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# Rapid adulteration detection of cold pressed oils with their refined versions by UV–Vis spectroscopy

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The aim of this study is the rapid detection of food pressed oils adulteration with their refined versions, using UV–Vis spectroscopy. The study investigates some common oil physico-chemical parameters such are: density, viscosity, refractive index, acid index, peroxide value, saponification index, to detect differences between cold pressed oils versus refined ones, for some food-grade oils found on Romanian market, as well as FT-IR spectroscopy and GC–MS analytical method, obtaining similar results to those presented in the literature data. The difference between some of the obtained results is not relevant for telling the cold-pressed oils from their refined version for adulteration investigation purpose. Colour analysis instead is a very good method to differentiate a cold pressed oil from a refined one. Taking this into account, the cold pressed oils and their refined versions were mixed in different proportions, and their colour properties were analyzed, obtaining linear dependences for  $a^*$  and  $b^*$  CIE  $L^*a^*b^*$  parameters with cold pressed oil content in the mixture. Dependence equations were proposed.

Beans and seeds are the most important vegetable oil sources. Some oils are obtained from cold pressing technology, which is environmentally friendly, preserves the nutrients in the oils and is easy to perform. Cold pressed oils contain natural ingredients with numerous health benefits. There are different methods to obtain refined oils in food industry. By refining technologies, the undesirable materials may be removed along with some valuable components<sup>1,2</sup>. Quality differences of cold pressed vs. refined oils was reported<sup>3,4</sup>. Some physico-chemical parameters, such are: density, acid index, peroxide value, viscosity, or GC–MS (gas chromatography mass spectrometry) detection may be used to distinguish a cold pressed oil from a refined one<sup>3</sup>. Fatty acids composition of vegetable oils<sup>5–9</sup>, influences the human health, lipids being among fundamental nutrients<sup>10</sup>.

Price of food oils depends on their quality and purity, cold pressed oils being more expensive, that is why labels on oil bottles must mention whether it is a crude or a refined oil. Cold pressed oils are frequently subjected to fraud by mixing with different other seeds oils with inferior quality. Standards and rules are introduced by international quality control organizations to detect and prevent food oil falsification<sup>11</sup>. Some authors proposed methods to investigate these adulterations, such are: three-dimensional fluorescence spectroscopy<sup>12</sup>, UV–IMS and chemometric analysis<sup>13</sup>, fluorescence quenching method with aqueous CTAB-coated quantum dots<sup>14</sup>, confocal X-ray scattering analysis with coherent/incoherent scattered X-rays<sup>15</sup>, near-infrared spectroscopy and chemometric techniques<sup>16</sup>, optical thin-film biosensor chips<sup>17</sup>, or stimulated Brillouin scattering in combination with visible absorption spectroscopy<sup>18</sup>.

Oil colour is an important property for consumers<sup>19</sup> and a good indicator of oil quality. Yet, this feature was presented only in few articles<sup>7,20</sup>.

To the best of our knowledge, there are no reports on detecting adulteration of cold pressed oils with their refined versions, or the use of CIE  $L^*a^*b^*$  colour space in UV–Vis spectroscopy method for adulteration detection.

The purpose of this paper is to highlight that UV–Vis spectroscopy is a rapid and facile method for detecting adulteration of some cold pressed oils with their refined versions in different proportions, comparing to some other known and used methods.

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	Density, 20 °C g/cm <sup>3</sup>	Viscosity, cP, at 25 °C	Refractive index, at 25 °C	Acid index, mgKOH/g	Peroxide value, mEqO <sub>2</sub> /kg	Saponification index, mgKOH/g
<b>Coconut oil</b>						
CP	0.921 <sup>a</sup>	0.446 <sup>a</sup>	1.449 <sup>a</sup>	1.23 ± 0.07	7.89 ± 0.08	266 ± 3
R	0.918 <sup>a</sup>	0.367 <sup>a</sup>	1.450 <sup>a</sup>	0.50 ± 0.04	5.76 ± 0.05	256 ± 2
<b>Sunflower oil</b>						
CP	0.919	0.508	1.476	0.99 ± 0.02	4.95 ± 0.04	192 ± 3
R	0.917	0.497	1.477	0.60 ± 0.03	2.15 ± 0.03	188 ± 2
<b>Grapeseed oil</b>						
CP	0.929	0.464	1.478	1.86 ± 0.04	6.38 ± 0.06	192 ± 1
R	0.926	0.443	1.479	0.62 ± 0.02	2.82 ± 0.02	189 ± 2
<b>Canola oil</b>						
CP	0.921	0.563	1.474	1.32 ± 0.03	1.75 ± 0.04	191 ± 2
R	0.915	0.546	1.475	0.48 ± 0.02	0.68 ± 0.02	187 ± 1

**Table 1.** Physico-chemical properties of the cold pressed (CP) and the refined (R) oils. <sup>a</sup>Values measured at 40 °C.

Type of oil	Content [mmol/L]				
	Methyl laurate (t <sub>R</sub> = 19.32 min)	Methyl miristate (t <sub>R</sub> = 25.31 min)	Methyl oleate (t <sub>R</sub> = 37.80 min)	Methyl linoleate (t <sub>R</sub> = 39.09 min)	Methyl linolenate (t <sub>R</sub> = 41.00 min)
<b>Coconut</b>					
CP	41.17	122.85	8.31	3.65	0.00
R	40.51	116.21	4.86	4.37	0.00
<b>Sunflower</b>					
CP	2.22	0.00	17.17	56.40	0.00
R	29.63	88.14	7.52	3.59	0.00
<b>Grapeseed</b>					
CP	0.00	0.00	22.12	147.61	6.89
R	0.00	0.00	11.42	27.85	2.86
<b>Canola</b>					
CP	0.00	0.00	55.13	16.74	7.65
R	0.00	0.00	6.19	4.13	2.85

**Table 2.** Fatty oil composition of cold pressed (CP) and refined (R) oils.

## Results and discussion

**Physico-chemical parameters.** Table 1 shows that physico-chemical parameters regarding density, viscosity, acid index, peroxide value, saponification index for the oils are similar to other results presented in literature<sup>3,4,7,20–24</sup>. Refractive indexes differ only for the last decimal between the cold pressed oils and the refined ones, being higher for the refined oils, all the other physico-chemical parameters are higher for cold pressed oils.

