Proceedings of the 7th Congress on Plant Protection

Доклады 7-ого Конгресса по защите растений

Plant Protection Society of Serbia
Общество по защите растений Сербии

International Organization for Biological Control
-East Palearctic Regional Section (IOBC-EPRS)
-West Palearctic Regional Section (IOBC-WPRS)

Международная организация по биологической борьбе
- Восточно палеарктическая региональная секция (МОББ-ВПРС)
- Западно палеарктическая региональная секция (МОББ-ЗПРС)

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„Integrated Plant Protection – a Knowledge-Based Step towards Sustainable Agriculture, Forestry and Landscape Architecture“

(November 24-28, 2014, Zlatibor, Serbia)

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„Интегрированная защита растений - научно обоснованный шаг к устойчивому развитию сельского хозяйства, лесоводства и пейзажной архитектуры“

(24-28 ноября 2014 года, Златибор, Сербия)

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PREFACE

The Plant Protection Society of Serbia (PPSS) and two regional sections of the International Organization for Biological and Integrated Control (IOBC-EPRS and IOBC-WPRS), on the occasion of the 60th anniversary of the PPSS organized VII Congress on Plant Protection with a motto: *"Integrated Plant Protection – a Knowledge-Based Step towards Sustainable Agriculture, Forestry and Landscape Architecture"* (November 24-28, 2014, Zlatibor, Serbia). The Congress enabled exchange of up-to-date scientific and technical information on plant protection in Agriculture, Forestry and Landscaping among researchers, teachers, experts in extension and public services and the business community, and promoted international cooperation. The Congress focused on basic knowledge and management practices established in plant protection, as well as on the development of alternative and innovative approaches. In addition, biological control as an important tool for the control of the harmful organisms with a minimal risk for ecosystems was discussed. A total of 209 contributions was presented - 8 keynote presentations, 28 oral presentations and 173 poster presentations - prepared by 467 authors from 26 countries. The Congress Proceedings comprise 65 contributions - 5 keynote presentations and 60 oral and poster presentations in six sessions, prepared by the authors from 18 countries (Algeria, Austria, Bosnia-Herzegovina, France, Georgia, Hungary, Italy, Kazakhstan, Montenegro, Poland, Russia, Rwanda, Serbia, Slovenia, Switzerland, Turkey, Uganda, USA). All contributions were reviewed by members of the Scientific Committee and other reviewers selected and invited by the editors of this publication.

Belgrade, November 2015 Editors

ПРЕДИСЛОВИЕ

Общество по защите растений Сербии (ОЗРС), Международная организация по биологической борьбе с вредными животными и растениями - Восточно палеарктическая региональная секция (МОББ-ВПРС) и Международная организациая по биологической борьбе и интегрированной системе защиты растений - Западно-палеарктическая региональная секция (МОББ-ЗПРС), по поводу 60-летия ОЗРС организировали VII Конгресс по защите растений, под девизом: *"Интегрированная защита растений - научно обоснованный шаг к устойчивому развитию сельского хозяйства, лесоводства и пейзажной архитектуры"* (24-28 ноября 2014 года, Златибор, Сербия). Цель Конгресса была обеспечение континуитета взаимообмена научно-техническими информациями, отвечающими современным требованиям защиты растений в сельском хозяйстве, лесоводстве и пейзажной архитектуре, которые представляют интерес для ученых, исследователей, преподавателей, экспертов-советников в области сельского хозяйства, лесоводства и пейзажной архитектуры, специалистов государственных и коммунальных служб, деловых кругов и средств массовой информации. Целью Конгресса является и продолжение содействия развитию и популяризации международного сотрудничества. Конгресс был концентрирован на основные знания и практический менаджмент в защите растений, а также на развитие алтернативних и новых подходов. Биологическая защита каторая представляет значительный способ для безопасной борьбы с вредними организмими была тоже рассмотривана. На конгрессе представлено 209 презентаций - 8 докладов по приглашению, 28 устных и 173 постер презентаций - которые подготовило 467 авторов из 26 стран. Сборник имеет 65 докладов - 5 докладов по приглашению и 60 устных и постер презентаций, распределенных в шести секциях. Авторы докладов приехали из 18 стран (Алжир, Австрия, Босния-Герцеговина, Франция, Грузия, Венгрия, Италия, Казахстан, Черногория, Польша, Россия, Руанда, Сербия, Словения, Швейцария, Турция, Уганда, США). Рецензенты всех опубликованных докладов в сборнике – члены Научного совета и другие рецензенты, выбранные редакторам этого издания.

