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Original research paper

THE CHROMATOGRAPHIC ANALYSIS OF THE STAR ANISE ESSENTIAL OIL AS THE POTENTIAL BIOPESTICIDE

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ABSTRACT: Since the main components of essential oils are considered responsible for their biological activity, the chromatographic analysis of star anise essential oil was done. The identification of the essential oil compounds was carried out by matching their linear retention indices and MS spectra with those of authentic standards from MS library data bases. The main constituent was *trans*-anethole with 84.79% while 24 other constituents in total made less than 15.00% of the studied essential oil.

Key words: *biological activity, star anise, essential oil, trans-anethole, GC-MSD*

INTRODUCTION

The attention drawn by essential oils due to their biological effect as potential agents in pest control is evidently growing from year to year. They are considered to be the by-products of plant metabolism and regarded to as evaporable secondary metabolites of plants which are the mixture of mono and sesquiterpenes (Stojanović et al., 2018). The biological activity of essential oils depends on their chemical composition, the part of the plant they have been extracted from, phenological phase of the plant, environmental conditions and the extraction methods (Ukeh and Umoetok, 2011). A large number of isolated allelochemicals show their bioactivity in low (10^{-5}–10^{-6} mol/L) or extremely low concentrations (10^{-10} mol/L) (Šućur, 2015).

The star anise (Image 1) is an evergreen aromatic plant from *Schisandraceae* family native to southwest China. It is widely cultivated in tropical and subtropical regions and is used as food as well as a medicine, which indicates small or no toxic effects on humans (Zhou et al., 2016). The essential oil of star anise is applied locally for rheumatism and externally after childbirth, it is also used as an antiseptic, as well as for the treatment of emesis, stomach ache, pain and insomnia. The content of essential oil in fresh fruits is 2.5-3.5% while in case of dried material is between 8 and 9 percent (Wang et al., 2011).

Since the main components of essential oils are considered responsible for their biological activity the objective was to carry out the chromatographic analysis of star anise essential oil obtained in the distillation by water vapor.

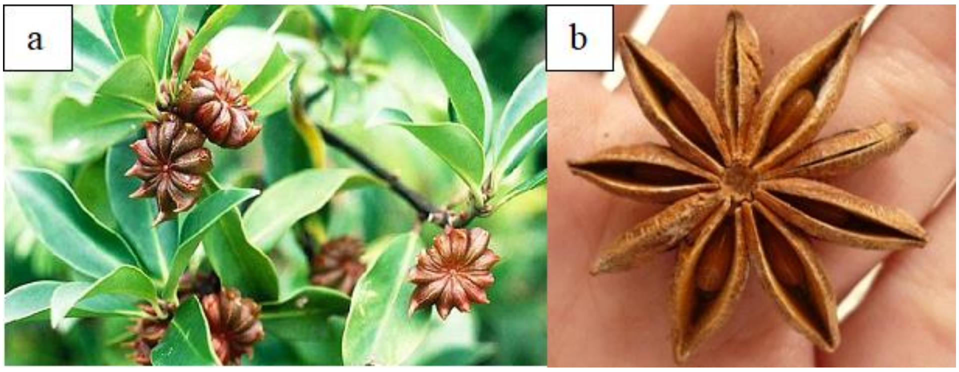


Image 1. The plant (a) and the fruit (b) of star anise
(a) https://balconygardenweb.com. (b) original image (Stojanović, T.)

MATERIAL AND METHODS

Chemicals and equipment

All chemicals of analytical grade and reagents used in this study were purchased from Sigma Chemical Co. (USA). Gas chromatograph-mass spectrophotometer was from Agilent Technologies (USA), model 7890A.

Essential oil extraction

The essential oil of star anise was extracted from commercially available fruit (fructus) by hydrodistillation (HD) with n-hexane as an organic solvent/recipient. Thirty grams of star anise fruit were subjected to hydrodistillation for 3 hours using a Clevenger-type apparatus according to the method outlined by the European Pharmacopoeia (Council of Europe. 2010). The essential oil was collected over water, separated, dried over anhydrous sodium sulphate and stored in the dark at 4 °C.

