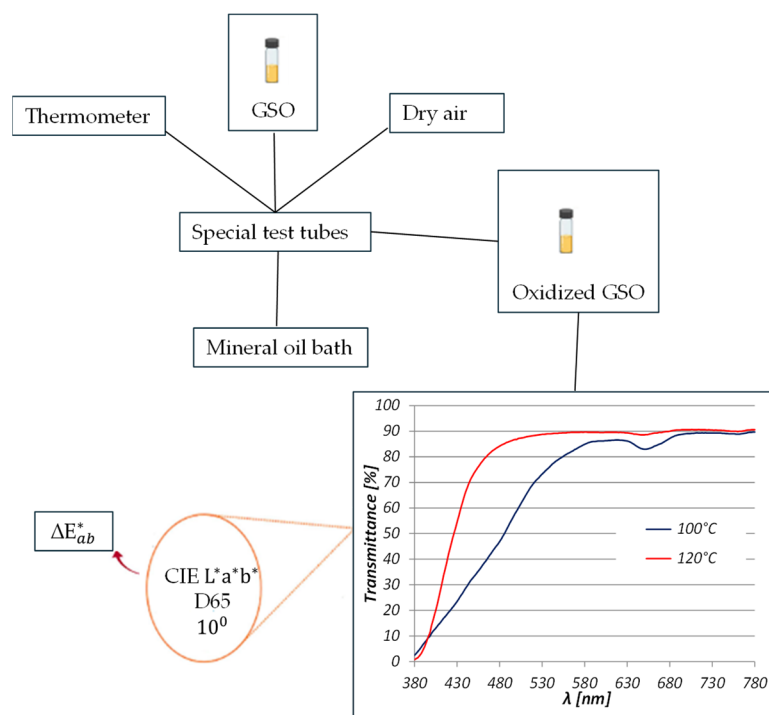


Figure 1. Scheme of the GSO oxidation process.

The statistical analysis was performed using Statistical Product and Service Solutions software (SPSS, Version 10, IBM, Chicago, IL, USA). Pearson’s correlation coefficient was used to determine the correlations between the variants.

The flammability tests were conducted on an installation belonging to the Lubricant Analysis Laboratory of the Faculty of Engineering of the “Dunarea de Jos” University of Galati. The setup and working procedures were detailed in several papers [53–58]. The schematic diagram of the experimental setup is shown in Figure 2.

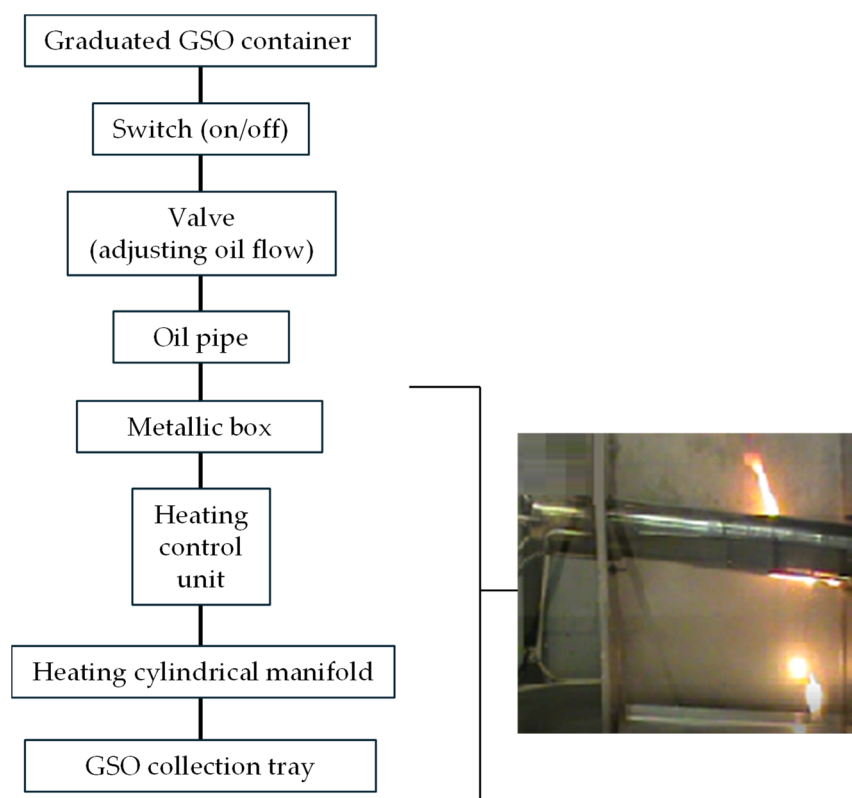


Figure 2. Schematic diagram of the experimental equipment for testing the GSO flammability with a hot surface as the heat source.

On the surface of the cylinder heated to various temperatures, a quantity of 10 ± 0.5 mL of oil is dripped for 55 s. During the entire test period, the temperature of the cylinder remains constant.

3. Results and Discussion

It is known that unsaturation can irreversibly affect the stability of oils, and, as a result, this will subsequently lead to their degradation effects [59].

Thus, in this work, grape seed oils were subjected to heat treatment at temperatures of 100 °C and 120 °C, maintained for 4, 8 and 10 h, with an air intake having a constant flow of 30 L/min. After this treatment, the oils were analyzed with the help of a spectrophotometer and the evolution of transmittances was determined. Based on the determination of the transmittance values, the trichromatic components and coordinates were calculated, as well as the color differences of the heat-treated grape seed oils. In Figures 1–3, the variations of the transmittances of the oxidized and heat-treated oils at the temperatures of 100 °C and 120 °C, for 4, 8 and 10 h, were represented.

The transmittance curves of the grape seed oils oxidized for 4, 8 and 10 h at 100 °C temperature compared to the reference sample (non-oxidized) are shown in Figure 3a. Moreover, the transmission curves were also plotted at 120 °C temperature (Figure 3b).

Analyzing the transmittance curves obtained for non-oxidized oil and oxidized oil for 4 h, it is observed that there are no significant differences between their transmittance values, over the entire range of wavelengths analyzed (Figure 3a), which indicates that the oxidative effect is weak and that no obvious structural changes were revealed in the composition of the tested oil.

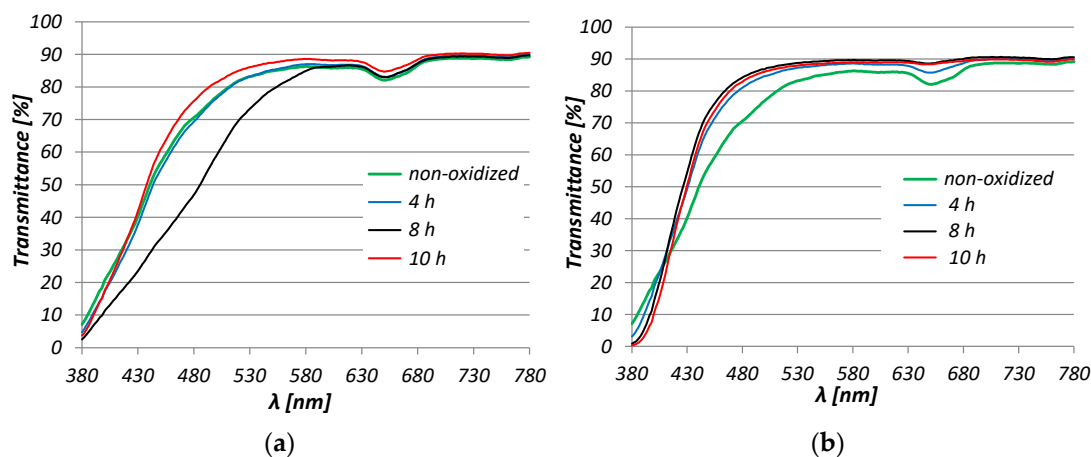


Figure 3. Spectral transmittance curves of oxidized grape seed oils at 100 °C (a) and 120 °C (b) for 4, 8 and 10 h.

Increasing the oxidation time to 8 h, in the range of wavelengths 400–580 nm, a significant decrease in transmittance values is observed compared to the standard sample, followed by insignificant differences in transmittances in the last range of wavelengths. This can be attributed to structural changes that occur during oxidation processes, the main reason being the resulting peroxides from unsaturated fatty acids (C12:2, C16:1, C18:1 cis, C18:2 and C18:3, C20:1, C20:4, C22:1) present in the composition of the analyzed oil and their decomposition [12,17,60–62].

