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# Bioactive Compounds, Nutritional Quality and Oxidative Stability of Cold-Pressed Camelina (*Camelina sativa* L.) Oils

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**Abstract:** In this study, 29 cold-pressed camelina (*Camelina sativa* L.) oils, pressed from seeds grown in Poland and purchased directly from local producers, were analyzed. The degree of change in the tested oils' characteristic hydrolytic and oxidative lipid values was determined. Oxidative stability was determined using the Rancimat and PDSC methods. Fatty acid and phytosterol contents were determined by GC-FID, and tocopherols by HPLC. The analyzed oils were characterized by good, but variable, quality, and met the requirements specified for cold-pressed edible oils. Highly desirable fatty acid composition, low SFA content (about 6%), high α-linolenic acid content (34.7–37.1%), and optimal PUFA *n*-3 to PUFA *n*-6 ratio (1.79–2.17) were shown. The high nutritional value of camelina oils was confirmed on the basis of high contents of tocopherols (55.8–76.1 mg/100 g), phytosterols (331–442 mg/100 g), and carotenoids (103–198 mg of β-carotene/kg). The optimal nutritional quality indices were as follows: 0.05–0.07 for the atherogenicity index (AI), and 0.1–0.2 for the thrombogenicity index (TI). The significant impact of primary (PV) and total oxidation (TOTOX) of camelina oil on oxidative stability was evaluated using Rancimat and PDSC methods. Both methods were also confirmed to be appropriate for the assessment of the oxidative stability of camelina oils.

**Keywords:** *Camelina sativa* oil; carotenoids; chlorophyll pigments; fatty acid composition; oxidative stability; phytosterols; tocopherols

## 1. Introduction

Recently, there has been a significant increase in consumers' interest in low-processed food of high nutritional value. These products include cold-pressed oils, which are obtained not only from many typical oilseeds, such as sunflower, rape, and flax, but also from less known sources, e.g., borage, milk thistle, poppy, pumpkin, and camelina seed (*Camelina sativa* L.). Camelina is an oily plant belonging to the *Brassicaceae* family, also known as false flax, gold-of-pleasure, leindotter or camelina. *Camelina sativa* was already being cultivated around 3000 years ago. The history of camelina cultivation in Europe dates back to the Bronze Age, and the minimal climatic and soil requirements made the plant very popular in many countries—Russia, Ukraine, Croatia, the Czech Republic, Germany, Austria, Switzerland, Poland, England and Hungary. However, since the middle of the nineteenth century, camelina began to be replaced by other oilseeds [1–4]. In recent years, the interest in this plant has been growing again, though. Camelina is used as a rich source of essential n-3  $\alpha$ -linolenic acid in the production of health-promoting foods and supplements [2,5], as animal feed (for cows, chicken-broilers,

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turkey, sheep and fish) [5–7], and as feedstock for biodiesel manufacture and jet fuel [8,9], as the crop is low-input, and hence economical to produce [1,10–13].

Camelina is a short-season crop (the vegetation period of the plant lasts from 85 to 100 days). Therefore, it is ideal for growing in temperate climates. Soil requirements are also not demanding, so camelina can be cultivated on soils with a low nitrogen content—semiarid, marginal and saline soils. The crop is also resistant to pests; therefore, it does not require the use of a large amount of plant protection products [3,4,10,11,14].

Camelina sativa L. seeds contain about 33-47% fat [1,2,11,12]. The high nutritional value of camelina oil is primarily related to its fatty acid profile. It contains only about 10% saturated fatty acids (SFA), mainly palmitic (16:0), stearic (18:0) and eicosanoic (20:0) acids. Oleic (18:1) (14-16%) and eicosenoic acids (20:1) (12–15%) are predominant in monounsaturated fatty acids. Over half of the total fatty acid pool is constituted of polyunsaturated fatty acid (PUFA), of which the highest proportions are  $\alpha$ -linolenic acid (ALA) (18:3) (31–40%) and linoleic acid (LA) (18:2) (15–23%). Of particular importance is the high content of ALA, which is considered an *n*-3 essential fatty acid, and whose supply in the diet is very often insufficient.  $\alpha$ -Linolenic acid is very important in the prevention and treatment of cardiovascular diseases, hypertension, and in ensuring proper vision and the general functioning of the human organism [15-17]. In addition, Rahman et al. [18] showed that Camelina sativa L., upon consumption, may exert anti-obesity and antidiabetic effects, and may also inhibit human low-density lipoprotein (LDL) oxidation and DNA damage induced by peroxyl and hydroxyl radicals. The seed extracts of camelina showed significant antioxidant activity and effectively inhibited pancreatic lipase and  $\alpha$ -glucosidase activities. These enzymes in the digestive tract are associated with lipid and carbohydrate digestion, respectively, and control obesity and blood glucose level in the human body.

The nutritional benefits of camelina oil have been known and appreciated for many years, as demonstrated by its use in herbal medicine (for wounds, treatment of burns, stomach ulcers, and eye inflammations). The plant is often recommended as a dietary oil to supplement deficiencies of the n-3 acids family [1–4,19–21]. Cold-pressed camelina oil is also rich in non-glycerols—the content of tocopherols varies between 55.6–99.4 mg/100 g [3,4,18], and sterols from 360 to 590 mg/100 g [19,22].

Camelina oil is characterized by a very high nutritional value; therefore, the consumption of this oil can improve human health, especially in industrialized countries [1–5,18,20–22]. Nevertheless, the high nutritional value of this oil is limited by relatively low oxidative stability. Camelina oil was found to have higher oxidative stability than linseed, but lower stability than olive, sesame and rapeseed oils [20–23]. Additionally, it should be remembered that the technology used for obtaining cold-pressed camelina oil (mechanical cleaning processes only) means that the composition and quality of the oil depend highly on the quality of the raw material. Therefore, it is crucial to assess the safety of market camelina oils, and to monitor the quality parameters of these oils, such as the degree of hydrolysis, primary and secondary oxidation state or oxidative stability.

Therefore, this study aimed (i) to analyze the quality and quantify the bioactive compounds in camelina oils pressed from seeds grown in Poland and purchased directly from local producers; and (ii) to explore the impact of the analyzed parameters on the oxidative stability of camelina oils.

## 2. Materials and Methods

## 2.1. Materials

The research materials consisted of 29 cold-pressed camelina oils (CO): 10 oils from year 2016 (samples 1–10), 10 oils from year 2017 (samples 11–20) and 9 oils from year 2018 (samples 21–29), purchased directly from producers—small local manufactures. The oils were cold-pressed from Polish raw materials according to the declaration of producers. All oils were stored in brown, dark glass bottles. All chemical analyses were performed within 10 days of oil pressing, and during analysis, the

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samples of oils were stored under refridgerated conditions. All the determined values were recorded for two samples and analyzed in triplicate.

#### 2.2. Chemicals

All reagents and solvents used were of analytical grade and purchased from POCH S.A. (Gliwice, Poland).

#### 2.3. Determination Quality Parameters

The acid value (AV) was determined according to ISO 660:2009 [24], the results were expressed as mg KOH/g. Determination of the peroxide value (PV) was made by iodometric technique, applying the ISO 3960:2017 [25] and expressed in meq O<sub>2</sub>/kg. According to ISO 660:2009 [26], the content of secondary oxidation products, known as p-anisidine value (p-AnV), was determined. The overall rate of oxidation measured as the TOTOX indicator (TOTOX = 2PV + p-AnV) was also calculated. Determination of ultraviolet absorbance expressed as specific UV extinction  $E_{1cm}^{1\%}$  at  $\lambda_{max}$  232 and 286 nm was determined according to ISO 3656:2011 [27].

# 2.4. Determination of Fatty Acid Composition

Samples of tested oils were prepared for the GC-flame ionization detector (FID) analysis as fatty acid methyl esters according to the AOCS Official Method Ce 2–66 [28]. Determination of esterified fatty acids in camelina oils was performed according to the AOAC Official Method 996.06 [29] with modifications described by Symoniuk et al. [30]. Methyl esters were separated on a TRACE 1300 (Thermo Fisher Scientific, Inc., Wilmington, DE, USA) gas chromatograph equipped with a Restek BPX70 GC capillary column (60 m in length  $\times$  0.22 mm I.D., 0.25  $\mu$ m film thickness). The fatty acids were identified by comparison of their retention times with those of authentic standards (Restek, Bellefonte, PA, USA), and the results were reported as weight percentages of total fatty acids.

