Effects of the Contents of Impurities and Seed Hulls on the Quality of Cold-Pressed Sunflower Oil

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Abstract

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The effects of different contents of impurities and seed hulls in the raw material on the sensory characteristics, chemical quality, and oxidative stability of sunflower oil prepared by the procedure of cold pressing on a screw press were investigated. It was found that the presence of impurities (up to 10%) and hulls (up to 32%) had an adverse effect on the sensory and chemical characteristics of the oil. The adverse influence on the oils colour was also evidenced from the results of measuring their transparency, which ranged from 14.75% to 43.60%. The presence of impurities and seed hulls caused also a decrease in the oxidative stability of oils, as the values of the induction period ranged from 3.63 h to 4.63 h, while the Totox values were in the range from 2.25 to 5.87.

Keywords: seed hulls; dehulled kerner; sensory characteristics; chemical quality; oxidative stability; cold-pressed sunflower oil

The growing awareness of people of the importance of healthy way of life and the consumption of foods that have positive effects on their health, i.e. foods rich in protective components, has been accompanied by a constant improvement on the process of edible oil production. In line with this, there appeared the need for the production of unrefined cold-pressed oil, aiming at the preservation and protection of their natural and highly valuable components (Tuberoso *et al.* 2007; Matthäus & Spener 2008).

Edible unrefined oils are produced in small plants, the so-called mini oil mills, using equipment of small or medium capacity and raw materials from the region, and applying simple technology. This assumes oil preparation solely by mechanical pressing on a screw press. After pressing, the oil is additionally purified by filtration or sedimentation, while no further quality improvement by refining is allowed (De Leonardis *et al.* 2003; Matthäus & Spener 2008). Recent technological developments have enabled the manufacture of high-quality unrefined oils to be economically justified (Lanzani *et al.* 1988; Contini & Moresi 1994).

The quality of unrefined oils depends primarily on the nature and quality of the raw material. The production of such oils of especially high quality is considered to be a real challenge to the manufacturers (Matthäus & Brühl 2008). One of the main problems in the production of cold-pressed sunflower oil is the variable quality of the starting raw material (Matthäus 2008) and the presence of hull, which makes about 20–30% of the seed mass (Isobe *et al.* 1992). Sunflower hull is a complex cellulosic material containing also waxes, which pass to the oil in the process of pressing (Turkulov *et al.* 1986; Martini & Añón 2000), and cause visible turbidity during oil storing at lower temperature. Turbid oils are not acceptable in the market and because of that

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they have to be subjected to cold filtration or the starting material has to be dehulled prior to pressing. In the technological procedure of the production of cold-pressed sunflower oil, seed dehulling is considered to be a critical operation. However, there are presently commercially available edible unrefined sunflower oils produced from both nondehulled and dehulled seeds. Since more recently, such oils have often been produced by pressing partly dehulled seed, which causes not only the appearance of oil turbidity, but also affects the sensory properties of the resulting oil, whose aroma is described as "bitter" or "woody". A larger content of seed hulls in the pressed material may also lead to an increase in the temperature during the pressing (Isobe *et al*. 1992; Rass *et al*. 2008).

In the assessment of the quality of edible unrefined oils, of special importance are their sensory characteristics. In contrast to the refined oils, the sensory properties of cold-pressed oils preserve some of the characteristics of the source raw material and thus contribute to the formation of a specific aroma of the food, providing a special gastronomic pleasure (Dimić 2000; De Leonardis *et al*. 2003). Sensory quality of unrefined oils is of essential significance as it points out to the potential omissions, errors, or irregularities in the oil production process ("burned" aroma), due to inappropriate storage of the material to be pressed ("mouldy", "musty" aroma) or prolonged oil storing ("rancid" aroma) (Rass *et al*. 2008).