GC-MSD analysis

Gas chromatography (GC) and gas chromatography–mass spectrometric (GC–MS) analyses were performed using an Agilent 7890A GC equipped with an inert 5975C XL EI/CI mass spectrometer detector (MSD) and flame ionisation detector (FID) connected by capillary flow technology 2-way splitter with make-up. The HP-5MS capillary column (30 m×0.25 mm×0.25 μm) was used. The GC oven temperature was programmed from 60 to 300 °C at a rate of 3 °C min^{-1} and held for 15 min. Helium was used as the carrier gas at 16.255 psi (constant pressure mode). An auto-injection system (Agilent 7683B Series Injector) was employed to inject 1 μL of sample. The sample was analyzed in the splitless mode. The injector temperature was 300 °C and the detector temperature 300 °C. MS data were acquired in the EI mode with scan range of 30–550 m/z, source temperature of 230 °C, and quadruple temperature of 150 °C; the solvent delay was 3 min.

Compound identification

Identification of all compounds in the analyses was match by comparison of their linear retention indices (relative to C8-C36 n-alkanes on the HP-5MSI column) and MS spectra with those of authentic standards from NIST11 and homemade MS library data bases (Petrović et al., 2019).

RESULTS AND DISCUSSION

From the obtained results it could be concluded that the main constituent of the star anise essential oil is *trans*-anethole with 84.79% in content, which can be evidently seen in the chromatogram (Figure 1). Beside *trans*-anethole there were 24 constituents which in total make less than 15.00% of the studied essential oil (Table 1).

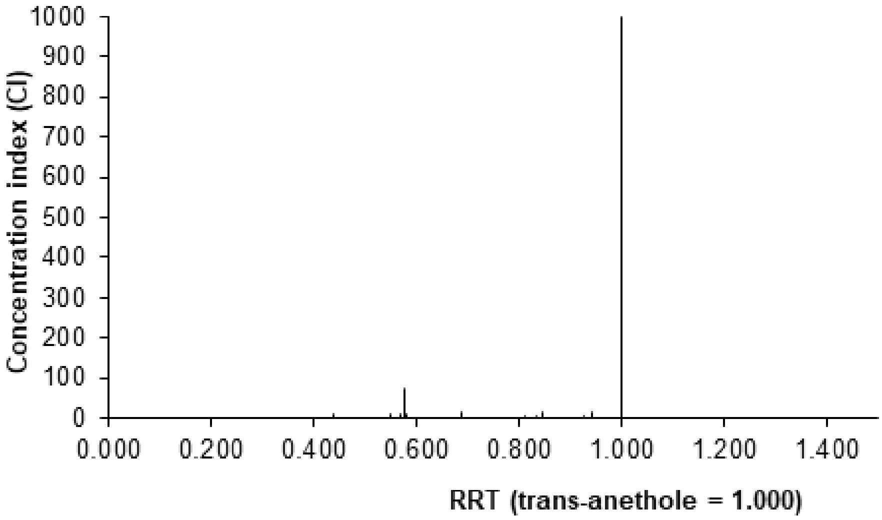
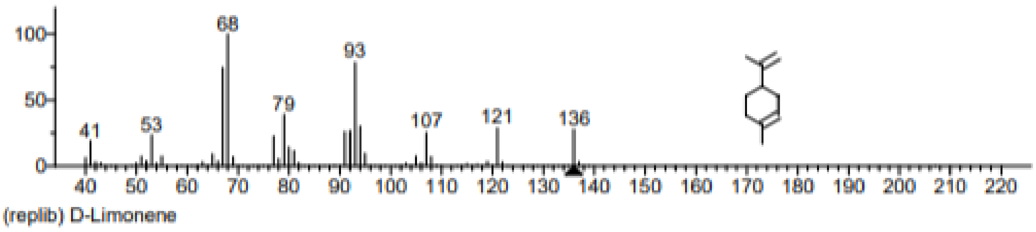


Figure 1. Chromatogram of the essential oil of star anise

Based on the chromatographic analysis of the star anise essential oil obtained in the distillation by water vapor it can be concluded that the main constituent of the star anise essential oil is *trans*-anethole with 84.79% in content, while the limonene (Figure 2) was the second present substance in the essential oil with 6.18% (Table 1).



