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Effect of sterilization of sweet fennel fruits (*Foeniculum vulgare* var. *dulce* Mill.) on essential oil yield and composition

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Abstract. The sweet fennel fruits (*Foeniculum vulgare* var. *dulce* Mill.) are processed to obtain their essential oil. It is part of the composition of various products from the food industry, pharmacy, medicine and cosmetics. The scope is to investigate the influence of the fruits sterilization with superheated water steam on the yield and composition of the essential oil, before the fruits processing by water distillation. The main components in the essential oil, obtained from fruits before sterilization, are trans-anethole (67.10%), fenchone (21.58%), methyl chavicol (3.14%), and α-pinene (2.31%). The essential oil, obtained from fruits after sterilization, has the same main components in its composition but their amount is different: trans-anethole (71.38%), fenchone (16.75%), methyl chavicol (2.89%), and α-pinene (2.26%).

1 Introduction

Fennel (*Foeniculum vulgare* Mill.) is part from the Apiaceae family. Two types are grown from it: sweet (var. *dulce* (Mill.) Thell.) and bitter (var. *vulgare* (Mill.) Thell). Also known as var. *azoricum* (Mill.) Thell., as well as another subspecies ssp. *piperitum* (Ugria) Cout., but they have no practical significance [1].

The essential oil is localized in endogenous containers, a type of essential oil channel, in an amount of 1.5 to 3.5% in the sweet and 2.5 to 6.0% in the bitter variant. Today, different varieties are selected in order to increase the oil content.

The fruits are harvested when they ripen and after storage are processed by distillation. The essential oil is liquid with a typical aniseed, slightly spicy smell, with a light earthy-camphor note [1]. *Trans*-anethol is the main component in the essential oil of sweet fennel (from 65 to 91%) depending on the country of origin [2-4]. The other compounds are fenchone (1.0-12.0%), limonene (2-7%), and methyl chavicol (2.3-7.3%) [5-8].

The essential oil finds application in the medicine [9-11], food industry [12-15], in cosmetics [16], *etc*. [17].

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Very often the raw plant material, when collected from the field, is contaminated with various impurities - mechanical or organic, which create conditions for a rise in temperature during storage, due to the occurrence of self-heating and subsequent development of microorganisms. This is an undesirable process, especially for those of them that have single-gene containers and can be stored for several months to a year under suitable storage conditions. For this reason, various methods are often used to destroy the unwanted microflora - sterilization, irradiation, *etc*. [18].

There is no data in the literature regarding the sterilization of fennel fruits before their processing by distillation and its influence of the essential oil, which is also is scope of the work.

2 Materials and methods

2.1 Plant materials

Sweet fennel fruits were studied, harvest 2020. The fruits were grounded for 30 s to a size of 0.5 mm.

To remove the unwanted microflora on the surface of the fruit, sterilization with superheated steam was carried out under the following conditions: equipment consisting of a feed screw, a screw sterilizer, a steam boiler, a steam heater and a cooling screw. The necessary steam for sterilization is obtained from potable water, which is heated in a boiler until steam is formed. By means of pipelines, it enters a superheater for additional heating in order to produce superheated steam. The superheated steam, with a temperature of 180°C, is fed into the screw sterilizer, in which the raw material is fed with the help of an inclined screw. The raw material moves in countercurrent to the steam and stays in the sterilizer for 4 s. After passing through the sterilizer, the raw material is fed into the cooling screw, after which it is processed.

The moisture of the fruits is determined [19].

2.2 Isolation of the essential oil by hydrodistillation

The fruits were processed by water distillation under laboratory conditions [20].

