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Influence of Drying, Pressing, and Antioxidants on Yield and Oxidative Stability of Cold Pressed Oils

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Abstract

Investigated were the influence of drying and pressing parameters of rapeseed, sunflower, and safflower seeds on oil yield, as well as antioxidant addition on the oxidative stability of cold pressed oils. The parameters of the oilseeds and cold pressed oils quality (moisture content, iodine value, saponification value, peroxide value, free fatty acids, and insoluble impurities content) were determined using standard methods. In terms of oxidative stability, rosemary, green tea, and pomegranate extracts were used as natural antioxidants, and propyl gallate was used as synthetic antioxidant. The results showed that the moisture content of the oilseeds, as well as nozzle size and electromotor frequency of the press had an effect on the yield of cold pressed oil. By reducing the nozzle size and electromotor frequency, the volume of produced cold pressed oil increased. Rosemary extract effectively increased the oxidative stability of sunflower and safflower oils, and green tea extract increased the stability of rapeseed oil. Greater stabilisation of the tested oils was achieved with the addition of 0.4 % of natural antioxidants compared to the addition of 0.2 %. The synthetic antioxidant, propyl gallate, failed to stabilise safflower oil, and unlike the natural antioxidants, it was less protective for rapeseed and sunflower oil.

Keywords

Drying, pressing, cold pressed oil, oxidative stability, antioxidants

1 Introduction

Rapeseed, sunflower, and safflower are widespread oilseeds. While sunflower is mostly used for the production of edible refined oil, and rapeseed for biodiesel production, safflower is best known as a spice. In addition to the mentioned applications, the production of cold pressed oils from these oilseeds is becoming more frequent due to their beneficial effects. In the production of cold pressed oils, the moisture content of the raw oilseeds is of great importance for oil yield. Before pressing or any other process, oilseeds are dried in order to prolong preservation during storage. Sun drying is a process that has been used to preserve food since ancient times. Today, in addition to natural sun drying, industrially controlled drying is more commonly used. However, drying is one of the most energy-intensive processes in industry, the complexity of which is manifested in the simultaneous transfer of mass and heat.[1] For this reason, new, energy-saving drying technologies have been designed. According to *Strømmen et al.*[2], batch-type heat pump dryer can save up to 80 % energy compared to conventional drying at same temperatures. Drying different materials, such as agricultural materials,[3] vegetables and fruits,[4–8] seafood,[9] different seeds,[10,11] *etc.*, using a heat pump, has not only proven energy-efficient but also with no quality change or even better quality of the material itself, compared to conventional drying. However, like almost all technologies, this one has some disadvantages, such as required additional space, demanding maintenance, potential refrigerant leakage, and capital costs.[12,13] The production of cold pressed oil is achieved by using a screw press without conditioning of the raw material or using an organic solvent. Cold pressing of oilseeds enables maximum retention of bioactive compounds, such as essential fatty acids, phenolic and flavonoid substances, tocopherols, tocotrienols, phytosterols, *etc.*,[14] as well as of the characteristic sensory properties of the oils. The by-product of pressing is a cake in which part of the oil, proteins, minerals, fibres, and other ingredients remain.[15,16] Sunflower and safflower oils are quality vegetable oils due to the high content of essential ω-6 linoleic fatty acid (75 and 80 %, respectively), but due to their fatty acid profile they are subject to rapid oxidative spoilage and need to be stabilised. The quality of rapeseed oil is attributed to the essential ω-3 α-linolenic fatty acid whose content of 2–14 % leads to oxidative instability of the oil.[17] Edible vegetable oils are quickly subject to undesirable changes resulting in oil spoilage. A common type of oil spoilage is autoxidation, the rate of which depends on the production process, oil composition, storage conditions, and the presence of ingredients that can accelerate (pro-oxidants) or slow down (antioxidants) oxidation reaction.[18] Primary and secondary oxidation products are formed during oxidative spoilage of the oil,[19,20] and in small quantities give off an unpleasant odour, which impairs the sensory properties of the oil.[21] Knowing the stability of vegetable oils is important to be able to determine the time over which the oil can be preserved from pronounced oxidation, and its shelf life. Today, many methods are used to determine the