The results being very close to one another, and requiring time and chemical reagents, these physico-chemical parameters may not be used successfully for distinguishing a cold pressed oil from a refined one, and for the adulteration detection purposes.

**FT-IR spectra.** FT-IR spectral correlations for different oils are not reported in the literature. That is why we wanted to emphasize if this analysis is suitable for differentiating the cold-pressed and refined oils. The results of FT-IR spectral analyses for the cold pressed and refined oils are as follows:

*Coconut oil* is a rich source of short- and medium-chain saturated fatty acids account for 70% of these fatty acids and it has a low content of unsaturated fatty acids with a negligible content of both ω-6 and ω-3 polyunsaturated fatty acids<sup>7</sup>.

Due to the low content of oleic acid (<5%) and linoleic acid (<1%), in the coconut oil, the carbon-carbon double bonds characteristic of the FT-IR spectroscopy at 1654 cm<sup>-1</sup> assigned to stretching vibration ν<sub>C=C</sub> could not be revealed. However, a small shoulder may be observed at 3,008 cm<sup>-1</sup> that can be assigned to stretching vibration of the olefinic carbon-hydrogen bond ν<sub>C-H</sub><sup>25,26</sup>.

*Sunflower oil* are rich in mono- (19.5%) and polyunsaturated (65.7%) fatty acids<sup>3,7</sup>. The FT-IR spectra of sunflower oils present the same characteristic bands of glycerol fatty saturated esters found in the coconut.

*Grapeseed oil* are rich in mono- (16.1%) and polyunsaturated (69.9%) fatty acids.

*Canola oil* are rich in mono- (63.3%) and polyunsaturated (28.1%) fatty acids<sup>27</sup>.

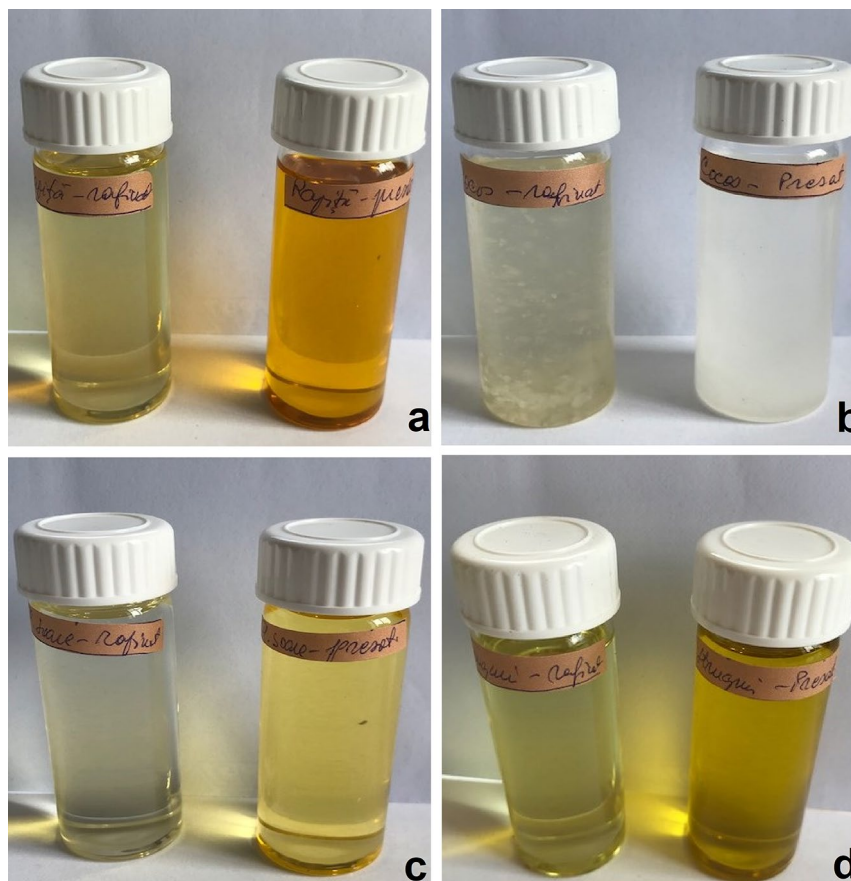
Illuminant	Canola oil	$L^*$	$a^*$	$b^*$
D65	R	88.89	-2.34	8.86
	CP	83.99	3.63	33.02
A	R	89.23	0.12	8.39
	CP	86.31	6.32	36.38
F2	R	89.16	-1.68	10.07
	CP	86.33	0.81	36.97
Illuminant	Coconut oil	$L^*$	$a^*$	$b^*$
D65	R	90.34	-0.78	2.91
	CP	90.96	-0.26	0.61
A	R	90.46	0.07	2.76
	CP	90.72	-0.06	0.56
F2	R	90.44	-0.6	3.33
	CP	90.97	-0.22	0.71
Illuminant	Sunflower oil	$L^*$	$a^*$	$b^*$
D65	R	90.5	-0.88	2.29
	CP	90.23	-1.39	14.35
A	R	90.56	-0.18	2.09
	CP	91.02	0.92	15.29
F2	R	90.56	-0.66	2.64
	CP	90.95	-1.38	15.51
Illuminant	Grapeseed oil	$L^*$	$a^*$	$b^*$
D65	R	90.51	-2.51	7.24
	CP	87.2	-1.55	23.23
A	R	90.72	-0.38	6.64
	CP	88.47	1.9	24.53
F2	R	90.7	-1.77	8.29
	CP	88.38	-1.67	25.37

**Table 3.** Colour parameters of all refined (R) and cold pressed (CP) oils for different illuminants.

The FT-IR spectra of sunflower, grapeseed and canola oils present the same characteristic bands of glycerol fatty saturated esters found in the coconut.