## 2.5. Calculated Oxidizability (COX) Value, and Nutritional Quality Index

The calculated oxidizability value (COX) of the cold-pressed oils was calculated by applying the formula proposed by Fatemi and Hammond [31]:

Equation (1)—calculated oxidizability value (COX)

$$COX = \frac{C18:1 + 10.3xC18:2 + 21.6xC18:3}{100}$$
 (1)

The atherogenic index (AI) (Equation (2)) and the thrombogenic index (TI) (Equation (3)) of the cold-pressed camelina oils were evaluated following, from Ulbricht and Southgate [32]:

Equation (2)—Atherogenicity index (AI)

$$AI = \frac{C12 : 0 + 4xC14 : 0 + C16 : 0}{\sum MUFA + \sum (\omega 3) + \sum (\omega 6)}$$
(2)

Equation (3)—Thrombogenicity index (TI)

$$TI = \frac{C14 : 0 + C16 : 0 + C18 : 0}{0.5xMUFA + 0.5x\sum(\omega 6) + 3x\sum(\omega 3) + (\omega 3/\omega 6)}$$
(3)

The ratio of hypocholesterolemic to hypercholesterolemic FA (HH) was calculated based on Santos, Bessa, and Santos [33].

Equation (4)—Ratio of hypocholesterolemic to hypercholesterolemic FA (HH)

$$HH = \frac{C18:1 + C18:2 + C18:3 + C18:4 + C20:4}{C14:0 + C16:0}$$
(4)

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## 2.6. Determination of Chlorophyll and Carotenoid Pigments Content

The content of chlorophyll pigments in oils was determined according to the AOCS Official Method Cc 13i-96 [34]. The results of the measurements are expressed in mg of pheophytin/kg. In accordance with BS 684 Section 2.20 [35], carotenoid content was determined, and is expressed as  $\beta$ -carotene in mg per kg of oil.

# 2.7. Tocochromanol Determination by NP-HPLC

Each oil (200 mg) was dissolved in n-hexane, made up to 10 ml, and transferred to vials for analysis. Tocopherols were determined using a Waters HPLC system (Waters, Milford, MA, USA) consisting of a fluorometric detector (Waters 474), a photodiode array detector (Waters 2998 PDA), an autosampler (Waters 2707), a column oven (Waters Jetstream 2 Plus), and a LiChrosorb Si 60 column (250  $\times$  4.6 mm, 5  $\mu$ m) from Merck (Darmstadt, Germany). To detect the fluorescence of the tocopherols and PC-8, the excitation wavelength was set to  $\lambda$  = 295 nm and the emission wavelength to  $\lambda$  = 330 nm [36,37].

# 2.8. Determination of Phytosterols

Phytosterol contents were determined by GC following the procedure described by AOCS Official Method Ch 6-91 [38]. Briefly, 50 mg of lipids were saponified with 1 M methanolic KOH for 18 h at room temperature; water was then added, and the unsaponifiables were extracted three times with hexane/methyl tert-butyl ether (1:1, v/v). Derivatives of the sterols that had been silylated by Sylon BTZ were separated on a HP 6890 series II Plus gas chromatograph (Hewlett Packard, Palo Alto, CA, USA) equipped with DB-35MS capillary column (25 m × 0.20 mm, 0.33  $\mu$ m; J&W Scientific, Folsom, CA, USA). The sample was injected in splitless mode. An internal standard, 5 $\alpha$ -cholestane, was used for sterol quantification. Phytosterols were identified by comparing the retention data with the standards.

# 2.9. Oxidative Stability by Rancimat Method

The oxidative stability of tested camelina oils was determined with 743 Rancimat apparatus from Metrohm, Switzerland, according to ISO 6886:2016 [39], utilizing a sample of 2.50 g  $\pm$  0.01 g, under a constant air flow (20 L/h), in temperature 100 °C. The induction times (in hours) were printed automatically by the apparatus software with the accuracy of 0.01.

# 2.10. Oxidative Stability by Pressure Differential Scanning Calorimetry (PDSC)—Isothermal Measurements

The oxidative stability was determined using differential scanning calorimeter (DSC Q20P, TA Instruments, New Castle, DE, USA) coupled with a high-pressure cell (series DSC Pressure Cell, TA Instruments). Oil samples of 3–4 mg were weighed in open aluminum pans and placed in the sample chamber under an oxygen atmosphere with an initial pressure of 1400 kPa (filling the chamber with an oxygen flow rate of 100 mL/min), at a temperature of 100 °C. The obtained diagrams were analyzed using TA Universal Analysis 2000 software. For each sample, the output was automatically recalculated and is presented as amount of energy per 1 g. The maximum PDSC oxidation rate time ( $\tau_{max}$ ) was determined based on the maximum rate of oxidation (maximum rate of heat flow) with the accuracy of 0.01. The result of PDSC measurement was expressed in minutes.

# 2.11. Statistics Analyses

Measurements were done in triplicate, results in tables are the average of replications with deviation standard. Results were subjected to analysis of variance (ANOVA). Significant means were subjected to analysis by post hoc Tukey's multiple range test ( $\alpha = 0.05$ ). Pearson correlation coefficients, principal component analysis (PCA) was performed using XLSTAT (Addinsoft, Paris, France, 2017).

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#### 3. Results and Discussion

## 3.1. Physicochemical Quality Parameters of Camelina Oils

Lipid oxidation and hydrolysis are the main processes leading to deterioration of edible oils; therefore, it is very important that the degree of these changes be as low as possible. The degree of hydrolytic changes, indicating the free fatty acid content of tested camelina oils, was evaluated on the basis of the acid value. The content of primary oxidation products was determined by peroxide value (PV), and the secondary oxidation products—carbonyl compounds—were determined by anisidine value (p-AnV). The oxidation state of the tested oil was determined using the TOTOX indicator. UV-specific absorbance was also determined:  $K_{232}$  (the level of primary oxidation products and conjugated dienes) and  $K_{268}$  (the level of secondary oxidation products and conjugated trienes). The results obtained are provided in Table 1.

