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Determination of Physico-Chemical Characteristics of Walnut (*Juglans regia* L.) Oil From Cultivar Sampion

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Abstract: The aim of this work was separation and determination of oil from the cultivar Sampion. Two techniques of oil extraction were employed: cold pressing and organic solvent extraction. Composition of walnut kernels and basic physico-chemical parameters of walnut oil were determined, respectively. The oil content was 69.07 %. The fatty acid composition was determined by gas chromatography and the main components were as follows: linoleic acid 58.0%, oleic acid 20.7% and linolenic acid 9.8% for sample obtained by cold pressing and 60.9%, 19.0%, 13.6%, respectively, for sample obtained by solvent extraction.

Key words: walnut oil, physico-chemical characteristics, composition of fatty acids.

Introduction

Increasing attention is paid today to preservation of natural and healthy food.

Due to certain health disorders and risks, mostly caused by inadequate nourishment, the current world trends are imposing a new concept of development in technology of edible oils as well.

Favourable health effects are attributed to a large number of lypid substances today, and the majority of scientific studies and researches refer to essential fatty acids, especially those from ω-3 and ω-6 series, conjugated linoleic acid, phospolypids, herbal

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sterols, natural antioxidants and other ingredients (Savage *et al*. 1999, Cunnane *et al*. 1993).

Walnut oil is characterised by a high level of ω-3 and ω-6 essential fatty acids, which affects its nutritive value. Use of walnut oil has an important preventive role in modification of lypoprotein profile and a protective role in cardiovascular diseases and cancer (Corridan *et al*. 1998, Sabate *et. al*. 1993).

The primary goal of this study was to characterise walnut oil of the domestic cultivar Sampion and investigate the effect of the types of oil separation procccess on the content of essential fatty acids.

Materials and Methods

Herbal material. A nutshell walnut harvested in 2007 was obtained from the Centre of Fruit Growing and Viticulture in Čačak, and it was preserved in jute sacks, in a cellar, at a temperature of 4°C, until analysis.

Nutshell was hand-removed. Obtained kernels used for oil extraction, were of proper colour, yeasty, without signs of herbal diseases and pests, ripe, without strange swell and taste. The kernels were preserved in closed glass bowls at t=1 ^{0}C, in the dark, until use.

Procedure of oil extraction. For oil extraction from walnut kernels two procedures were performed: cold pressing, involving mechanical oil extraction by hydraulic strainer (inox), under the pressure of 150 bars, and classical procedure of oil extraction with a suitable organic dissolver, the so-called Soxhlet method (Karlović *et al*.1996).Both procedures were performed at a temperature of 50 °C in order to preserve the nutritive value of walnut oil.

Composition of walnut kernels. Chemical characteristics of walnut kernels: Content of oil was determined by Soxhlet; content of nitrogen by Kjeldahl; humidity, and content of cellulose were determined by official SRPS ISO method (Karlović *et al*.1996), and mineral matters by AOAC method (AOAC 2000). Content of carbohydrates was determined by mathematical calculation based on the formula:

carbohydrate content = 100 % – (% of humidity + % of proteins + % of oil + % of ashes + % of cellulose) - Grosso *et al*. (2000)

Three measurements were repeated, and the results obtained are presented as average values.

Determination of physico-chemical characteristics of extracted oil. Acid number (AN), iodine number (IN), saponificational number (SN), peroxide number (PN) and the index of refraction were determined by official JUS ISO methods (Dimić *et al*. 2000).

Composition and content of fatty acids were determined by gas chromatography developed by Christie (1973). Content of fatty acids was evaluated using the Varian apparatus, model 1400, with an ionizing flame detector and metal colons of 300x0.32 cm size used. Determination was performed under following conditions: stationary phase LAC- 3R-728 (20 %); support of stationary phase was Chromosorb W/AW, 80-100 mesh; mobile phase was nitrogen with a flow rate of

24 ml/min; temperature of injector block and detector was 200 °C. 1 µl of methylestre of fatty acids sample was directly injected in a gas chromatograph.

Result and Discussion

Chemical characteristics of the test walnut kernels are shown in Table 1.

Tab. 1. Chemical composition of Sampion walnut cultivar

Humidity (%)	4.88
Oil (% d.m.)*	67.07
Mineral matters (%d.m.)	2.18
Cellulose (%d.m.)	1.65
Proteins (%d.m.)	14.22
Carbohydrates (%d.m.)	13.95

* % dry matter

The results given above suggest that the walnut kernel is characterised by a high content of oil, 67.07 %, due to which it can be classified into a group of herbal species that can be used for commercial oil extraction.

Apart from the high oil content, the walnut kernel contains a high percentage of proteins (14.22 %) and carbohydrates (13.95 %). Owing to the level of the above components, cake which remains after the oil extraction can be used in other food technologies (for example, confectionary industry).

Physico-chemical characteristics of walnut oil are shown in Table 2.

Tab. 2. Physical and Chemical Characteristics of walnut oil separated by cold pressing and solvent extraction method

Parameters	Cold pressing	Extracted
Refraction index 20 ^{o}C	1.475	1.476
Saponification number	188.5	188.0
Iodine number	145.1	150.1
FFA (% oleic.acid.)	0.18	0.20
Peroxide number (mmolO_2/mg)	0	0.1

Low content of free fatty acids (FFA) of 0.18 or 0.20 %, regardless of the type of separation process, indicates that the starting substance was of high quality, well preserved, and that during extraction process, hydrolysis did not take place.

In cold pressed oil, immediately after separation, there were no primary products of oxidation (PN is zero), indicating that walnut kernels were fresh and well preserved.