- Characteristic bands for esters groups: intense band at  $1746-7\text{ cm}^{-1}$  assigned to stretching vibration of the carbon-oxygen double bond  $\nu_{\text{C=O}}$  in aliphatic esters; medium band at  $1237-8$  and  $1163\text{ cm}^{-1}$  assigned to stretching vibration of the carbon-oxygen single bond  $\nu_{\text{O=C-O}}$  in aliphatic esters; medium band at  $1099-1119\text{ cm}^{-1}$  assigned to stretching vibration of the aliphatic tetrahedral carbon-oxygen bond  $\nu_{\text{C-O}}$  in aliphatic esters<sup>25,26</sup>.
- Characteristic bands for alkyl groups ( $\text{CH}_2$  and  $\text{CH}$  in glycerol;  $\text{CH}_2$  and  $\text{CH}_3$  in saturated fatty acids): intense bands at  $2924-7\text{ cm}^{-1}$  and a shoulder at  $2953-5\text{ cm}^{-1}$  assigned to antisymmetric/symmetric stretching vibration of the aliphatic tetrahedral carbon-hydrogen bond  $\nu^{\text{as}}_{\text{C-H}}$  and  $2854-5\text{ cm}^{-1}$   $\nu^{\text{s}}_{\text{C-H}}$  aliphatic; medium bands at  $1463-5\text{ cm}^{-1}$  and a weak band at  $1416-9\text{ cm}^{-1}$  assigned to an antisymmetric bending in-plane deformation of the aliphatic tetrahedral carbon-hydrogen bond  $\delta^{\text{as}}_{\text{C-H}}$ , and at  $1376\text{ cm}^{-1}$  assigned to a symmetric bending in-plane deformation of the aliphatic tetrahedral carbon-hydrogen bond  $\delta^{\text{s}}_{\text{C-H}}$  aliphatic; the weak bands at  $1031-4$ ,  $962-7$ ,  $912\text{ cm}^{-1}$  are not characteristic ones, being attributed to stretching vibration of the aliphatic tetrahedral carbon-carbon bond,  $\nu_{\text{C-C}}$ , to skeletal vibrations for aliphatic groups, and a medium band at  $722-3\text{ cm}^{-1}$  assigned to scissoring out-of-plane deformation for the aliphatic groups  $\text{CH}_2$ ,  $\text{CH}_3$  and  $\text{CH}$ ,  $\gamma_{\text{CH}}$ <sup>26,28</sup>.
- In addition, the absorption bands characteristic of (*cis*) carbon-carbon double bonds of mono- and poly-unsaturated fatty acids present in higher concentrations are also highlighted: weak-medium band at  $3007-9\text{ cm}^{-1}$  assigned to stretching vibration of the olefinic carbon-hydrogen ( $=\text{CH}-$ ) bond  $\nu_{\text{C-H}}$ ; weak band at  $1652-4\text{ cm}^{-1}$  assigned to stretching vibration of the carbon-carbon double bond  $\nu_{\text{C=C}}$  *cis*-olefinic; a weak band at  $1416-9\text{ cm}^{-1}$  assigned to bending in-plane deformation of the olefinic carbon-hydrogen bond  $\delta_{\text{C-H}}$ , and a weak band at  $722-3\text{ cm}^{-1}$  assigned to scissoring out-of-plane deformation for the olefinic group  $\text{CH}$ ,  $\gamma_{\text{CH}}$ <sup>25,26</sup>.

With the exception of grape seed oil, both in sunflower oil and canola oil, comparing the intensity of the two characteristic bands typical for olefinic groups: at  $3007\text{ cm}^{-1}$   $\nu_{\text{C-H}}$  olefinic and  $1653\text{ cm}^{-1}$   $\nu_{\text{C=C}}$ , FT-IR spectra indicate a lower overall concentration of unsaturated fatty acids of refined oils compared to those cold pressed.



**Figure 1.** Visual colour difference between refined (left) and cold pressed (right) oils: (a) Canola; (b) coconut; (c) sunflower; (d) grapeseed.

The purpose of this paper was to check if using this analytical method, notable differences between cold pressed and adulterated oils with refined oils by the same type may be observed, and not to quantitatively identify the content in saturated and unsaturated fatty acids (respectively the ratio between them). It may be noticed that the informations provided by the FT-IR spectra cannot be used in establishing adulteration of a cold-pressed oil with its refined one.

**GC-MS analysis.** GC-MS analysis is widely used to determine fatty acid composition of different oils<sup>7,10,23,27-33</sup>.

By the hydrolysis of oils, followed by the derivatization of fatty acids as methyl esters<sup>34,35</sup>, the oil composition of cold pressed (CP) and refined (R) ones was determined by GC-MS (Table 2).