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oxidative stability of vegetable oils based on accelerated oil oxidation.[22–25] The stability of vegetable oils can be improved with the addition of antioxidants, synthetic or natural substances that slow down the autoxidation process.[26,27] Today, a variety of plant materials are being investigated that contain bioactive ingredients, such as phenolic compounds, and show significant antioxidant activity in vegetable oils.[28,29] Thus, to protect against oxidative spoilage, the stabilisation of cold pressed oils is aimed at the application of various natural antioxidants (extracts of herbs like green tea, rosemary, sage, *etc.*).[30,31] *Erkan et al.*[32] investigated the antioxidant activity of rosemary extract on oil stabilisation. *Gramza et al.*[33] reported high antioxidant activity of ethanolic green tea extract and black tea extract in sunflower oil. *Hraš et al.*[34] indicated an antioxidant and synergistic effect of rosemary and alpha tocopherol extracts in stabilising sunflower oil. The aim of this study was to examine the influence of drying temperature and cold pressing parameters (nozzle size, electromotor frequency) of rapeseed, sunflower and safflower seeds on oil yield. Additionally investigated was the influence of natural (rosemary, green tea, pomegranate extract), and synthetic (propyl gallate) antioxidants on oxidative stability of the cold pressed oils.

2 Experimental

2.1 Materials

The effects of drying and pressing parameters on the yield of cold pressed oil were determined for rapeseed, sunflower, and safflower seeds obtained from a local family farm. Chloroform and *n*-hexane were purchased from Carlo Erba (France). Potassium iodide and acetic acid were purchased from LabExpert (Slovenia). Iodine monobromide, sodium hydroxide, and ethanol were purchased from Kefo (Slovenia). Sodium thiosulphate, potassium hydroxide, hydrochloric acid, and quartz sand were purchased from Gram Mol (Croatia). Starch was purchased from Kemika (Croatia). Phenolphthalein was purchased from VWR Chemicals (United Kingdom). Diethyl ether was purchased from J. T. Baker (Poland). The effect of antioxidant addition on the oxidative stability of the produced cold pressed oils was determined using natural antioxidants: rosemary extract (type Oxy'Less® CS), green tea extract, and pomegranate extract, manufactured by Naturex (France), and synthetic antioxidant: propyl gallate, purchased from Danisco (Denmark).

2.2 Methods

2.2.1 Determination of initial oil and moisture content

The initial oil content in rapeseed, sunflower, and safflower seeds, as well as the residual oil in cakes was measured by Soxhlet extraction with *n*-hexane as solvent.[35] Moisture content of the oilseeds was determined before and after drying by the thermogravimetric method using moisture analyser MOC-120H, and once again before pressing according to AOAC Official Method 925.40.[36] The measurements were done in triplicate.

2.2.2 Oilseeds drying

Prior to drying, the initial moisture content of the oilseeds was determined. To monitor moisture loss during drying, a moisture analyser MOC-120H was used. Drying was carried out in a low-energy air dryer (detailed characteristics described by *Budžaki et al.*[13]) with 5 kg of each oilseed, at two temperatures, 35 and 45 °C, with a total drying time of one hour. Dryer energy consumption [kW] was recorded during each drying.

2.2.3 Cold pressing

Crude oil was produced from 1.5 kg of rapeseed, sunflower or safflower seeds by cold pressing using a continuous screw press. The pressing parameters, nozzle size (size of the holes of the press head for the cake outlet), and the electromotor frequency were changed to examine the impact on the oil yield. The crude oil was precipitated for 14 days in the dark, followed by vacuum filtration to remove as many insoluble impurities as possible. The amount of obtained cold pressed oil (U, %) was calculated according to Eq. (1)[17].

$$U = U_0 - U_P\left(\frac{a}{b}\right) \qquad (1)$$

U_0 is oil content of the raw material (%), U_P is oil content of the cake (%), a is dry matter of the raw material (%), and b is dry matter of the cake (%). Efficiency of pressing, P (%) was calculated according to Eq. (2):

$$P = \left(\frac{U}{U_0}\right)\cdot 100\ \% \qquad (2)$$

2.2.4 Oil quality parameters determination

Iodine value, saponification value, and free fatty acids of cold pressed oils were determined according to AOAC official methods 920.185, 920.160, and 940.28,[37] and expressed as g I_2/100 g, mg KOH/g, and % of oleic acid. Peroxide value was determined according to ISO 3960[38] and expressed as mmol O_2/kg. Insoluble impurities and moisture in obtained cold press oils were determined according to ISO 663[39] and ISO 662[40], and expressed as %. All determinations were carried out in triplicate.