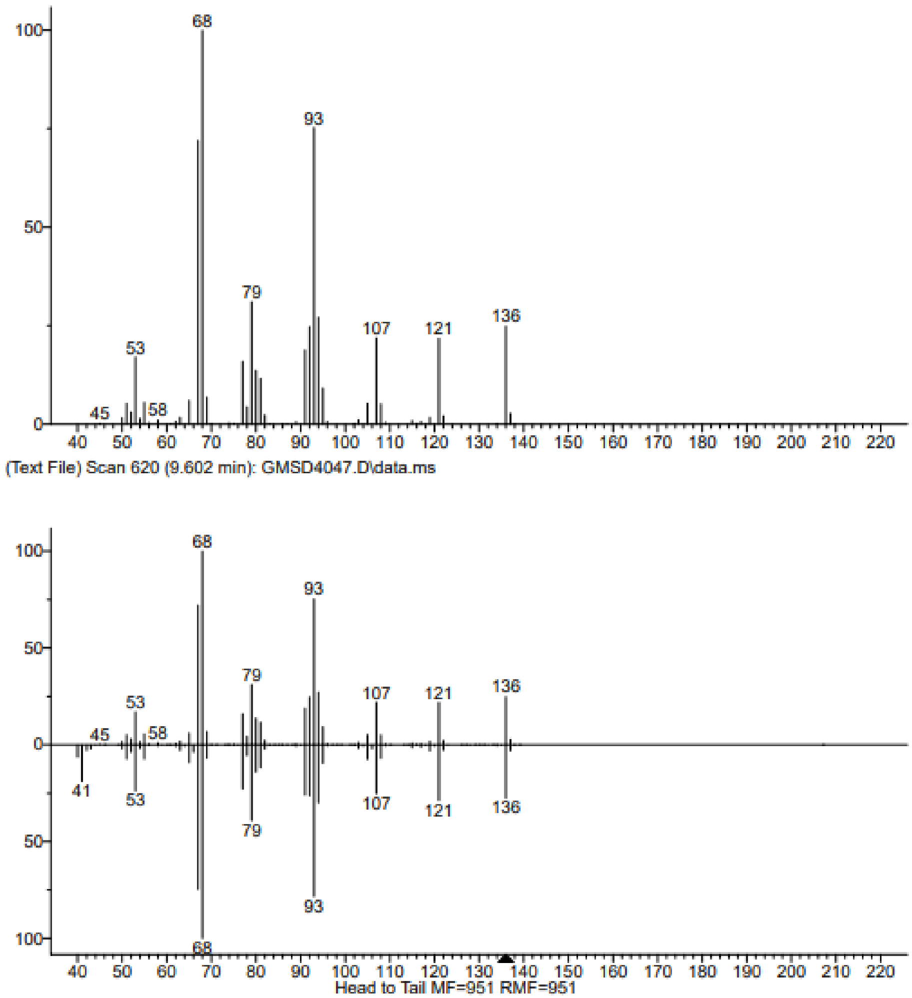


Figure 2. Limonene MS spectar

Beside *trans*-anethole there were 24 constituents which in total make less than 15.00% of the studied essential oil.

A number of studies led to the similar findings. Zhou et al. (2016) also identified *trans*-anethole as the main constituent of the star anise essential oil. The same group of authors stated that numerous studies showed the insecticidal effect of *trans*-anethole in case of German cockroach (*Blattella germanica*), Brown-tail moth (*Euproctis chrysorrhoea*) and the Mediterranean fruit fly (*Ceratitis capitata*).