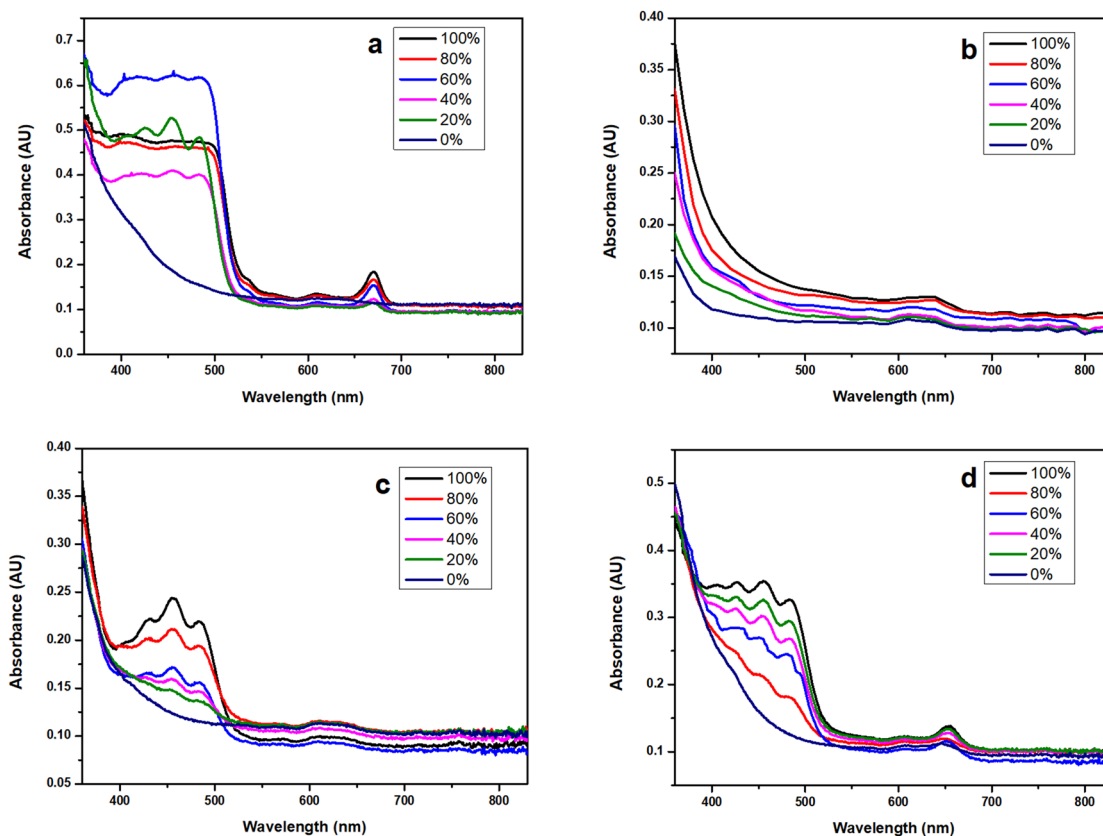
For the three types of oils rich in unsaturated fatty acids: sunflower, grapeseed and Canola oils, concentrations in oleic acid ( $\omega$ -9), linoleic acid ( $\omega$ -6) and linolenic acid ( $\omega$ -3) were significantly lower in refined oils compared to cold pressed ones, so this method may be used for differentiating a cold pressed oil from a refined one, but it is a complex method, requiring derivatization of oils, and calculation of fatty oil composition from chromatograms.

**Oil colour and metamerism effect.** As known, colour sensation results from combining the following factors, which are a lighting source, an object and an observer. The lighting source physically exists, and its spectral energetic distribution may be measured. CIE (International Illuminating Committee) introduced some standard illuminants, among them being CIE A – incandescent light, CIE D65 – white natural light and CIE F2 – cold fluorescent.

Two coloured surfaces can stimulate all three centers of excitation of the eye to the same extent under a specific illumination, but not under a different light source, appearing identical in the first case, and different in the second case, the phenomenon being called metamerism<sup>36,37</sup>.

Food oils may be observed under different illuminants, depending on where they are displayed in the supermarkets, resulting different colour parameters (Table 3), fact that indicates that food oils present the metamerism effect.

When using the D65 illuminant (assimilated to natural open-air daylight), all the refined oils except the coconut oil, present higher values for lightness than cold pressed ones, which may also be observed by the naked eye (Fig. 1).



**Figure 2.** Absorbance spectra of cold pressed oils adulterated with refined ones (percentage of cold pressed oil): (a) Canola; (b) coconut; (c) sunflower; (d) grapeseed.

Regarding the  $a^*$  parameter value, for the cold pressed Canola oil it is in the red domain but for the refined one is in the green domain; for all the other cold pressed and refined oils,  $a^*$  parameter value is in the green domain. The values for the  $a^*$  parameter are three times higher for the refined coconut oil than for the cold pressed one, two times higher for the refined grapeseed oil than for the cold pressed one, and double for the cold pressed sunflower oil as compared to that of the refined one.

All oils have the  $b^*$  parameter value in the yellow domain, presenting great differences in value between cold pressed oils and the refined ones: four times higher for the cold pressed Canola oil, six times higher for the refined coconut oil, seven times higher for the cold pressed sunflower oil, and three times higher for the cold pressed grapeseed oil.

**Colour study of adulteration of food oils with the refined ones.** Colour measure being a good method for differentiating cold pressed oils and refined ones we propose it as a rapid detection method for adulteration of cold pressed oils with their refined versions in different proportions (mass%).

Colour analyses of this adulteration reveal that the absorbance spectra (Fig. 2) and the CIE  $L^*a^*b^*$  parameters (Fig. 3) differ with the cold pressed oil content in the mixture.