**Table 1.** Physicochemical quality parameters and oxidative stability of camelina oils.

Oil Samples	AV (mg KOH/g)	PV (meq O <sub>2</sub> /kg)	p-AnV	тотох	K <sub>232</sub>	K <sub>268</sub>	OIT (h)	PDSC τ <sub>max</sub> (min)
CO 1	0.56 a	0.98 a	0.55 abcd	2.51 ab	1.29 a	0.12 bcd	5.87 <sup>abc</sup>	158.3 <sup>cdef</sup>
CO 2	0.78 <sup>abc</sup>	1.17 <sup>a</sup>	0.32 abc	2.66 bc	1.33 <sup>ab</sup>	0.11 <sup>abc</sup>	6.18 <sup>c</sup>	165.2 g
CO3	0.68 ab	2.11 <sup>d</sup>	0.36 abc	4.58 <sup>ij</sup>	1.54 abcd	0.09 ab	4.31 ab	146.7 a
CO 4	0.91 <sup>abc</sup>	0.93 <sup>a</sup>	1.01 <sup>cde</sup>	2.87 <sup>cd</sup>	1.36 abc	0.13 <sup>cd</sup>	6.12 <sup>c</sup>	162.3 fg
CO 5	0.73 ab	1.34 <sup>ab</sup>	1.12 <sup>de</sup>	3.80 gh	1.48 abcd	0.15 <sup>de</sup>	5.48 abc	154.6 bcde
CO 6	1.09 abcd	1.12 <sup>a</sup>	0.99 bcde	3.23 dfg	1.43 abcd	0.17 ef	5.96 <sup>abc</sup>	159.4 defg
CO 7	0.85 abc	1.26 <sup>ab</sup>	1.02 <sup>cde</sup>	3.54 ab	1.40 abcd	0.15 <sup>de</sup>	4.96 abc	152.5 abc
CO8	0.83 abc	0.89 a	1.48 <sup>e</sup>	3.26 dfg	1.27 <sup>a</sup>	0.18 ef	5.84 <sup>abc</sup>	156.7 cdef
CO 9	0.98 <sup>abc</sup>	1.46 abc	0.94 abcde	3.86 gh	1.44 abcd	0.13 <sup>cd</sup>	5.39 abc	154.3 bcde
CO 10	1.04 <sup>abc</sup>	2.42 <sup>d</sup>	0.55 abcd	5.39 <sup>j</sup>	1.55 abcd	0.09 ab	5.11 abc	154.1 bcde
CO 11	0.96 <sup>abc</sup>	1.06 <sup>a</sup>	0.32 abc	2.44 <sup>a</sup>	1.31 <sup>ab</sup>	0.12 bcd	5.87 <sup>abc</sup>	157.7 cdef
CO 12	0.89 abc	1.87 <sup>bcd</sup>	0.41 abcd	4.15 hi	1.54 <sup>abcd</sup>	0.11 abc	5.03 abc	151.9 abc
CO 13	0.99 <sup>abc</sup>	1.13 <sup>a</sup>	0.97 bcde	3.23 dfg	1.31 <sup>ab</sup>	0.10 abc	5.38 abc	151.6 abc
CO 14	1.23 bcd	3.49 <sup>e</sup>	1.12 <sup>de</sup>	$8.10^{1}$	2.01 <sup>f</sup>	0.19 <sup>f</sup>	4.26 <sup>a</sup>	149.3 <sup>ab</sup>
CO 15	1.12 abcd	1.09 a	0.86 abcde	3.04 df	1.28 <sup>a</sup>	0.12 bcd	5.63 abc	157.5 <sup>cdef</sup>
CO 16	1.07 <sup>abcd</sup>	2.28 <sup>d</sup>	0.97 bcde	5.53 <sup>jk</sup>	1.68 <sup>de</sup>	0.13 <sup>cd</sup>	5.12 abc	158.6 cdef
CO 17	1.62 <sup>d</sup>	3.41 <sup>e</sup>	1.01 <sup>cde</sup>	7.83 <sup>kl</sup>	1.87 <sup>ef</sup>	0.13 <sup>cd</sup>	4.65 abc	152.3 <sup>abc</sup>
CO 18	0.89 <sup>abc</sup>	3.17 <sup>e</sup>	0.94 abcde	7.28 <sup>kl</sup>	1.84 <sup>ef</sup>	0.13 <sup>cd</sup>	4.69 abc	153.2 abcd
CO 19	0.95 <sup>abc</sup>	1.05 <sup>a</sup>	0.42 abcd	2.52 ab	1.34 <sup>abc</sup>	0.08 a	6.01 bc	162.4 fg
CO 20	1.21 bcd	1.42 <sup>abc</sup>	0.32 <sup>abc</sup>	3.16 dfg	1.51 abcd	0.10 <sup>abc</sup>	5.42 abc	155.3 bcde
CO 21	0.80 <sup>abc</sup>	1.26 <sup>ab</sup>	0.22 a	2.74 abc	1.44 abcd	0.11 <sup>abc</sup>	5.57 <sup>abc</sup>	155.1 bcde
CO 22	0.74 <sup>ab</sup>	1.04 <sup>a</sup>	0.32 abc	2.40 a	1.42 abcd	0.10 <sup>abc</sup>	5.97 <sup>abc</sup>	160.8 efg
CO 23	0.89 <sup>abc</sup>	1.17 <sup>a</sup>	0.30 abc	2.64 bc	1.40 abcd	0.09 ab	5.68 abc	158.2 cdef
CO 24	0.81 <sup>abc</sup>	1.31 <sup>ab</sup>	0.41 abcd	3.03 df	1.43 abcd	0.11 <sup>abc</sup>	5.68 <sup>abc</sup>	157.6 <sup>cdef</sup>
CO 25	0.78 <sup>abc</sup>	1.16 <sup>a</sup>	0.38 <sup>abc</sup>	2.70 abc	1.38 abc	0.08 a	5.60 abc	157.7 cdef
CO 26	0.95 <sup>abc</sup>	1.29 <sup>ab</sup>	0.36 <sup>abc</sup>	2.94 <sup>df</sup>	1.45 abcd	0.10 <sup>abc</sup>	5.59 abc	155.9 bcde
CO 27	1.15 bcd	2.03 <sup>cd</sup>	0.29 abc	4.35 <sup>ij</sup>	1.62 <sup>cde</sup>	0.09 ab	5.01 abc	153.1 abcd
CO 28	1.23 bcd	1.37 <sup>ab</sup>	0.27 ab	3.01 <sup>cd</sup>	1.48 abcd	0.10 abc	5.52 abc	156.8 cdef
CO 29	1.35 <sup>cd</sup>	2.46 <sup>d</sup>	0.45 abcd	5.37 <sup>j</sup>	1.59 bcde	0.12 bcd	4.69 abc	152.9 abcd
min	0.56	0.89	0.22	2.40	1.27	0.08	4.26	146.7
max	1.62	3.49	1.48	8.10	2.01	0.19	6.18	165.2
mean	0.97	1.61	0.64	3.87	1.48	0.12	5.38	156.3

Means within each column with different superscript letters (a–l) are significantly ( $\alpha$  = 0.05) different. Each value in the table represents the mean of three measurements.

The acid value (AV) of the tested oils, ranged from 0.56 to 1.62 mg KOH/g (Table 1). The analyses performed exhibited a large variation in the obtained results, and thus a differentiated degree of hydrolysis of the tested oils. Nevertheless, all tested *Camelina sativa* oils met the quality requirements provided by Codex Alimentarius (<4 mg KOH/g) for cold-pressed oils. Marszałkiewicz et al. [22], when testing camelina oil pressed from seeds of winter and spring conventional crops, obtained similar AV values (1.34–1.49 mg KOH/g). Differentiation of AV camelina oils was also reported by

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Ratusz et al. [20] (0.78–2.35 mg KOH/g). Slightly lower results were presented by Raczyk et al. [21] (0.51–0.89 mg KOH/g). The hydrolytic changes occurring in cold-pressed oils may result from either the endogenous lipolytic enzymes that can be found in the raw material or the influence of water contained in the raw material. Furthermore, the hydrolytic change rate is also affected by impurities, degree of damage to the seed structure, and technological processes, particularly the process of oil extraction and treatment [1,20,40].

Peroxide value (PV) determines the content of primary oxidation products, and in cold-pressed oils intended for consumption, this should not exceed 15 meq  $O_2/kg$ , as specified in Codex Alimentarius (Table 1). The PV of tested oils ranged from 0.89 to 3.49 meq  $O_2/kg$ . Similar results were obtained by Ratusz et al. [20] (0.79–2.04 meq  $O_2/kg$ ), slightly higher by Symoniuk et al. [23], (2.37–3.00 meq  $O_2/kg$ ). Hrastar et al. [3] demonstrated greater variation in this parameter in oils from Slovenia in the growing seasons 2007–2009 (0.74–8.85 meq  $O_2/kg$ ). Dissimilarities in the oxidation state of the investigated oils can be explained by the differences in the quality of the raw material, as well as the conditions of technological processes and the storage of the finished product. It has to be stressed here again that the oils tested were simply filtered after pressing, not refined [1,40].

The anisidine value (p-AnV), which indicates the secondary oxidation products—carbonyl compounds—in the tested oils, was low but variable, and ranged from 0.22 to 1.48 (Table 1). Lower values of p-AnV of market camelina oils (0.45–0.67) were obtained by Symoniuk et al. [23]. Low p-AnV values are characteristic for oils pressed at low temperatures, while the refining process, and in particular the deodorization step, promotes the formation of secondary oxidation products [23,40].

The TOTOX index was calculated to determine the overall oxidation state. In the tested camelina oils, the TOTOX index varied significantly, ranging from 2.40 to 7.83 (Table 1). This variability results from both the initial quality of the raw material, and the means and conditions of oil pressing and purification, as well as the conditions and duration of storage [1,4,20,21,23,40]. Therefore, the TOTOX index value can oscillate within wide limits, which is also indicated by the testing of commercial cold-pressed oils conducted by Symoniuk et al. [23].

The characteristics of the tested oils were also based on the results of UV extinction, i.e., the specific extinction measured at two wavelengths, 232 nm and 268 nm.  $K_{232}$  determines the level of primary oxidation products and conjugated dienes, and is proportional to the content of hydroxyl peroxide compounds; whereas  $K_{268}$  determines the level of conjugated trienes and secondary oxidation products, indicating the content of carbonyl compounds in the tested oils.  $K_{232}$  of camelina seed oils tested ranged from 1.27 to 1.87, while  $K_{268}$  ranged from 0.08 to 0.19 (Table 1). The UV extinction coefficient should reach  $\leq$ 2.50 at a wavelength of 232 nm and  $\leq$ 0.20 at a wavelength of 268 nm; hence, the analyzed oils were characterized by a low degree of primary and secondary oxidation, which was correlated, respectively, with low PV and p-AnV of the oils. Literature works and standards lack guidelines concerning values of this parameter for cold-pressed oils. Given the similarity of cold-pressed oils to extra virgin olive oil, however, the levels stipulated for olive oil were used for data interpretation, which demonstrated that the analyzed oils met the requirements specified in the UE Regulation for extra virgin olive oil. Similar results were achieved for virgin rapeseed oil by Wroniak [40] ( $K_{232}$  from 1.31 to 2.04, and  $K_{268}$  from 0.07 to 0.26).