Table 1. Constituents of the star anise essential oil

No	Constituents	Kie	Kil	Rt/ms	Rt/fid	Area	%. m/m	% id	Rrt	Ci
1	*A*-pinene	927.0	932	6.58	10.776	368.2	0.76	0.76	0.437	9
2	Camphene	941.3	946	7.00	11.295	15.1	0.03	0.03	0.458	0
3	*B*-pinene	970.1	974	7.87	12.301	38.1	0.08	0.08	0.499	1
4	Myrcene	991.6	988	8.58	12.763	72.5	0.15	0.15	0.517	2
5	*A*-phellandrene	1001.0	1002	8.80	13.283	94.6	0.20	0.20	0.538	2
6	*D*^{3}-carene	1005.1	1008	8.94	13.504	331.7	0.69	0.69	0.547	8
7	*A*-terpinene	1013.1	1014	9.21	13.741	51.2	0.11	0.11	0.557	1
8	*P*-cymene	1020.4	1020	9.46	14.067	343.9	0.71	0.71	0.570	8
9	Limonene	1023.5	1024	9.56	14.257	2975.6	6.18	6.18	0.578	73
10	1.8-cineole	1024.9	1026	9.61	14.340	468.5	0.97	0.97	0.581	11
11	*G*-terpinene	1056.5	1054	10.71	15.346	61.8	0.13	0.13	0.622	2
12	N.i.	1071.8		11.22	15.922	17.1	0.04		0.645	0
13	Terpinolene	1086.3	1086	11.72	16.506	88.1	0.18	0.18	0.669	2
14	Linalool	1099.3	1095	12.16	16.990	540.4	1.12	1.12	0.689	13
15	Terpinen-4-ol	1173.4	1174	14.73	20.038	179.5	0.37	0.37	0.812	4
16	*A*-terpineol	1187.9	1186	15.24	20.572	192.1	0.40	0.40	0.834	5
17	Estragole	1198.7	1195	15.62	20.817	511.1	1.06	1.06	0.844	13
18	*Cis*-anethole	1255.3	1249	17.56	22.882	235.1	0.49	0.49	0.927	6
19	*P*-anis aldehyde	1261.3	1251	17.74	23.254	524.1	1.09	1.09	0.942	13
20	*Trans*-anethole	1288.4	1282	18.65	24.673	40843.0	84.79	84.79	1.000	1000
21	*A*-copaene	1366.7	1374	21.20	27.359	33.1	0.07	0.07	1.109	1
22	*Cis-a*-bergamotene	1406.3	1411	22.48	28.632	41.0	0.09	0.09	1.160	1
23	*Trans*-caryophyllene	1408.5	1417	22.54	28.915	25.8	0.05	0.05	1.172	1
24	*Trans-a*-bergamotene	1427.0	1432	23.11	29.324	91.8	0.19	0.19	1.188	2
25	*Trans-b*-farnesene	1449.8	1454	23.82	30.104	25.1	0.05	0.05	1.220	1
	SUM					48168.6	100.00	99.96		

Kim and Ahn (2001) determined the *trans*-anethole toxic activity towards the adults of rice weevil (*Sitophilus oryzae*) and cigarette beetle (*Lasioderma serricorne*). In the studies conducted by Lopez et al. (2008) and Ebadollahi (2013) *trans*-anethole showed the toxic effect against the lesser grain borer (*Rhyzopertha dominica*).

CONCLUSIONS

The identification of the essential oil compounds from the star anis was carried out by matching their linear retention indices (relative to C8-C36 *n*-alkanes on the HP-5MSI column) and MS spectra with those of authentic standards from NIST11 (2011) and homemade MS library data bases. The major detected compounds were *trans*-anethole (84.79%) and limonene (6.18%).

Considering the literature data essential oil of star anise has a potential as a biopesticide. Based on the findings of this study and the assumption that the main components affect the biological activity of essential oil *trans*-anethole (propenylphenol synthesized via the shikimic acid-phenylpropanoid pathways) is considered responsible for biological activity of the star anise essential oil and its biopesticide potential.

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