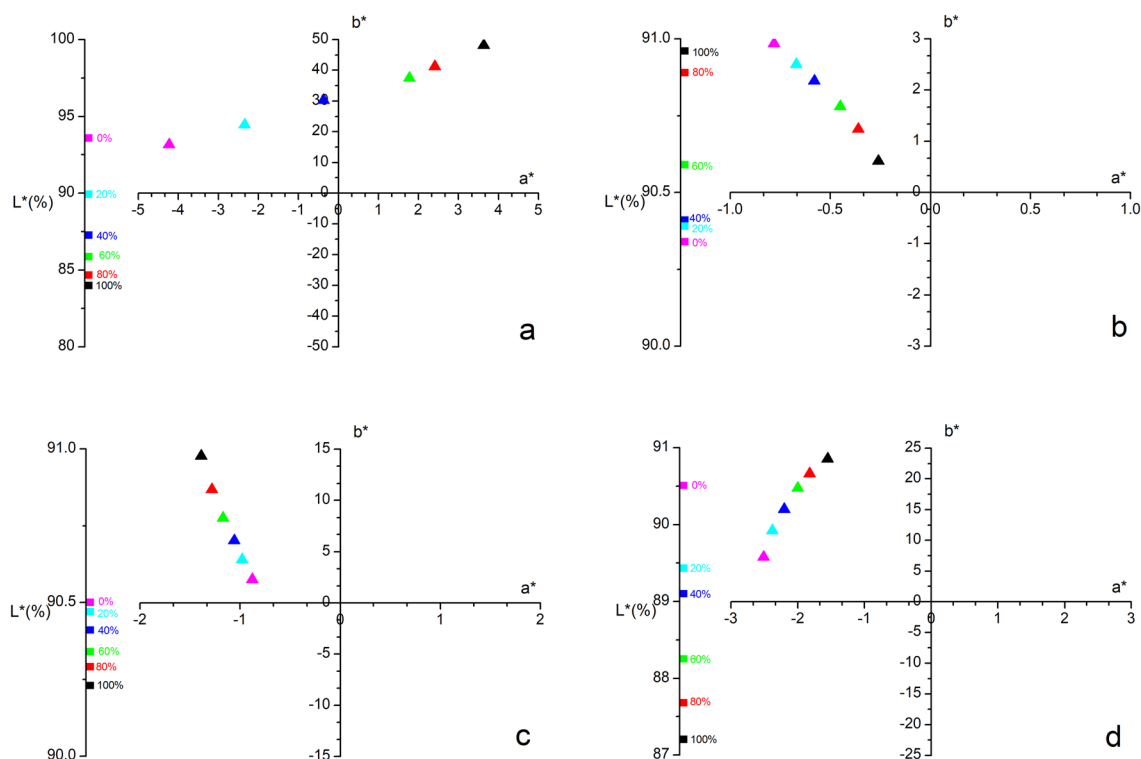
Absorbance spectra of cold pressed oils present a maximum at about 650 nm for all oils, and, except coconut oil, triplets at 450–500 nm<sup>36</sup>. These maxima do not appear in any of the refined oils. When adulterating cold pressed oils with refined ones, these maxima appear on the absorbance spectra, but they fade out as the percentage of refined oil adulteration increases.

CIE  $L^*a^*b^*$  parameters of all oil mixtures (Fig. 3) show that the lightness increases when adding the refined oil to the cold pressed one, as expected, except for the coconut oil (Fig. 1). The  $a^*$  and  $b^*$  parameters have a linear dependence for all the studied oils (Fig. 4), with the equations presented in Table 4. The correlation coefficient  $R^2$  is 0.99 for all equations.

With these established equations, the amount of cold pressed oil in a product found in the supermarkets may be calculated, after determining the CIE  $L^*a^*b^*$  parameters by UV–Vis spectroscopy.

## Materials and methods

Four different cold pressed oils and their refined versions were purchased from the Romanian market: coconut oil, sunflower oil, grapeseed oil and Canola oil, for a period of three years. Every year we purchased all oils under the same brand names, in order to compare the results. Because the properties of the oils were similar, in this paper we only presented one example for each oil purchased in the last year of the research. The chemicals used in this study were of analytical grade.



**Figure 3.** CIE  $L^*a^*b^*$  parameters of cold pressed oils adulterated with refined ones (percentage of cold pressed oil): (a) Canola; (b) coconut; (c) sunflower; (d) grapeseed.

Density was determined using pycnometer method.

Refraction index was determined using an Abbe-Zeiss refractometer.

Acid number was determined according to ISO 660: 2009 method.

Peroxide value was determined according to ISO 3960: 2017 method.

Viscosity was determined using a Brookfield CAP 2000<sup>+</sup> L viscosimeter.

FT-IR spectra were recorded by the film working technique, with KBr pellets, using a Jascow FT-IR-430 spectrophotometer, at a resolution of  $4\text{ cm}^{-1}$ .

The chromatograms and the mass spectra corresponding to the chromatographic peaks were recorded using a GC-MS Thermo Scientific System TRACE 1310, ITQ 1100 Ion Trap MS. The column used was TG-WAXMS  $30 \times 0.25\text{ mm} \times 0.5\text{ }\mu\text{m}$ , Thermo Scientific. Temperature program:  $80\text{ }^\circ\text{C}$  (1 min)  $150\text{ }^\circ\text{C}$  (0.5 min) -  $240\text{ }^\circ\text{C}$  (0.5 min)/ $3\text{ }^\circ\text{C}$ ,  $240\text{--}300\text{ }^\circ\text{C}$  (2 min)/ $7\text{ }^\circ\text{C}/\text{min}$ . Injector temperature  $250\text{ }^\circ\text{C}$ , helium flow rate  $1\text{ mL}/\text{min}$ . MS parameters were: transfer line temperature at  $310\text{ }^\circ\text{C}$ , reading range  $30\text{--}700\text{ m/z}$ . Hexadecane was used as internal standard.