In the context of these discussions, according to a recent study [63], oxidative degradation, correlated with complex implications of lipid oxidation, is associated with the formation of new organic compounds, such as, for example, n-hexanal, as a result of the degradation of the most abundant unsaturated fatty acid in the GSO composition, namely linoleic acid. The same study claims that other derivatives of associated aldehydes and ketones result as products of lipid oxidation, as well as furan derivatives resulting from thermal oxidative degradation of unsaturated fatty acids.

Thus, along with the thermal oxidative processes, various compounds associated with GSO degradation result, which, in our opinion and as shown by the results of the present study, induce changes in the chromatic parameters.

Moreover, our results show that keeping the oil at a temperature of 100 °C for 10 h causes an insignificant decrease in the transmittance values in the first wavelength range (380–418 nm) followed then, until the end of the range, by a slight increase in transmittance values compared to the non-oxidized reference sample (Figure 3a).

Furthermore, it is shown that there is a non-monotonicity of the spectral transmittance curves that occurs at 652 nm. This can probably be attributed to minor components (non-saponifiable materials) in the GSO composition, such as the chlorophyll fraction and pheophytins a. Also, the data presented in Figure 3 show one small area (632 ÷ 694 nm) induced due to the presence of these types of pigments (chlorophylls and pheophytins) [64].

In addition, the hypochromic effect is noticed, probably because of pigment degradation that occurs during the oxidation process. This area was found in the green color transmission zone and is mainly due to the pheophytins (Q band). The brown color of this pigment induces the GSO color change with its oxidation [65].

More than that, from our results, a transmittance increase can be observed from 8 to 10 h (Figure 3), and from 100 to 120 °C (Figure 4). This variation in transmittance can be most likely associated with the formation of oxidation products. Under the experimental conditions, important pigments in the GSO composition, such as carotenoids, can undergo significant degradation. This inevitably leads to an increase in their discoloration, which induces an accentuation of the hypo-chromic effect. Therefore, the changes observed in these situations during oxidative and thermal exposure of GSO can be attributed to

the loss of carotenoid conjugation. In this context, major changes are occurring in the blue region of the spectral curves. Therefore, these transformations in terms of chemical composition during oxidation at increasing temperature can be associated with a decrease in the iodine index (a measure of the degree of unsaturation), respectively with a reduction in the number of double bonds. According to studies realized by Arab et al. [66], the iodine value decreases as the temperature of the natural oil used in the study increases.

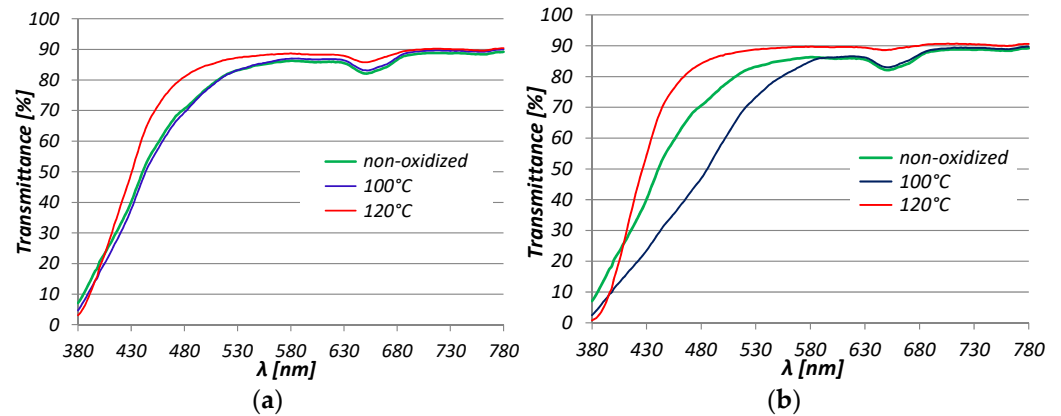


Figure 4. Spectral transmittance curves of oxidized grape seed oils for 4 h (a) and 8 h (b) at 100 °C and 120 °C.

These results indicate that transmittance measurements can provide useful information on the degree of resistance of such an oil to oxidation processes that may occur during lubrication.

On the other hand, testing oils at a temperature of 120 °C causes an increase in transmittance values, compared to the non-oxidized oil sample, for all oxidation periods, in the range of wavelengths 416–530 nm (Figure 3b).

The increase in the oxidation temperature to 120 °C causes an increase in the transmittance values for the oil tested for 4 h, compared to the oil oxidized at the temperature of 100 °C, especially in the range of wavelengths 416–500 nm (Figure 4a).

In the range of wavelengths 400–580 nm, for the oil tested at 100 °C temperature, for 8 h, there were decreases in the transmittance values, compared to the standard sample, while for the oil tested at 120 °C temperature, the transmittance values increased (Figure 4b).

Finally, our results show that in the case of the oils tested for 10 h (Figure 5), the increase in the oxidation temperature causes an increase in the transmittance values, compared to the non-oxidized oil sample, in the range of wavelengths 400–700 nm.

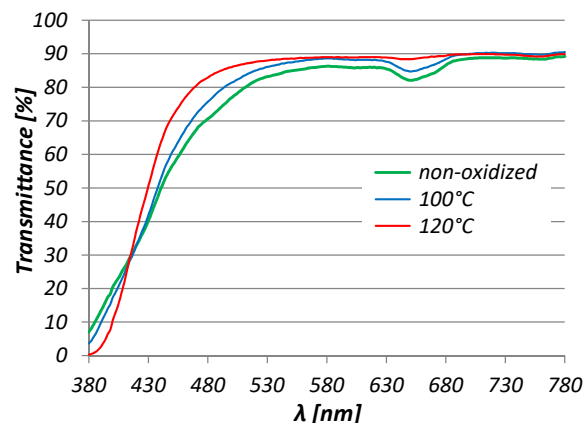


Figure 5. Spectral transmittance curves of oxidized grape seed oils for 10 h at 100 °C and 120 °C.

Testing oils at 120 °C causes an important increase in transmittance values for all oxidation periods, in the wavelength range 410–680 nm.

On the other hand, it is fundamental to correlate these changes in the composition of the analyzed oil over the course of and during the oxidation processes with the variation of color shades throughout these stages of the oxidation process.

In this context, based on the transmittance spectra, the trichromatic components and coordinates were calculated, as well as the color differences for GSO oxidized at 100 °C and 120 °C, respectively, for 4, 8 and 10 h (Tables 1–6).

Table 1. Experimental results for grape seed oils oxidized at a temperature of 100 °C, obtained according to the color system (x, y) CIE 1964 (CIEXYZ)/Illuminant D65/10° [51,52].

Grape Seed Oils 100 °C		Non-Oxidized	4 h Oxidized	8 h Oxidized	10 h Oxidized
Trichromatic components *	X	76.986 ± 0.684	77.011 ± 0.685	70.581 ± 0.627	79.510 ± 0.707
	Y	79.735 ± 0.708	79.775 ± 0.709	72.490 ± 0.644	82.344 ± 0.732
	Z	68.268 ± 0.607	66.057 ± 0.587	42.744 ± 0.380	72.817 ± 0.647
Trichromatic coordinates	x	0.342 ± 0.003	0.346 ± 0.004	0.380 ± 0.004	0.339 ± 0.003
	y	0.354 ± 0.003	0.358 ± 0.004	0.390 ± 0.004	0.351 ± 0.003
	z	0.303 ± 0.003	0.296 ± 0.003	0.230 ± 0.002	0.310 ± 0.003

* X, Y, Z—tristimulus values. Values are mean ± standard deviation (n = 3).

Table 2. Chromatic coordinates (system: CIELAB *) for grape seed oils oxidized at 100 °C/Illuminant D65/10°.

