Srpsko hemijsko društvo
Serbian Chemical Society

Sekcija za hemiju i zaštitu životne sredine
Chemistry and Environmental Protection Division

6. simpozijum

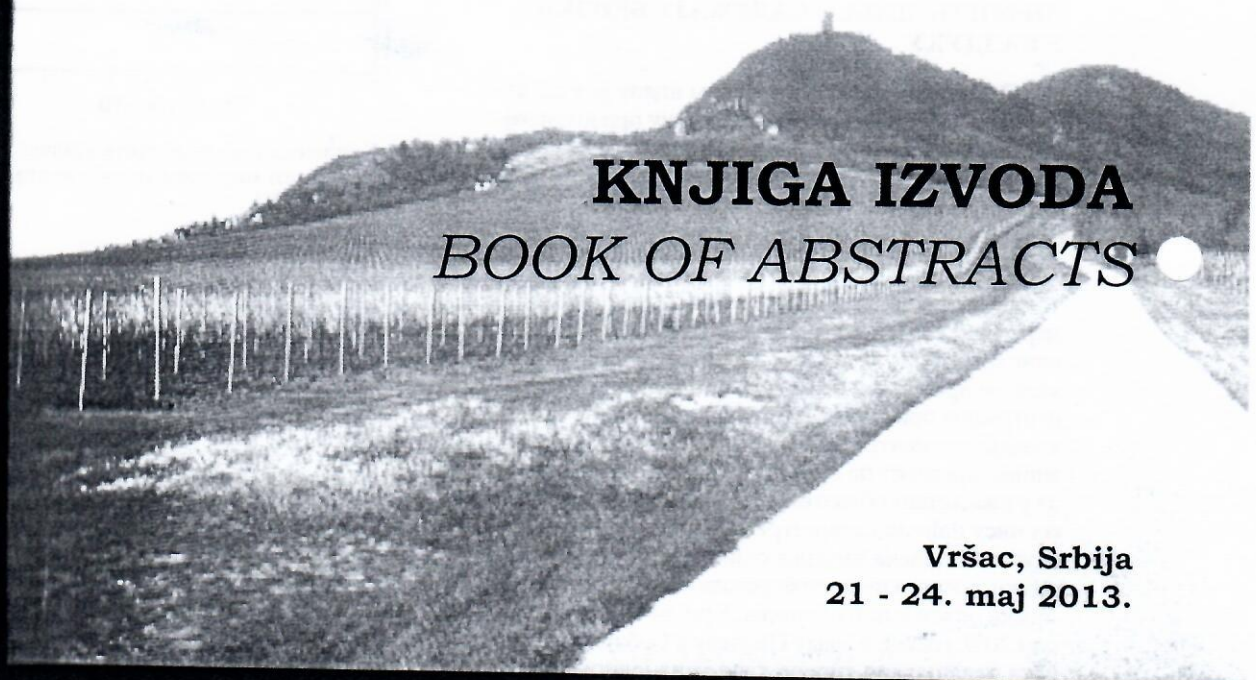
Hemija i zaštita životne sredine EnviroChem 2013

sa međunarodnim učešćem

6th Symposium

Chemistry and Environmental Protection EnviroChem 2013

with international participation



KNJIGA IZVODA

BOOK OF ABSTRACTS

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Solid-phase extraction followed by high-performance liquid chromatography with diode array detection for screening of dicamba herbicide in water

