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ANNUAL CARAWAY ESSENTIAL OIL COMPOSITION GROWN IN ORGANIC AND CONVENTIONAL GROWING SYSTEMS

ABSTRACT

Caraway is grown for its essential oil content, which is present in the whole plant, but the concentration is highest in the fruits. The annual caraway seed (*Carum carvi* L.) was grown in different environmental conditions on three localities in the Vojvodina province, Serbia, during the year 2011. The field experiment in a randomized block design included: control (without applying fertilizers), organic (Slavol, Bactofil B-10, Royal Ofert biohumus and vermicompost) and conventional (NPK) crop systems. Ripe dried seed was crushed and distilled for its essential oil by a Clevenger-type apparatus. The oil quality was assessed through analysis by combined gas chromatography and mass spectrometry. Caraway fruit contains 2.66-4.94% essential oil consisting of 22 compounds, from which limonene and carvone account for the main portion (above 98%). The highest content of limonene was from the field fertilized by vermicompost (57.26%), and the lowest from the control field (54.14%). Conversely, the highest content of carvone was from the control field (44.31%), and the lowest from the field fertilized by vermicompost (40.78%). From this experiment it can be concluded that the relationship between limonene and carvone has a negative correlation.

Keywords: caraway, essential oil, limonene, carvone

INTRODUCTION

Caraway (*Carum carvi* L.) is an annual or a biennial herb that is widely cultivated in many parts of the temperate zone. The biennial type is under cultivation in northern Europe and the annual type is cultivated in the Mediterranean area. The dried fruits, which are known commercially as seeds, are used in many types of bread, cheese, canned vegetables and confections (Putievsky, 1978). Also, caraway is grown for its essential oil content which is present in the whole plant, but the concentration is highest in the fruits (Sedláková et al., 2003a). Caraway fruits contain from 1 to 6% essential oil that

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gives caraway its characteristic aroma. There are approximately 30 compounds contained in this plant species, while carvone and limonene account for about 95% of them. The essential oil amount and composition is genetically conditioned and depends on climatic conditions during fruit formation and ripening (Sedláková et al., 2003a).

The cultivation of medicinal plants is an important part of agriculture with special crops cultivated organically on arable land. The main goal of the organic production of medicinal plants is to produce material with better quality as well as to sustain the quality of natural resources and secure economic and social awareness (Habán and Otepka, 2007).

Regarding environmentally friendly farming methods, there is more focus on the activation of the natural ecosystem in the soil, mainly of microorganisms. Applying bacterial fertilizers by adding, from the aspect of nutrient supply, important bacterial species to the soil, the microbial population of the soil can be richer and more complete, and therefore the mobilization processes in the soil will be accelerated. So plant producers use these special bacterial fertilizers on even larger areas without, or with a reduced amount of chemical fertilizers (Kincses and Sipos, 2008).

The processing of organic waste from animal farms created specific fertilizers like Royal Ofert biohumus (organic waste from poultry and pig farms inoculated with larval domestic fly) and vermicompost (modified cattle manure with *Lumbricus terrestris*).

MATERIALS AND METHODS

Annual caraway seeds (*Carum carvi* L.) obtained from the herbal farm of Jan Selešćanski from Kulpin, were sown at the beginning of April 2011, on three localities in the Vojvodina province, Serbia, in continuous rows at a distance of 35 cm, and with a seedling depth of 3-4 cm. Sowing was done by hand, directly in the soil. After germination, the plants were thinned. The plots were kept weed-free by hand weeding and hoeing. Seeds were harvested manually at the full maturity stage during August.

Field experiments in a randomized block design included: control (without applying fertilizers), organic (Slavol, Bactofil B-10, Royal Ofert biohumus and vermicompost) and conventional (NPK) crop systems.

Seed materials were analysed 3 months from harvesting in order to determinate the essential oil content. Ripe dried seed was crushed and distilled for its essential oil by a Clevenger-type apparatus. The oil quality was assessed through analysis by combined gas chromatography and mass spectrometry. GC-MS analysis was performed using an Agilent 6890 gas chromatograph coupled to an Agilent 5973 Network mass selective detector (MSD), in positive ion electron impact (EI) mode. The separation was achieved using an Agilent 19091S-433 HP-5MS fused silica capillary column, 30m x 0.25mm i.d., 0.25 μm film thickness. The GC oven temperature was programmed from 60 °C to 285 °C at a rate of 4.3 °C/min. Helium was used as a carrier gas, the inlet pressure was 25

kPa and the linear velocity was 1 ml/min at 210 °C. Injector temperature: 250 °C; injection mode: splitless. MS scan conditions: source temperature, 200 °C; interface temperature, 250 °C; energy, 70 eV; mass scan range, 40-350 amu.

Identification of the components was done on the basis of the retention index and the comparison with reference spectra (Wiley and NIST databases).

RESULTS AND DISCUSSION

The content of essential oils, the amount of carvone and limonene in the oil and the ratio of both substances are the main quality criteria determined in caraway production (Sedláková et al., 2003b). In our experiment, caraway fruit contained 2.66-4.94% essential oil consisting of 22 compounds, from which limonene and carvone accounted for the main portion (above 98%). The seeds contained other compounds such as: mircene (0.30%), trans dihydro carvone (0.21%), trans carveole (0.20%), cis limonene oxide (0.18%), trans limonene oxide (0.17%), γ-terpinene (0.16%), muurola-4(14),5-diene trans (0.16%), E-caryophillene (0.15%) and trace amounts of other compounds (results not shown).