PN value of the extracted oil was 0.1 mmol O_2 / kg which implies that the extraction process did not provoke the increase of PN value, or the change of oxidative stability of oil.

Values of the RI, SN and IN are in accordance with previous literature data (Savage 2001, Shijie *et al*. 2002, Amaral *et. al*. 2003) where the extraction process did not have any effect.

Composition and content of fatty acids is shown in Table 3.

Tab. 3. Fatty acid composition of investigated walnut oil

Fatty acids (%)	Cold pressed	Extracted
C16:0	7.5	7.1
C16:1	0.4	0.4
C18:0	1.7	1.6
C18:1	20.7	19.0
C18:2 (ω-6)	59.8	60.9
C18:3 (ω-3)	9.8	11.0
PUFA	69.6	71.9

The oil separation process did not affect significantly the content of fatty acids. Dominant is the linoleic fatty acid (C 18:2), containing 59.8 % of cold pressed oil, and 60.9 % of extracted oil, 20.7 % and 19.0 % of oleic acid (C 18:1), α – linoleic acid (C 18:3) 9.8 % and 11.0 %, and of palmitic acid (C 16:0) 7.5 % and 7.1 %. The content of stearic acid (C 18:0) is low, ranging from 1.7 % or 1.6 %, and the palmitooleic acid (C 16:1) is present only in traces.

From the nutritive point of view, the walnut oil content of essential fatty acids, linoleic (ω-6) and α-linolenic (ω-3) is of high importance.

These polysaturated fatty acids (PUFA) are the dominant group of fatty acids in the samples, and their total content was 69.6 % for the cold pressed oil, and 71.9 % for extracted oil. High content of PUFA contributes to a high nutritive value of the examined walnut oil, but occasionally leads to low stability of walnut oil.

Owing to the high value of oil content of 67.07 %, walnut kernel can be classified into a group of herbal species which can be used for commercial oil extraction. It is characterised by a high content of essential fatty acids, linoleic and ά-linolenic fatty acids that contribute to the nutritive value of oil, and also to its susceptibility to oxidative changes.

Conclusion

The results obtained suggest the following:

- starting substance, the walnut kernels cultivar Sampion (Center for Fruit Growing, Čačak) was of high quality, well preserved (without primary oxidation products, with peroxide number PN equal to zero, immediately after oil extraction by cold pressure);

- types of oil separation (cold pressure or solvent extraction) do not affect the oxidation stability of walnut oil, and during oil separation process hydrolysis does not take place;

- the values of IR, SR, IN are similar for both oil separation types, and are in accordance with literature data;

- walnut kernels, cultivar Sampion, possesses a high content of oil (67.07 %), therefore it can be classified into a group of herbal species which can be used for commercial oil extraction;

- high content of essential fatty acids, the linoleic ω-6 (59.8 % for cold pressured, and 60.9 % extracted by solvent) and α-linoleic ω-3 (9.8 % for cold pressured and 11 % extracted by solvent) is very important from the nutritive point of view; however, high PUFA content (69.6 % of cold pressured and 71.9 % extracted by solvent) indicates its low oxidative stability;

- owing to the high content of proteins (14.22 %) and carbohydrates (13.95 %) in cake, which remains after the oil extraction, it can be used in other food technologies (confectionary industry).

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ODREĐIVANJE FIZIČKO-HEMIJSKIH KARAKTERISTIKA ULJA ORAHA (*Juglans regia* L.) SORTE ŠAMPION

- orginalni naučni rad -

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Rezime

Danas se u svetu sve više pažnje posvećuje očuvanju prirodne i zdrave hrane. Zbog određenih zdravstvenih poremećaja i rizika, uslovljenih pre svega neadekvatnom ishranom, aktuelni svetski trendovi neminovno nameću novi koncept razvoja i u tehnologiji jestivih ulja.

Povoljni zdravstveni efekti se pripisuju većem broju lipidnih supstanci, pri čemu se najveći broj radova i istraživanja odnosi na esencijalne masne kiseline, posebno one iz ω-3 i ω-6 serije, konjugovanu linolnu kiselinu, zatim fosfolipide, biljne sterole, prirodne antioksidanse i druge sastojke.

Ulje oraha se odlikuje visokim sadržajem ω-6 i ω-3 esencijalnih masnih kiselina, što utiče na njegovu visoku nutritivnu vrednost. Upotreba orahovog ulja ima značajnu preventivnu ulogu u modifikaciji lipoproteinskog profila i zaštitnu ulogu kod kardiovaskularnih oboljenja i kancera.

Istraživanja u ovom radu su imala za cilj da se uradi karakterizacija ulja oraha domaće sorte Šampion (Centar za voćarstvo, Čačak) i ispita uticaj postupka izdvajanja ulja na sadržaj esencijalnih masnih kiselina.

Izdvojeno je ulje iz jezgra oraha domaće sorte Šampion primenom dve tehnike: hladno ceđenje i ekstrakcija organskim rastvaračem. Određeni su sastav i hemijske karakteristike jezgra oraha kao i fizičko-hemijske karakteristike izdvojenog ulja. Sadržaj ulja je iznosio 69.07 %. Sastav i sadržaj masnih kiselina je određen metodom gasne hromatografije. Dominantne masne kiseline su: linolna sa sadržajem od 58.0 %, zatim oleinska sa sadržajem od 20.7 % i linolenska 9.8 % u ulju dobijenom hladnim ceđenjem, odnosno 60.9%, 19.0%, 8.7% u ulju dobijenom ekstrakcijom organskim rastvaračem.