

## Relationship between the fat and oil composition and their initial oxidation rate during storage

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**Abstract:** Until now, the relationship between the fat and oil composition and their oxidation stability has been studied only at elevated temperatures (typically above 100 °C). Therefore, the initial oxidation rates of 19 edible fats and oils were determined as an increase in the peroxide value during storage in the dark at 35 °C with free access to air (oxygen). The initial oxidation rates of fats and oils were compared with parameters characterising these fats and oils (peroxide value, acid value, fatty acid composition, antioxidant capacity, and tocopherol content). Using a simple correlation analysis, the initial oxidation rate correlated the most strongly with the peroxide value of the analysed fats and oils ( $P < 0.01$ ). A highly reliable model ( $P < 0.0001$ ) was obtained by multivariate statistical analysis. According to this model, the initial oxidation rate is affected mainly by the peroxide value and then by total *trans* fatty acid content, and antioxidant capacity.

**Keywords:** tocopherols; antioxidant capacity; fatty acids; peroxide value; acid value

The oxidative stability is an important parameter in the quality assessment of individual fats and oils, particularly when determining their usefulness in food production (Choe & Min 2006). However, methods used to determine the oxidative stability of fats and oils are relatively time consuming, especially under real storage conditions. Therefore, an association between the oxidative stability of fats and oils and their composition is documented in many studies (Méndez et al. 1996; Savage et al. 1999; Arranz et al. 2008; Dabbou et al. 2010; Yun & Suhr 2012; Gao & Birch et al. 2016; Sabolová et al. 2017; Redondo-Cuevas et al. 2018).

The majority of these studies have been performed using a Rancimat apparatus (Méndez et al. 1996; Savage et al. 1999; Arranz et al. 2008; Dabbou et al. 2010; Yun & Suhr 2012; Redondo-Cuevas et al. 2018),

an Oxipres apparatus (Sabolová et al. 2017), by thermogravimetric analysis (Gao & Birch et al. 2016), or a combination thereof, which are accelerated methods used for determining the oxidative stability of fats and oils at elevated temperatures (typically above 100 °C). Oxidative stability of fats and oils depends on a number of internal factors including triacylglycerol and fatty acid composition; composition of minor components, for example antioxidants such as tocopherols, carotenoids and phenolic compounds; an occurrence of prooxidants, etc. Moreover, oxidative stability is affected by various external factors, such as exposure to atmospheric oxygen, light, moisture and temperature (Choe & Min 2006). As the temperature increases, the oxidative stability of fats and oils significantly decreases, and the extent

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Table 1. Description of fats and oils

Sample abbreviation	Sample description
LO	Linseed oil, unrefined
SoO	Soybean oil, unrefined
CO	Corn oil, refined
WO	Walnut oil, unrefined
RO1	Rapeseed oil, unrefined
PO	Palm oil, unrefined
HOSO	High oleic sunflower oil, refined
OpO	Olive pomace oil, i.e. commercial mixture of refined olive pomace oil and virgin olive oil
SO1	Sunflower oil, refined
PL	Pork lard
RO2	Rapeseed oil, refined
EVOO	Extra virgin olive oil
OO	Olive oil, i.e. commercial mixture of refined olive oil and virgin olive oil
PnO	Peanut oil, refined
GO	Grapeseed oil, unrefined
CF	Coconut fat, unrefined
RiO	Rice oil, unrefined
SeO	Sesame oil, unrefined
SO2	Sunflower oil, refined

of this temperature effect is different for various fats and oils (Sabolová et al. 2017).

Therefore, the aim of this study was to provide the comparison between the chemical composition of fats and oils and their oxidative stability during storage at only slightly elevated temperature, which is more representative of real storage conditions. The oxidative stability of edible fats and oils was indicated by the initial oxidation rate as measured by the initial increase in peroxide value because peroxide value limits (for olive oil Commission Regulation No. 2568/91 and for lard Commission Regulation No. 853/2004) are achieved significantly earlier in the process than at the end of an induction period (Yanishlieva-Maslarova 2001).

## MATERIAL AND METHODS

**Fats and oils.** Used fat and oil samples (see Table 1 for their descriptions) were purchased at local markets. The fatty acid compositions of these fats and oils are presented in Table 2, and other determined chemical parameters are given in Table 3.