2.2.5 Oxidative stability of cold pressed oils

The oxidative stability of cold pressed oils was determined by Schaal Oven rapid oil oxidation test.[41] The cold pressed oil samples, with and without added antioxidant, were heated in a thermostat at a constant temperature of 63 °C for four days. During the test, a change in the peroxide value (every 24 h) was observed as a result of accelerated oxidation of the tested oils. Natural antioxidants (rosemary, pomegranate, and green tea extracts) were used in concentrations of 0.2 and 0.4 %, and synthetic (propyl gallate) at a concentration of 0.01 %.

3 Results and discussion

3.1 Oilseeds drying results

According to *Matthäus and Brühl*[42], it is very important to determine the temperature and duration of drying in order to prevent the oilseeds deterioration caused by overheating. Therefore, applying lower drying temperatures can prevent changes in the colour, smell, and taste of the seeds, as well as increase energy savings. According to the literature, the maximum allowable drying temperature for rapeseed and sunflower is 60 °C.[43,44] Two lots of each oilseed, rapeseed, sunflower, and safflower, of different initial moisture contents were dried at temperatures of 35 and 45 °C. Monitored were the times required to dry the oilseeds to the desired final moisture, and the dryer energy consumption. Table 1 shows the drying results, initial and final moisture values of each oilseed, drying temperature, required drying time, and the dryer energy consumption.

Rapeseed, sunflower, and safflower were dried separately at 35 °C to the final moisture content of 6.5, 5.5, and 6.5 %, respectively. The drying results (Table 1) showed that rapeseed, from the initial moisture of 7.34 % to the desired moisture content of 6.32 %, and sunflower, from the initial moisture of 6.58 % to the desired moisture content of 5.63 %, required 1 h of drying. On the other hand, safflower required 0.5 h of drying from the initial moisture of 8.15 % to the desired moisture content of 6.33 %. These results suggest that safflower seeds required only half an hour, or twice less time to lower the moisture to the same content as in rapeseed (6.33 and 6.32 %, respectively), while the initial moisture content in safflower was higher (8.15 and 7.34 %, respectively). The reason for this could be the size of the seed, *i.e.,* the size of the evaporated surface. Namely, safflower seeds are more than twice the size of rapeseed. By drying at 35 °C, safflower seeds lost the largest share of moisture in the shortest time. For all three oilseeds, the consumption of drying energy at 35 °C was 4.4 kW. Subsequently, rapeseed, sunflower, and safflower, of initial moisture of 7.41, 6.02, and 7.48 % respectively, were dried separately at 45 °C to the final moisture of 5.50, 4.50, and 5.00 %. The drying results (Table 1) showed that rapeseed, from the initial moisture of 7.41 % to the desired moisture content of 5.52 %, and sunflower, from the initial moisture of 6.02 % to the desired moisture content of 4.63 %, required 1 h of drying. Safflower required 0.5 h of drying from the initial moisture of 7.48 % to the desired moisture content of 4.87 %. In addition, when dried at 45 °C, safflower seeds lost the highest moisture content in the shortest drying time. For all three oilseeds, the consumption of drying energy at 45 °C was 5.6 kW, which is higher for 35 °C (4.4 kW). Therefore, it can be concluded that it is more energy-efficient to carry out drying at a lower temperature, which is more desirable given the quality of the final dried material. In addition, it can be seen that the drying time in such set conditions is not significantly longer with respect to the desired final moisture content of the oilseeds.

3.2 Influence of pressing parameters

Before pressing, determined were the moisture and oil contents of the dried rapeseed, sunflower and safflower seeds. Table 2 shows the results of monitoring the effect of rapeseed moisture content (7.50 %, 6.62 %, 5.52 %), as well as press nozzle size (7 mm, 11 mm) on oil yield during cold pressing. The pressing of rapeseed with moisture content of 7.50 % and oil content of 35.32 % under pressing conditions: electromotor frequency F = 25 Hz, temperature of press head heater T = 90 °C, and nozzle size N = 11 mm, yielded 380 ml of crude oil, and after precipitation and vacuum filtration, 330 ml of cold pressed oil, while the residual content of oil in the cake was 21.53 %. By reducing the moisture content of the seeds to 6.62 %, with oil content of 35.10 %, and pressing under same pressing conditions, a higher amount of cold pressed rapeseed oil (345 ml) was produced with a smaller oil residue in the cake (20.48 %). Further reduction of the moisture content to 5.52 %, with oil content of 34.75 %, and pressing under the same conditions, yielded a lower amount of crude oil (240 ml) and cold pressed oil (200 ml). The results further showed that the highest volume of cold pressed rapeseed oil (400 ml) and the lowest oil residue in the cake (16.10 %) was achieved at a seed moisture of 5.52 % with a 7-mm nozzle size. Therefore, it could be said that the moisture content of rapeseed as well as the nozzle size had an effect on the oil yield. By pressing of rapeseed using a smaller nozzle at all three initial moisture contents, a higher amount of crude oil and cold pressed oil was produced. Thus, it is evident that the highest oil yield