Белград, Ноября 2015 Редакторы

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DETERMINATION OF PESTICIDE RESIDUES IN WATERMELONS BY LC-MS/MS

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ABSTRACT

The LC–MS/MS was applied for the detection of pesticide residues in watermelons. Since watermelons are predominantly used as fresh food and to a lesser extent in food processing there is a justified concern that, due to treatments, they can contain pesticide residues above the maximum residue levels – MRLs. The pesticide extraction was carried out by QuEChERS method. The samples were tested regarding the content of 55 pesticides with the carbofuran-D3 as internal standard. The linearity was studied in the range of 0.01–0.50 mg/kg and the determination coefficients (R^2) were higher than 0.99 for all the investigated pesticides. The calibration was performed as matrix calibration, by means of spiking the calibration samples before the extraction and preparing them in the same way as the test samples. The recovery data were obtained by spiking blank samples at three concentration levels (0.01, 0.05 and 0.1 mg/kg) yielding recoveries in the range of 61.0–114.2% with the relative standard deviation (RSD) less than 13%. The limits of quantification (LOQs) were established as 0.01 mg/kg. The multiple detections were confirmed in the analysed samples. The most frequently detected pesticides were carbendazim, acetamiprid, tefluthrin and fenpropimorph. In only one sample the concentrations of carbendazim and tefluthrin were above the MRLs.

Key words: Pesticide residues, QuEChERS, watermelons, LC-MS/MS

INTRODUCTION

The watermelon (*Citrulus vulgaris* sin. *Citrulus lanatus Thumb.*) is a sweet, juicy, rich in β-carotene (Đurovka and Ilin, 2002) and very nutritious fruit, that is packed with some of the most important antioxidants in nature. In addition to vitamin C and A, the watermelon harbors lycopene, an efficient oxygen radical scavenger, which can protect against chronic diseases such as cancers, cardiovascular diseases, and osteoarthritis inflammation (Park et al., 2010). Since watermelon is farmed primarily *via* protected and successive cultivation techniques, it is far more susceptible to physiological disorder and damage due to pests and diseases than are plants raised under usual cultivation conditions (Nguyen et al., 2008). A number of pesticides need to be used to control pests and diseases in order to increase watermelon production. On our market there are 7 compounds registered for the use in watermelon protection out of which 1 is an insecticide, 4 are herbicides and 4 fungicides (Sekulić and Jeličić, 2013). Additionally, many chemicals deriving from indirect sources such as soil, contaminated with agro-inputs, drift from adjoining crop fields, *etc. and* may contaminate the edible inner part of the

watermelon, and may affect human health (Park et al., 2010). Since watermelons are predominantly used as fresh food and to a lesser extent in food processing there is a justified concern that, due to treatments, they can contain pesticide residues above the maximum residue levels – MRLs (Bursić et al., 2014).

The health safety of food is of great significance for consumers, food industry and economy (Jevšnik et al., 2008). Thus our county adapted the MRLs values to the current MRLs in the European Union (Off. Gazette RS 29/2014; Regulation EC No 396/2005).

Therefore, to protect human health and control the environmental pollution, sensitive and efficient analytical methods for the determination of pesticide residues at trace levels are desirable (Wang et al., 2011). Nowadays, many analytical methods reported in the literature for the determination of pesticides involve gas chromatography (GC) equipped with most commonly used detectors such as electron capture detectors (ECD), nitrogen phosphorus detectors (NPD) or mass spectrometers (MS) which have been used broadly for many years to monitor volatile and thermally stable pesticides. The liquid chromatography-mass spectrometry (LC-MS) is an ideal technique for the analysis of residues of non-volatile and thermally unstable pesticides. It has been recently reported that LC-MS/MS is capable of analyzing pesticide multiresidues more efficiently than LC-MS (Park et al., 2010). The LC/MS-MS method has high selectivity and sensitivity, simplicity of sample cleanup, and easy and reliable indentification and quantification, even at trace levels of pesticides (Vuković, 2012).

Nevertheless, there are some difficulties for pesticide residues direct determination due to their low concentration in most cases. So, to obtain accurate and sensitive results, the determination of the pesticides is usually accomplished by many preliminary steps like sampling, extraction, and clean-up for interference removal and analyte concentration before chromatographic analysis (Wang et al., 2011). The trends in recent years have been directed towards the decrease in sample amounts for the analysis with the approach which is safe and less harmful to the environment and at the same time implies a quicker, simpler method for sample preparation with simultaneously providing high recovery and good precision (Bursić et al., 2013). Anastasiades et al. (2003) developed a quick, essential, cheap, efficient, robust and safe method (QuEChERS) so as to overcome the limitations of the existing preparation methods.