Accelerated shelf life estimates may be made using sensory or chemical techniques. However, any chemical method used to evaluate the shelf life must be closely correlated with the sensory analysis. Only sensory analysis can detect the flavours resulting from oxidative and nonoxidative degradation processes. No instrumental or chemical analysis can detect all off-flavours. The sensory analysis is ultimately the best method of determining an oil quality and stability due to its unique sensitivity and is critical to realistic shelf life evaluations and accelerated shelf life studies (Warner 1995).

Apart from the quality of the seed, the quality of cold-pressed sunflower oils that are widely present on the market is especially influenced by the presence of impurities and hull in the pressed material (Rass *et al*. 2008). Bearing in mind the fact that sensory and chemical characteristics are related to the oil oxidative stability (Frega *et al*. 1999; Broadbent & Pike 2003), the knowledge of the corresponding parameters is of crucial significance from the point of view of both the producer and the consumer of oil. While, on the other hand, the bad sensory characteristics must not imply a reduced oxidative stability of cold-pressed oils (Dimić 2005). In view of this, the objective of the present study was to investigate the sensory properties, chemical quality, and oxidative stability of cold-pressed sunflower oil depending on the presence of different amounts of impurities and seed hulls in the starting material.

MATERIAL AND METHODS

Material. The investigation was carried out on five samples of cold-pressed oil prepared by pressing domestic linoleic type sunflower hybrid Cepko. The dominant fatty acids contents in the oil composition were as follows: palmitic 5.44%, stearic 4.08%, oleic 32.92%, and linoleic 55.61% wt of total fatty acids. The sunflower seeds were the products of conventional cultivation, and they were stored before processing in silo cell under the conditions of low-temperature and good ventilation for 30 days.

The experiments were carried out in a small scale oil factory equipped with a seed cleaner, a dehuller, and a screw press for processing cold-pressed sunflower oil. Dehulling of the seeds is carried out by an impact dehuller with the removal of the hulls by airflow and gravity. The resulting mixture of seed hulls, dehulled kernels and intact seeds is separated. The hulls are removed by airflow and the dehulled kernels are separated from the intact and broken seeds by gravity.

The oils were produced by pressing the dehulled kernels and the dehulled kernels with the addition of certain amounts of impurities and hulls according to the following experimental plan:

Sample 1: dehulled kernels with 0% impurities and 16% hulls;
Sample 2: dehulled kernels with 5% impurities and 0% hulls;
Sample 3: dehulled kernels with 5% impurities and 32% hulls;
Sample 4: dehulled kernels with 10% impurities and 16% hulls;
Sample 5: dehulled kernels with 0% impurities and 0% hulls.

The impurities consisted of usual admixtures, mainly of organic origin, and fatty dust present in

Table 1. Basic characteristics of sunflower dehulled kernel and impurities

Parameter	Kernel	Impurities
Moisture content (%)	6.90 ± 0.08	11.01 ± 0.11
AV* (mgKOH/g)	0.54 ± 0.01	38.75 ± 0.13
PV* (mmol/kg)	0.27 ± 0.01	15.72 ± 0.80
p-AnV*	0.00 ± 0.00	32.76 ± 0.60

*quality parameters related to oil obtained by cold solvent extraction; AV – acid value; PV – peroxide value; *p*-AnV – *p*-anisidine value

the seed mass. Their basic characteristics along with those of dehulled kernels are given in Table 1. The impurities were taken from the silo and seed cleaner.

Each of the samples of the above composition was pressed in duplicate using 5 kg of the material each time. The pressing was performed on a screw press (Anton Fries, Niedernberg, Germany), capacity of 6–9 kg/h, at the rotation speed of 30–45 rpm. The temperature of the oils at the press outlet was 55–60°C. The pressed oils were kept at room temperature (20–25°C) for 24 h for sedimentation of the residues, then the upper layer was decanted and filtered through ordinary laboratory filter paper. The oil samples were stored in glass bottles at 4°C until their analysis.

