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INVESTIGATION ON THE *RUBUS IDAEUS* L. SEED OIL (ROSACEAE)

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Abstract: The seed oil from a local variety *Rubus idaeus* L. (family Rosaceae) was isolated and the physical and chemical characteristics were determined. The oil content of *Rubus idaeus* seeds ranged from 8-15%. Standard procedures were applied to determine specific gravity, refractive index, acid value, saponification value, iodine value and peroxide value. The fatty acid composition was identified by GL chromatography and the main components were as follows: 9.9% oleic, 59.2% linoleic and 26.4% α-linoleic acid (ALA). Furthermore, *Rubus idaeus* tocopherol composition (mg/100g) was identified by HPLC, as: α-tocopherol 154.0, γ-tocopherol 192.0 and β-tocopherol 12.0. The seed oil contained 0.24 mg/L of carotene, 1.12 mg/L of chlorophyll *a* and 0.63 mg/L of chlorophyll *b*.

Key words: *Rubus idaeus* seed oil, fatty acids, tocopherols.

Introduction

The *Rubus idaeus* L. plant species, family Rosaceae, is a native floral element of central and eastern European regions, but in some cases can be also found in East Asia and North America. *Rubus idaeus* has several traditional names, as follows: jagodnjak, malinjak, crvena kupina, crvena jagoda i sunica. Indeed, *Rubus idaeus* is extensively used for preparing juices and syrups, namely *Sirupus rubi idaei.* Especially, the fruit generally has high content of

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C-vitamine (12-15 mg/%) and organic acids, i.e., citric and malic acids (Tucakov, J., 1996). In Serbia several species grow, but the best known cultivars of the *Rubus species* are as follows: *Malling promise, Malling exploit, Malling exploit* x *Rubin, Rote Wadenswiter* x *Letham* and *Willamette,* which are used as a very quality fruit (Mratinić, E., 1988). The fruit juice of *R. idaeus* is used in traditional medicine because of its cardiotonic properties, for the improvement of immune injury system and in the treatment of constipation. In addition, leaves tea is also used to relieve stomach problems, as well as astringent for inflammation in the treatment for colds and bronchial and stomach problems (Serbian Academy of Sciences and Arts, 1989 and Wichtl, M., 1984).

Moreover, several chemical constituent studies have been made on carbohydrates, organic acids, vitamins and essential oils from the fruit (Guichard, 1982 and Honkanen, 1980) and the tannins and flavonoids content of the leaves, namely *Rubi idaei folium,* are also included. No medicinal use has been reported for this plant species seed oil. Thus, the seed oil composition was evaluated in the current study, which has not been previously reported. These data may help in the selection of *R. Idaeus* seed oil for future commercial production.

Material and Methods

Rubus idaeus seed oil. - Fresh seeds of *Rubus idaeus* (cultivar *Malling promise*) were collected in the vicinity of Beska, near the Mt. Fruška Gora, with the identity authenticated by the Department of Horticulture, Faculty of Agriculture, University of Belgrade. A voucher specimen was deposited in the herbarium of this institution. The seeds were taken from ripened fruit of *R. Idaeus.* The seeds were dried at room temperature and ground in an electric blender. The sample of seeds (100 g) was extracted with petroleum ether (Merck, 40-60 °C) using a Soxhlet apparatus for 6 hr. The extract was desolventized *in vacuo* on a rotary evoporator at 35 °C, yielding lipid samples as the residue.

Physico-chemical characteristics of oil samples. - The ordinary oil constants, e.g. acid value (AV), iodine (IV), saponification (SV) and peroxide (PV) values, specific gravity and the refractive index were estimated according to the AOCS method (Official Methods of Analysis of AOAC, 1995). The crude chlorophyll sample was prepared by isolation of the 85% acetone extracts from the seed oil and stored in the dark at -15 °C. A qualitative evaluation of the chlorophyll precipitate by thin layer chromatography indicated that it consisted predominantly of chlorophyll *a* and a minor amount of chlorophyll *b.* The chlorophyll was determined spectrophotometrically according to the AOCS method (Official Methods of Analysis of AOAC, 1995). The carotene content was carried out, using the spectrometric method (Cocks et al. 1966).