Grape Seed Oils 100 °C		Non-Oxidized	4 h Oxidized	8 h Oxidized	10 h Oxidized
Chromatic coordinates	L*	91.57 ± 0.814	91.58 ± 0.814	88.20 ± 0.784	92.73 ± 0.824
	a*	2.82 ± 0.025	2.79 ± 0.025	4.00 ± 0.036	2.86 ± 0.025
	b*	13.44 ± 0.119	15.35 ± 0.136	32.50 ± 0.289	11.71 ± 0.104
	a*/b*	0.21	0.18	0.12	0.24
	(a*/b*) ²	0.04	0.03	0.02	0.06
	C _{ab} *	13.74 ± 0.122	15.60 ± 0.139	32.75 ± 0.291	12.05 ± 0.107
	h _{ab}	78.15 ± 0.001	79.70 ± 0.002	82.98 ± 0.003	76.26 ± 0.001

* Symbols: CIE L*a*b* system; L*—lightness; a*—redness/greenness; b*—yellowness/blueness; C*—chroma (C_{ab}* = $\sqrt{a^{*2} + b^{*2}}$); h_{ab}—hue angle (h_{ab} = $\tan^{-1}(b^*/a^*)$); D65—CIE standard illuminant [52].

Table 3. Experimental values of color differences for grape seed oils oxidized at 100 °C.

Grape Seed Oils 100 °C	ΔL^*	Δa^*	Δb^*	ΔC_{ab}^*	Δh_{ab}
4 h oxidized	0.02	−0.03	1.91	1.87	1.54
8 h oxidized	−3.36	1.18	19.06	19.01	4.84
10 h oxidized	1.16	0.04	−1.74	−1.69	−1.88

Table 4. Experimental results for grape seed oils oxidized at 120 °C, obtained according to the color system (x, y) CIE 1964 (CIEXYZ)/Illuminant D65/10°.

Grape Seed Oils 120 °C		Non-Oxidized	4 h Oxidized	8 h Oxidized	10 h Oxidized
Trichromatic components	X	76.986 ± 0.684	81.526 ± 0.725	83.661 ± 0.744	82.597 ± 0.734
	Y	79.735 ± 0.709	84.163 ± 0.748	86.153 ± 0.766	85.126 ± 0.757
	Z	68.268 ± 0.607	81.483 ± 0.724	87.065 ± 0.774	84.205 ± 0.748
Trichromatic coordinates	x	0.342 ± 0.003	0.330 ± 0.003	0.326 ± 0.003	0.328 ± 0.003
	y	0.354 ± 0.003	0.341 ± 0.003	0.335 ± 0.003	0.338 ± 0.003
	z	0.303 ± 0.003	0.330 ± 0.003	0.339 ± 0.003	0.334 ± 0.003

Table 5. Chromatic coordinates (systems: CIELAB) for grape seed oils oxidized at 120 °C/Illuminant D65/10°.

Grape Seed Oils 120 °C		Non-Oxidized	4 h Oxidized	8 H Oxidized	10 h Oxidized
Chromatic coordinates	L*	91.57 ± 0.557	93.52 ± 0.569	94.38 ± 0.574	93.94 ± 0.571
	a*	2.82 ± 0.017	3.39 ± 0.021	3.81 ± 0.023	3.67 ± 0.022
	b*	13.44 ± 0.082	6.36 ± 0.039	3.77 ± 0.023	5.07 ± 0.031
	a*/b*	0.21	0.53	1.01	0.72
	(a*/b*) ²	0.04	0.28	1.02	0.52
	C _{ab} *	13.74 ± 0.084	7.21 ± 0.044	5.36 ± 0.033	6.26 ± 0.038
	h _{ab}	78.15 ± 0.475	61.98 ± 0.377	44.66 ± 0.272	54.14 ± 0.329

Table 6. Experimental values of color differences for grape seed oils oxidized at 120 °C.

Grape Seed Oils 120 °C	ΔL*	Δa*	Δb*	ΔC _{ab} *	Δh _{ab}
4 h oxidized	1.96	0.57	−7.08	−6.53	−16.18
8 h oxidized	2.81	0.99	−9.68	−8.38	−33.49
10 h oxidized	2.37	0.85	−8.37	−7.48	−24.02

The evolution of the following specific parameters is of major importance to evaluate the color of the oil due to oxidative processes: brightness parameter (L*), yellowness parameter (b*) and, respectively, color differences (Δ_{ab}*).

For the brightness parameter (L*), in the case of oils oxidized at a temperature of 100 °C, for 4, 8 and 10 h, it is observed that the oil oxidized for 4 h records approximately the same value compared to the non-oxidized oil sample (Table 2), which denotes a good oxidation behavior of GSO under the aspect of this chromatic parameter. Regarding non-oxidized oil color, measurements have shown that the value of lightness was 91.57 (the average measured values). In the context of the few research studies on the present topic, Masan et al. (2016) [67] reported comparable results between 77.64 and 90.52 units. Moreover, our results regarding this chromatic parameter are similar to those realized on other types of vegetable oil. In this context, the studies carried out by Moyano et al. (2008) [68] on some samples of virgin olive oil indicated brightness values between 49.88 and 99.28 units. We believe that these results are very useful for future research. A recent study [26] proposes the possibility of using refined olive pomace oil as a base stock for the formulation of a totally biodegradable chainsaw lubricant. Furthermore, Ramos-Escudero et al. [69] reported values of lightness of commercial Sacha inchi oil originating from Peruvian cultivars from 91 to near 100 units. These comparable results indicate that GSO is similar in lightness to other vegetable oils.

However, upon increasing the oxidation time to 8 h, there is a decrease in this parameter, followed by an increase with the corresponding increase in the oxidation time to 10 h. Thus, after 10 h of forced oxidation, the brightness parameter records an increase compared to non-oxidized oil of 1.27%. In the context in which the oxidation temperature increased to 120 °C, a tendency to increase the brightness parameter values is observed (Table 5), the increase of this parameter, after 10 h of oxidation, being 2.59% compared to the non-oxidized oil sample.

Regarding the next parameter that was particularly monitored, the degree of yellowness (b*), at the oxidation temperature of 100 °C (Table 2), it was found that it shows a slightly increasing trend after 4 h, followed by a sharp increase after 8 h, while after 10 h of oxidation, this parameter records an equally pronounced decrease. The value of the parameter b*, recorded after 10 h of forced oxidation, decreases by approximately 13% compared to the non-oxidized standard sample. Moreover, increasing the oxidation temperature to 120 °C causes a sharp decrease in the values of the analyzed parameter (b*) for all oxidation periods, compared to the non-oxidized sample. Thus, after 10 h of oxidation,

there is a 62.27% decrease in the value of the b^* parameter compared to the non-oxidized reference sample. This variation of the GSO color depending on the temperature and the time during which it is subjected to forced oxidation is also supported by the other chromatic parameters, such as chroma, C_{ab}^* and the hue angle, h_{ab} (Tables 4 and 5). The colorimetric analyses carried out using chromatic parameters indicated that the effect of oxidation on the oil results in a significant decrease in the chromatic parameters. Moreover, the variation of the hue angle is correlated with the chromatic tonality. The effect of oil oxidation on its color variation is expressed much more precisely through the differences of these chromatic parameters reported with respect to the reference (non-oxidized oil). The color differences corresponding to these color coordinates are presented in Tables 3 and 6.

Also, Figure 6 shows the color differences (ΔE_{ab}^*) recorded between the oxidized samples and the non-oxidized reference sample. Oil oxidized at 100 °C for 4 h produced the least color difference, probably because GSO is characterized by a high total phenolic content and a good antioxidant capacity [70].

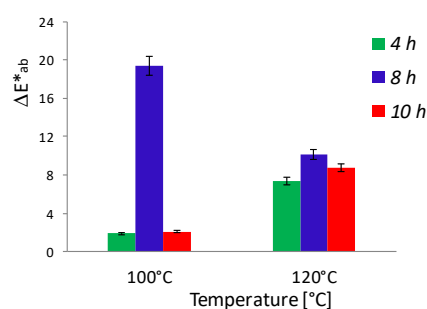


Figure 6. Total color differences (ΔE_{ab}^*) for oxidized grape seed oils for 4, 8 and 10 h at 100 °C and 120 °C. $\Delta E_{ab}^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$ [52] where: ΔL^* , Δa^* , and Δb^* are the differences of these values between the samples just after oxidation and non-oxidized samples.