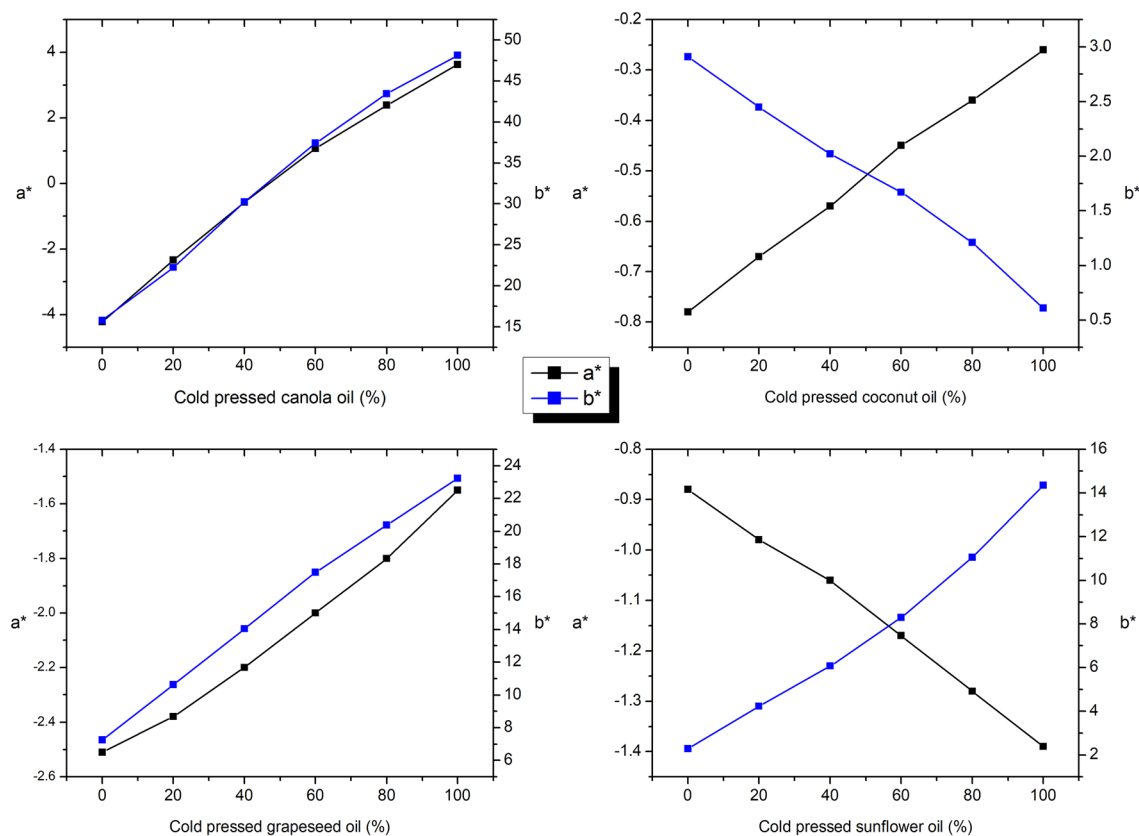
Colour analysis was conducted using a Cary-Varian 300 Bio UV-VIS colourimeter with integrating sphere, using a Spectralon standard and three illuminants: D65, A and F2. All colour data were expressed by  $L^*$ ,  $a^*$ ,  $b^*$  coordinates, where  $L^*$  corresponds to lightness;  $a^*$  corresponds to the transition from green ( $-a^*$ ) to red ( $+a^*$ ); and  $b^*$  corresponds to the transition from blue ( $-b^*$ ) to yellow ( $+b^*$ ).

**Additional Information.** In order to verify our theory, colour analysis was also performed on the same four different cold pressed oils (coconut oil, sunflower oil, grapeseed oil and Canola oil) and their refined versions, which were purchased under different brand names. All the results are presented in the Supplementary Information.

The proposed equations for the  $a^*$  and  $b^*$  parameters for the oils purchased under the same brand names are similar to the ones proposed for the oils purchased under different brand names.

## Conclusions

This study describes some methods for differentiating cold pressed food-grade oils from the refined ones. Classical physico-chemical properties, such as density, viscosity, acid index, saponification index, peroxide value, or refractive index present very similar values for the tested cold-pressed oils and their refined versions, so these physico-chemical parameters may not be successfully used for distinguishing a cold pressed oil from a refined one, and for the adulteration detection purposes. FT-IR cannot be used in establishing adulteration of cold-pressed oil with a refined one, because there are no noticeable differences between FT-IR spectra of the cold-pressed oils and of their refined versions. GC-MS analysis may be used for differentiating a cold pressed oil from a refined one, but it is a complex method, requiring derivatization of oils, and calculation of fatty oil composition. Colour difference between the cold pressed oils and the refined ones may be visually appreciated



**Figure 4.** Dependence of  $a^*$  and  $b^*$  parameters on cold pressed oil content (mass%).

Cold pressed oil	$a^*$		$b^*$	
	Intercept	Slope	Intercept	Slope
Canola	$-3.95 \pm 0.11$	$0.079 \pm 0.003$	$16.25 \pm 1.32$	$0.332 \pm 0.002$
Coconut	$-0.78 \pm 0.07$	$0.005 \pm 0.001$	$2.92 \pm 0.91$	$-0.022 \pm 0.012$
Sunflower	$-0.87 \pm 0.05$	$-0.005 \pm 0.002$	$1.79 \pm 0.4$	$0.119 \pm 0.021$
Grapeseed	$-2.55 \pm 0.7$	$0.010 \pm 0.0005$	$7.45 \pm 0.63$	$0.161 \pm 0.0005$

**Table 4.** Dependence equations for  $a^*$  and  $b^*$  on cold pressed oil content (mass%) in the mixtures.

and determined by UV–Vis spectroscopy. For this reason, this last investigation technique was proposed as a rapid method for appreciating adulteration of cold pressed oils with refined ones. When adulterating cold pressed oils with refined ones, the maxima in the absorbance spectra fade out as the percentage of refined oil adulteration increases.