Sensory evaluation. The samples were prepared according to the recommended practise for panel sensory evaluation of edible vegetable oils by AOCS Official Method (Cg 2-83:1983). Oil samples (20 ml) were kept in 50-ml closed beakers in an oven at 50 ± 1°C for 30 min, after which a three-member panel of experienced assessors evaluated the oil sensory characteristics (colour, odour, taste, and flavour) for this kind of oil, according to the standard (ISO 5492:1992). The emphasis was put on the intensity of aroma and the development of an atypical flavour. The aroma intensity evaluation was expressed on a 0–5 scale (0 – not perceivable and 5 – strongly perceivable). Descriptive sensory characteristics of the oils were also given. In the frame of the sensory analysis, the visual appearance of the oils was assessed at 22°C.

The turbidity of the oil samples was assessed by the cold test according to the AOCS Official Method (Cc 11-53:1973) and by the clarity test at 4°C. The clarity test was performed as follows: The oil samples were heated to 130°C, then cooled to 0°C and kept at this temperature for 5.5 hours. Immediately after that, the oil samples were heated to 4°C and kept at this temperature for seven days with visual inspection of their clarity after three and seven days.

In addition to the sensory testing, the oil colour was also determined by measuring the transparency at 455 nm (UV/VIS spectrophotometer model T80+; PG Instruments Ltd., London, UK) against carbon tetrachloride as the blank (Dimić & Turkulov 2000).

Moisture content (%) was determined by the conventional method with the sample heating at 103 ± 2°C to the full elimination of water and volatile matter, i.e. to the constant mass of the dry residue (ISO 660:2000).

The peroxide value (PV) (ISO 3960:2001) expressed in mmol/kg, was determined by the reaction of oil and 3:2 chloroform:acetic acid mixture with potassium iodide in darkness. The free iodine was then titrated with a standard thiosulfate solution.

The acid value (AV) (ISO 660:2000), expressed in mg KOH/g, was determined by titration of a solution of oil in 1:1 ethanol:ether with ethanolic solution of potassium hydroxide.

The *p*-anisidine value (*p*-AnV) was determined following the AOCS Official Method (Cg 18-90: 1994), on a UV/VIS spectrophotometer, model T80+ (PG Instruments Ltd., London, UK).

The Totox value was calculated as twice PV plus *p*-AnV.

Determination of oxidative stability. The oxidative stability of oils was investigated by determining the induction period (IP) on a Rancimat 743 apparatus (Metrohm, Herisau, Switzerland) at 110°C and an air flow of 18 l/h (ISO 6886:1996). The portions of oil (2.5 g) were carefully weighed into each of 12 reaction vessels and analysed simultaneously.

The influence of heat on the stability of the oil samples was investigated using Schaal test (Pokorny *et al*. 1985). The amount of 50 g of each oil sample was placed in two open *Petri* dishes, stored in an oven at 63 ± 2°C in the dark, and tested for deterioration after 96 h, whereby the PV was determined. Stability was expressed in % as the change in the peroxide value (CPV) according to the formula (Diraman & Dibeklioğlu 2009): $CPV(\%) = 100\ (PV_2 - PV_1)/PV_1$, where: PV_1 – peroxide value of the sample at the beginning; PV_2 – peroxide value at the end.

All reagents used were of analytical grade (Merck, Darmstadt, Germany).

Statistics. The experimental values were expressed as the means of four determinations (two oil samples in two replicates). Statistical analysis was performed using the Statistica 8 software package. Statistical differences between the oil samples were estimated by applying two-way ANOVA and using the Tukey test at a significance level of 5% ($P < 0.05$)

RESULTS AND DISCUSSION

The influence of different contents of impurities and hulls in the cold-pressed material on the sensory characteristics of oils was evaluated using quantitative descriptive analysis (QDA). The results are graphically displayed as the spider diagrams in Figure 1.