Table 1 – Oilseeds drying results
Tablica 1 – Rezultati sušenja uljarica

Oilseed Uljarica	Drying temperature Temperatura sušenja / °C	Initial moisture Početna vlaga / %	Final moisture Konačna vlaga / %	Time Vrijeme / h	Energy consumption Potrošnja energije / kW
Rapeseed Uljana repica	35	7.34	6.32	1	4.4
	45	7.41	5.52	1	5.6
Sunflower Suncokret	35	6.58	5.63	1	4.4
	45	6.02	4.63	1	5.6
Safflower Šafranika	35	8.15	6.33	0.5	4.4
	45	7.48	4.87	0.5	5.6

Table 2 – Influence of rapeseed moisture and press nozzle size on oil yield during pressing
Tablica 2 – Utjecaj vlage sjemena uljane repice i veličine otvora glave preše na iskorištenje ulja tijekom prešanja

Moisture content of seed Udio vlage sjemena / %	Oil content of seed Udio ulja u sjemenu / %	*N* / mm	*T* / °C	*F* / Hz	Crude oil Sirovo ulje / ml	Temp. of crude oil Temp. sirovog ulja / °C	Oil Ulje CP-R / ml	Mass of cake Masa pogače / g	Oil content of cake Udio ulja u pogači / %	Moisture content of cake Udio vode u pogači / %	Efficiency of pressing Stupanj djelovanja preše / %
7.50	35.32	11	90	25	380	39	330	1151	21.53	8.38	39.04
		7	90	25	400	42	350	1134	20.27	8.08	42.61
6.62	35.10	11	90	25	395	42	345	1142	20.48	7.08	42.02
		7	90	25	432	42	370	1068	17.58	6.19	50.23
5.52	34.75	11	90	25	240	50	200	1246	26.71	4.83	24.38
		7	90	25	460	54	400	1099	16.10	5.22	54.42

CP-R – cold pressed rapeseed oil/hladno prešano repičino ulje; *N* – nozzle size (defines the diameter of the cake)/veličina otvora glave preše (definira promjer pogače) (mm); *T* – temperature of the press head heater at the outlet of the cake /temperatura grijača glave preše kod izlaza pogače (°C); *F* – frequency regulator (regulates the speed of the press)/frekventni regulator (regulira brzinu pužnice preše) (Hz).

was obtained with a smaller nozzle size precisely because of the higher process pressure during pressing, compared to the larger nozzle size. Additionally, the lowest moisture content (5.52 %) showed the best oil yield, although, according to the literature, the optimum moisture for rapeseed pressing is 6.5–7.5 %, and lower seed moisture may result in lower oil yield.[45] On the other hand, *Siger and Józefiak*[46] carried out cold pressing of rapeseed with initial moisture of 5, 7.5, and 10 %, and recorded the highest oil yield at the lowest seed moisture.

Table 3 shows the results of the influence of moisture in sunflower seeds (6.77 %, 5.60 %, 4.63 %) and the nozzle size (12 mm, 10 mm) during cold pressing on oil yield. Pressing of sunflower seeds with a moisture content of 6.77 % and oil content of 45.17 % with process parameters N = 12 mm, F = 25 Hz, T = 90 °C, yielded 625 ml of crude oil and 520 ml of cold pressed oil, with 18.81 % of residual oil content of the cake. By reducing the seeds moisture content to 5.60 %, with oil content of 43.95 %, and pressing under the same conditions, a lower amount of cold pressed oil (370 ml) was produced with a higher oil residue in the cake (29.99 %). Further reduction of sunflower moisture content to 4.63 %, with oil content of 42.66 %, and pressing under same conditions, yielded an even lower amount of oil (300 ml) and a higher proportion of residual oil in the cake (35.48 %). The highest amount of cold pressed sunflower oil (550 ml) was obtained at seed moisture of 6.77 % and 10-mm nozzle size. The obtained results indicated that the moisture content in sunflower seeds and the nozzle size had an effect on the oil yield. By increasing the moisture content of sunflower seeds, the yield of cold pressed oil also increased. Thus, it could be concluded that, unlike rapeseed, the higher moisture content of sunflower contributed to a higher yield of cold pressed oil. *Bambgoye and Adejumo*[47] reported that higher moisture content leads to greater oil yield from sunflower; however, the pressing process, hot or cold, was not