Regarding CIE  $L^*a^*b^*$  parameters, dependence equations for  $a^*$  and  $b^*$  on cold pressed oil content (mass%) were proposed, that may be used to calculate the amount of cold pressed oil in a product.

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## References

- Gunstone, F. D. Production and trade of vegetable oils. In *Vegetable Oils in Food Technology: Composition, Properties and Uses* (ed. Gunstone, F. D.) 1–17 (CRC Press, Boca Raton, 2002).
- Pal, U. S., Patra, R. K., Sahoo, N. R., Bakhara, C. K. & Panda, M. K. Effect of refining on quality and composition of sunflower oil. *J. Food Sci. Technol.* **52**(7), 4613–4618. <https://doi.org/10.1007/s13197-014-1461-0> (2015).
- Pavlovskaa, G., Jankuloska, V., Knights, V. A. & Stojanova, E. Differences in chemical parameters of cold pressed oil and refined cooking oil. *Maced. J. Anim. Sci.* **6**(1), 47–50 (2016).
- Wroniak, M., Krygier, K. & Kaczmarczyk, M. Comparison of the quality of cold pressed and virgin rapeseed oils with industrially obtained oils. *Pol. J. Food Nutr. Sci.* **58**(1), 85–89 (2008).

5. Shinagawa, F. B., de Santana, F. C., Araujo, E., Purgatto, E. & Mancini-Filho, J. Chemical composition of cold pressed Brazilian grape seed oil. *Food Sci. Technol. Camp.* **38**, 164–171. <https://doi.org/10.1590/1678-457X.08317> (2018).
6. Kostadinovic-Velickovska, S. & Mitrev, S. Characterization of fatty acid profile, polyphenolic content and antioxidant activity of cold pressed and refined edible oils from Macedonia. *J. Food Chem. Nutr.* **01**(01), 16–21 (2013).
7. Muneeshwari, P. *et al.* Physico-chemical quality and stability of refined and virgin oils. *Int. J. Pure Appl. Biosci.* **5**(2), 1182–1191. <https://doi.org/10.18782/2320-7051.2731> (2017).
8. Vingerling, N., Oseredczuk, M., du Chaffaut, L., Ireland, J. & Ledoux, M. Fatty acid composition of commercial vegetable oils from the French market analysed using a long highly polar column. *Oilseeds Fats Crops Lipids* **17**(3), 185–192. <https://doi.org/10.1051/ocl.2010.0309> (2010).
9. Correa, E. C. *et al.* Optimal management of oil content variability in olive mill batches by NIR spectroscopy. *Sci. Rep.* **9**, 13974. <https://doi.org/10.1038/s41598-019-50342-6> (2019).
10. Ovcharova, T., Zlatanov, M. & Dimitrova, R. Chemical composition of seeds of four Bulgarian grape varieties. *Ciência e Técnica Vitivinícola* **31**(1), 31–40. <https://doi.org/10.1051/ctv/20163101031> (2016).
11. Dimia, C. & Dimia, S. Essential oils in foods: extraction, stabilization, and toxicity. *Curr. Opin. Food Sci.* **5**, 29–35 (2015).
12. Jing, X., Xiao-Fei, L. & Yu-Tian, W. A detection method of vegetable oils in edible blended oil based on three-dimensional fluorescence spectroscopy technique. *Food Chem.* **212**, 72–77. <https://doi.org/10.1016/j.foodchem.2016.05.158> (2016).
13. Garrido-Delgado, R. M., Muñoz-Pérez, E. & Arce, L. Detection of adulteration in extra virgin olive oils by using UV-IMS and chemometric analysis. *Food Control* **85**, 292–299. <https://doi.org/10.1016/j.foodcont.2017.10.012> (2018).
14. Xua, L., Xu, X., Xiong, H., Chen, L. & Li, Y. Rapid detection of vegetable cooking oils adulterated with inedible used oil using fluorescence quenching method with aqueous CTAB-coated quantum dots. *Sens. Actuators B* **203**, 697–704. <https://doi.org/10.1016/j.snb.2014.07.008> (2014).
15. Li, F., Liu, Z. & Sun, T. Authentication of vegetable oils by confocal X-ray scattering analysis with coherent/incoherent scattered X-rays. *Food Chem.* **210**, 435–441. <https://doi.org/10.1016/j.foodchem.2016.05.012> (2016).
16. Vanstone, N., Moore, A., Martos, P. & Neethirajan, S. Detection of the adulteration of extra virgin olive oil by near-infrared spectroscopy and chemometric techniques. *Food Qual. Saf.* **2**, 189–198. <https://doi.org/10.1093/fqsafe/fyy018> (2018).
17. Bai, S. *et al.* Rapid detection of eight vegetable oils on optical thin-film biosensor chips. *Food Control* **22**, 1624–1628. <https://doi.org/10.1016/j.foodcont.2011.03.019> (2011).
18. Shi, J. *et al.* Stimulated Brillouin scattering in combination with visible absorption spectroscopy for authentication of vegetable oils and detection of olive oil adulteration. *Spectrochim. Acta Part A Mol. Biomol. Spectrosc.* **206**, 320–327. <https://doi.org/10.1016/j.saa.2018.08.031> (2019).
19. Rehab, F. M. A. & El Anany, A. M. Physicochemical studies on sunflower oil blended with cold pressed tiger nut oil, during deep frying process. *J. Food Process. Technol.* <https://doi.org/10.4172/2157-7110.1000176> (2012).