2.3 Essential oil analysis

GC-FID. Apparatus Agilent 7890. Column 1: HP-Innowax – 60 m × 250 μm × 0,25 μm; carrying gas He - pressure 45 psi 35 psi, injector temperature 245.0°C; split 1:100; injected volume 1 μl; detector temperature – 260.0°C, Gases for FID detector H_2 – 35.00 ml/min, air – 350.0 ml/min, make up gas N_2 – 10 ml/min. Temperature mode of the column: 60°C – 2 min isotherms; Step I: rising the temperature with 10°C/min to 140°C; Step II: rising the temperature – 3°C/min to 230°C – 51 min isotherms; Step III: rising the temperature – 5/min to 250°C – 5 min isotherms. Transfering the stream to GC-FID-MS with additional gas He via Deans Switch Valve from 6 to 91 min; Transfer line to second gas chromatograph with temperature 260°C.

GC-FID-MS. Apparatus Agilent 7890 with MSD Agilent 5975C. Column 2: Cyclodex-B – 60 m × 250 μm × 0,25 μm; carrying gas He – pressure 35 psi (from GC_FID); detector temperature – 240.0°C, Gases for FID detector: H_2 – 35.00 ml/min, air – 350.0 ml/min, make up gas N_2 – 10 ml/min. Temperature mode of the column: 60°C – 3 min isotherms; Step I: rising the temperature with 80°C/min to 120°C; Step II: rising the temperature with 20°C/min to 220°C – 13 min isotherms; Step III: rising the temperature with 50°C/min to 230°C – 22.5

min isotherms. MS – detector: EI Energy 69.9; EM Voltage 1776; Scanning parameters - from mass 40 to 450 – 3.18 scan/s. Transfer line to mass-selective detector with temperature 200°C. Flow divider between FID-MS – capillary separator with additional gas He – flow split in ratio 2:1.

The data was processed with MS Chem Station v.E.02.01 software.

2.4 Statistical analysis

All the investigations were carried out three to five times, giving in the figures their mean values with the corresponding statistical error.

3 Results and discussion

Moisture and essential oil yield are presented in Fig. 1. The data shows that after sterilization the moisture of the material increases and the essential oil yield decreases.

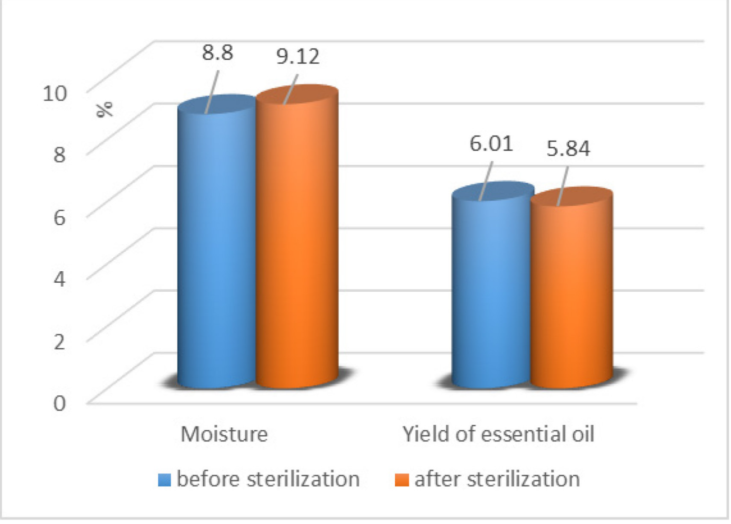

Fig. 1. Moisture and essential oil yield, %.

Table 1 presents the chemical composition of essential oils. The following chromatograms in Fig. 2 and Fig. 3 correspond to the main compounds in the composition.

During fruit sterilization, regardless whether the essential oil is deposited in endogenous containers, changes occur in its chemical composition – fenchone, which gives a bitter taste, decreases and *trans*-anethole, which is the main component, increases. In both oils, the amount of toxic *cis*-anethole is very low and does not change. The content of the compounds in the essential oil are related to the sterilization of the fruit with steam, in spite of the short time spent in the sterilizer.

According to Sarkis and Stappen [21], the following allergens were found in both oils – α- and β-pinene, limonene, camphor and anethole, with their amounts being higher, except for anethole in the oil obtained from fruits before sterilization.