To evaluate the negative effects of pesticides in watermelons and to ensure the consumers safety, the validated multiresidue LC/MS-MS method was used for the detection of pesticide residues. The pesticide extraction was carried out by the most promising sample preparation techniques QuEChERS (Vuković et al., 2012; Bursić et al., 2014). That is why in this study the purpose was to use QuEChERS method for the extraction and LC-MS/MS for the detection of 55 pesticides in watermelons in the control of human food safety.

MATERIAL AND METHODS

Materials

All solvents used were chromatography grade and were obtained from J.T. Baker (Deventer, Netherlands). Certified pesticide analytical standards were purchased from Dr. Ehrenstorfer (Augsburg, Germany), most of them of purity ≥98%. The internal standard carbofuran-D3 (99.7%) was purchased from Pestanal, Fluka (Germany).

QuEChERS Extract Tubes, EN Method, part No: 5982-5650 and Dispersive SPE 15 mL (High pigmented), part No: 5982-5356 were purchased from Agilent Technologies (USA).

Sample preparation

The samples were taken in accordance with the Regulation on methods of food sampling and testing aimd at the determination of plant protection products residues in food (Off. Gazzete RS No 110/2012) which defines the sampling methods and the minimum amount of laboratory samples. Three average samples of watermelon at the stage of technological maturity were taken from the fields. The samples were put into polyethylene bags and immediately transferred to the laboratory. On arrival each sample was homogenyzed and kept in a deep freeze at the temperature of -18 °C till being analyzed.

The extracts were obtained using the acetonitrile-based QuEChERS sample preparation technique (Figure 1.). The basic samples of waterelons, were collected from various field in Vojvodina. The sampling was carried out at the end of July 2013. All the samples were kept in polyethilene black bags in deep-freeze until being analyzed.

Analytical determination

Agilent 1100 Series HPLC system with Zorbax XDB C18 analytical column of 50×4.6mm and 1.8 mm particle size (Agilent Technologies) column was

used. For LC analysis, an Agilent 1200 HPLC system with a binary pump was used. For the mass spectrometric analysis, an Agilent 6410B Triple-Quad LC/MS system was used. Agilent MassHunter Workstation Software version B.04. QQQ Agilent Technologies, 2011 were applied for method development and data acquisition.

Validation: The method was validated according to SANCO/12571/2013. The limit of detection - LOD was determined as the lowest concentration giving a response of three times the average baseline. The ratio signal/noise in the obtained chromatograms for the LOD was calculated MassHunter Qualitative Software. The linearity was checked using matrix matched standards (MMS) at concentrations of 5.0, 10.0, 25.0, 50.0 and 100.0 ng/mL. The recovery was checked by enriching 10 g of a blank sample with the mixture of pesticide standard of 10 mg/ml in the amount of 100 and 50 µL (final mass concentration 0.10 and 0.05 mg/kg) and with the mixture of pesticide standard of 1 mg/mL in the amount of 100 µl (final mass concentration 0.01 mg/kg) with the addition of the internal standard carbofuran-D3.