By increasing the oxidation time to 8 h, an increase in color differences is recorded, the oil undergoing an intense oxidation process. After 10 h of oxidation, there is a decrease in this parameter, probably as a result of the accumulation of secondary compounds specific to lipid oxidation processes that have been shown to have a decisive impact on the sensory properties [71].

On the other hand, increasing the test temperature to 120 °C (Figure 6) and maintaining it for 4 h causes a much larger color difference than that obtained after 4 h at 100 °C, the increase being 286%. This shows that an increase in temperature by 20 °C generally leads to a considerable enhancement of the lipid oxidation process. However, after 8 h of oxidation, the color difference decreases significantly, probably because more peroxides are broken down than formed.

Increasing the oxidation time to 8 and 10 h, respectively, at 120 °C temperature, increases in the ΔE_{ab}^* parameter are observed, by 37.5% (after 8 h of oxidation) and by 18.6% (after 10 h of oxidation), compared to the oil sample oxidized for 4 h. Therefore, the data presented in Figure 6 highlight the color variation along the course of GSO oxidation depending on the temperature and the duration to which the oil is subjected to the forced oxidation process.

The observations regarding the relationship between the color variation and the degree of GSO deterioration during oxidation are similar to those made by Ma et al. [72] who carried out research on the deterioration of fully formulated aviation lubrication oils. Similarly, they concluded that the change in color coincides with the deterioration of oil quality and can be regarded as a significant clue to diagnose the quality of that lubricant. Moreover, this aspect is supported by the data presented in Table 7 according to which the correlation coefficients between the chromatic coordinate values and the corresponding GSO oxidation time show that, in general, there are significant correlations between them.

Table 7. Pearson linear correlation coefficients between the values of the chromatic coordinates (system CIELAB) in the case of oxidized grape seed oils ^{a,b} for 4, 8 and 10 h.

	L*	a*	b*	C _{ab} *	h _{ab}	t
^a Grape seed oils, 100 °C						
L*	1.000					
a*	−0.947	1.000				
b*	−0.985	0.979	1.000			
C _{ab} *	−0.985	0.980	0.998	1.000		
h _{ab}	−0.985	0.981	0.999	0.998	1.000	
t	−0.124	0.405	0.627	0.629	0.291	1.000
^b Grape seed oils, 120 °C						
L*	1.000					
a*	0.990	1.000				
b*	−0.999	−0.985	1.000			
C _{ab} *	−0.996	−0.977	0.999	1.000		
h _{ab}	−0.972	−0.988	0.962	0.948	1.000	
t	0.896	0.934	−0.892	−0.885	−0.888	1.000

t—oxidation time (hours).

According to Cohen [73], the small value of the correlation coefficient, *r*, indicates a small correlation only when the oil was oxidized at 100 °C in the case of two chromatic parameters: the brightness and the hue angle, respectively. In all the other situations, the Pearson coefficients indicate a significant correlation (strong positive and strong negative correlation).

Subsequently, grape seed oil was tested to determine the lowest temperature at which this oil ignites and the highest temperature at which the oil does not ignite when dropped on a heated cylindrical surface. Figure 7 shows the sequence and results of the flammability tests.

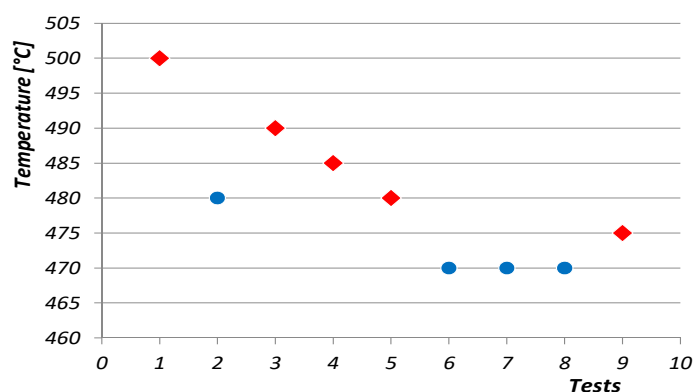


Figure 7. Flammability tests for grape seed oil (♦—it ignites; ●—it does not ignite).

The first test was performed at 500 °C temperature. At this temperature, the oil ignited after 5 s (Figure 8b) and immediately after, a strong burning of the oil on the surface of the cylinder started (Figure 8c captures the strongest burning). The oil does not burn in contact with the collector tray. Oil burning on the cylinder surface continues for 11 s after the drip time has expired (Figure 8d).

In the continuation of the experiment, the test temperature was lowered to 480 °C. Throughout the test, the grape seed oil did not ignite (Figures 9 and 10).

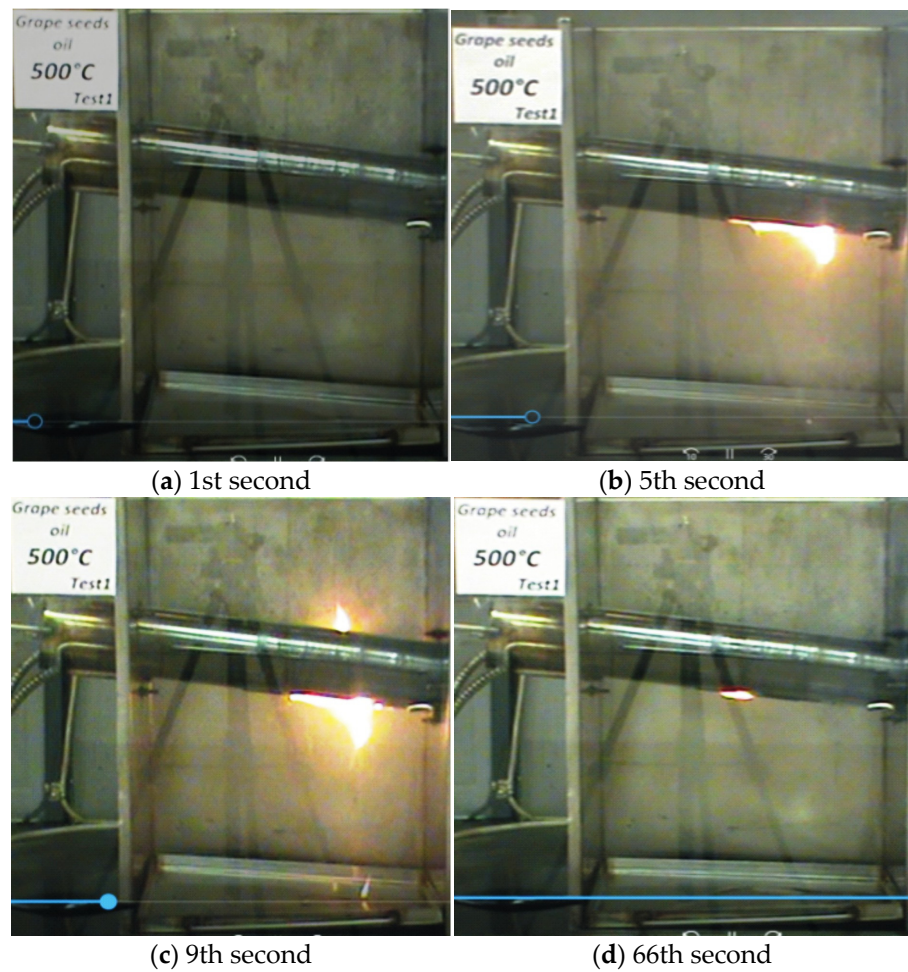


Figure 8. Grape seed oil tested at 500 °C.

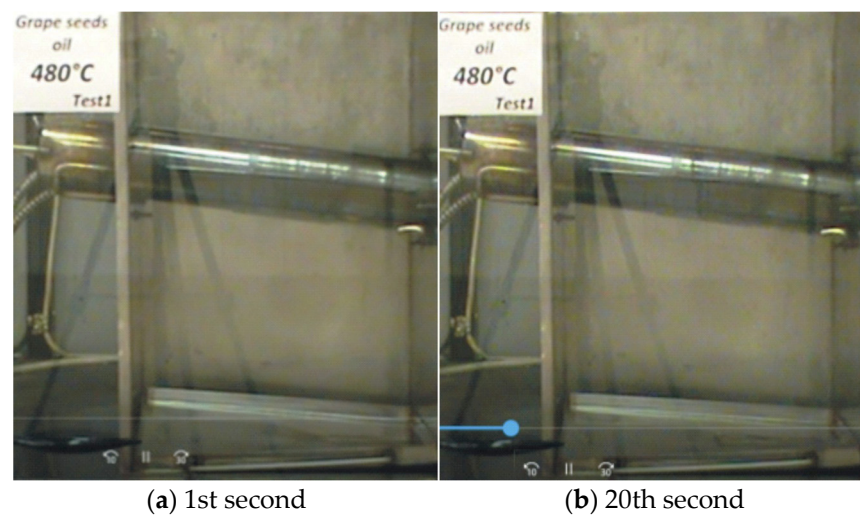


Figure 9. Grape seed oil tested at 480 °C (Test 1).