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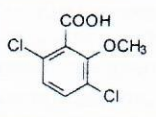
Chlorinated acids are selective agricultural herbicides which are widely employed in agriculture and gardening for control the growth of different unwanted vegetable species in crops. Because of high water solubility and toxicological risk of some acid herbicides and their metabolic products, monitoring of their concentration in surface and groundwater is very important task. The acidic herbicides are manufactured in formulation as free acids, as their alkaline salts or as esters. The unionized free acids vary in water solubility (Table 1), but the acidic herbicides most frequently exist in ionized form at environmental pH values. Acidic herbicides formulated as salts are water soluble, while those formulations prepared as esters are less water soluble. In the environment, acidic herbicides formulated as esters have short hydrolysis half-life time (24–48 h) and therefore they are generally present as ionized acids. For most analytes, especially for the acidic herbicides, solid phase extraction (SPE) is the choice of sample treatment, which is followed by appropriate chromatographic separation and sensitive determination of target components. For the acidic herbicides, combination of physico-chemical parameters influences their extraction from aqueous solution. Ionogenicity (pKa) and hydrophobicity (logKow) are especially important in determining the approach of SPE for efficient sample clean-up for further chromatographic analysis of chlorophenoxy acid herbicide in water samples.

Table 1. Physico-chemical properties of dicamba acidic herbicide

Common name/ molecular formula/ CAS No.	Systematic name	Structure	pKa	Aqueous solubility (mg/l)	$logK_{ow}$
Dicamba $C_8H_6Cl_2O_3$ (1918-00-9)	3,6-dichloro-2-methoxy-benzoic acid		1.9 [1]	4500 [2]	2.21 [3]

The acidic herbicides are polar and non-volatile compounds, and do not lend themselves to direct analysis by gas chromatography. Using high performance liquid chromatography (HPLC) the acidic herbicides can be analyzed in the ionic

form, the molecular (unionized acid) form or as the ester. Generally, the mechanism of separation/clean-up on SP extraction sorbent, used to extract the acidic herbicides from aqueous solution, is based on Van der Waals interactions (reversed phase bonded silica sorbents) or by electrostatic interactions (anion exchange). The present work describes screening method for efficient sample clean-up procedure for the determination of dicamba acid herbicide in water, using SPE. Methodology is based on the use of polymer-based weak anion exchange SPE sorbents (Strata X-AW) for fast extraction of the dicamba from the water samples and on optimised instrumental analytical method based on reversed-phase HPLC with diode-array detector (DAD, 210 nm) for determination of target analyte from the extract. The chromatographic separation was carried out on Zorbax C_{18} (50 mm × 4.6 mm, 1.8 μm) using an isocratic elution profile and mobile phase consisting of 13 mM phosphate buffer pH 3.4 and acetonitrile. Method validation was performed by analysing freshly spiked tap water samples with dicamba at levels between 0.5 and 5 μg/ml. Average recovery of the method ranged between 86.7-95.8%. Besides the regularly shaped and well-defined peaks belonging to the investigated dicamba pesticide, the SPE-HPLC-DAD chromatograms (Figure 1) contained of peaks which probably have origin from the solvent/mobile phase. There were no significant interfering peaks in the elution region of dicamba pesticide.

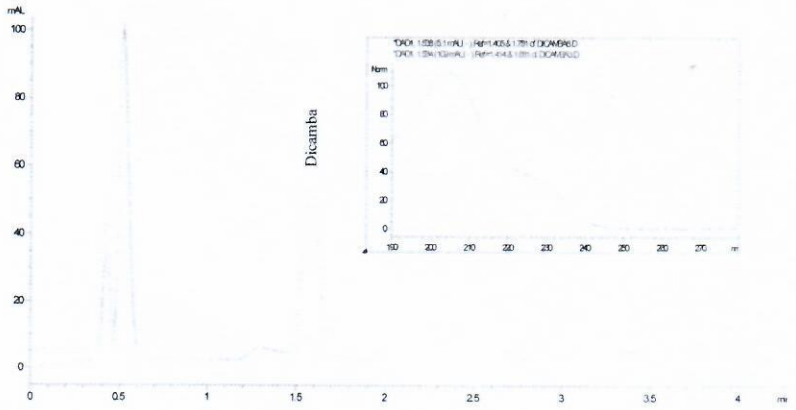


Figure 1. Comparison of chromatograms and the appropriate UV apex spectrums (as insets) of dicamba herbicide peaks of fortified tap water extract (lower) and standard dicamba solution (upper)

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