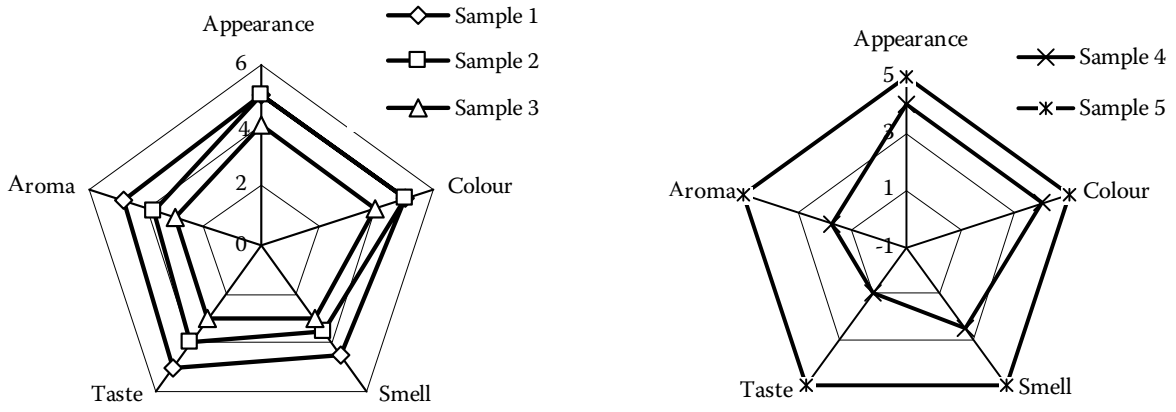
Judging from Figure 1, Sample 5 can be characterised as a "delicious oil" of exceptionally pleasant, specific "sunflower seed-like" smell and taste. This oil has a characteristic rich aroma with optimal note of fresh dried sunflower kernel, which could be expected since it was obtained from high quality dehulled kernels with no hulls and impurities.

Sample 1 was of a somewhat weaker taste and "nutty" aroma of raw sunflower dehulled kernel. Sample 2 had a characteristic aroma of "sunflower seed-like", however, due to the presence of impurities in the raw material for pressing, "sunflower seed-like" smell was poorly expressed, and poorly expressed "woody-like" taste was also detected.

The oils from seeds with hull had nice, pleasant and characteristic "sunflower seed-like" aroma, as indicated by Rass *et al.* (2008). These samples were also found to possess a mild "nutty" taste. However, as to the oil samples with hull (16–32%) and impurities (5–10%), samples 3 and 4, their sensory properties like taste and aroma could be described as exceptionally "bitter" and "woody-like", and they also revealed the occurrence of turbidity. Sample 4, apart from the worst aroma, was also characterised by "pungent" and "astringent" taste. This oil manifested very spurious smell and taste, which masked "sunflower seed-like" smell and taste, and which resulted from larger quantities of hulls and impurities. Having such characteristics, this oil could not be accepted for direct consumption as it does not meet the legislative requirements for the quality of edible oils (Regulation ... 2006).

Based on the results of the oil taste and aroma examination, of it can be concluded that the simultaneous presence of impurities and hull in the starting material for pressing had a markedly pronounced adverse effect on the sensory characteristics of the oils. Samples 3 and 4 had the poorest aroma and worst taste, which is understandable because the presence of certain amounts of impurities and hull in the material for pressing causes the appearance of intensive and unpleasant "bitter" and "woody-like" taste and aroma of cold-pressed sunflower oil, as was already pointed out in the literature (Šmit *et al.* 2005; Rass *et al.* 2008).

As evident from Figure 1, Samples 1, 2, and 5 scored maximum points when the appearance as a parameter of the oil sensory quality was concerned. In contrast to these oils, which were "brilliantly"



Sensory quality of analysed oils had no negative sensory attributes like "roasty", "burned" and "fusty", as it was expected, pointing out that dehulled kernel for pressing was of good quality and that pressing was performed under low temperatures. All oils have characteristic sensory attributes of the raw material used for the preparation of the oils

Figure 1. QDA diagrams of sensory quality of cold-pressed sunflower oils

Table 2. Results of the Cold test and Clarity test of cold pressed sunflower oils