In this context, a temperature higher than 480 °C is chosen, located in the middle of the interval between the two temperatures at which the oil was tested; in this case, 490 °C. At this temperature, the oil ignites 16 s after the start of the test (Figure 11b) and continues to burn for the entire test duration and 7 s after the experiment is over (Figure 11d). Figure 11c shows the most intense burning during the test.

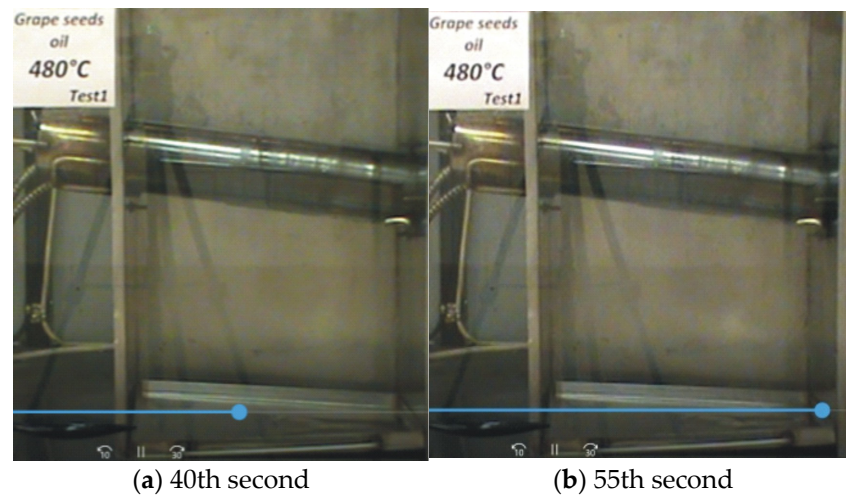


Figure 10. Grape seed oil tested at 480 °C (Test 1).

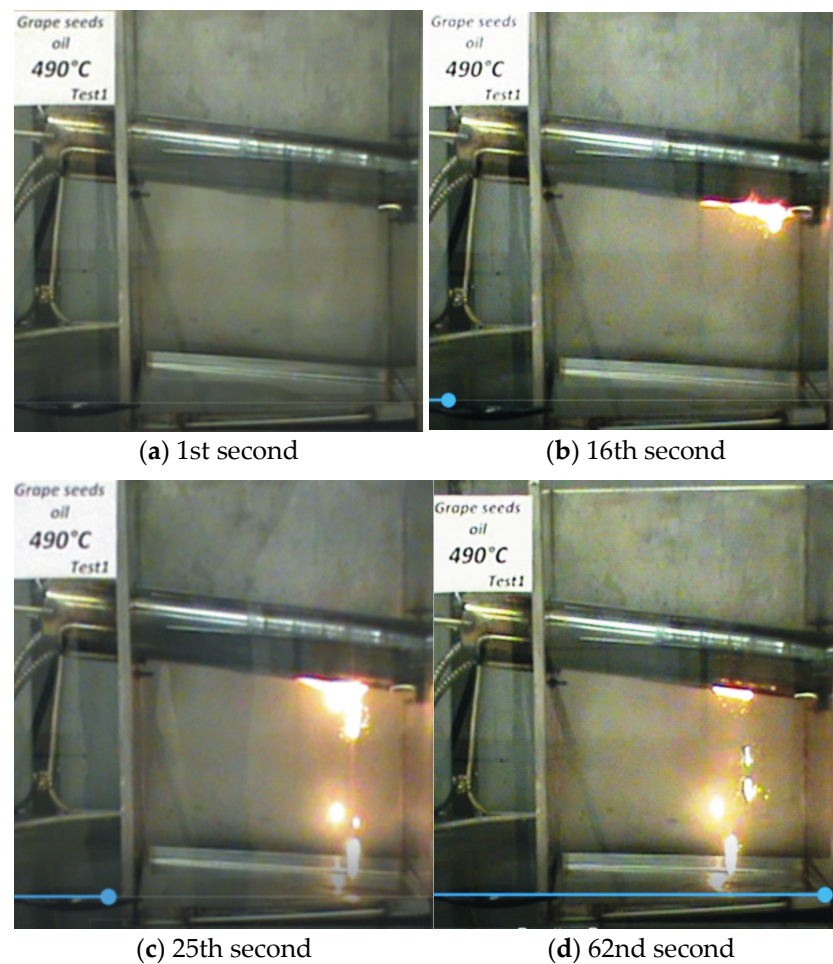


Figure 11. Grape seed oil tested at 490 °C.

The next test was performed at 485 °C temperature (in the middle of the 480–490 °C range). In this case, values close to those recorded at the 490 °C temperature were obtained.

The grape seed oil ignited after 19 s (Figure 12b), maintaining a pronounced burn throughout the test period (Figure 12c).

The experimental data show that, after the completion of the test, the oil still burns for 9 s on the surface of the cylinder (Figure 12d).

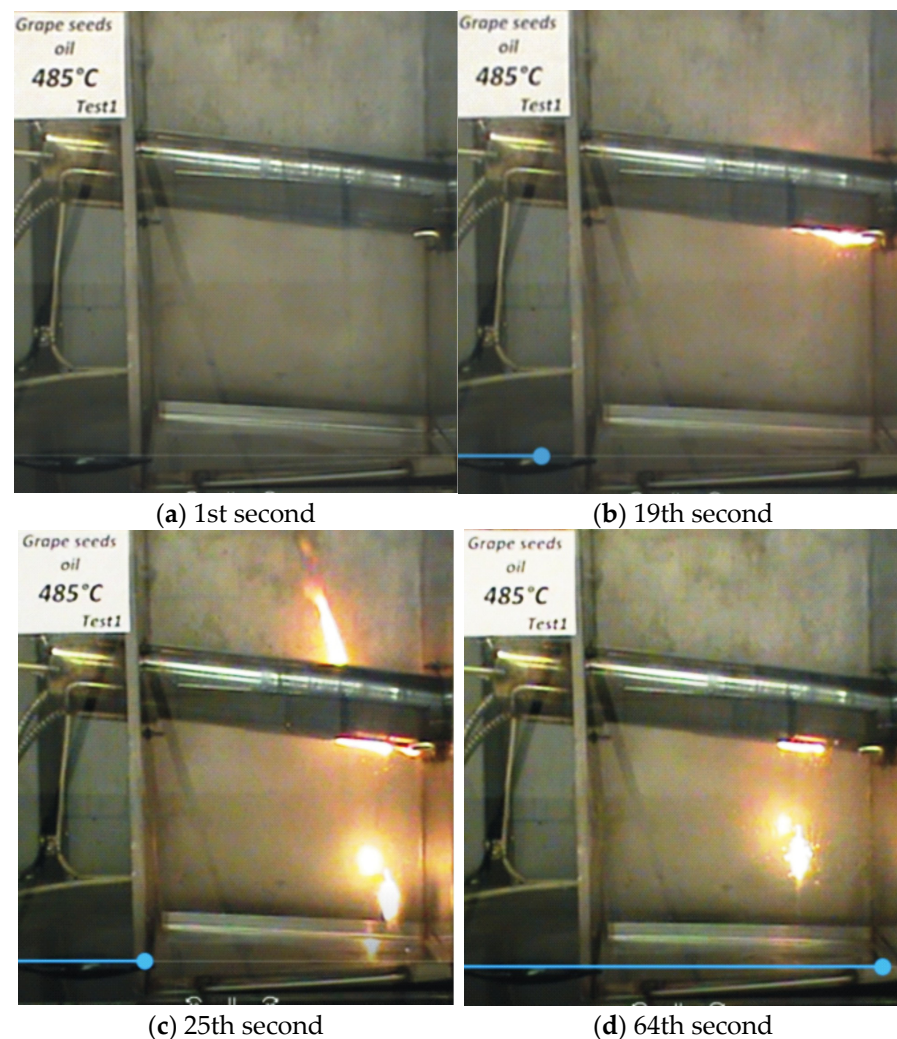


Figure 12. Grape seed oil tested at 485 °C.