**Determination of initial oxidation rate.** The oxidative stability of edible fats and oils was determined

Table 2. Fatty acid composition of fats and oils

Sample	Fatty acid composition (%)								Total <i>trans</i> isomer content	Iodine value (g I <sub>2</sub> 100 g <sup>-1</sup> )
	Palmitic	Stearic	Other saturated	Oleic	Other mono-unsaturated*	Linoleic	α-Linolenic	Other poly-unsaturated†		
LO	6.03	4.00	0.49	15.73	1.00	44.01	28.57	0.17	0.19	165.7
SoO	9.85	5.21	1.11	22.63	1.58	52.81	6.75	0.06	0.10	130.0
CO	11.54	1.88	0.88	30.68	1.10	52.77	0.92	0.23	0.25	121.6
WO	6.96	2.71	0.27	13.68	1.69	61.01	12.50	1.18	1.21	154.2
RO1	4.46	1.65	1.29	61.73	4.59	18.62	7.59	0.07	0.11	109.2
PO	37.16	3.91	2.04	42.63	1.33	12.51	0.37	0.05	0.07	60.5
HOSO	4.61	3.03	1.74	72.14	1.26	16.91	0.12	0.19	0.24	93.0
OpO	12.22	2.95	0.95	67.81	3.93	11.33	0.73	0.08	0.38	83.3
SO1	6.21	3.25	1.55	33.67	1.04	53.73	0.08	0.47	0.51	123.9
PL	24.63	13.38	2.35	40.45	6.47	11.95	0.68	0.09	0.35	63.0
RO2	4.55	1.58	1.28	60.57	5.29	18.86	7.47	0.40	0.49	109.6
EVOO	9.49	3.38	0.82	74.53	2.79	8.21	0.75	0.03	0.07	82.6
OO	12.88	2.74	0.76	69.78	4.34	8.76	0.70	0.04	0.13	80.8
PnO	5.05	1.92	6.39	72.94	3.28	10.07	0.25	0.11	0.11	83.6
GO	7.03	4.79	0.58	21.51	1.14	64.20	0.34	0.42	0.42	132.2
CF	8.24	3.23	81.83	5.51	0.08	1.07	0.03	0.01	0.01	6.8
RiO	15.13	2.38	2.13	43.58	1.62	33.78	0.69	0.69	0.75	100.3
SeO	9.07	5.53	0.89	38.68	1.06	44.38	0.31	0.08	0.08	111.9
SO2	6.22	3.55	1.48	28.92	0.98	58.70	0.05	0.11	0.12	127.7

†Including *trans* isomers; for abbreviations see Table 1

Table 3. Chemical characterisation of fats and oils

Sample	PV (mEq O <sub>2</sub> kg <sup>-1</sup> )	AV (mg KOH g <sup>-1</sup> )	AOC (mg a-T kg <sup>-1</sup> )	α-T (mg kg <sup>-1</sup> )	β- and γ-T (mg kg <sup>-1</sup> )	δ-T (mg kg <sup>-1</sup> )	α-T3 (mg kg <sup>-1</sup> )	β- and γ-T3 (mg kg <sup>-1</sup> )	δ-T3 (mg kg <sup>-1</sup> )
LO	1.0 ± 0.0	2.15 ± 0.02	729 ± 18	20.1 ± 2.1	488.9 ± 0.2	7.1 ± 1.1	ND	<3	< 3
SoO	6.3 ± 0.3	1.18 ± 0.02	1 174 ± 12	168.7 ± 3.7	779.2 ± 8.9	254.9 ± 3.6	ND	ND	ND
CO	4.4 ± 0.1	0.20 ± 0.00	1 144 ± 37	183.7 ± 0.5	673.5 ± 7.2	29.5 ± 4.2	11.0 ± 1.1	9.6 ± 0.3	ND
WO	7.0 ± 0.0	0.75 ± 0.02	533 ± 31	39.5 ± 0.1	285.5 ± 11.0	37.4 ± 2.5	ND	ND	ND
RO1	9.6 ± 0.1	1.07 ± 0.04	961 ± 12	271.2 ± 8.9	456.0 ± 6.7	12.6 ± 2.0	ND	ND	ND
PO	2.6 ± 0.1	0.69 ± 0.03	811 ± 24	118.0 ± 7.1	81.5 ± 6.8	60.2 ± 2.7	205.2 ± 2.5	231.6 ± 2.9	53.7 ± 3.5
HOSO	3.4 ± 0.0	0.11 ± 0.00	628 ± 15	576.9 ± 11.9	25.6 ± 0.2	18.7 ± 0.2	ND	ND	ND
OpO	2.3 ± 0.0	0.50 ± 0.02	1 358 ± 74	460.6 ± 15.6	17.3 ± 6.1	5.0 ± 1.3	ND	ND	ND
SO1	3.9 ± 0.0	0.31 ± 0.31	837 ± 11	718.0 ± 12.6	34.9 ± 0.5	29.0 ± 2.3	ND	ND	ND
PL	3.2 ± 0.0	1.18 ± 0.05	45 ± 0	7.1 ± 0.7	ND	ND	ND	ND	ND
RO2	1.2 ± 0.0	0.30 ± 0.01	977 ± 44	289.9 ± 3.8	341.9 ± 4.0	7.8 ± 0.2	ND	ND	ND
EVOO	10.1 ± 0.0	0.98 ± 0.01	1 079 ± 18	284.2 ± 7.8	14.4 ± 3.3	< 3	ND	ND	ND
OO	11.4 ± 0.0	0.52 ± 0.01	302 ± 28	154.2 ± 2.8	10.1 ± 0.6	< 3	ND	ND	ND
PnO	0.5 ± 0.1	0.05 ± 0.00	324 ± 17	149.2 ± 0.1	127.9 ± 0.5	ND	ND	ND	ND
GO	4.1 ± 0.2	0.18 ± 0.02	569 ± 33	122.8 ± 0.8	20.4 ± 1.1	ND	93.6 ± 2.5	83.0 ± 0.7	ND
CF	0.1 ± 0.1	0.24 ± 0.00	ND	ND	ND	ND	5.5 ± 0.0	< 3.0	ND
RiO	4.0 ± 0.1	1.04 ± 0.01	2 145 ± 22	147.9 ± 6.3	157.3 ± 2.4	2.7 ± 0.4	43.1 ± 2.8	183.5 ± 6.0	17.4 ± 6.2
SeO	1.5 ± 0.1	1.18 ± 0.02	594 ± 15	ND	655.1 ± 0.6	ND	ND	< 3.0	ND
SO2	2.7 ± 0.1	0.05 ± 0.01	831 ± 38	671.6 ± 7.9	ND	28.0 ± 1.4	< 3.0	< 3.0	ND