Test	Sample				
	1	2	3	4	5
Cold test at 0°C, 5.5 h	negative	negative	positive	negative	negative
Clarity test					
– at 4°C, 3 days	turbidity* in the form of tiny crystals	totally clear	intense turbidity in the form of large crystals	turbidity in the form of tiny crystals	totally clear
–at 4°C, 7 days	turbidity in the form of tiny crystals	totally clear	intense turbidity in the form of large crystals	turbidity in the form of tiny crystals	totally clear

*term turbidity refers to the visible turbidness characteristic of waxes

clear and transparent, Samples 3 and 4 were slightly turbid, and hence the number of points for their appearance was lower. The comparison of the appearance of the investigated oil samples showed that the contents of 5% impurities and 16% hull had no adverse effect on the appearance of the oil, whereas the simultaneous presence of their higher amounts caused its slight turbidity.

It is known that the processes of cold sedimentation and filtration allow the removal of turbidity-causing matter, and that the content of waxes as the main causative agent of oil turbidity is determined by the content of the hull in the raw material (Careli *et al*. 2002). The presence of this matter in the investigated oil samples could be judged from the results of the Cold test and Clarity test (Table 2). Under the conditions of the Cold test, Samples 1, 2, 4, and 5 remained clear, whereas the result for Sample 3 was positive. In this sample, waxes were probably present at a level exceeding 70 mg/kg, which was the consequence of a high content of hull in the starting material (32%). Namely, according to the own previous results (Turkulov *et al*.1986), the limit of wax content in the sunflower oil that remains clear under the conditions of the Cold test is 70 mg/kg. Irrespective of the fact that particular samples remained clear during the Cold test, the results obtained suggest that, in contrast to impurities, the presence of hull caused oil turbidity after three and seven days at 4°C (Samples 1, 3, and 4), and the intensity of turbidity was proportional to the hull content in the starting material. The oil obtained by pressing pure kernel (Sample 5) remained clear under all conditions of the tests.

The values of the basic indicators of chemical quality of the oils along with the corresponding transparency are listed in Table 3.

Apart from the visual assessment (Figure 1), oil colour was also determined by measuring transparency at 455 nm. Statistical analysis of the results of transparency measurements indicates the existence of significant differences (^{a}) between Samples 1 and 2, prepared from the raw material containing only either hull or impurities, Samples 3 and 4 (^{b}), containing both impurities and hull, and Sample 5 (^{c}), prepared from pure sunflower kernel. The values of the investigated samples transparency varied very much, ranging from 14.75% for the darkest oil (Sample 4, dehulled kernel with 10% of impurities and 16% of hulls) to 43.60% for the most transparent oil (Sample 5, oil from dehulled kernel).

Based on the presented results, a positive correlation can be seen between the transparency values

Table 3. Basic chemical quality parameters and transparency of oil samples

Sample	Transparency (%)	Moisture (%)	AV (mgKOH/g)	PV (mmol/kg)	*p*-AnV	Totox
1	32.76 ± 0.15^{a}	0.03 ± 0.01^{a}	0.26 ± 0.00^{a}	1.26 ± 0.03^{a}	0.00 ± 0.00^{a}	2.52 ± 0.00^{a}
2	36.21 ± 0.08^{a}	0.03 ± 0.01^{a}	0.49 ± 0.01^{b}	1.73 ± 0.02^{b}	0.16 ± 0.01^{b}	3.61 ± 0.01^{b}
3	16.37 ± 0.11^{b}	0.05 ± 0.02^{b}	0.63 ± 0.02^{c}	1.97 ± 0.05^{b}	0.27 ± 0.01^{c}	4.20 ± 0.12^{c}
4	14.75 ± 0.04^{b}	0.08 ± 0.02^{c}	0.74 ± 0.03^{d}	2.17 ± 0.06^{c}	1.53 ± 0.02^{d}	5.87 ± 0.07^{d}
5	43.60 ± 0.10^{c}	0.01 ± 0.00^{d}	0.21 ± 0.00^{e}	1.13 ± 0.00^{a}	0.00 ± 0.00^{a}	2.25 ± 0.01^{a}