Table 1. Chemical composition of the essential oils (% of TIC[a]).

Nº	RI [b]	Compounds	Before sterilization	After sterilization
1.	933	*α*-Pinene [c]	2.31 ± 0.02	2.26 ± 0.02
2.	947	Camphene	0.21 ± 0.0	0.18 ± 0.0
3.	979	*β*-Pinene [c]	0.25 ± 0.0	0.20 ± 0.0
4.	988	Myrcene	1.08 ± 0.01	0.96 ± 0.0
5.	1001	*α*-Phellandrene	0.42± 0.0	0.36 ± 0.0
6.	1024	Limonene [c]	1.62 ± 0.01	1.45 ± 0.01
7.	1026	*β*-Phellandrene	- [d]	0.85 ± 0.0
8.	1054	*γ*-Terpinene	0.69 ± 0.0	0.64 ± 0.0
9.	1085	Fenchone	21.58 ± 0.20	16.75 ± 0.15
10.	1165	Camphor [c]	0.40 ± 0.0	0.37 ± 0.0
11.	1195	Methyl chavicol	3.14 ± 0.03	2.89 ± 0.20
12.	1290	*cis*-Anethole [c]	0.11 ± 0.0	0.11 ± 0.0
13.	1291	*trans*-Anethol [c]	67.10 ± 0.60	71.38 ± 0.70

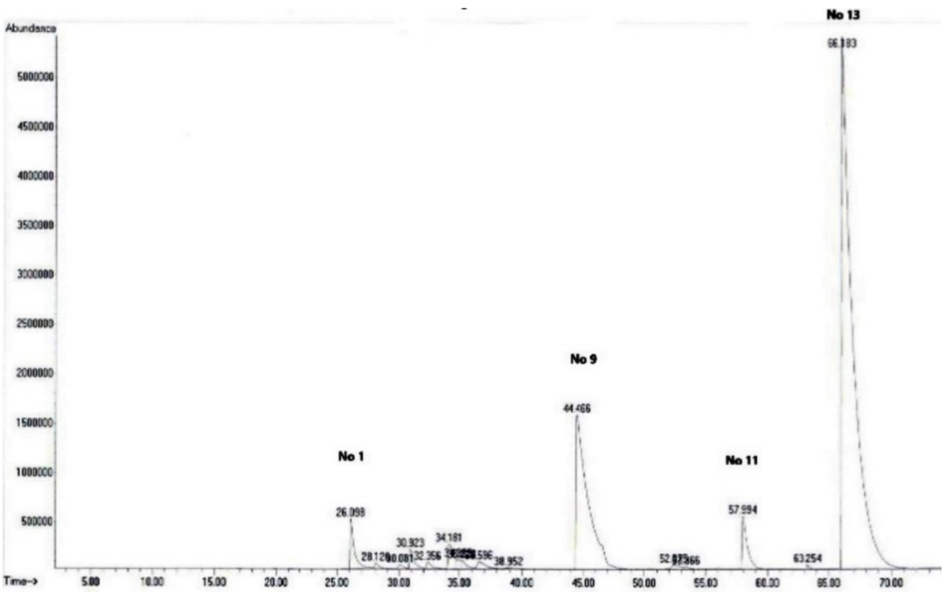

Fig. 2. Main components in essential oil, obtained from fruits prior to sterilization (peak numbers correspond to those in Table 1).

The distribution of components by functional groups is presented in Fig. 4. The data shows that:

- in the essential oil obtained from fruits prior to sterilization phenyl propanoids predominate (71.12%), followed by oxygenated monoterpenes (22.22%), and monoterpene hydrocarbons (6.65%).

- in the essential oil obtained from fruits after sterilization phenyl propanoids predominate (75.59%), followed by oxygenated monoterpenes (17.40%), and monoterpene hydrocarbons (7.01%).