Later, the second test was performed at 480 °C, a temperature at which, in the first test, the grape seed oil did not ignite (Figures 9 and 10). This time, as can be seen (Figure 13a), the oil ignites 15 s after starting the test.

After 41 s from the start of the test, the grape seed oil is no longer burning on the surface of the cylinder (Figure 13c) and continues not to burn until the test is stopped (the oil burned on the surface of the cylinder for 25 s).

Figure 13d shows the moment of the end of the test (second 55).

To further analyze the behavior of the oil, the test temperature was lowered to 470 °C.

Following the tests performed, it was highlighted that the oil did not burn in three tests performed consecutively (Figures 14–16).

At this point, a temperature (470 °C) at which the oil does not burn was obtained in three consecutive tests and an immediately higher temperature (480 °C) at which the oil ignites. To refine the temperature range, the test temperature is increased to 475 °C (the middle of the 470–480 °C range). At this temperature, the grape seed oil ignited very late, more precisely in the 54th second (Figure 17b), the oil continuing to burn on the surface of the cylinder for 8 s after the dripping stopped (Figure 17d).

In the conditions where grape seed oil is dripped onto a heated cylindrical surface, it can be highlighted that the lowest temperature at which the oil ignites is 475 °C and the highest temperature at which the oil does not ignite is 470 °C (the test at 470 °C was repeated 3 times).

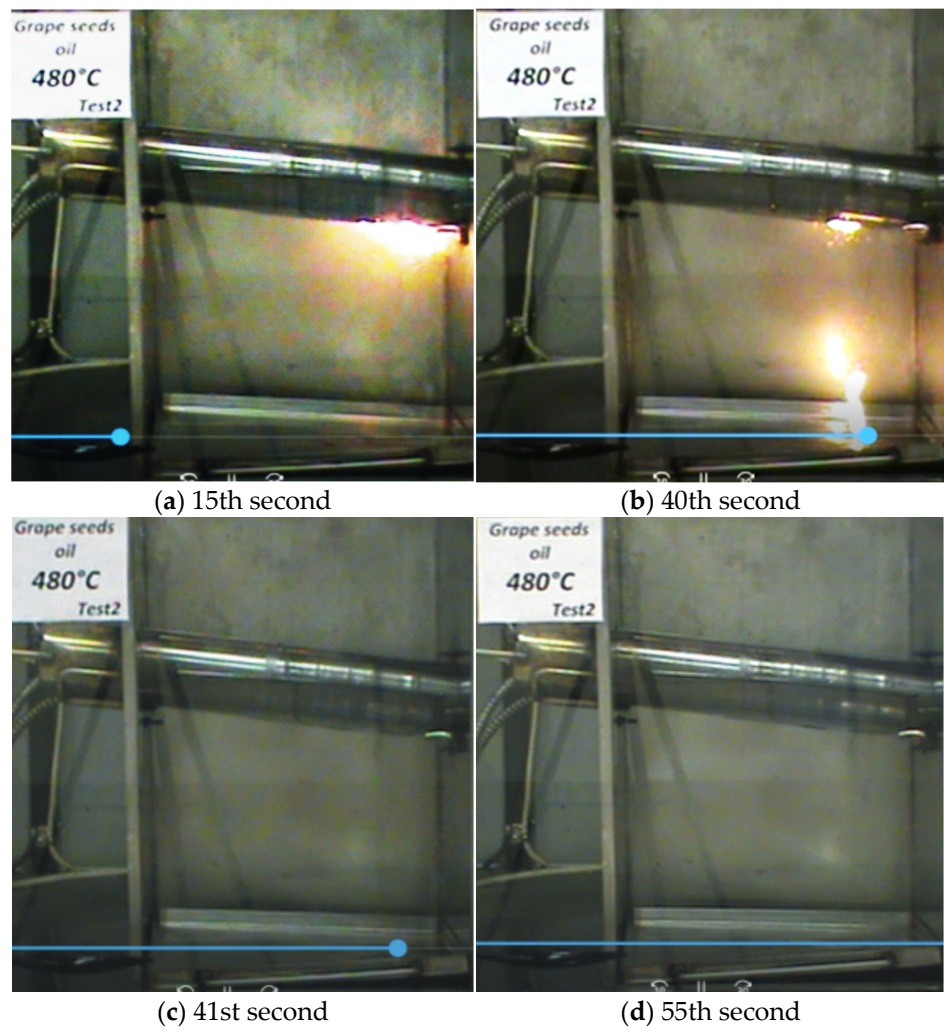


Figure 13. Grape seed oil tested at 480 °C (Test 2).

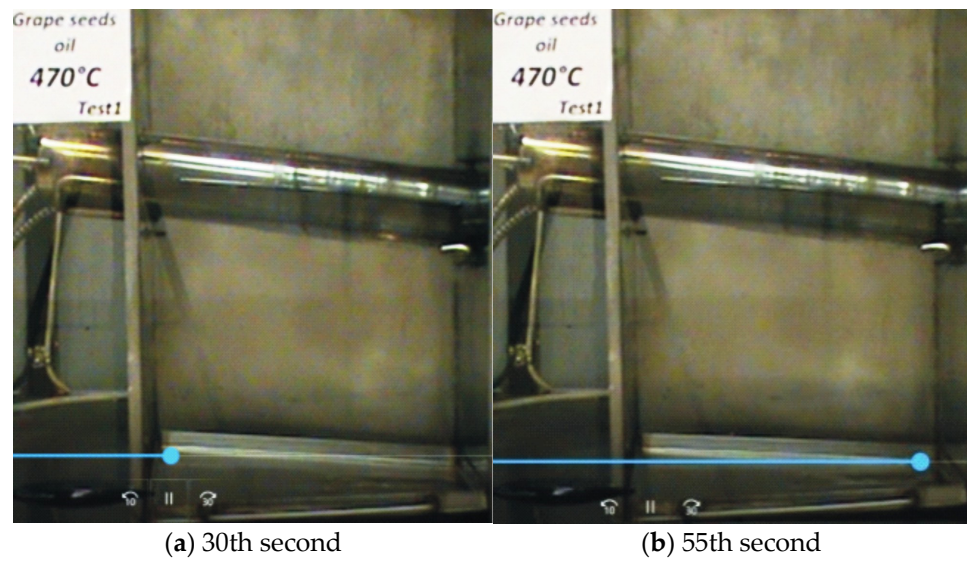


Figure 14. Grape seed oil tested at 470 °C (Test 1).

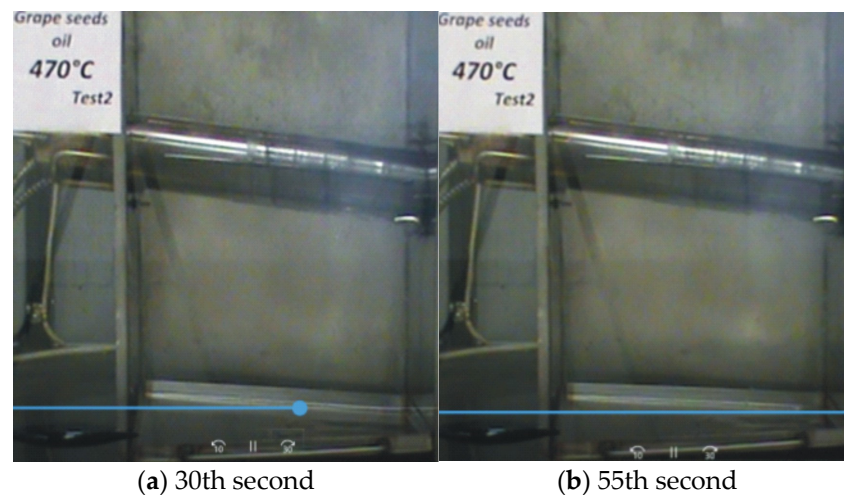


Figure 15. Grape seed oil tested at 470 °C (Test 2).