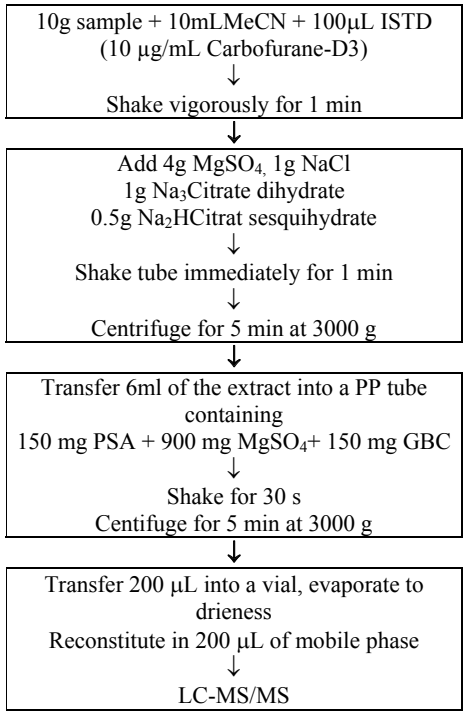

Figure 1. QuEChERS extraction

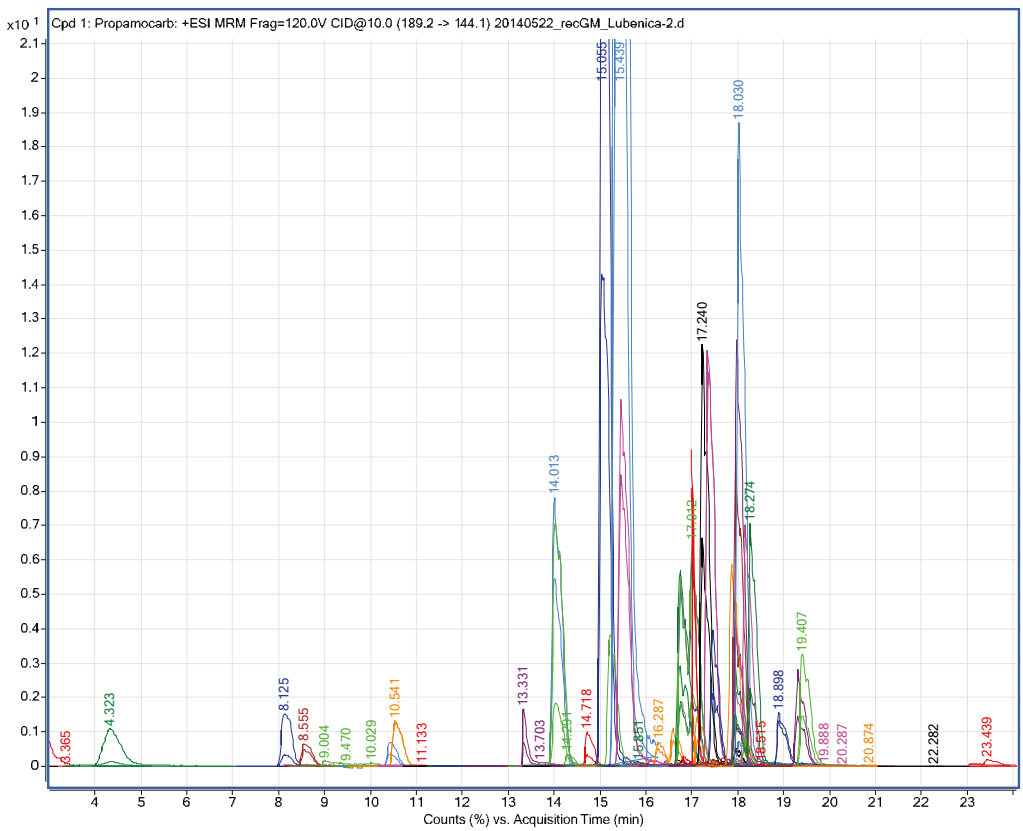

Figure 2. Overlaid MRM chromatograms of 55 pesticides standard in watermelon sample spiked at 10.0 ng/mL

RESULTS

The LC-MS/MS was used for the simultaneous residue determionation of 55 pesticides (acetamiprid, azoxystrobin, bupirimate, carbendazim, carbofuran, carbosulfan, chlorpyrifos, clothianidin, cyproconazole 1&2, cyprodinil, clethodim, difenconazol, dimethomorph, endosulfan alpha, epoxiconazole, fenhexamide, fenoxycarb, fenpropathrin, enpropimorph, fenvalerate, flusilazole, flutriafol, hexaconazol, imidacloprid, indoxacarb, krezoxym-methyl, metalaxyl-M, metconazol, methomyl, methoxyfenozide, methydathion, myclobutanil, oxadixyl, penconazol, pencycuron, pirimicarb, pirimifos-methyl, propamocarb, propoxur, propyconazol, pyraclostrobin, pyrimethanil, pyriproxifen, spiroxamine, tebuconazol, tebufenpyrad, tefluthrin, thiabendazole, thiacloprid, thiodicarb, triadimefon, triadimenol, trifloxystrobin, trifluralin and zoxamide) in the watermelon. Most of the studied pesticides are comprised by the monitoring programme of Serbia, regarding the substances whose presence and residue levels are studied in the food of plant origin (Off. Gazzet RS 58/2014). The active substances which were analyzed i.e. added to the list are bupirimate, clethodim and propamocarb as they are registered in the application with watermelons (Sekulić and Jeličić, 2011). The extraction was done using QuEChERS extraction kits with pre-weighed anhydrous salts in sealed packets which make it possible to add salts after adding organic solvents to samples, and to avoid an exothermic reaction that can compromise analyte recovery. Dispersive kits with sorbents and salts supplied in 15 mL centrifuge tubes accommodate the aliquot volumes specified by current AOAC and EN methodologies. These dispersive kits provide excellent recoveries and reproducibility for all types of fruits and vegetables.