Data are reported as means ± SD (n = 4); AV – acid value; PV – peroxide value; *p*-AnV – *p*-anisidine value

$^{a–e}$different superscript letters within columns indicate significant differences ($P < 0.05$) among oil samples

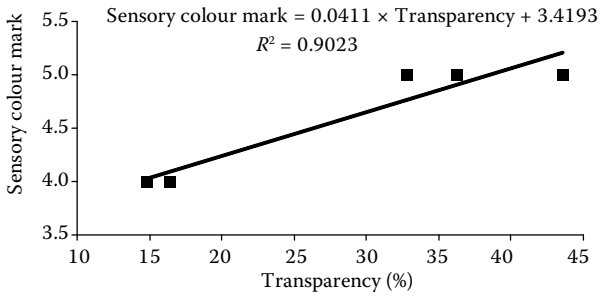


Figure 2. Correlation between the sensory colour mark and oil transparency

(Table 3) and sensory colour mark (Figure 1), with a high value of the correlation coefficient (R^2= 0.9023) – Figure 2.

The moisture contents of the investigated oil samples (Table 3) were in the range from 0.01% in Sample 5 (dehulled sunflower kernel oil) to 0.08% in Sample 4 (dehulled kernel with 10% impurities and 16% hulls), which is far below the maximum tolerable value prescribed by the legislation (Codex 1999).

The acid value is an important parameter of oil quality as it indicates the possible damage of sunflower seed before pressing. The AV of the investigated oils (Table 3) was low, ranging from 0.21 to 0.74 mg KOH/g, which is significantly lower compared to the literature reports quoting the AV for edible unrefined sunflower oil to be in the range from 0.42 to 3.85 mg KOH/g (Cvengroš 1995; De Panfilis *et al*. 1998).

The analysis of the AV results shows that a statistically significant difference ($P < 0.05$) exists between all oil samples. The presence of both impurities and seed hulls in the pressed material exerts an adverse effect as these components cause an increase in the oil acidity. The lowest AV was measured in the oil prepared from dehulled sunflower kernel (0.21 mg KOH/g – Sample 5) and the highest in the oil sample obtained from the material with 10% impurity and 16% hulls (0.74 mg KOH/g – Sample 4). A significant effect of higher contents of impurities in the pressed material on oil acidity could be expected since the AV of impurites was extremely high, 38.75 mg KOH/g.

The effect of the presence of seed hulls on the acidity of pressed oils was reported in the literature (Zheng *et al*. 2003; Šmit *et al.* 2005).

Based on the data obtained for PV, which ranged from 1.13 mmol/kg to 2.17 mmol/kg (Table 3), it may be concluded that the investigated samples of fresh cold-pressed sunflower oil were of a good quality. However, statistical analysis of the PV data showed the existence of significant differences between the investigated oil samples. It was found that the hull content of 16% (Sample 1) had no significantly adverse effect on PV (^{a}), that the presence of 5% impurities (Sample 2) had equally significant (^{b}) adverse effect as the simultaneous presence of 5% impurities and maximum content of 32% hull (Sample 3) (^{b}), as well as that the simultaneous presence of impurities at a level of 5–10% and hulls of 16–32% (Samples 3 and 4) (^{c}) exerted equally significant (^{c}) and as the same time, the strongest effect on the increase of PV.

The *p*-AnV data for the investigated oil samples were up to 1.53 (Table 3), which is significantly lower compared to sunflower oils obtained by extraction, for which this parameter is in the range from 1.46 (Perez *et al*. 2004) to 4.99 (Anjum *et al*. 2006).

According to the results of the Rancimat test, the IP values (at 110°C) of the investigated oil samples (Table 4) were in the range from 3.63 h to 4.63 h, which is somewhat higher compared to the findings of De Leonardis *et al*. (2003), who reported a value of 2.63 hours.