Table 3 – Influence of sunflower seed moisture content and press nozzle size on oil yield during pressing
Tablica 3 – Utjecaj vlage sjemena suncokreta i veličine otvora glave preše na iskorištenje ulja tijekom prešanja

Moisture content of seed Udio vlage sjemena / %	Oil content of seed Udio ulja u sjemenu / %	*N* / mm	*T* / °C	*F* / Hz	Crude oil Sirovo ulje / ml	Temp. of crude oil Temp. sirovog ulja / °C	Oil Ulje CP-R / ml	Mass of cake Masa pogače / g	Oil content of cake Udio ulja u pogači / %	Moisture content of cake Udio vode u pogači / %	Efficiency of pressing Stupanj djelovanja preše / %
6.77	45.17	12	90	25	625	47	520	936	18.81	7.87	58.36
		10	90	25	640	49	550	926	17.80	8.08	60.59
5.60	43.95	12	90	25	500	53	370	1066	29.99	4.74	33.61
		10	90	25	520	53	420	1028	29.80	4.75	34.03
4.63	42.66	12	90	25	380	51	300	1061	35.48	4.15	21.45
		10	90	25	430	51	320	1108	35.33	4.32	21.78

CP-SF – cold pressed sunflower oil/hladno prešano suncokretovo ulje; *N* – nozzle size (defines the diameter of the cake)/veličina otvora glave preše (definira promjer pogače) (mm); *T* – temperature of the press head heater at the outlet of the cake /temperatura grijača glave preše kod izlaza pogače (°C); *F* – frequency regulator (regulates the speed of the press)/frekventni regulator (regulira brzinu pužnice preše) (Hz).

Table 4 – Influence of safflower seed moisture content, press nozzle size, and electromotor frequency on oil yield during pressing
Tablica 4 – Utjecaj vlage sjemena šafranike, veličine otvora glave preše i frekvencije elektromotora na iskorištenje ulja tijekom prešanja

Moisture content of seed Udio vlage sjemena / %	Oil content of seed Udio ulja u sjemenu / %	*N* / mm	*T* / °C	*F* / Hz	Crude oil Sirovo ulje / ml	Temp. of crude oil Temp. sirovog ulja / °C	Oil Ulje CP-R / ml	Mass of cake Masa pogače / g	Oil content of cake Udio ulja u pogači / %	Moisture content of cake Udio vode u pogači / %	Efficiency of pressing Stupanj djelovanja preše / %
8.37	25.35	11	90	30	290	50	225	1207	10.69	7.42	57.83
		16	90	30	280	50	220	1221	11.89	8.03	53.10
6.41	24.49	16	90	38	285	45	220	1156	10.71	6.16	57.75
		16	90	30	330	48	260	1134	10.33	5.58	59.25
4.87	22.93	16	90	38	270	52	200	1239	15.69	3.42	38.11
		16	90	30	–	–	–	–	–	–	–

CP-SA – cold pressed safflower oil/hladno prešano ulje šafranike; *N* – nozzle size (defines the diameter of the cake)/veličina otvora glave preše (definira promjer pogače) (mm); *T* – temperature of the press head heater at the outlet of the cake /temperatura grijača glave preše kod izlaza pogače (°C); *F* – frequency regulator (regulates the speed of the press)/frekventni regulator (regulira brzinu pužnice preše) (Hz).

specified. *Pedretti et al.*[48], reported the yield of sunflower oil extraction in the range 33–39 % of seed weight at 9 % moisture content. Examining the influence of the nozzle size (12 mm, 10 mm) on the yield of produced cold pressed sunflower oil, it was found that a smaller nozzle size produced a greater amount of sunflower oil at all three moisture contents, which could be explained by the higher pressure generated with a smaller nozzle size.