20. Aydeniz Güneşer, B., Yılmaz, E. & Ok, S. Cold pressed versus refined winterized corn oils: quality, composition and aroma. *Grasas Aceites* **68**(2), e194. <https://doi.org/10.3989/gya.1168162> (2017).
21. Gopala, K. A. G., Gaurav, R., Ajit, S. B., Prasanth, K. P. K. & Preeti, C. Coconut oil: chemistry, production and its applications—a review. *Indian Coconut J.* **53**, 15–27 (2010).
22. Marina, A. M., Che Man, Y. B., Nazimah, S. A. H. & Amin, I. Chemical properties of virgin coconut oil. *J. Am. Oil Chem. Soc.* **86**, 301–307. <https://doi.org/10.1007/s11746-009-1351-1> (2009).
23. Shi, L. *et al.* Physicochemical property, chemical composition and free radical scavenging capacity of cold pressed kernel oils obtained from different *Eucommia ulmoides*. *Oliver Cult. Ind. Crops Prod.* **124**, 912–918. <https://doi.org/10.1016/j.indcrop.2018.08.070> (2018).
24. Theeraphol, S. & Soottawat, B. Chemical compositions and properties of virgin coconut oil extracted using protease from hepatopancreas of Pacific white shrimp. *Eur. J. Lipid Sci. Technol.* **118**(5), 761–769. <https://doi.org/10.1002/ejlt.201400655> (2016).
25. Silverstein, R. M., Webster, F. X., Kiemle, D. J. & Bryce, D. L. *Spectrometric Identification of Organic Compounds* 78–125 (Wiley, New York, 2015).
26. Kamel, B. S., Dawson, H. & Kakuda, Y. Characteristics and composition of melon and grape seed oils and cakes. *J. Am. Oil Chem. Soc.* **62**(5), 881–883. <https://doi.org/10.1007/BF02541750> (1985).
27. Siger, A., Józefiak, M. & Górnaś, P. Cold-pressed and hot-pressed rapeseed oil: the effects of roasting and seed moisture on the antioxidant activity, canolol, and tocopherol level. *Acta Sci. Pol. Technol. Aliment.* **16**(1), 69–81. <https://doi.org/10.17306/J.AFS.2017.2017.0458> (2017).
28. Konuskan, D. B., Arslan, M. & Oksuz, A. Physicochemical properties of cold pressed sunflower, peanut, rapeseed, mustard and olive oils grown in the Eastern Mediterranean region. *Saudi J. Biol. Sci.* **26**(2), 340–344. <https://doi.org/10.1016/j.sjbs.2018.04.005> (2019).
29. Celenk, V. U., Gumus, Z. P., Argon, Z. U., Buyukhelvacigil, M. & Karasulu, E. Analysis of chemical compositions of 15 different cold-pressed oils produced in Turkey: a case study of tocopherol and fatty acid analysis. *J. Turk. Chem. Soc. Sect. A Chem.* **5**(1), 1–18. <https://doi.org/10.18596/jotcsa.335012> (2018).
30. Kostadinovic-Velickovska, S. & Mitrev, S. Characterization of fatty acid profile, polyphenolic content and antioxidant activity of cold pressed and refined edible oils from Macedonia. *J. Food Chem. Nutr.* **01**(01), 16–21 (2013).
31. Makala, H. Cold-pressed oils as functional food. *Plant Lipids Science, Technology, Nutritional Value and Benefits to Human Health* (ed. Budryn, G. & Żyżelewicz, D.) 185–200 (Research Signpost, Kerala, 2015).
32. Vingerling, N., Oseredczuk, M., du Chaffaut, L., Ireland, J. & Ledoux, M. Fatty acid composition of commercial vegetable oils from the French market analysed using a long highly polar column. *Oilseeds Fats Crops Lipids* **17**(3), 185–192. <https://doi.org/10.1051/ocl.2010.0309> (2010).
33. Seiichi, S., Akiko, S., Yasue, S. & Chiemi, S. A rapid method for trans-fatty acid determination using a single capillary GC. *J. Oleo Sci.* **56**, 53–58 (2007).
34. Griffiths, J. Colour and measurement. In *Colour and Constitution of Organic Molecules* (ed. Arrowsmith, J.) 1–17 (Academic Press, New York, 1976).
35. MacAdam, D. L. Colours of objects. In *Colour Measurement* (eds Enoch, J. M. *et al.*) 106–128 (Springer, Berlin, 1981).
36. Yildirim, K. & Melek Kostem, A. A technical glance on some cosmetic oils. *Eur. Sci. J.* **2**, 1857–7431. <https://doi.org/10.19044/esj.2014.v10n10p%25p> (2014).
37. Shirasawa, S., Sasaki, A., Saida, Y. & Satoh, C. A rapid method for trans-fatty acid determination using a single capillary GC. *J. Oleo Sci.* **56**(2), 53–58. <https://doi.org/10.5650/jos.56.53> (2007).

## Author contributions

Each author has made significant contributions to this work. S.B., G.E.M and C.V. performed the analysis regarding the physico-chemical parameters, S.V.N. performed the GC–MS and FT–IR analysis, M.S.M. interpreted the FT–IR and GC–MS analysis, S.P. and R.I.L. performed and interpreted the colour analysis. S.P. designed the work and wrote the main text of the manuscript. S.P. and R.I.L. prepared the Figures and Tables and put the paper



together. S.B. prepared the Reference chapter. R.I.L. submitted the paper to the journal. All authors reviewed the manuscript.

### Competing interests

The authors declare no competing interests.

### Additional information

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