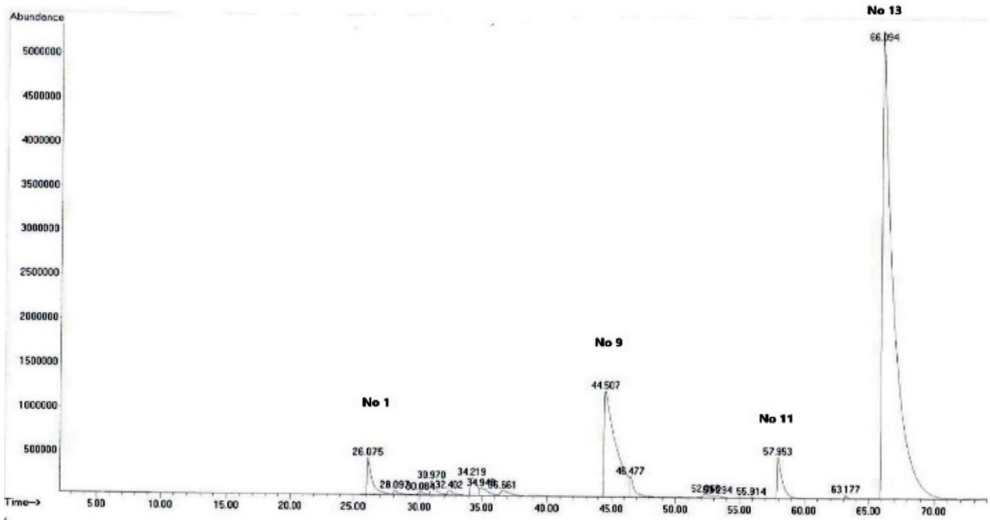

Fig. 3. Main components in essential oil, obtained from fruits after sterilization (peak numbers correspond to those in Table 1).

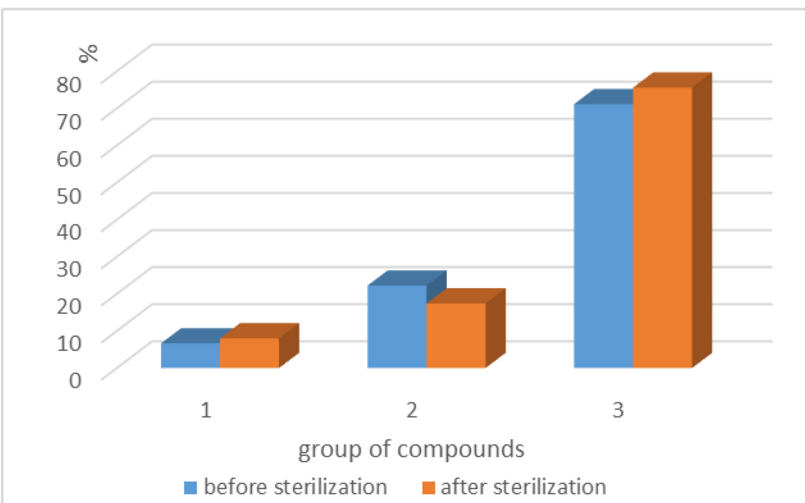

Fig. 4. Group of components in the essential oils, %: 1 – monoterpene hydrocarbons; 2 – oxygenated monoterpenes; 3 – phenyl propanoids.

4 Conclusion

The paper monitored the effect of superheated steam sterilization of sweet fennel fruits on the amount and the change in the components in the essential oil, obtained by hydro distillation under laboratory conditions. This treatment reduces the amount of fenchone (from 21.58 to 16.75%), which gives the essential oil a bitter taste, and increases the content of anethole (from 67.10 to 71.38%), which is its main component.

The concentration of the components depending on the functional groups contained in them shows that in the essential oils phenyl propanoids predominate (between 71.12 and

75.59%), followed by oxygenated monoterpenes (between 17.40 and 22.22%), and monoterpene hydrocarbons (between 6.65 and 7.01%).

The application of superheated steam sterilization to fennel fruits before their industrial processing by steam distillation, is a subject for future research.

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