Extraction of pyrethrins from Dalmatian pyrethrum (*Tanacetum cinerariifolium* /Trevir./ Sch. Bip.) by matrix-solid phase dispersion

Martina Biošić[1]
Ines Topalović[1]
Dario Dabić[1]
Filip Varga[2,3]
Martina Grdiša[2,3]

[1]Department of Analytical Chemistry, Faculty of Chemical Engineering and Technology, University of Zagreb, Marulićev trg 19, Zagreb, Croatia
[2]Department of Seed Science and Technology, Faculty of Agriculture, University of Zagreb, Svetošimunska 25, Zagreb, Croatia
[3]Centre of Excellence for Biodiversity and Molecular Plant Breeding (CoE CroP-BioDiv), Svetošimunska cesta 25, Zagreb. Croatia

Introduction

Fig. 1. Dalmatian pyrethrum (*Tanacetum cinerariifolium* /Trevir./ Sch. Bip*)*

Dalmatian pyrethrum (*Tanacetum cinerariifolium* /Trevir./Sch. Bip.) is a plant species of the Asteraceae family, restricted to the eastern coast of the Adriatic Sea. It is a perennial plant species known as the source of natural insecticide pyrethrin. The term pyrethrin refers to the six insecticide active components: pyrethrin I and II, cinerin I and II, jasmolin I and II. Insecticidal potential of Dalmatian pyrethrum is measured by the content and composition of pyrethrins which are mainly concentrated in the flower heads.

In order to obtain deeper knowledge of its insecticidal potential, content and composition of pyrethrins was assessed by matrix-solid phase dispersion (MSPD). MSPD, a process allowing simultaneous extraction and clean-up of analytes from solid samples, is used as an alternative technique to classical extraction methods with significant reduction in solvent consumption. The performance of MSPD is based on blending analyzed samples (solid or semi-solid) with a suitable adsorbent to form a homogenous packing material. After successful packing, the sample/adsorbent column is eluted by a stepwise solvent program.

Materials and methods

(I) the flower heads are blended with the dispersant material in a mortar with a pestle

(II) the homogenized powder is transferred in a solid-phase extraction cartridge, and compressed

(III) elution with a suitable solvent or solvent mixture is performed by the aid of a vacuum pump

Fig. 2. Main steps of matrix-solid phase dispersion extraction procedure

<u>1st set of experiments:</u>

(I) flower heads:florisil = 1:2
flower heads:C18 = 1:2
flower heads:silikagel = 1:2

(III) 10 mL of ethanol
10 mL of acetone
10 mL of acetonitrile
10 mL of ethyl acetate
10 mL of dichloromethane

<u>2nd set of experiments:</u>

(I) flower heads:florisil = 1:2
flower heads:silikagel = 1:2

(III) 10 mL of acetone:ethyl-acetate = 1:1 (*v/v*)
10 mL of acetone:ethyl-acetate = 1:2 (*v/v*)
10 mL of acetone:ethyl-acetate = 1:4 (*v/v*)
10 mL of acetone:ethyl-acetate = 2:1 (*v/v*)
10 mL of acetone:ethyl-acetate = 4:1 (*v/v*)

Results

Table 1. Comparison of recoveries obtained using different sorbents and solvents (*n* = 3) in the first set of experiments

pyrethrins	Recovery (%)											
	FLORISIL				SILIKAGEL				C18			
	ethanol	acetone	acetonitrile	ethyl-acetate	ethanol	acetone	acetonitrile	ethyl-acetate	ethanol	acetone	acetonitrile	ethyl-acetate
cinerin II	40.77	76.41	80.61	83.26	21.93	92.59	63.68	109.14	24.42	33.69	70.16	81.91
pyrethrin II	53.60	83.39	66.96	79.54	49.32	91.47	66.94	106.69	57.39	73.85	70.08	73.26
jasmolin II	58.95	88.07	93.32	80.75	34.29	85.30	58.26	58.19	58.79	70.22	77.74	81.90
cinerin I	48.49	91.28	82.94	88.91	22.59	83.95	60.08	108.30	20.33	26.48	23.01	30.11
pyrethirn I	50.89	83.09	73.19	85.46	52.04	84.44	68.35	99.79	51.14	62.49	59.11	67.22
jasmolin I	50.44	91.05	88.12	75.90	45.16	95.35	57.54	102.38	58.16	69.88	38.75	78.93

*Recoveries obtained using dichloromethane as elution solvent were very low for all six pyrethrins (data not shown)

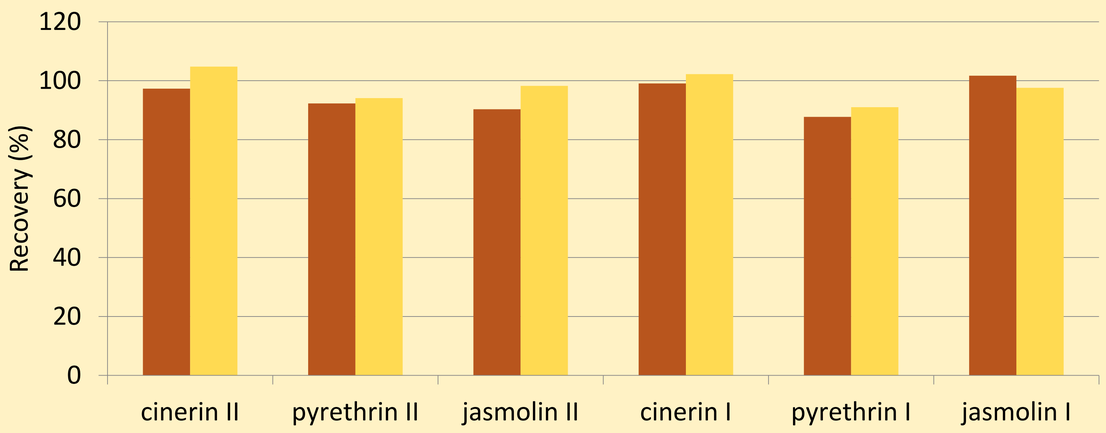

Fig. 3. Recoveries obtained using mixture of acetone:ethyl-acetate = 1:1 (*v/v*) and florisil (lighter column) or silikagel (darker column) sorbent (*n* = 3) in the second set of experiments

All six pyrethrins were isolated from flower heads of Dalmatian pyrethrum using MSPD followed by HPLC-DAD. Results showed that pyrethrins I and pyrethrins II were successfully extracted by florisil as a sorbent using 10 mL of acetone:ethyl-acetate = 1:1 (*v/v*) as an elution solvent using 1:2 ratio of flower heads and sorbent. Presented results prove that MSPD can be attractive, affordable and effective method for the extraction of pyrethrins from Dalmatian pyrethrum.

Conclusion

10th Conference on Medicinal and Aromatic Plants of Southeast European Countries, Split, Croatia

Acknowledgement: The research has been fully supported by the Croatian Science Foundation under the project "Genetic background of Dalmatian pyrethrum (*Tanacetum cinerariifolium* /Trevir./ Sch. Bip.) insecticidal potential" – (PyrDiv) (IP-06-2016-9034).