Table 4 shows the results of testing the effect of safflower seeds moisture content (8.37 %, 6.41 %, 4.87 %), nozzle size (16 mm, 11 mm), and electromotor frequency (30 Hz, 38 Hz) during pressing on oil yield. Pressing of safflower seeds with a moisture content of 8.37 % and oil content of 25.35 %, at N = 16 mm, T = 90 °C, and F = 30 Hz, yielded 280 ml of crude oil and 220 ml of cold pressed oil, with residual oil in the cake of 11.89 %. By reducing the moisture in the seeds to 6.41 %, with oil content of 24.49 %, and pressing under the same conditions, a higher amount of crude oil (330 ml) and cold pressed oil (260 ml) was obtained with a lower proportion of residual oil in the cake (10.33 %). However, when pressing saffron with a moisture content of 4.87 % and oil content of 22.93 %, the press became clogged and stopped working. *Ovsyannikov et al.*[49] reported that, at moisture content less than 8.7 %, the cellular structure of the membrane destroyed by the screw, adsorbed a certain amount of oil, which could not be separated at appropriate pressures. By pressing safflower with a moisture content of 8.37 % at N = 11 mm, F = 30 Hz, and T = 90 °C, a higher amount of cold pressed oil (225 ml) was produced than with the 16-mm nozzle size, which, as with the other two oilseeds, could be explained by increase in pressure. When examining the influence of the electromotor frequency during pressing on the yield of safflower oil, it was observed that, at 38 Hz, a lower amount of oil (220 ml) was produced compared to application of 30 Hz (260 ml). The highest amount of cold pressed safflower oil (260 ml) was obtained at seed moisture of 6.41 %, 16-mm nozzle size, and 30 Hz electromotor frequency. However, according to *Ergönül and Özbek*[50], the optimum safflower seed moisture content for cold pressing is 12 %. *Ovsyannikov et al.*[49] pressed safflower seeds with moisture content of 4.2–16.3 %, and stated that moisture content of the initial seeds had a significant effect on the oil yield; as the moisture content increased, the oil yield increased as well, while the residual oil content of the cake decreased.

3.3 Quality of the produced oil

Chemical characteristics and standard quality parameters of produced cold pressed oils are shown in Table 5.

Table 5 – Quality of the produced oil
Tablica 5 – Kvaliteta proizvedenog ulja

Quality parameter Parametar kvalitete	Rapeseed oil Repičino ulje	Sunflower oil Suncokretovo ulje	Safflower oil Ulje šafranike
Peroxide value Peroksidni broj / mmol O_2/kg	0.46	0.48	3.62
Free fatty acids Slobodne masne kiseline / %	1.37	0.23	9.58
Moisture Vlaga / %	0.14	0.09	0.14
Insoluble impurities Netopljive nečistoće / %	0.44	0.46	0.51
Iodine value Jodni broj / g I_2/100 g	118.95	132.44	143.15
Saponification value Saponifikacijski broj / mg KOH/g	189.87	190.91	187.86

The results obtained by analytical methods showed that the oils were of good quality, which parameters were mostly in accordance with the values prescribed by the Regulations on Edible Oils and Fats.[51] According to the mentioned Regulation, the peroxide value of cold pressed oils must not exceed 7 mmol O_2/kg of the oil, and therefore all three oils meet the standard. Likewise, all three oils meet water content, which must not exceed 0.4 %. Since the maximum allowed value for free fatty acids is 2 %, only safflower oil failed to meet the Regulation, indicating higher acidity of the oil. This acidity is the result of oil spoilage (hydrolytic decomposition) during storage of oilseeds. *Frega et al*.[52] found that the free fatty acids in vegetable oil act as prooxidants, which accelerate the oxidative spoilage of oil, and reduce the stability of the oil to a greater extent. In addition, in all three oils, a higher value of insoluble impurities was observed compared to the Regulations.[51] This proportion of insoluble solid particles from the oil can be reduced by filtration. *Matthaus*[53] indicates that the content of individual ingredients affects the stability of sunflower, rapeseed, and walnut oil. The iodine and saponification values are well in accordance with results in available literature.[54–57]

3.4 Oxidation stability of the produced oil

Table 6 shows the analysis results of the effect of antioxidant addition on the change in oxidative stability of cold pressed rapeseed, sunflower, and safflower oil. The accelerated oil oxidation test was performed by the Schaal Oven test, and the degree of oxidative spoilage expressed as peroxide value. As natural antioxidants, 0.2 and 0.4 % of green tea, rosemary, and pomegranate extracts were used,

Table 6 – Influence of the addition of natural and synthetic antioxidants on the oxidative stability of cold pressed rapeseed, sunflower, and safflower oils

Tablica 6 – Utjecaj dodatka prirodnog i sintetskog antioksidansa na oksidacijsku stabilnost hladno prešanog repičinog, suncokretovog i ulja šafranike