The fatty acids profiles were determined by GLC. The methyl esters of the fatty acid were prepared by the method of Christie (Christie, 1973) and analysed by a Hewlett-Packard (Avondale, PA) 571017 gas chromatograph, fitted with a flame ionisation detector. A 152.4 cm x 0.317 cm glass column packed with 10% DEGS on Chromosorb VV 80/100 mesh was used for the analysis. Samples were run isothermally at 190 °C with injector and detector ports at 200 °C. Helium carrier gas flow was 30 mL/min. The peaks obtained by injecting 20 μl methyl esters were identified by running a standard fatty acid mixture and comparing the R values.

Tocopherols, α, β, γ and δ were analyzed by HPLC. A Perkin-Elmer (Norwalk, CT) LC-55 spectrometer was used as a detector at an excitation of 295 nm. The operation conditions were similar to those of Carpenter (Carpenter, 1979). Chromatography of tocopherols was done on a reversed phase μ-porasil (C_{18}) column (30 cm x 4 mm) and eluted with a solvent mixture of isopropyl/n-hexane (1.5:98.5) at flow rate of 1.5 mL/min.

Results and Discussion

The oil isolated from seeds of *Rubus idaeus* was greenish-brown in colour. It had a strong odour and characteristic taste. The total oil content of the *Rubus idaeus* seeds ranged from 12-15%. Physico-chemical characteristics of the seed oil are summarised in Table 1.

Tab. 1. - Physical and chemical characteristic of *Rubus idaeus* seed oil

Specific gravity 20 °C kg/m³	0.911
Refractive index 20 °C	1.4735
Acid value	1.89
Saponification value	189
Iodine value	139
Peroxide value mmol O_2/kg	0.22
Carotene content mg/L	0.24
Chlorophyll *a* content mg/L	1.12
Chlorophyll *b* content mg/L	0.63

The table 1 clearly shows that the iodine (IV) and saponification (SV) values are especially high. The acid value (AV) and peroxide value (PV) are relatively low. The specific gravity and refractive index of the oil are also relatively high. Table 1 indicates that these properties of *Rubus idaeus* seed oil are similar to oils rich in linolenic acid, such as the linseed and hempseed oils (Sonntag, 1979). Content of carotene was low, while the content of chlorophyll was very high, e.g. two times higher than to that in the corresponding soybean oil (Sonntag, 1979).

The fatty acids composition was also investigated and the obtained data are presented in Table 2.

Tab. 2. - Fatty acids content and summary of their important parameters of *Rubus idaeus* seed oil

Fatty acid	%
16:0	0.42
16:1	t
18:0	0.2
18:1	9.9
18:2	59.2
18:3	26.4
PUFA*	85.6
Total saturated acids	4.4
Total unsaturated acids	95.5
Ratio unsaturated/saturated	21.7

t-trace; *-polyunsaturated fatty acids

The table 2 demonstrates that the major fatty acids in *Rubus idaeus* seed oil are linoleic and α-linolenic acids. Low molecular weight (C_{10}-C_{12}) and high molecular weight (C_{20}-C_{22}) fatty acids were not detected in the investigating seed oil. The total saturated acids made up a small proportion of the total fatty acids content of the seed oil. Oleic acid accounting for 9.9% of the total fatty acid content *R. Idaeus* seed oil was characterised by a high content of polyunsaturated essential fatty acids: linoleic acid -18:2 ω-6, (52.2%) and linolenic acid 18:3 ω-3, (26,4%). It is known that fish oils are rich source of ω-3 polyunsaturated acids (PUFA), but fish oil can be high in cholesterol content, so plant sources provide a way to obtain ω-3 fatty acids without increasing dietary cholesterol intake (Haumann, 1988). α-Linolenic acid (ALA) can be converted through desaturation and elongation in the body to the long chain ω-3 fatty acids eicosapentaenoic acid (EPA 20:5 ω-3) and decosahexaenoic acid (DHA 22:6 ω-3) (Haumann, 1997). This elongation and desaturation process is influenced by many factors. One is the prevalence of ω-6 fatty acids in the diet. (Lands, et al. 1997). From a health point of view ω-3 fatty acids are of interest because they are important constituents of membranes in brain cells and heart muscle cells.