## 3.2. Bioactive Compounds of Camelina Oils

## 3.2.1. Fatty Acid Composition and Nutrient Values

The composition of individual fatty acids is the main determinant of the quality, nutritional value and oxidative stability of edible oils. Table 2 presents the percentage fatty acid composition of the investigated oils.

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**Table 2.** Fatty acid composition and nutritional value of analyzed camelina oils.

Oil	Fatty Acids																	
Samples	C16:0	C18:0	C18:1	C18:2	C18:3	C20:0	C20:1	C20:2	C20:4	C22:1	SFA	MUFA	PUFA	- n-3/n-6	COX	AI	TI	НН
CO 1	5.7 <sup>i</sup>	2.4 <sup>i</sup>	16.5 fg	18.1 <sup>1</sup>	36.2 gh	1.2 <sup>j</sup>	13.7 df	1.8 <sup>e</sup>	1.3 f	2.0 i	9.3 <sup>e</sup>	32.2 bc	57.4 fg	1.9 <sup>c</sup>	9.9 d	0.06 a	0.1 <sup>a</sup>	12.7 f
CO 2	4.8 a	2.2 g	16.5 fg	15.6 <sup>b</sup>	35.6 <sup>ef</sup>	1.2 <sup>j</sup>	16.7 <sup>k</sup>	$1.4$ $^{\rm b}$	$0.8^{b}$	$3.1 ^{\text{m}}$	8.2 bc	36.3 <sup>g</sup>	53.4 a	2.2 h	9.5 a	0.05 a	0.1 a	14.3 <sup>k</sup>
CO3	5.9 <sup>k</sup>	2.5 <sup>j</sup>	16.6 <sup>gh</sup>	17.4 hi	35.4 <sup>cd</sup>	$1.5^{1}$	13.6 df	1.8 <sup>e</sup>	$0.8^{b}$	2.9 <sup>1</sup>	9.9 g	33.1 ef	55.4 <sup>b</sup>	2.0 e	9.6 <sup>b</sup>	0.07 a	0.1 <sup>a</sup>	11.9 <sup>c</sup>
CO 4	6.2 <sup>n</sup>	2.7 <sup>1</sup>	14.8 a	17.7 <sup>k</sup>	36.1 <sup>gh</sup>	1.2 <sup>j</sup>	15.5 <sup>j</sup>	2.0 g	0.9 <sup>c</sup>	1.2 <sup>c</sup>	10.1 <sup>h</sup>	31.5 ab	56.7 <sup>e</sup>	1.9 <sup>de</sup>	9.8 <sup>cd</sup>	0.07 a	0.1 <sup>a</sup>	11.2 <sup>a</sup>
CO 5	5.0 <sup>c</sup>	1.6 a	15.4 <sup>c</sup>	18.2 lm	36.7 i	1.3 <sup>k</sup>	15.0 <sup>i</sup>	1.9 <sup>f</sup>	1.0 <sup>d</sup>	1.7 <sup>f</sup>	7.9 b <sup>c</sup>	32.1 bc	57.8 g	1.9 <sup>de</sup>	10.0 <sup>de</sup>	0.06 a	0.1 a	14.3 <sup>k</sup>
CO 6	6.0 <sup>1</sup>	2.1 <sup>f</sup>	15.3 bc	17.3 <sup>gh</sup>	36.3 <sup>gh</sup>	1.1 <sup>i</sup>	15.0 <sup>i</sup>	1.8 <sup>e</sup>	1.0 <sup>d</sup>	2.0 i	9.2 <sup>e</sup>	32.3 bc	56.4 <sup>de</sup>	2.0 fg	9.8 <sup>cd</sup>	0.07 a	0.1 a	11.7 <sup>b</sup>
CO 7	6.1 <sup>m</sup>	2.1 f	16.1 <sup>e</sup>	18.3 <sup>m</sup>	36.2 gh	1.3 <sup>k</sup>	14.7 <sup>h</sup>	1.9 <sup>f</sup>	0.8 b	1.9 h	9.5 <sup>f</sup>	32.7 <sup>d</sup>	57.2 <sup>f</sup>	1.9 <sup>d</sup>	9.9 d	0.07 a	0.1 a	11.7 <sup>bc</sup>
CO8	6.1 <sup>m</sup>	2.5 <sup>j</sup>	16.6 gh	17.7 <sup>k</sup>	36.1 gh	1.2 <sup>j</sup>	13.6 df	1.8 <sup>e</sup>	1.3 <sup>f</sup>	2.0 i	9.8 g	32.2 bc	56.9 <sup>e</sup>	1.9 <sup>d</sup>	9.8 <sup>cd</sup>	0.07 a	0.1 a	11.8 bc
CO9	6.0 <sup>1</sup>	2.0 e	16.4 <sup>f</sup>	17.5 <sup>ij</sup>	35.5 <sup>ef</sup>	1.2 <sup>j</sup>	14.0 g	1.8 <sup>e</sup>	1.1 <sup>e</sup>	2.0 i	9.2 <sup>e</sup>	32.4 <sup>c</sup>	55.9 <sup>bc</sup>	1.9 <sup>d</sup>	9.6 bc	0.07 a	0.1 a	11.8 bc
CO 10	5.9 k	2.0 e	15.2 b	18.8 <sup>n</sup>	37.1 <sup>j</sup>	1.1 <sup>i</sup>	14.4 gh	1.7 <sup>d</sup>	0.3 a	1.8 g	9.0 <sup>de</sup>	31.4 ab	57.9 g	1.9 <sup>de</sup>	10.1 <sup>e</sup>	0.07 a	0.1 a	12.1 <sup>d</sup>
CO 11	5.7 i	1.8 <sup>c</sup>	17.0 <sup>j</sup>	18.2 lm	36.3 gh	1.0 h	13.8 <sup>f</sup>	1.6 <sup>c</sup>	$0.8^{b}$	1.9 h	8.5 <sup>cd</sup>	32.7 <sup>d</sup>	56.9 <sup>e</sup>	1.9 <sup>d</sup>	9.9 d	0.06 a	0.1 a	12.7 <sup>f</sup>
CO 12	5.0 <sup>c</sup>	2.0 e	17.8 <sup>1</sup>	17.6 <sup>jk</sup>	36.8 <sup>i</sup>	0.8 f	13.5 <sup>cd</sup>	1.7 <sup>d</sup>	1.0 d	1.7 f	7.8 b	33.0 <sup>e</sup>	57.1 <sup>f</sup>	2.0 fg	9.9 de	0.06 a	0.1 a	14.6 <sup>1</sup>
CO 13	6.0 <sup>1</sup>	2.1 f	16.7 hi	16.9 <sup>df</sup>	36.1 gh	1.0 h	13.7 <sup>d</sup>	$1.4^{b}$	1.6 <sup>i</sup>	1.5 <sup>e</sup>	9.1 <sup>de</sup>	31.9 b	56.0 <sup>c</sup>	2.0 <sup>e</sup>	9.7 <sup>c</sup>	0.07 a	0.1 a	11.9 <sup>c</sup>
CO 14	5.1 <sup>d</sup>	2.4 i	18.0 <sup>m</sup>	18.1 <sup>1</sup>	35.0 ab	0.9 g	12.9 <sup>c</sup>	1.7 <sup>d</sup>	1.5 h	1.9 h	8.4 <sup>c</sup>	32.8 <sup>de</sup>	56.3 <sup>de</sup>	1.8 b	9.6 b	0.06 a	0.1 a	14.2 <sup>k</sup>
CO 15	4.9 b	1.9 <sup>d</sup>	15.8 <sup>d</sup>	17.5 <sup>ij</sup>	36.7 i	1.1 i	14.3 gh	1.7 <sup>d</sup>	2.0 m	1.2 <sup>c</sup>	7.9 bc	31.3 a	57.9 g	1.9 <sup>c</sup>	9.9 d	0.06 a	0.1 a	$14.7^{1}$
CO 16	5.8 <sup>j</sup>	2.3 h	16.8 <sup>i</sup>	17.7 <sup>k</sup>	36.1 gh	0.6 <sup>e</sup>	13.6 df	1.6 <sup>c</sup>	1.7 <sup>j</sup>	1.8 g	8.7 d	32.2 bc	57.1 <sup>f</sup>	1.9 bc	9.8 cd	0.07 a	0.1 a	12.5 <sup>e</sup>
CO 17	5.3 e	2.9 n	17.2 k	15.9 <sup>c</sup>	35.5 ef	0.8 f	15.0 <sup>i</sup>	1.9 f	1.9 <sup>1</sup>	1.4 <sup>d</sup>	9.0 <sup>de</sup>	33.6 f	55.2 b	2.0 g	9.5 a	0.06 a	0.1 a	13.3 hi
CO 18	5.1 <sup>d</sup>	2.8 <sup>m</sup>	15.9 <sup>d</sup>	18.1 <sup>1</sup>	35.8 fg	0.9 g	14.0 g	1.8 <sup>e</sup>	1.2 e	2.0 i	8.8 de	31.9 b	56.9 <sup>e</sup>	1.9 bc	9.8 cd	0.06 a	0.1 a	13.9 <sup>j</sup>
CO 19	5.1 <sup>d</sup>	2.3 h	17.3 <sup>k</sup>	17.3 gh	35.9 fg	1.1 <sup>i</sup>	14.2 g	1.9 <sup>f</sup>	1.3 <sup>f</sup>	1.7 f	8.5 <sup>cd</sup>	33.2 ef	56.4 <sup>de</sup>	1.9 <sup>de</sup>	9.7 <sup>c</sup>	0.06 a	0.1 a	14.1 <sup>j</sup>
CO 20	5.7 i	1.7 b	16.6 gh	17.7 <sup>k</sup>	37.1 <sup>j</sup>	1.0 h	13.6 df	1.9 f	1.7 <sup>j</sup>	1.1 b	8.4 °	31.3 a	58.4 h	1.9 <sup>de</sup>	10.0 e	0.06 a	0.1 a	12.8 <sup>fg</sup>
CO 21	5.0 c	2.1 <sup>f</sup>	19.1 <sup>r</sup>	17.0 <sup>f</sup>	36.8 i	0.3 b	12.0 ab	2.0 g	2.3 °	1.7 f	7.4 <sup>a</sup>	32.8 <sup>de</sup>	58.1 gh	1.9 <sup>de</sup>	9.9 d	0.06 a	0.1 a	15.0 m
CO 22	5.1 <sup>d</sup>	2.6 k	18.6 °	15.3 a	37.1 <sup>j</sup>	0.2 a	11.9 a	2.0 g	1.8 <sup>k</sup>	2.2 k	7.9 bc	32.7 <sup>d</sup>	56.2 <sup>d</sup>	2.2 h	9.8 cd	0.06 a	0.1 a	14.9 <sup>k</sup>
CO 23	5.6 h	2.1 f	18.5 °	17.0 <sup>f</sup>	36.7 i	0.3 b	12.0 ab	1.8 e	2.0 m	1.8 g	8.0 bc	32.3 bc	57.5 f <sup>g</sup>	1.9 <sup>de</sup>	9.9 d	0.06 a	0.1 a	13.3 hi
CO 24	5.5 g	2.3 h	19.0 pr	17.2 g	35.7 ef	0.3 b	11.7 a	1.9 <sup>f</sup>	2.1 <sup>n</sup>	2.0 i	8.1 bc	32.7 <sup>d</sup>	56.9 e	1.9 bc	9.7 bc	0.06 a	0.1 a	13.5 <sup>i</sup>
CO 25	5.6 h	2.5 <sup>j</sup>	18.6 °	16.8 <sup>d</sup>	35.7 ef	0.4 <sup>c</sup>	12.0 a	2.0 g	1.8 k	2.0 i	8.5 <sup>cd</sup>	32.6 <sup>d</sup>	56.3 <sup>de</sup>	1.9 <sup>de</sup>	9.6 bc	0.06 a	0.1 a	13.0 g
CO 26	5.5 g	2.7 1	18.3 <sup>n</sup>	17.3 gh	35.1 bc	0.5 d	12.6 <sup>c</sup>	1.9 f	2.1 <sup>n</sup>	1.9 h	8.7 d	32.8 <sup>de</sup>	56.4 <sup>de</sup>	1.8 b	9.6 ab	0.06 a	0.1 a	13.2 hi
CO 27	5.4 f	1.7 b	16.6 gh	19.3 °	34.7 a	1.1 <sup>i</sup>	15.9 <sup>j</sup>	1.2 a	1.6 <sup>i</sup>	0.7 a	8.2 bc	33.2 ef	56.8 e	1.7 a	9.7 bc	0.06 a	0.1 a	13.4 <sup>i</sup>
CO 28	6.0 <sup>1</sup>	2.4 <sup>i</sup>	18.9 P	17.4 hi	35.0 ab	0.6 e	12.0 ab	1.7 <sup>d</sup>	1.6 <sup>i</sup>	2.1 <sup>j</sup>	8.9 de	33.0 e	55.8 bc	1.8 bc	9.5 ab	0.07 a	0.1 a	12.2 <sup>d</sup>
CO 29	5.4 <sup>f</sup>	2.2 g	18.0 m	16.9 <sup>df</sup>	36.0 g	0.9 g	13.0 <sup>cd</sup>	1.7 <sup>d</sup>	1.4 g	1.5 e	8.6 d	32.5 °	56.0 °	2.0 f	9.7 °	0.06 a	0.1 <sup>a</sup>	13.4 <sup>i</sup>
min	4.8	1.6	14.8	15.3	34.7	0.2	11.7	1.2	0.3	0.7	7.4	31.3	53.5	1.7	9.5	0.05	0.1	11.2
max	6.1	2.9	19.1	19.3	37.1	1.5	16.7	2.0	2.3	3.1	10.0	36.3	58.4	2.2	10.1	0.07	0.1	15.0
mean	5.5	2.2	17.0	17.4	36.0	0.9	13.7	1.8	1.4	1.8	8.7	32.6	56.7	1.9	9.8	0.06	0.1	13.1