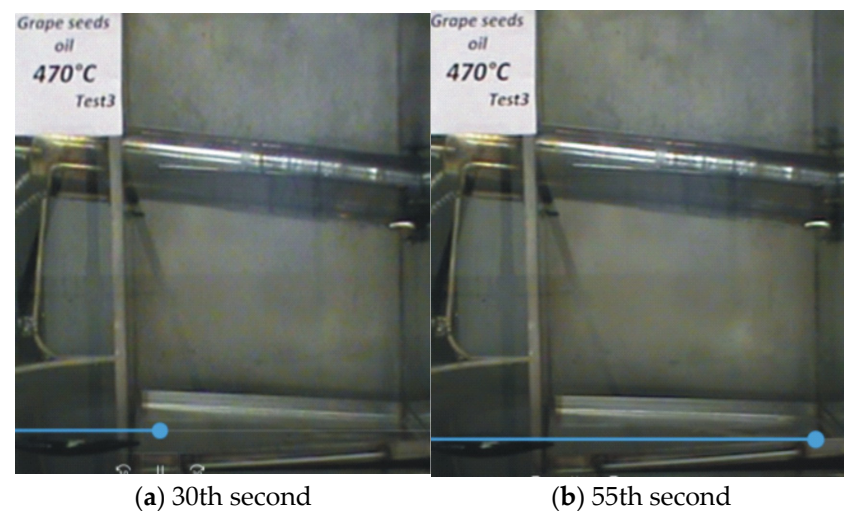


Figure 16. Grape seed oil tested at 470 °C (Test 3).

The experimental results obtained in this study are in agreement with those obtained by Georgescu et al. [54], which show that vegetable oils, such as olive oil, soybean oil, corn oil, and rapeseed oil, have ignition temperatures on hot surfaces between 495 and 510 °C. On the other hand, according to these authors, the respective values of ignition temperatures proved to be superior to mineral oils. Thus, for the hydraulic oil tested as a reference fluid, the ignition temperature on a hot surface is 425 °C. Also, the tests carried out in our study show that GSO presents a value of the ignition temperature on a hot surface superior to the limits of the tests realized by Yuan [74] on some mineral oils. According to his studies, when a hot surface was used as the heat source, in the case of the hydraulic oils (AW 32, AW 46, AW 68, compressor oil, etc.), the minimum hot surface ignition temperatures ranged from 350 to 440 °C. In addition, the results of our studies support the research conducted by Javier et al. [40]. In this case, the authors concluded that GSO and GSO enhanced with nanoparticles have the potential for use as good environmentally friendly lubricants. Moreover, recent studies have highlighted the possibility of increasing the resistance of GSO to oxidation processes in a similar way to other natural oils by formulating multilayer emulsions [75] and nanoemulsions [76]. At the same time, in their studies, Li et al. [77] show that the quality of grapes can be substantially improved when they are treated with biological control agents based on natural substances. Thus, it can be concluded that by reducing the degradation of grapes, the quality of the obtained oil increases substantially.

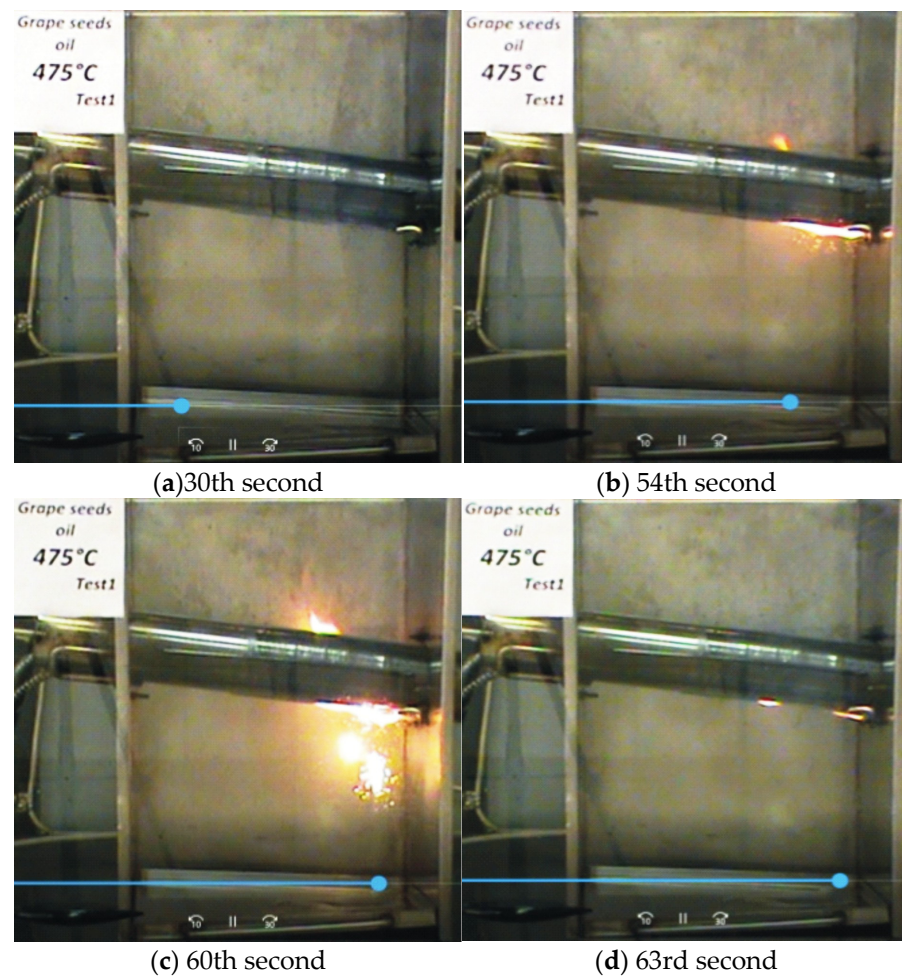


Figure 17. Grape seed oil tested at 475 °C.

In our study, the oil samples with representative results collected in the collecting tray of the oil testing facility for flammability were analyzed against the reference oil used by the spectrophotometer, determining the transmittance evolutions. Thus, Figure 18 shows the evolution of transmittances for the following samples: the untested oil, the oil that did not ignite at 470 °C temperature (test 1), the oil that ignited at 475 °C temperature (test 1), the oil which ignited at 480 °C (test 2) and for the oil sample that ignited at 500 °C (test 1).

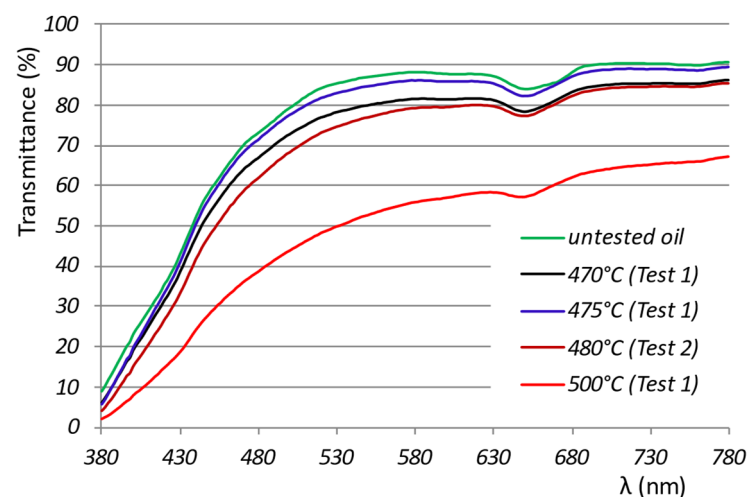


Figure 18. Spectral transmittance curves of grape seed oils tested at flammability.

From the data presented (Figure 18), a decrease in the transmittance values can be observed in the case of all oil samples, compared to the reference sample (untested), over the entire range of analyzed wavelengths.

It is highlighted that a sharp decrease in the transmittance value is recorded for the oil sample that ignited at 500 °C temperature.