Table 1 presents the essential oil, limonene and carvone content in three localities depending on the application of fertilizer. As can be seen, the highest content of essential oil was obtained on the experimental plot with the Royal Ofert biohumus and vermicompost (4.06%), and a slightly lower essential oil content was from the plot with the conventional growing system (4.04%).

The main components of caraway fruit essential oil are limonene and carvone. Limonene is common in cosmetic products, and is used in food manufacturing and some medicines, e.g. as a flavouring to mask the bitter taste of alkaloids, and as a fragrant in perfumery. It is also used as a botanical insecticide. Carvone is used as a fragrance and flavour, potato sprouting inhibitor, antimicrobial agent, building block and biochemical environmental indicator (Carvalho and Fonseca, 2006).

Bouwmeester et al. (1998) explain that carvone arises from limonene. During fruit development the content of carvone increases at the expense of limonene. In a previous study Bouwmeester and Kuijpers (1993) point out that annual caraway has a lower essential oil content than the fruits of the biennial varieties, and also that annual caraway usually has a lower carvone to limonene ratio.

The highest content of limonene was from the field fertilized by vermicompost (57.26%), and the lowest from the control field (54.14%). Conversely, the highest content of carvone was from the control field (44.31%), and the lowest from the field fertilized by vermicompost (40.78%). From this experiment it can be concluded that the relationship between limonene and carvone has a negative correlation.

The limonene to carvone ratio in this experiment ranged from 1.2-1.4% depending on the application of fertilizer. Bouwmeester et al. (1995), in essential oil obtained from hydrodistillation, determined the limonene/carvone ratio to be

1.49%, but from a hexane extract this ratio was lower (0.865%). Toxopeus and Lubberts (1994), in an experiment conducted in the Netherlands with biennial caraway, got an L/C ratio from 0.77-0.94% depending on the harvest time and soil type. The authors pointed out that the carvone content was substantially affected by the time of harvesting, i.e. 10-15% lower carvone content from the second harvest time, 7 days after the first harvest.

Table 1: Essential oil, limonene and carvone content depend on the application of fertilizer:

		Control	Slavol	Bactofil	Royal Ofert	Vermicompost	NPK
Essential oil content (%)	Mošorin	2.75	3.12	2.96	3.66	3.26	3.30
	Ostojićevo	3.24	3.28	2.66	3.87	4.05	3.87
	V.Radinci	4.39	4.79	4.32	4.65	4.87	4.94
	Average	**3.46**	**3.73**	**3.31**	**4.06**	**4.06**	**4.04**
Limonene content (%)	Mošorin	52.06	56.04	57.18	58.28	58.23	60.49
	Ostojićevo	51.49	55.85	50.64	56.03	55.33	54.88
	V.Radinci	58.87	57.38	54.39	55.65	58.23	52.62
	Average	**54.14**	**56.42**	**54.07**	**56.65**	**57.26**	**56.00**
Carvone content (%)	Mošorin	46.92	42.43	41.20	40.27	40.08	38.30
	Ostojićevo	47.68	42.66	47.91	42.25	43.16	43.37
	V.Radinci	38.33	40.78	43.87	41.44	39.10	45.86
	Average	**44.31**	**41.96**	**44.33**	**41.32**	**40.78**	**42.51**
L/C (%)		**1.2**	**1.3**	**1.2**	**1.4**	**1.4**	**1.3**

CONCLUSION

The content of essential oils, the amount of carvone and limonene in the oil and the ratio of both substances are the main quality criteria determined in caraway production. Caraway fruit contains 2.66-4.94% essential oil consisting of 22 compounds, from which limonene and carvone account from the main portion (above 98%). The highest content of limonene was from the field fertilized by vermicompost (57.26%), and the lowest from the control field (54.14%). Conversely, the highest content of carvone was from the control field (44.31), and lowest from the field fertilized by vermicompost (40.78%). From this experiment it can be concluded that the relationship between limonene and carvone has a negative correlation. The limonene to carvone ratio ranged between 1.2-1.4%.

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SASTAV ETARSKOG ULJA JEDNOGODIŠNJEG KIMA GAJENOG U ORGANSKOM I KONVENCIONALNOM SISTEMU

SAŽETAK

Kim se gaji zbog njegovog etarskog ulja, koje je prisutno u celoj biljci, ali je njegova koncentracija najveća u plodovima. Jednogodišnji kim (*Carum carvi* L.) gajen u različitim ekološkim uslovima na tri lokaliteta u Vojvodini, Srbija, tokom 2011. godine. Poljski ogled postavljen po slučajnom blok sistemu uključuje: kontrolu (bez primene đubriva), organska đubriva (Slavol, Bactofil B-10, Royal Ofert biohumus i glistenjak) I konvencionalni (NPK) system gajenja. Zreli, suvi plodovi nakon mlevenja su destilisani aparatom Clevenger-tipa radi dobijanja etarskog ulja. Kvalitet etarskog ulja je analiziran gasnom hromatografijom i masenom spektrometrijom. Plodovi kima sadrže 2.7-4.9% etarskog ulja sa 21 komponentom, od kojih su limonene i karvon najzastupljeniji, preko 98%. Najveći sadržaj limonene je na parcelama đubrenim sa glistenjakom (57.26%), a najmanji na kontroli (54.14%). Nasuprot tome, najveći sadržaj karvona je na kontroli (44.31), a najmanji na parceli đubrenoj glistenjakom (40.78%). Iz ovog ogleda se može zaključiti da postoji veza između limonene i karvona u negativnoj korelaciji.

Ključne riječi: kim, etarsko ulje, limonene, karvon