Means within each column with different superscript letters (a–r) are significantly ( $\alpha$  = 0.05) different. Each value in the table represents the mean of three measurements.

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The analyzed camelina oils were characterized by a very beneficial fatty acid nutritional profile—about 90% were unsaturated acids (UFA), and the saturated acids (SFA) content was only 7.4 to 10.1%. SFA acids include palmitic, stearic, arachidic and docosanoic acids, of which the palmitic acid is predominant (about 6%). The obtained results are similar to those presented by other authors [1–5,20–23].

Between polyunsaturated acids (PUFA),  $\alpha$ -linolenic acid C18:3 n-3 (ALA) was predominant. This acid is essential for the proper functioning of the body, and because it is not synthesized by the human body, it must be delivered by the diet [15,16]. It constituted 34.7 to 37.1% of the total fatty acids (Table 2). Comparable results have been presented by Abramovic and Abram [1] (35.2%), Imbrea et al. [41] (35.6%), Raczyk et al. [21] (35.4–36.1%) and Symoniuk et al. [23] (31.62–34.32%). Belayneh et al. reported a slightly lower share [19] (32.8%). Literature data show a significant advantage of camelina oil in terms of ALA content in relation to many other edible oils. By comparison, Wroniak [40] found 8.07–9.95%  $\alpha$ -linolenic acid in cold-pressed rapeseed oil, and Symoniuk et al. [23] found 0.33–3.81% in sunflower oil. The oil with the highest content of this acid is linseed oil, in which the level of ALA varies from 49.2 to 64.6% [30].

The next acid, included in the essential fatty acids, is linoleic acid C18:2 *n*-6 (LA). Its share ranged from 15.3 to 19.3%. Comparable values have been obtained by Berti et al. [5] and Balayneh et al. [19] (18.5%) and Hrastar et al. [3] (16.1–18.6%), who studied camelina oil from seeds grown in the Korosča region (Slovenia). A similar share of linoleic acid in *Camelina sativa* seed oils cultivated in the Wielkopolska region (Poland) was also reported by Ratusz et al. [20] (18.0–18.9%). Marginally lower values were obtained by Symoniuk et al. [23] (16.5–18.0%), who also investigated market oils. The eicosadienoic acid (C20:2) content in the analyzed oils was around 1.2–2.0% in all the camelina oils. Similar results (1.3–1.4%) were obtained by Marcheva [42], who studied genetic resources of *Camelina sativa* from Poland, Bulgaria and Netherlands.

Acids from the *n*-3 and *n*-6 families are extremely important for both the proper development of the body and for keeping it in good condition. The ratio of fatty acids of the *n*-3 to *n*-6 families is, from a nutritional point of view, paramount, because both of these essential fatty acid families compete for the same enzymes during metabolic processes in the human body [15,16,32]. In the camelina oils investigated in this work, the ratio of the abovementioned acids ranged from 1.79 to 2.17, which again proves the high nutritional value of the camelina oils. This ratio is definitely better than that found in popular vegetable oils, e.g., rapeseed, sunflower, soybean oil [23,43,44].