The tendency of color variation compared to untested oil (expressed both by the trichromatic coordinates x and y , shown in Table 8, and by using the chromatic parameters L^* , a^* , b^* , C_{ab}^* and h_{ab} , as shown in Table 9) was somewhat similar to that of the oil that did not ignite at the temperature of 470 °C (Test 1), and that of the oil that ignited at the temperature of 475 °C (Test 1). In both cases, as shown in Table 9, the tested oils were darker than the reference oil, with the L^* parameter having lower values (89.56 and 91.53, respectively). The brightness decreased by 3.19% in the case of the test performed at 470 °C, and by 1.06% in the case of the test performed at 475 °C. This variation was a result of chemical transformations that occurred during the testing process.

Table 8. Experimental results for grape seed oils tested at flammability, obtained according to the color system (x, y) CIE 1964 (CIEXYZ)/Illuminant D65/10°.

Grape Seed Oils		Untested Oil	470 °C (Test 1) (It Does Not Ignite)	475 °C (Test 1) (It Ignites)
Trichromatic components	X	79.002 ± 0.702	72.930 ± 0.648	76.997 ± 0.684
	Y	81.857 ± 0.728	75.355 ± 0.670	79.661 ± 0.708
	Z	71.418 ± 0.635	64.971 ± 0.577	69.259 ± 0.616
Trichromatic coordinates	x	0.340 ± 0.003	0.342 ± 0.003	0.341 ± 0.003
	y	0.352 ± 0.003	0.353 ± 0.003	0.353 ± 0.003
	z	0.307 ± 0.003	0.305 ± 0.003	0.307 ± 0.003

Table 9. Chromatic coordinates (CIELAB system) for grape seed oils tested at flammability/Illuminant D65/10°.

Grape Seed Oils		Untested Oil	470 °C (Test 1) (It Does Not Ignite)	475 °C (Test 1) (It Ignites)
Chromatic coordinates	L^*	92.51 ± 0.557	89.56 ± 0.545	91.53 ± 0.563
	a^*	2.78 ± 0.017	3.13 ± 0.019	2.99 ± 0.018
	b^*	12.47 ± 0.076	12.80 ± 0.078	12.56 ± 0.076
	a^*/b^*	0.22	0.24	0.24
	$(a^*/b^*)^2$	0.05	0.06	0.057
	C_{ab}^*	12.78 ± 0.078	13.18 ± 0.080	12.91 ± 0.079
	h_{ab}	77.43 ± 0.471	76.26 ± 0.464	76.63 ± 0.466

The degree of redness (a^*) contributes considerably to these changes in oil color during the flammability tests (increase by 12.59% and 7.55%, respectively). The degree of yellowness, evaluated by the term b^* , shows a less significant contribution (2.65% increase when the oil did not ignite, and 0.72% in the case of tests performed at the last temperature at which the oil ignited, 475 °C).

This shows that the color parameter b^* contributes to small changes in the oil color during the tests, but the determined role belongs to the parameter a^* . In addition, the same tendency of slight increase is also noted in the case of the parameter C_{ab}^* , which, in the case of the test carried out at 470 °C, when the GSO did not ignite, had an increase of over 3%, and in the case of the test at 475 °C, when the oil ignited, the chroma increased by only 1.02%. This behavior of GSO indicates that, probably due to the large number of volatile organic compounds that are formed [78] and that degrade during the thermal process, the intensity of the color of the oil varies less (ΔC_{ab}^* , Table 10) as the temperature approaches the lowest value at which the oil ignites. The same can be seen in the case of variations in the other chromatic parameters. These results are also supported by the hue

angle, h_{ab} , which decreases by 1.51% at 470 °C and 1.03% at 475 °C, noting the extremely small variation of this parameter (Δh_{ab} , Table 10) in the case of the lowest temperature at which GSO ignited.

Table 10. Experimental values of color differences for grape seed oils tested at flammability.

Grape Seed Oils	ΔL^*	Δa^*	Δb^*	ΔC_{ab}^*	Δh_{ab}	ΔE_{ab}^*
470 °C (Test 1) (it does not ignite)	−2.95	0.35	0.33	0.40	−1.17	2.9
475 °C (Test 1) (it ignites)	−0.98	0.21	0.09	0.13	−0.80	1.01

Finally, according to the data presented in Table 10, the color differences obtained in this study at the lowest temperature at which GSO ignited (475 °C) show that this oil can be recommended for use as a lubricant, especially when the oxidative stability can be considerably improved, for example, by adding extracts also obtained from grape seeds that have been shown to have a good antioxidant character [78]. Moreover, the Pearson coefficients determined for the chromatic parameters and the lowest temperature at which this oil ignites showed that there are significant correlations, showing a strong positive correlation in the case of the degree of red, a^* , and a strong negative correlation in the case of the hue angle, h_{ab} (Table 11).

Table 11. Pearson linear correlation coefficients between the values of the chromatic coordinates (systems: CIELAB) in the case of grape seed oils tested at flammability.

	L^*	a^*	b^*	C_{ab}^*	h_{ab}	FT
L^*	1.000					
a^*	−0.998	1.000				
b^*	−0.992	0.991	1.000			
C_{ab}^*	−0.993	0.992	0.998	1.000		
h_{ab}	0.207	−0.213	−0.083	−0.089	1.000	
FT	−0.749	0.914	0.704	0.743	−0.948	1.000

FT—flammability temperature, °C.

The results outlined in Tables 7 and 11 show that using statistical analysis methods to evaluate the color variation during GSO heating can be effective.

4. Conclusions

The modification of the GSO color along with its degree of oxidation represents an important signal to identify timely the quality of the oil used as an alternative to mineral and synthetic lubricants. Therefore, an important indicator for determining the stability and oil degradation due to oxidation is its color intensity. The results of this study provide a better understanding of the stability of the chromatic intensities upon oxidation of GSO. Therefore, the direct determination of trichromatic parameters of oxidized oil samples could be a much faster alternative to measuring the GSO performance. Also, it could be a much easier alternative to measure the degradation degree and the lifetime of this oil.

By testing the behavior of GSO under forced oxidation at temperatures of 100 °C and 120 °C, it has been shown that this oil exhibits a low color variation associated with the degree and temperature of oxidation, which recommends it for successful use in lubrication processes.

The analysis of the chromatic parameters revealed that the brightness of GSO subjected to oxidation denotes its good stability throughout the experiments. The smallest color difference in the degree of red was obtained in the case of the oil oxidized for 4 h at 100 °C temperature, while the highest value of this parameter was recorded in the case of the oil oxidized for 8 h at the same temperature. Increasing the oxidation temperature to 120 °C

induces small, relatively similar, although contradictory, variations in the color differences regarding the degrees of red and yellow of the tested oils. However, our results show that the largest variation in GSO color (expressed by ΔE_{ab}^*) occurs after 8 h of oxidation.

Testing the oil for flammability on a cylindrical surface revealed that the lowest temperature at which grape seed oil ignites is 475 °C, and the highest temperature at which the oil does not ignite after three consecutive tests is 470 °C.

Based on the obtained results, it can be concluded that this type of oil can be used safely in machines whose surfaces can reach temperatures of a maximum of 470 °C during their operation.

In this context, if, in the case of the highest temperature at which the oil does not ignite after three consecutive tests, the total variation in the color of GSO is perceptible with the naked eye ($\Delta E > 2$), in the case of the lowest temperature at which the oil ignites, the color variation cannot be detected with the naked eye ($\Delta E < 2$).

The results obtained through the research carried out in this study on the chromatic parameters demonstrate that GSO can be considered resistant to oxidative processes, although, due to its content of unsaturated fatty acids [62,79,80], it is still prone to such processes [18,62].

As a result, considering the antioxidant-rich composition [25,81,82] and the reduced color variation during oxidative processes that occur at the lowest temperature at which this oil ignites, we believe that GSO can be recommended for use as an eco-friendly lubricant. In light of the results of the present work, GSO offers a renewable, sustainable, and low-cost alternative to mineral and synthetic lubricants.

The knowledge about the performance of GSO as a lubricant still requires greater understanding of anti-wear properties. This research is part of a larger study plan, which includes research of much longer duration, e.g., that on anti-wear properties. Obviously, this research on the performance of this type of oil should be deepened and improved. Therefore, we realize that these preliminary tests on GSO open new frontiers for researchers. This attempt is to establish whether GSO can be regarded as an efficient eco-friendly lubricant.

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