Sample Uzorak	Antioxidant Antioksidans		Peroxide value Peroksidni broj / mmol O_2/kg				
	/%		Day 0 0. dan	Day 1 1. dan	Day 2 2. dan	Day 3 3. dan	Day 4 4. dan
	–			0.48	1.23	1.91	5.97
Rapeseed oil Repičino ulje	Green tea extract Ekstrakt zelenog čaja	0.2	0.46	0.50	1.00	1.49	2.46
		0.4		0.49	0.98	1.21	2.00
	Pomegranate extract Ekstrakt nara	0.2		0.49	1.22	1.96	6.47
		0.4		0.49	1.22	1.95	6.28
	Rosemary extract Ekstrakt ružmarina	0.2		0.49	1.00	1.49	3.40
		0.4		0.49	1.21	1.92	2.89
	Propyl gallate Propil galat	0.01		0.49	0.99	1.19	3.30
Sunflower oil Suncokretovo ulje	–		0.48	1.72	4.87	9.80	17.02
	Green tea extract Ekstrakt zelenog čaja	0.2		1.50	3.42	5.54	7.69
		0.4		1.45	2.88	4.25	6.69
	Pomegranate extract Ekstrakt nara	0.2		1.47	4.70	9.70	14.70
		0.4		1.47	4.90	11.00	15.84
	Rosemary extract Ekstrakt ružmarina	0.2		1.22	2.44	3.41	3.90
		0.4		1.22	1.94	2.71	3.78
	Propyl gallate Propil galat	0.01		1.45	3.18	3.96	6.46
Safflower oil Ulje šafranike	–		3.62	5.00	7.47	8.46	9.95
	Green tea extract Ekstrakt zelenog čaja	0.2		5.00	5.10	6.10	6.66
		0.4		4.78	4.78	4.93	5.18
	Pomegranate extract Ekstrakt nara	0.2		4.98	6.77	9.55	11.65
		0.4		5.02	6.96	8.62	10.78
	Rosemary extract Ekstrakt ružmarina	0.2		4.66	4.99	5.06	5.29
		0.4		4.27	4.37	4.71	5.00
	Propyl gallate Propil galat	0.01		4.33	8.17	10.84	14.36

and as synthetic antioxidant, 0.01 % propyl gallate. Rapeseed oil without added antioxidants (control sample) after 4 days of testing had a peroxide value of 5.97 mmol O_2/kg, indicating good resistance of the oil to oxidative spoilage. The stability of this oil is affected by the fatty acid composition and the high content of γ-tocopherol (423 mg kg^{-1}), which protects against oxidation.[58] *Farahmandfar et al*.[59,60] reported that plant extracts, as well as rice bran extract successfully stabilise rapeseed oil. The addition of 0.2 % green tea extract achieved greater protection of the oil from oxidative spoilage. When using green tea extract, peroxide value after 4 days was 2.46 mmol O_2/kg, a lower value compared to the use of rosemary extract where peroxide value was 3.40 mmol O_2/kg, and propyl gallate where peroxide value was 3.30 mmol O_2/kg. *Gramza et al*.[33] indicated good antioxidant activity of green tea extract in oil stabilisation. Increasing the content of natural antioxidants to 0.4 % resulted in even greater oil stability. The application of pomegranate extract at a content of 0.2 and 0.4 % did not protect rapeseed oil from oxidation, since the peroxide value was higher compared to the control sample. In the case of sunflower oil (linoleic type), after 4 days, a peroxide value of 17.02 mmol O_2/kg was obtained, indicating lower resistance of the oil to oxidative spoilage. This could be due to the high content of linoleic fatty acid (60–70 %) and α-tocopherol (608 mg kg^{-1}).[58] Effective stabilisation of this oil was achieved by applying all tested antioxidants. Rosemary extract added in concentrations of 0.2 and 0.4 % showed the best stabilisation of sunflower oil, where peroxide values of 3.90 and 3.78 mmol O_2/kg were obtained. Green tea extract showed better protection of this oil from oxidative spoilage compared to pomegranate extract, and slightly less than propyl gallate. The oxidative stability of safflower oil was low due to the high content of linoleic fatty acid (75–80 %) and the content of α-tocopherol (400 mg kg^{-1}).[17,23] *Aydeniz et al*.[54] reported that sunflower oil has the highest linoleic acid content among all commercial oils. During the stabilisation of safflower oil, it was observed that rosemary extract, at both concentrations, more effectively protected this oil from oxidation than green tea extract. *Abramović and Abram*[24] find that rosemary extract effectively stabilises undercoat oil. The addition of pomegranate extract and propyl gallate did not protect safflower oil from oxidation; after 4 days of the test, peroxide value was higher than the control sample.