Monoenoic acids (MUFA) accounted for more than 30% of all fatty acids. C18:1 oleic acid, the content of which was from 14.8 to 19.1%, and eicosenoic acid C20:1, were predominant among monoenoic acids. A typical trait of camelina oil is the presence of eicosenoic acid. Its content in the analyzed samples ranged from 11.7 to 16.7%. The measured contents of these acids are similar to those presented in other works: Abramovic and Abram [1] determined the content of C18:1 in cold-pressed camelina oils to be 17.4%, and C20:1 to be 14.9%; Berti et al. [5] determined them to be 15.7% and 15.1%, respectively.

Fatty acid composition has an immense impact on the dietary factors of fats and plant oils. The quality of dietary fat depends on its level of SFA, MUFA, PUFA, and especially on the ratio of *n*-6 to *n*-3 fatty acids [15,16,32,45]. Schwab et al. [46] summarized the effects of the quantity and quality of dietary fat on risk coronary heart disease (CHD), factors for type-2 diabetes, body weight, and cancer in healthy subjects and in subjects at risk of these diseases. In 1991, Ulbricht and Southgate [32] recognized seven dietary factors that can be associated with CHD. Saturated fatty acids, which raise cholesterol levels, and thrombogenic SFA are promoters of CHD, whereas the *n*-3 and *n*-6 isomers of PUFA, MUFA, and antioxidants have been described as protecting against CHD. For assessment of the nutritional value of oils, three nutritional quality indices were also determined: the atherogenicity index (AI), the thrombogenicity index (TI), and the hypocholesterolemic: hipercholesterolemic FA ratio (HH) were calculated on the basis of the fatty acid composition of the camelina oils. These indices are more useful than fatty acid composition in evaluating oils nutritionally. The AI values of all

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camelina oils amounted to between 0.05 and 0.07, and did not differ statistically at  $\alpha = 0.05$ . Ulbricht and Southgate [32] showed that the AI of oils from coconut, palm and olive are 13.63, 0.88 and 0.14, respectively. Ying et al. [37] determined AI value oils for avocado fruit (0.40) and borage (0.18), while similar values were shown for apricot kernel (0.05) and dill seed (0.08) oils. The studied camelina oils were also characterized by low TI (0.1), not statistically different. A similar TI value was provided by Ying et al. [37] for apricot (0.11), and for oils obtained from blackcurrant and hemp seed—0.23. Significantly higher TI values for coconut (6.81) and palm (2.07) oils were demonstrated by Ulbricht and Southgate [32]. The HH ratio of tested camelina oils ranged from 11.7 to 14.7 and was similar to oils obtained from dill (12.56), hemp (14.88), and blackcurrant seed (13.82) [37]. Therefore, enriching the diet with camelina oil, which is characterized by low AI and TI and a relatively high HH ratio, can make the diet less atherogenic without making it less thrombogenic, thus contributing to a decrease in the incidence of coronary heart diseases (CHD) [32,37,46].

## 3.2.2. Components of Unsaponifiable Fraction (Chlorophylls, Carotenoids, Tocopherols, Sterols)

The nutritional value of cold-pressed oils, including camelina oil, depends not only on the fatty acid profile, but also on the composition of the unsaponifiables fraction—the content of tocopherols, sterols, carotenoid pigments and chlorophylls. Table 3 presents the share of unsaponifiables in the studied camelina oils.

Pigments represent natural components of oilseeds, they are considered important factors because they can impart an undesirable dark color to vegetable oils or facilitate oxidation in the presence of light. The composition and content of chlorophyll pigments present in the seeds depends on seed maturity. The concentration of carotenoid pigments in analyzed oils ranged from 103 to 198 mg of  $\beta$ -carotene/kg and was variable. Raczyk et al. [21], when determining the total carotenoids in cold-pressed camelina oil, obtained values from 78 to 112 mg/kg. The results presented by Marszałkiewicz et al. [22] were significantly lower (8.11–54.07 mg/kg).

In the studied oils, the content of chlorophyll pigments ranged from 1.02 to 2.18 mg pheophytin/kg. Larger variability was shown by Symoniuk et al. [23] (0.60–2.60 mg/kg) in market camelina oils. The diversity in the composition of the unsaponifiables fraction in cold-pressed oils can be very large. According to Codex Alimentarius—Codex Stan 210:1999 [47], cold-pressed oils are obtained by mechanical procedures only, e.g., pressing or expelling, without the application of heat. They may have been purified by centrifuging, washing with water, filtering and settling only. Therefore, the content of carotenoid and chlorophyll pigments in cold-pressed oils depends largely on the quality of the raw materials, and the weather conditions during the growth and maturation of seeds, as well as the degree of maturity of the collected seeds [1–4,40].

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**Table 3.** The content of pigment, tocopherols and phytosterols in analyzed oils.