Results are expressed as the mean ± standard deviation ( $n = 3$ ); AOC – antioxidant capacity; AV – acid value; PV – peroxide value; α-T – α-tocopherol; β- and γ-T – β- and γ-tocopherol; δ-T – δ-tocopherol; α-T3 – α-tocotrienol; β- and γ-T3 – β- and γ-tocotrienol; δ-T3 – δ-tocotrienol; ND – not detected (the limit of detection was 1.0 mg kg<sup>-1</sup> for tocopherols and 4.0 mg kg<sup>-1</sup> for antioxidant capacity); for other abbreviations see Table 1

as the rate of the peroxide value increase at 35 °C in the dark with free access to air (oxygen) and expressed in mEq O<sub>2</sub> kg<sup>-1</sup> day<sup>-1</sup>. For this purpose, a large number of beakers (each of 100 mL in volume and with an internal diameter of 4.7 cm) were prepared for each fat and oil studied. The beakers were placed in a thermostatic drying oven, with a maximum temperature fluctuation of ± 0.1 °C. The increase in the peroxide value over 14 days was monitored in three replicates for all the analysed fats and oils. The entire beaker was always removed from the thermostatic drying oven for sampling. Thus, the oil volume in the beakers was constant throughout the heating.

**Peroxide value and acid value.** The peroxide value and acid value were determined using the appropriate IUPAC titrimetric methods (International Union of Pure and Applied Chemistry, 1987; 2.501).

**Fatty acid composition.** Fatty acids were determined by an Agilent Technologies 6890N gas chromatograph equipped with a flame ionisation detector (Agilent Technologies, Palo Alto, CA, USA) after derivatisation to the corresponding methyl esters. The GC analysis was performed under the conditions described

by Sabolová et al. (2017). A Supelco SP 2560 capillary column, 100 × 0.25 mm i.d., 0.2 µm thickness (Supelco, Bellefonte, PA, USA) was used for the chromatographic separation.

**Iodine value.** The official AOCS (American Oil Chemistry Society, 1995; Cd 1c-85) method was used for the calculation of the iodine value from the fatty acid composition.

**Antioxidant capacity.** Antioxidant capacity was determined spectrophotometrically, based on the ability of the analysed samples to scavenge free 2,2-diphenyl-1-picrylhydrazyl (DPPH) radicals (Sigma Aldrich, St. Louis, MO, USA), according to the method used by Espín et al. (2000) with modifications described by Sabolová et al. (2017). A Cary 60 spectrophotometer (Varian, Palo Alto, CA, USA) was used to measure the absorbance at 518 nm. The antioxidant capacity was quantified using the calibration curve of α-tocopherol (0 to 500 µg) and expressed as mg α-tocopherol equivalent per kg sample.