The tocopherols content in the investigated seed oil is presented in Table 3.

Tab. 3. - Tocopherol content of *Rubus idaeus* seed oil

	Mg/%
α - Tocopherol	154.0
β - Tocopherol	/
γ - Tocopherol	192.0
δ - Tocopherol	12.0
Total tocopherols	358.0

The total tocopherol content of *Rubus idaeus* seed oil had especially high value as 358.0 mg/100 g. γ-Tocopherol (192.0 mg/100 g) was a major constituent detected in the seed oil sample, followed by α-tocopherol in the significance yield of 154.0 mg/100 g and δ-tocopherol in the small yield of 12.0 mg/100 g. β-Tocopherol was not observed. Obviously, the high content of the tocopherols, especially the yield of

γ-tocopherol, probably may protect the seed oil from expected oxidation according to the high degree of PUFA (Pićurić-Jovanović, 1999). So, oxidative stability of *R. idaeus* seed oil has been the subject of our further investigation.

Conclusion

Results of the analyses of the tested oil for the content and composition of fatty acids indicated that further use in pharmaceutical and cosmetic industries may be warranted. *Rubus idaeus* seed oil contains 26.4% ALA. This oil probably has tendency to oxidise because of high content of PUFA, thus it must be stored under cold oxygen-free and light-free conditions. However *R. Idaeus* can not be used as a commodity seed in the production of edible oil, but can be useful as a medicinal or pharmaceutical product concerning that this oil is a rich, relatively pure source of ALA.

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ISPITIVANJE ULJA SEMENA *RUBUS IDAEUS* (ROSACEAE)

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Rezime

Seme biljne vrste *Rubus idaeus,* familija Rosaceae je ispitano kao izvor specifičnog ulja i određene su njegove fizičko-hemijske karatkeristike. Biljna vrsta *Rubus idaeus,* poznata kao malina, vrlo je rasprostranjena u Srednjoj i Istočnoj Evropi. Poznata su lekovita svojstva soka i sirupa maline, a u narodnoj medicini se najčešće koristi čaj lista maline u lečenju bronhijalnih oboljenja i stomačnih tegoba. Ulje semena maline do sada nije ispitivano, a nisu poznata ni njegova lekovita svojstva. Ulje izolovano iz semena maline je zelene boje i jakog karakterističnog mirisa. Sadržaj ulja se kreće u intervalu od 8-15%. Primenom standardnih metoda AOCS-a određena je specifična masa (0.911 kg/m^3), indeks refrakcije (1,4735 na 20^{0}C), kiselinski broj (1,89), saponifikacioni broj (189), jodni broj (139) i peroksidni broj (0,22 mmol O_2/kg). Sadržaj karotena je relativno nizak (0,24 mg/L), dok je sadržaj hlorofila vrlo visok (1,12 mg/L hlorofila *a* i 0,63 mg/L hlorofila *b*). Gasnom hromatografijom određen je sastav masnih kiselina. Ulje semena maline karakteriše izuzetno nizak sadržaj zasićenih masnih kiselina (4,4%) i veoma visok sadržaj nezasićenih masnih kiselina (95,5) pri čemu je značajan udeo polinezasićenih masnih kiselina (PUFA - 85,6%). Ulje semena *R. Idaeus* predstavlja značajan izvor α-linolenske kiseline (26,4%) (18:3, ω-3) koja je kao i ostale ω-3 masne kiseline neophodna u izgradnji membrana moždanih ćelija i mišićnog tkiva srca. Takođe je dokazano da ulje sadrži visok procenat tokoferola (358,0 mg%).

Usled visokog sadržaja polinezasićenih masnih kiselina ovo ulje je verovatno oksidativno nestabilno, zbog čega zahteva specijalne uslove čuvanja. Takođe, utvrđene osobine ulja određuju i mogućnost primene u medicini i farmakologiji, kao izvor ulja bogatih relativno čistom α-linolenskom kiselinom.

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