			Tocopherols [mg/100 g]					Sterols [mg/100 g]							
Oil Samples	Chlorophylls [mg/kg]	Carotenoids - [mg/kg]	Total	α-Т	β-Т	ү-Т	δ-Т	Total Sterols	Cholesterol	Brassicasterol	Campesterol	Campestanol	Stigmasterol	β-Sitosterol	Δ5-Avenasterol
CO 1	1.25 a	158 bcd	57.91 ab	1.89 ab	0.21 bc	54.62 ab	0.62 a	331 a	28 a	22 a	93 a	4 a	1 <sup>a</sup>	167 a	15 a
CO 2	1.11 <sup>a</sup>	137 abc	62.84 <sup>de</sup>	2.02 ab	0.20 b	59.10 <sup>de</sup>	0.99 ab	351 a	27 a	23 a	89 a	4 a	2 a	189 a	16 a
CO3	1.98 a	109 a	71.87 <sup>ijk</sup>	2.30 ab	0.09 a	67.80 <sup>ijk</sup>	1.50 c	368 a	30 a	25 a	75 <sup>a</sup>	4 a	2 a	217 a	16 <sup>a</sup>
CO 4	1.08 a	141 bcd	65.59 efg	2.14 ab	0.20 b	62.30 efg	1.02 ab	372 <sup>a</sup>	29 <sup>a</sup>	25 <sup>a</sup>	88 <sup>a</sup>	4 <sup>a</sup>	1 <sup>a</sup>	205 a	20 a
CO 5	1.16 a	193 efgh	68.23 gh	2.78 ab	0.18 b	63.60 gh	1.24 ab	376 a	26 a	26 a	74 <sup>a</sup>	4 a	3 a	225 a	18 <sup>a</sup>
CO 6	1.32 a	162 bcd	59.41 bc	2.17 ab	0.24 bc	54.80 bc	1.39 b	417 a	31 a	26 a	94 a	2 a	3 a	244 a	17 a
CO 7	1.49 a	169 bcde	64.14 <sup>def</sup>	1.27 ab	0.18 b	62.01 def	0.78 a	406 a	33 a	19 a	102 a	2 a	3 a	231 a	16 a
CO8	1.05 a	187 defg	67.90 gh	2.64 ab	0.28 c	64.10 gh	0.76 a	380 a	31 a	19 a	103 a	4 a	2 a	198 a	22 a
CO 9	1.18 a	186 defg	74.30 klm	2.12 ab	0.23 bd	69.50 klm	2.03 c	375 a	27 a	22 a	85 a	1 a	2 a	216 a	22 a
CO 10	1.51 a	106 a	72.10 <sup>ijk</sup>	2.05 ab	0.18 b	67.80 <sup>ijk</sup>	1.89 bc	382 a	30 a	23 a	89 a	2 a	2 a	216 a	12 a
CO 11	1.37 a	179 cdef	70.80 hij	1.74 ab	0.16 b	66.80 hij	1.29 ab	363 a	29 a	24 a	93 a	2 a	3 a	194 a	19 a
CO 12	1.59 a	126 abc	68.30 gh	1.56 ab	0.21 bc	64.80 gh	1.23 ab	415 a	30 a	27 a	87 a	2 a	2 a	248 a	18 <sup>a</sup>
CO 13	1.45 a	172 cdef	62.70 <sup>de</sup>	1.02 a	0.15 a	60.50 de	0.98 ab	407 a	28 a	21 <sup>a</sup>	86 a	3 a	3 a	246 a	23 a
CO 14	1.97 a	103 a	55.80 a	2.06 ab	0.12 a	52.41 a	0.63 a	346 a	32 a	20 a	93 a	3 a	2 a	179 a	17 a
CO 15	1.49 a	198 efgh	64.00 <sup>def</sup>	1.66 ab	0.14 a	61.20 <sup>def</sup>	0.81 ab	348 a	31 <sup>a</sup>	20 a	92 a	3 a	2 a	185 <sup>a</sup>	16 <sup>a</sup>
CO 16	1.74 a	107 a	59.70 bc	1.79 ab	0.11 a	56.40 bc	0.80 ab	393 a	29 a	23 a	87 a	2 a	2 a	231 a	18 a
CO 17	1.86 a	112 ab	61.90 <sup>cd</sup>	1.66 ab	0.09 a	58.46 <sup>cd</sup>	1.14 ab	423 a	26 a	21 a	102 a	1 a	2 a	252 a	19 a
CO 18	1.74 a	131 abc	66.10 <sup>fg</sup>	1.24 ab	0.13 a	63.50 <sup>fg</sup>	1.02 ab	363 a	25 a	21 <sup>a</sup>	92 a	2 a	2 a	199 a	20 a
CO 19	1.22 a	167 bcde	72.10 <sup>ijk</sup>	1.27 ab	0.15 a	68.90 <sup>ijk</sup>	1.24 ab	398 a	28 a	24 a	84 a	3 a	2 a	242 a	17 a
CO 20	1.59 a	164 bcd	70.30 hij	1.14 ab	0.18 b	66.90 hij	1.89 bc	422 a	28 a	23 a	93 a	2 a	3 a	256 a	18 <sup>a</sup>
CO 21	1.87 a	157 bcd	68.50 gh	1.12 ab	0.16 b	65.30 gh	1.56 b	332 a	26 a	30 ab	85 a	6 a	3 a	167 a	15 a
CO 22	1.71 a	183 defg	69.20 hi	0.98 a	0.16 b	65.90 <sup>hi</sup>	1.72 b	397 a	27 a	28 a	94 a	7 a	3 a	223 a	15 a
CO 23	2.15 a	168 bcde	70.60 hij	1.05 ab	0.12 a	66.10 hij	2.04 °	415 a	27 a	28 a	112 a	6 a	4 a	222 a	16 a
CO 24	1.26 a	174 <sup>cdef</sup>	71.80 <sup>ijk</sup>	1.05 ab	0.12 a	68.60 <sup>ijk</sup>	2.02 c	367 a	30 a	26 a	97 a	5 a	4 a	194 a	16 a
CO 25	1.02 a	189 <sup>defg</sup>	72.30 <sup>jkl</sup>	0.95 a	0.09 a	69.00 <sup>jkl</sup>	1.86 bc	358 a	29 a	23 a	72 a	2 a	1 a	214 a	16 a
CO 26	1.54 a	163 bcd	75.20 lm	1.03 a	0.18 b	72.10 lm	1.78 b	381 a	29 a	26 a	82 a	4 a	2 a	219 a	18 <sup>a</sup>
CO 27	1.89 a	109 a	69.90 hij	1.08 ab	0.10 a	66.90 hij	1.56 b	376 a	28 a	24 a	102 a	4 a	3 a	194 <sup>a</sup>	21 a
CO 28	1.85 a	129 abc	74.40 klm	1.41 ab	0.12 0.11 a	70.50 klm	1.99 °	430 a	33 a	24 a	95 a	3 a	2 a	252 a	20 a
CO 29	2.18 a	118 <sup>ab</sup>	76.10 <sup>m</sup>	1.51 ab	0.11 a	70.30 <sup>m</sup>	2.05 °	442 a	31 <sup>a</sup>	31 <sup>ab</sup>	97 a	2 a	3 a	262 a	16 <sup>a</sup>
min	1.02	103	55.80	0.95	0.12	52.41	0.62	331	25	19	72	1	1	167	15
max	2.18	198	76.10	2.78	0.09	72.30	2.05	442	33	31	112	7	4	262	23
mean	1.54	152	67.72	1.61	0.26	64.21	1.37	384	29	24	91	3	2	217	18

Means within each column with different superscript letters (a–m) are significantly ( $\alpha = 0.05$ ) different. Each value in the table represents the mean of three measurements.

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The content of tocopherols and sterols is also very important for the nutritional value and oil stability. The protection of PUFA, especially essential fatty acids, against peroxidation is believed to be the paramount biochemical role of tocopherols. However, individual tocopherol homologs differ in their biological and antioxidant activities. The greatest biological activity is that of  $\alpha$ -tocopherol  $(\alpha-T)$  [37,43,44]. In the oils tested, total tocopherol levels ranged from 55.8 to 76.1 mg/100 g (Table 3) and were comparable to those presented by Hrastar et al. [3] (55.6–87.3 mg/100 g), who studied Slovenian samples, but was slightly lower than those in the camelina oils tested by Zubr and Mätthaus [4] (69.7–99.4 mg/100 g) and Marszałkiewicz et al. [22] (66.5–78.9 mg/100 g). In the cold-pressed camelina oils from the seeds grown in Turkey, the content of tocopherols was markedly higher (144.1–168.7 mg/100 g) [11]. The total tocopherol content in camelina oils from our study was similar to the level measured in canola (71.4 mg/100 g) and sunflower oils (71.3 mg/100 g), but considerably higher than in olive oil (33.1 mg/100 g) as reported by Zaunschirm et al. [43]. In all of the camelina oils analyzed in this study, γ-tocopherol (γ-T) predominated, with more than 90%. This homologous form of tocopherol also predominates in other oils rich in ALA acid, e.g., linseed and perilla oil [44]. In the tested oils, the content of  $\gamma$ -T ranged from 52.41 to 72.30 mg/100 g. An analogous relationship was determined by other authors [3,22], who showed more than a 90% share of  $\gamma$ -T in relation to total tocopherol content in the studied oils. In addition to  $\gamma$ -T,  $\alpha$ -T,  $\delta$ -T and  $\beta$ -T were also identified, whose content ranged from 0.95 to 2.78, from 0.62 to 2.05 and from 0.09 to 0.28 mg/100 g, respectively.

Phytosterols are of great interest due to their health effects, particularly their cholesterol-lowering properties [19,22,37]. In the studied camelina oils, the total sterols ranged from 331 to 442 mg/100 g (Table 3). The major sterols in the tested oils were  $\beta$ -sitosterol (167–262 mg/100 g) and campesterol (74-112 mg/100 g). Campesterol,  $\beta$ -sitosterol and stigmasterol are generally found in high levels in plant oils and are structurally very similar to cholesterol except the side chain on C-17. In the oils analyzed in our study, the cholesterol level ranged from 25 to 33 mg/100 g. Similar levels of  $\beta$ -sitosterol, campesterol and cholesterol were determined by Marszałkiewicz et al. [22]. In the studied camelina oils, brassicasterol (19–31 mg/100 g), Δ5-avenasterol (16–23 mg/100 g) and traces of sitostanol and campestanol were also found. The sterol content in camelina oils reported by other researchers was between 193 and 590 mg/100 g [11,15,18]. For comparison, lower total levels of phytosterols were shown by Montesano et al. [48] in pumpkin (295 mg/100 g), and by Ying [37] in poppy seed (about 190 mg/100 g), avocado fruit (about 135 mg/100 g) and hemp (83 mg/100 g) oils. Lower and different levels of total sterols in goji berry (Lycium barbarum L.) produced in Mongolia (130.1 mg/100 g), China (55.1 mg/100 g) and Italy (42.8 mg/100 g) were determined by Cossignani et al. [49]. The content of tocopherols and sterols in vegetable oils decreases with heating and storage; therefore, storage conditions, as well as distribution and display in shops, is crucial [37,43].

# 3.3. Oxidative Stability by Rancimat and PDSC Method

Oxidative stability is a serious oil safety factor, used to estimate shelf life. The Rancimat test is often used for its determination. The studied camelina oils were characterized by oxidative induction time (OIT) at a temperature of  $100\,^{\circ}$ C from 4.26 to 6.18 h (Table 1). Under the same measurement conditions, Marszałkiewicz et al. [22] obtained results within 4.79-7.51 h, and Ratusz et al. [20] from 4.58 to 5.63 h. Abramovic and Abram [1], when testing camelina oils at  $110\,^{\circ}$ C, obtained OIT equal to 4.8 h. The oxidative induction time of the tested camelina oils was longer than that of linseed oil (from 3.67 to 4.65 h) [23], but shorter than that for cold-pressed rapeseed oils (12.96-13.98 h) [40].