**Tocochromanol content.** The oil samples and pork lard were dissolved in acetone (approximately 0.1 g mL<sup>-1</sup>). The determination of tocochromanol content was per-

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formed using reverse-phase HPLC with amperometric detection according to the method described by Sabolová et al. (2017). The analytical HPLC system consisted of a non-steel high-pressure pump (LCP 4020.31; Ecom, Prague, Czech Republic), a manual sample injector (Rheodyne 7725i; Oak Harbor, WA, USA), a column heater (LCO 101; Ecom, Prague, Czech Republic), an amperometric detector (HP 1049A series, Agilent Technologies, Santa Clara, CA, USA). A Hypersil ODS column, 4.6 × 200 mm, particle size 5 µm (Agilent Technologies) was used as a stationary phase. The individual tocochromanol contents were quantified using the corresponding calibration curves.

**Results processing.** Statistical analysis of the data was carried out using Microsoft® Excel 2010 software (Microsoft Office software package, Microsoft Corporation, Redmond, WA, USA) by simple correlation and regression analysis, and also by multivariate linear correlation and regression analysis.

## RESULTS AND DISCUSSION

The initial oxidation rate of the analysed fats and oils is summarised in Table 4. According to the initial oxidation rate, the tested fats and oils can be divided into

Table 4. Initial oxidation rate

Sample	Initial oxidation rate (mEq O <sub>2</sub> kg <sup>-1</sup> day <sup>-1</sup> )
LO	0.257 ± 0.011
SoO	0.136 ± 0.030
CO	0.039 ± 0.002
WO	1.641 ± 0.007
RO1	1.027 ± 0.018
PO	0.277 ± 0.003
HOSO	0.217 ± 0.003
OpO	0.068 ± 0.004
SO1	0.255 ± 0.005
PL	0.084 ± 0.019
RO2	0.110 ± 0.007
EVOO	0.544 ± 0.040
OO	0.413 ± 0.020
PnO	0.058 ± 0.006
GO	0.045 ± 0.006
CF	0.024 ± 0.003
RiO	0.045 ± 0.005
SeO	0.029 ± 0.007
SO2	0.095 ± 0.003

For abbreviations see Table 1

Table 5. Correlation coefficients for the association between the initial oxidation rate and selected parameters that describe the composition of fats and oils

Parameter	Pearson correlation coefficient
Peroxide value	0.582*
Acid value	0.193
Antioxidant capacity	−0.073
α-Tocopherol	−0.092
β- + γ-Tocopherol	0.071
δ-Tocopherol	0.013
Tocopherols	−0.003
Tocopherols + tocotrienols	−0.051
Saturated fatty acids	−0.242
Oleic acid	−0.032
Linoleic acid	0.123
α-Linolenic acid	0.368
Polyunsaturated fatty acid†	0.219
Oleic/linoleic ratio	−0.024
Polyunsaturated and saturated fatty acid ratio	0.395
Total <i>trans</i> isomer content	0.494**
Iodine value	0.309

†Including *trans* isomers; \**P* < 0.01; \*\**P* < 0.05

four groups. The relatively high initial oxidation rate was observed for walnut oil and unrefined rapeseed oil followed by the second group of oils including olive oil and extra virgin olive oil. A lower initial oxidation rate was found in the third group of oils: linseed, soybean, palm, refined rapeseed oil, and undefined, refined, and high oleic sunflower oil. Pomace olive oil, maize, peanut, rice and sesame oil, coconut fat, and pork lard represented the group with the lowest initial oxidation rate.

Using a simple correlation analysis, the oxidation rates were compared with parameters characterising the composition of fats and oils (see Table 5). As it is evident, the peroxide value has a dominant (and significant; *P* < 0.01) effect on the initial oxidation rate. This is consistent with the theory of lipid oxidation (hydroperoxides, because of their potential decomposition into free radicals, catalyse lipid oxidation) (Gordon 2001) and with the study of Sabolová et al. (2017), who compared the composition of fats and oils and their oxidative stability as determined by using an Oxipres apparatus at different temperatures. A similar study (Dabbou et al. 2010) did not show the effect of peroxide on the oxidation rate, probably because of the higher

temperature (120 °C) used to determine the oxidation rate. In these cases, the primary initiation caused by heat (higher temperature) exceeds the secondary initiation caused by the decomposition of hydroperoxides (Gordon 2001).

The oxidation rate was also significantly affected by the total content of *trans* fatty acids ( $P < 0.05$ ). This can be explained by the fact that during the formation of the *trans* isomers of polyunsaturated fatty acids, conjugated polyunsaturated fatty acids, which can have prooxidant activity (Chen et al. 1997; Haghighatdoost & Nobakht 2018), are also formed (Christy 2009).