4 Conclusion

Based on the results, it can be concluded that drying plays an important role in the preparation phase of oilseeds for the production of cold pressed oils, in order to achieve the desired moisture content of the material and, accordingly, higher oil yield. Since it is only possible to obtain a quality product from quality raw materials, research has shown that drying can be carried out at lower temperature (35 °C) without significantly more time and energy consumption. By pressing rapeseed dried to a moisture content of 5.52 % and using 7-mm nozzle size, a higher amount of oil was produced with a lower proportion of residual oil in the cake compared to pressing rapeseed of higher moisture contents and using larger nozzle size. By pressing sunflower dried to a moisture content of 6.77 %, and using 10-mm nozzle size, a higher oil yield was obtained in comparison with pressing sunflower with lower moistures and using larger nozzle size. By drying safflower to 6.41 % moisture and pressing at 16-mm nozzle size, and 30 Hz, a higher amount of oil was produced in relation to the higher and lower moisture contents, smaller nozzle size and higher frequency. Generally, higher oil yield was obtained from all three oilseeds by using a smaller nozzle size, precisely due to achieving higher process pressure. On the other hand, regarding the influence of the oilseeds initial moisture content, rapeseed and safflower showed that reduced moisture content resulted in a higher amount of cold pressed oil, while sunflower had a higher yield of cold pressed oil with higher moisture content. The influence of the electromotor frequency was examined only for safflower, where it was observed that a higher amount of oil was obtained at lower electromotor frequency. Natural antioxidants proved better at protecting the oxidative stability of cold pressed oils at higher concentrations, 0.4 %, compared to synthetic propyl gallate, which failed to show satisfactory results in relation to its applied concentration. Rosemary extract proved to be the best at protecting the antioxidant stability of cold pressed sunflower and safflower oils, while green tea yielded the best results in the case of cold pressed rapeseed oil.

List of abbreviations and symbols
Popis kratica i simbola

CP-R – cold pressed rapeseed oil
– hladno prešano repičino ulje

CP-SF – cold pressed sunflower oil
– hladno prešano suncokretovo ulje

CP-SA – cold pressed safflower oil
– hladno prešano ulje šafranike

N – nozzle size, mm
– veličina otvora glave preše, mm

T – temperature of the press head heater at the outlet of the cake, °C
– temperatura grijača glave preše kod izlaza pogače, °C

F – frequency regulator, Hz
– frekventni regulator, Hz

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SAŽETAK

Utjecaj sušenja, prešanja i antioksidansa na prinos i oksidativnu stabilnost hladno prešanih ulja

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U ovom radu istražen je utjecaj parametara sušenja (temperatura) i prešanja (veličina otvora glave preše, frekvencija elektromotora) sjemena uljane repice, suncokreta i šafranike na prinos ulja te dodatka antioksidansa (prirodnog, sintetičkog) na oksidacijsku stabilnost hladno prešanih ulja. Standardnim metodama određeni su parametri kvalitete uljarica (sadržaj vlage) i hladno prešanih ulja (jodni broj, saponifikacijski broj, peroksidni broj, slobodne masne kiseline, vlaga i sadržaj netopljivih nečistoća). Što se tiče oksidacijske stabilnosti, kao prirodni antioksidansi upotrijebljeni su ekstrakti ružmarina, zelenog čaja i nara, a kao sintetski propil galat. Rezultati su pokazali da vlažnost uljarica te veličina otvora glave preše i frekvencija elektromotora utječu na iskorištenje hladno prešanog ulja. Smanjenjem veličine otvora glave preše i frekvencije elektromotora povećava se volumen proizvedenog hladno prešanog ulja. Ekstrakt ružmarina učinkovito je povećao oksidacijsku stabilnost ulja suncokreta i šafranike, a ekstrakt zelenog čaja povećao je stabilnost repičina ulja. Veća stabilizacija ispitivanih ulja postignuta je dodatkom 0,4 % prirodnih antioksidansa u odnosu na dodatak od 0,2 %. Sintetski antioksidans propil galat nije stabilizirao ulje šafranike, a za razliku od upotrijebljenih prirodnih antioksidansa, manje štiti repičino i suncokretovo ulje od oksidacije.

Ključne riječi
Sušenje uljarica, prešanje, hladno prešano ulje, oksidativna stabilnost, antioksidansi

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