The oxidative stability of oils can also be well characterized by the induction time  $\tau_{max}$  determined on the basis of PDSC technique [20,23,30,50]. In the studied oils, PDSC  $\tau_{max}$  ranged from 146.7 to 165.2 min (Table 1). Similar values under the same measurement conditions are presented in Ratusz et al. [20] (151.9–172.3 min). Symoniuk et al. [23], when testing camelina oils at 120 °C, obtained substantially lower values (17.8–28.8 min).

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## 3.4. Statistical Analyses

To investigate the influence of the analyzed parameters (LOO, LK, p-AV, TOTOX,  $K_{232}$ ,  $K_{268}$ , fatty acids composition, content of chlorophylls, carotenoids, sterols and tocopherols) on the oxidation stability of camelina oils, a statistical analysis was performed. Analysis of the obtained results indicates that the oxidation induction times of the investigated oils determined using the Rancimat (OIT) and PDSC ( $\tau_{max}$ ) methods were strongly correlated, and the correlation coefficient was r = 0.88 (Table 4). The high correlation coefficient value allows us to conclude that these methods can be used interchangeably.

<b>Quality Feature</b>	OIT	PDSC $\tau_{max}$
AV	-0.40 *	-0.24
PV	-0.86 *	-0.60 *
p-AnV	-0.16	-0.16
TOTOX	-0.83 *	-0.60 *
K <sub>232</sub>	-0.80 *	-0.54 *
K <sub>268</sub>	-0.14	-0.14
Chlorophylls	-0.65 *	-0.52 *
Carotenoids	0.64 *	0.38 *
SFA	-0.10	-0.14
MUFA	0.07	0.22
PUFA	0.05	-0.10
COX	0.14	0.00
Tocopheols	0.06	-0.04
α -tocopherol	-0.03	-0.05
β-tocopherol	0.49 *	0.29
δ-tocopherol	0.05	-0.01
γ-tocopherol	0.05	-0.05
Sterols	-0.15	-0.11
OIT	-	0.88 *

Table 4. Correlation coefficient for oxidative stability and different quality features of oil.

The oxidation stability of cold-pressed oils, due to their diverse and rich chemical composition, can be determined by many factors. Table 4 presents the correlation coefficients for individual quality and oil stability determinants defined using the Rancimat and PDSC methods. The obtained results make it possible to conclude that a strong correlation occurs between the OIT and the primary (PV) and total oxidation state of the sample (TOTOX) (r = -0.86 and -0.83, respectively). A strong correlation was also found for OIT and  $K_{232}$  (r = -0.80). A slightly lower correlation value was demonstrated for chlorophyll and carotenoid pigments (r = -0.65 and 0.64, respectively). For the PDSC method, statistical analysis of the obtained results confirmed that the PV and TOTOX of oil had the highest impact on its stability (r = -0.60). The correlations between PDSC ( $\tau_{max}$ ) and  $K_{232}$  (r = -0.54) and between chlorophyll and carotenoid pigments (r = -0.52 and 0.38) were also slightly lower. However, their values were lower for the PDSC method than for Rancimat; the same relationship was noted in studies by Symoniuk et al. [23]. Using both the Rancimat and PDSC methods, the influence of the composition of fatty acids and the content of tocopherols and sterols did not prove to have any influence on the stability of camelina oil. This may be due to the small variation in the samples tested in this respect [23].

The oxidative stability of cold-pressed camelina oils is not only dependent on initial raw material chemical composition. The degree of seed maturity, mechanical damages, contaminations, moisture, seed processing conditions, heating and cold-pressing conditions, the temperature of the pressed oil, press pressure, and time and temperature of sedimentation and filtration are significant factors, as well [1,4,20,23].

To verify the determined regularities and correlations, analysis of the main components (PCA) was carried out. PCA was prepared for average measures for each camelina oil with regard to 20 attributes

<sup>\*—</sup>correlation statistically significant for  $\alpha = 0.05$ .

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(Table 5). As shown in Figure 1, the first two components represented 52.86% of the variability (30.22 and 22.64%, respectively).

<b>Table 5.</b> Principal	l component analy	ysis (PCA) factor [	loadings for the qual	ty factors of analyzed oils.

Quality Feature	PC1	PC2
AV	0.361	0.040
PV	0.890	0.030
p-AnV	0.124	0.600
TOTOX	0.916	0.000
K <sub>232</sub>	0.839	0.028
K <sub>268</sub>	0.137	0.572
Chlorophylls	0.359	0.329
Carotenoids	0.571	0.054
SFA	0.025	0.123
MUFA	0.000	0.010
PUFA	0.014	0.000
COX	0.012	0.004
Tocopherols	0.151	0.527
a-tocopherol	0.030	0.429
β-tocopherol	0.170	0.386
γ-tocopherol	0.148	0.555
δ-tocopherol	0.079	0.610
Sterols	0.012	0.115
OIT	0.796	0.069
PDSC $\tau_{max}$	0.462	0.047

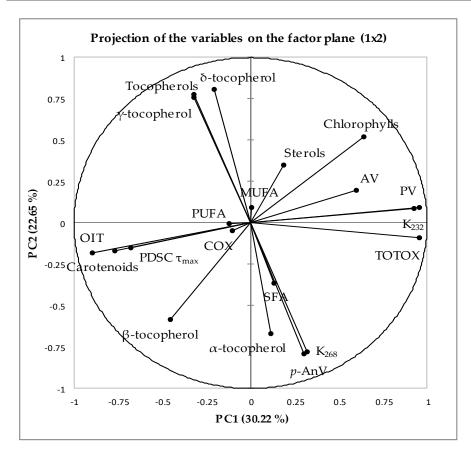


Figure 1. Principal component analysis (PCA) based on quality features of analyzed camelina oils.

The following quality features contributed highly to PC1: TOTOX (0.916), PV (0.890),  $K_{232}$  (0.839), OIT (0.796), carotenoids (0.571), PDSC  $\tau_{max}$  (0.462), AV (0.361), and chlorophylls (0.359). On the

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other hand,  $\delta$ -tocopherol (0.610), K<sub>268</sub> (0.572), p-AnV (0.600),  $\gamma$ -tocopherol (0.555), tocopherols (0.527)  $\alpha$ -tocopherol (0.429) and  $\beta$ -tocopherol (0.386) contributed highly to PC2 (Table 5).

## 4. Conclusions

The results reported in this study confirm that cold-pressed *Camelina sativa* oils are very interesting, with high nutritional value. The high nutritional value of camelina oils was confirmed owing to the optimal composition of fatty acids (low content of SFA, high content of  $\alpha$ -linolenic acid, optimal PUFA n-3 to PUFA n-6 ratio, high content of tocopherols, phytosterols and carotenoids, and optimal nutritional quality indices: the atherogenicity index (AI), the thrombogenicity index (TI), and the hypoto hypercholesterolemic FA ratio (HH)). A significant impact was found for primary (PV) and total oxidation (TOTOX) of camelina oil on oxidative stability evaluated by both Rancimat test and PDSC methods. Both the methods were also confirmed to be appropriate for the assessment of oxidative stability of camelina oils. To the best of our knowledge, this is the first time comprehensive analysis data of camelina oil from Poland have been reported. Information obtained from this research could help to assess the potential of cold-pressed camelina oil to be commercially exploited for different applications, e.g., cosmetics, nutraceutical application, and incorporations into food formulations to benefit human health. A more extensive sampling for a better characterization of camelina seed oils and for authentication purposes is necessary.

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## **Abbreviations**

 $\alpha$ -T alpha ( $\alpha$ )-tocopherol AI atherogenicity index ALA  $\alpha$ -linolenic acid p-AnV p-anisidine value AV acid value

 $\beta$ -T beta ( $\beta$ )-tocopherol CHD coronary heart diseases

CO camelina oil COX oxidizability value  $\gamma$ -T gamma ( $\gamma$ ) tocopherol  $\delta$ -T delta ( $\delta$ ) tocopherol fatty acids

GC-FID gas chromatography with flame ionisation detector
HH ratio of hypocholesterolemic to hypercholesterolemic FA

HPLC high-performance liquid chromatography

OIT oxidative induction time

 $K_{232}$  level of primary oxidation products and conjugated dienes  $K_{268}$  level of secondary oxidation products and conjugated trienes

LA linoleic acid
Max the highest value
Mean the mean value

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Min the lowest value

MUFA monounsaturated fatty acid PCA principal component analysis

PDSC pressure differential scanning calorimetry

PDSC  $\tau_{max}$  maximum induction time PUFA polyunsaturated fatty acid

PV peroxide value
SFA saturated fatty acid
TI thrombogenicity index
TOTOX total oxidation index
UFA unsaturated fatty acids

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