As presented in Figure 1, a simple regression analysis does not provide a sufficiently reliable model for predicting oxidative stability, which was also suggested by other authors (Dabbou et al. 2010). Therefore, multivariate linear correlation and regression analysis was also applied to the obtained data. Based on the results of a simple correlation analysis and from the results of previous studies (Méndez et al. 1996; Apparicio et al. 1999; Arranz et al. 2008; Sabolová et al. 2017),  $\alpha$ -tocopherol content; total tocopherol content; antioxidant capacity; peroxide value; acid value; the proportion of oleic acid, linoleic acid,  $\alpha$ -linolenic acid, and polyunsaturated fatty acids; oleic and linoleic acid ratio; polyunsaturated and saturated fatty acid ratio, and total *trans* fatty acid content were chosen as parameters characterising the composition of the analysed fats and oils.

Using multivariate statistical methods, the following model was obtained ( $P < 0.0001$ ;  $R = 0.783$ ): initial oxidation rate =  $(0.0694 \pm 0.0171) \times$  peroxide value

+  $(0.7131 \pm 0.2138) \times$  total *trans* fatty acid content –  $(0.0003 \pm 0.0001) \times$  antioxidant capacity. This means that, in addition to the above-mentioned parameters [i.e. peroxide value ( $P < 0.001$ ) and total *trans* fatty acid content ( $P < 0.005$ )], the oxidation rates of fats and oils under the storage conditions used were also influenced by the antioxidant capacity ( $P < 0.05$ ). This is consistent with a number of analogous studies (Apparicio et al. 1999; Arranz et al. 2008; Sabolová et al. 2017; Redondo-Cuevas et al. 2018). However, there are significant discrepancies in the results of those studies, and in some cases, the effect of total antioxidant content on the oxidative stability of fats and oils was not demonstrated (Dabbou et al. 2010).

According to the obtained results, other than *trans* fatty acids, the fatty acid composition has no significant effect on the initial oxidation rate of fats and oils under the storage conditions used in this study. This is a surprising finding because, according to most of the previously published studies (Savage et al. 1999; Dabbou et al. 2010; Sabolová et al. 2017; Redondo-Cuevas et al. 2018), fatty acid composition affects the oxidative stability of fats and oils. However, in all of those studies, accelerated methods and temperatures above 100 °C were used for the determination of the oxidation rate of fats and oils. It can be assumed that under these conditions, the primary initiation of lipid oxidation (strongly related to the fatty acid composition) by heat predominates, while under storage at a slightly elevated temperature, the secondary initiation of lipid oxidation predominates, influenced mainly by the hydroperoxide content (Gordon 2001).

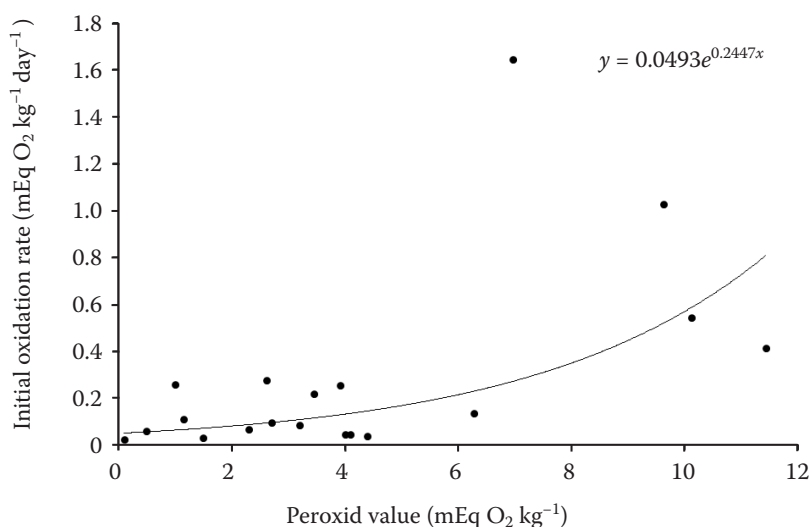


Figure 1. Possibility of predicting the initial oxidation rate of fats and oils from their initial peroxide value

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## CONCLUSION

The peroxide value is a characteristic that is the best indicator of the oxidative stability of fats and oils under real storage conditions. For a more accurate prediction of the oxidative stability, total *trans* fatty acid content (which characterises previous thermal damage to the fat or oil) and total antioxidant content must also be taken into account. This information can particularly help in the selection and quality control of fats and oils used in further food production.

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