The calibration was carried out in the watermelon matrix in order to overcome the matrix effect. The R^2 were >0.99 for all the studied pesticides ranging from 0.01 to 0.25 mg/mL. The obtained mean values of the responses were in the range from 61.0 to 114.2% with RSD <20.00%. The LOQs were 0.01 mg/kg (Bursić et al., 2014).

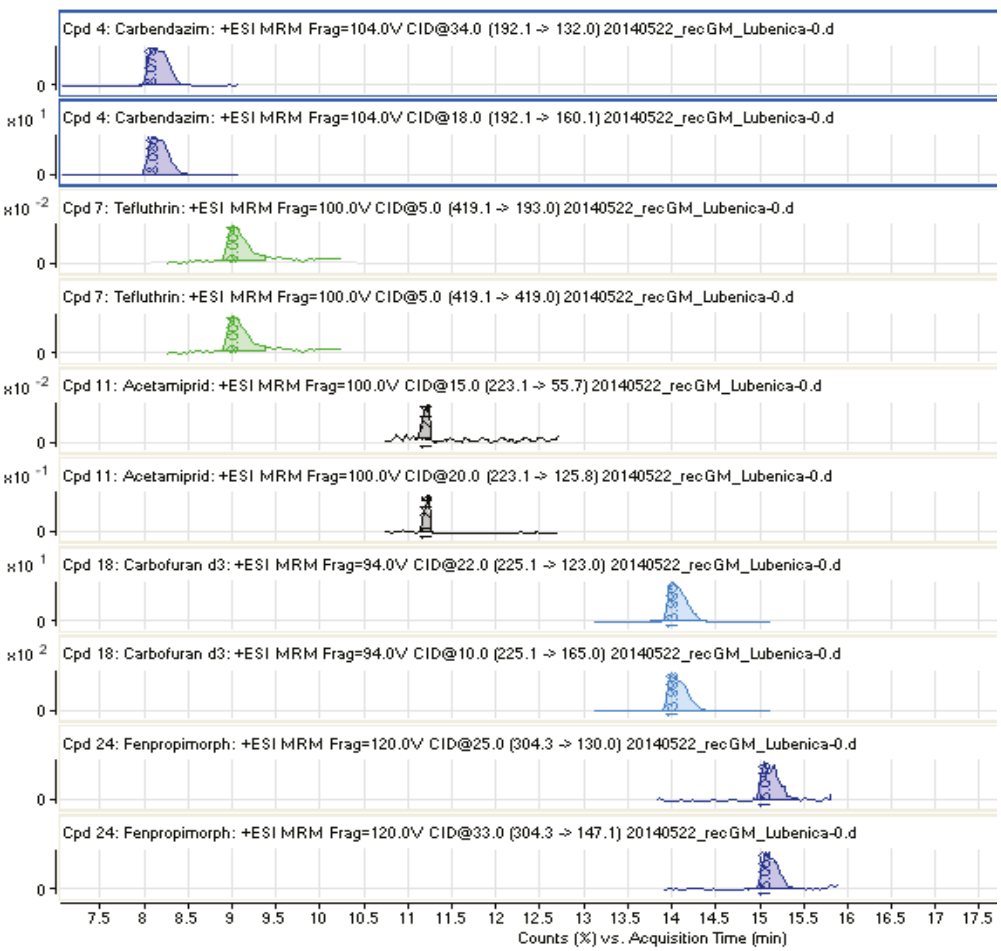

Figure 3. Pesticide residues detections in watermelon sample

DISCUSSION

The validated method which uses a liquid chromatography tandem mass spectrometry provides a very high sensitivity, good reproducibility, appropriate linearity and can be applied with the high reliability to the analysis of investigated pesticide residues in watermelon samples. The LOQs of 0.01 mg/kg confirm that the method is appropriate for the determination of pesticide residues in watermelon samples according to the regulations of the Serbian and EU MRLs.

The multiple detections were confirmed in the analysed samples. The most frequently detected pesticides were carbendazim, acetamiprid, tefluthrin and fenpropimorph. In only one sample the concentrations of carbendazim (0.134 mg/kg) and tefluthrin (0.304 mg/kg) were above the MRLs. The MRLs for this pesticide are 0.1 and 0.02 mg/kg, respectively. In the same sample the acetamiprid (0.005 mg/kg) and fenpropimorph (0.004 mg/kg) were detected, in the concentrations, which are much below the MRLs.

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