By comparing the IP values of the investigated oil samples, it can be concluded that the presence of impurities and hull in the starting material had a markedly adverse effect on the oxidative stability of cold-pressed sunflower oils. The longest induction period of 4.63 h and 4.60 h was found with Samples 5 and 1, respectively, i.e. the oils obtained by pressing the material containing no impurities. On the other hand, the shortest IP of 3.63 h was measured for Sample 3, with 5% impurities and maximum content of hull, 32%. The observed unfavourable effect of hull on the oxidative stability of cold-pressed sunflower oil is probably a result of the interaction with impurities present in the raw material for pressing, which is in agreement with the literature findings (Šmit *et al.* 2005). However, there are also opposite opinions, e.g. De Leonardis *et al*. (2005) reported that sunflower hull had a positive effect on the oxidative stability of the resulting oil.

The oxidative stability of the oil samples increased with the decrease in the contents of free fatty acids and primary oxidation products, which is supported by the values of the coefficients of correlation between the IP and AV (R^2 = 0.62) and between IP and PV (R^2 = 0.64) (Tables 3 and 4). The obtained values of the correlation coefficients agree well with those reported by Vidrih *et al*. (2010).

The results of the Schaal test (Table 4) indicate that the thermostating of the oils at 63 ± 2°C for

Table 4. Oxidative stability of investigated oil samples

Sample	Rancimat test IP (h)	Schaal-test		
		PV* (mmol/kg)	CPV × 10^2 (%)	Totox*
1	4.60 ± 0.04[a]	13.99 ± 0.46[a]	10.10	27.97 ± 0.03
2	4.00 ± 0.22[b]	14.36 ± 0.10[a]	7.30	29.39 ± 0.19
3	3.63 ± 0.06[c]	15.48 ± 0.23[b]	6.86	31.82 ± 0.46
4	4.15 ± 0.14[d]	18.13 ± 0.04[c]	7.35	38.21 ± 0.04
5	4.63 ± 0.03[a]	12.36 ± 0.08[d]	9.94	24.72 ± 0.16

*values determined after holding the oil at 63 ± 2°C for 96 h

[a–d]different superscript letters within columns indicate significant differences ($P < 0.05$) among oil samples

96 h caused a significant increase in the PV. The final peroxide values were found to be 6.86-fold to 10.10-fold higher than the initial PV. The CPV data ranged from 686% to 1010%. The CPV data show that the least change in the PV was observed for Sample 3, obtained by pressing the material having 5% impurities and 32% hulls, whereas the highest increase was found for the oil sample originating from the material without impurities and with 16% hulls. It should be pointed out that the data for the oxidative stability of the investigated oils obtained by the Schaal and Rancimat tests (Table 4) are in correlation ($R^2 = 0.51$).

A good positive correlation was also found between the data for the oxidative stability obtained by Schaal test and the results of sensory analysis. Namely, the oil samples having best sensory characteristics exhibited at the same time the highest oxidative stability.

An insight into the oxidative state of the oil samples was obtained based on the Totox values. The initial Totox values were in the range from 2.25 (Sample 5) to 5.87 (Sample 4) (Table 3). The Totox values after the four-day oil thermostating at 63 ± 2°C ranged from 24.72 (Sample 5) to 38.21 (Sample 4) (Table 4). By comparing the Totox values before and after thermostating, a good positive correlation can be seen between them, i.e. the oil sample exhibiting the highest oxidative stability was also most stable on heating.

CONCLUSION

The contents of both impurities and hull in the pressed material have a very adverse effect on the sensory characteristics and chemical quality of the cold-pressed sunflower oils, the effect of impurities being much more pronounced. Simultaneous presence of impurities and hull is the most unfavourable. The oil aroma (i.e. the smell and taste) depends far more on the presence of impurities and hull than the appearance and colour of the oil. The latter two parameters are almost identically influenced by the presence of impurities and hull in the starting material.

The results obtained show that the presence of impurities and hulls had also a negative effect on the oxidative stability of the investigated oil samples.

The highest quality of the cold-pressed sunflower oil was obtained by pressing the dehulled kernel with no impurities and hulls.

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