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ORIGINAL ARTICLE

OCCURRENCE OF PESTICIDE RESIDUES IN FRUITING VEGETABLES FROM PRODUCTION FARMS IN SOUTH-EASTERN REGION OF POLAND

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ABSTRACT

Background. Considering the fact that pesticides are commonly used in agriculture, continuous monitoring of these substances in food products is of great significance. Residues of these substances can be present in crops after harvest.

Objective. The aim of this study was to evaluate presence of pesticide residues in fruiting vegetables from production farms in south-eastern region of Poland in 2012–2015.

Material and methods. 138 samples were tested using accredited test methods. The monitoring programme covered determination of 242 pesticides. The tests covered tomato, cucumber and pepper crops. The test results were interpreted in accordance with criteria included in the European Commission recommendations published in the document SANCO/12571/2013 (now superseded by Document SANTE 2015), as well as on a basis of the maximum residue levels in force in the EU Member States.

Results. Pesticide residues were found in 47 samples, representing 34% of all tested samples. 17 active substances were found, belonging to fungicides and insecticides. Azoxystrobin (38%), boscalid (28%) and chlorothalonil (21%) were most commonly found in fruiting vegetables testing samples. Non-compliances related to use of plant protection product not authorized for protection of a given crop were observed in 6% of analysed samples. However, pesticide residues of fruiting vegetables in quantities that exceed the maximum residue levels (NDP, ang. MRLs), as well as substances which use for plant protection is forbidden were no found.

Conclusions. Crops monitoring is used to determine to what extent such products are contaminated with pesticide residues, and ensures protection of customer health.

Key words: *fruiting vegetables, pesticide residue analysis, maximum residue levels*

STRESZCZENIE

Wprowadzenie. Ze względu na powszechne stosowanie pestycydów w rolnictwie bardzo ważne jest prowadzenie stałej kontroli tych substancji w produktach spożywczych. Ich pozostałości mogą znajdować się w płodach rolnych nawet po zbiorze.

Cel badań. Celem pracy była ocena występowania pozostałości pestycydów w warzywach owocowych pochodzących z gospodarstw produkcyjnych z terenu Polski południowo-wschodniej w latach 2012–2015.

Materiał i metoda. Przebadano 138 próbek stosując akredytowane metody badawcze. Program kontroli obejmował oznaczenie 242 pestycydów. Badaniami objęto uprawy pomidora, ogórka, papryki i 242 substancje czynne. Wyniki badań interpretowano według kryteriów zawartych w zaleceniach Komisji Europejskiej i opublikowanych w dokumencie SANCO/12571/2013 (obecnie zastąpionym przez dokument SANTE 2015), a także w oparciu najwyższe dopuszczalne pozostałości pestycydów obowiązujących w państwach UE.

Wyniki. Pozostałości pestycydów wykryto w 47 próbkach warzyw owocowych, co stanowi 34% ogółu przebadanych próbek. Wykryto 17 substancji czynnych należących do fungicydów oraz insektycydów. W próbkach warzyw owocowych najczęściej wykrywano: azoksystrobinę (38%), boskalid (28%) i chlorotalonil (21%). W 6% analizowanych próbek stwierdzono nieprawidłowości związane z zastosowaniem preparatów niezarejestrowanych do ochrony danej uprawy. Natomiast w żadnej próbce warzyw owocowych nie stwierdzono pozostałości środków ochrony roślin w ilości, która przekraczałyby najwyższe dopuszczalne poziomy pozostałości (NDP, ang.MRLs), jak również substancji, których stosowanie w ochronie roślin zostało zabronione.

Wnioski. Kontrola płodów rolnych pozwala stwierdzić w jakim stopniu są one zanieczyszczone pozostałościami stosowanych pestycydów oraz zapewnia ochronę zdrowia konsumentów.

Słowa kluczowe: *warzywa owocowe, analiza pozostałości środków ochrony roślin, najwyższe dopuszczalne pozostałości*

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INTRODUCTION

Pesticides are chemicals used in agriculture to protect crops against insects, fungi, weeds and other pests. Although highly selective formulations are used acting on a specific agrophage or a specific group agrophages, in modern agriculture there is a risk that also humans will be affected. According to WHO, pesticides are also potentially toxic to humans. They may induce adverse health effects including cancer, effects on reproduction, immune or nervous systems. Before they can be authorized for use, pesticides should be tested for all possible health effects and the results should be analysed by experts to assess any risks to humans [23]. Considering the fact that pesticides are commonly used in agriculture, continuous monitoring of these substances in food products is of great significance. Their residues can be present in crops even after harvest.

Since 2004, Poland has been participating in the European monitoring system for pesticide residues in food and conducting tests for those substances as a part of official monitoring programme. Such tests facilitate quality assessment of plant products for correct use of plant protection agents in accordance with current legislation, as well as verification whether food products available in the market contain pesticide residues at a level exceeding current maximum residue levels (MRLs) [17], established in the European Commission Regulation No. 396/2005 [12].

The Laboratory of Pesticide Residue Analysis in Rzeszów conducts analyses of pesticide residues in fruit, vegetables and cereals as a part of official monitoring under the long-term programme for 2011–2015. This study covers results of tests conducted in 2012–2015 on a selected group of plants, i.e. fruiting vegetables (cultivated in a field or under shelter).

The aim of this study was to evaluate presence of pesticide residues in fruiting vegetables from production farms in south-eastern region of Poland in 2012–2015.

MATERIALS AND METHODS

Monitoring tests covered fruiting vegetables including tomatoes, cucumbers and peppers. Samples were collected randomly from farms by inspectors of the Inspectorates of Plant Health and Seed Inspection according to the Regulation of the Minister of Agriculture and Rural Development of 27 November 2013 [13]. All tested samples were raw products. The range of delivered samples resulted from the official monitoring programme, considering both production and consumption of relevant products. Vegetables were collected from the south-eastern region of Poland, from Podkarpackie, Małopolskie, Lubelskie and Świętokrzyskie voivodeships.

The monitoring program covered 242 substances. Table 1 presents classification of all active substances according to biocidal effects, with a quantification limit specified.

Accredited methods of gas chromatography and spectrophotometry were used for identification and quantification of tested active substances.

A multiresidue chromatographic method was based on residue extraction with an organic solvent and further purification of the extract with column chromatography. Final determination of residues was performed on gas chromatographs Agilent 7890 and Agilent 6890 equipped with electron capture (ECD) and nitrogen–phosphorus detectors (NPD) [7, 15].

Dithiocarbamate fungicides were analysed by a spectrophotometric method, based on their decomposition to CS_2 in acid environment and transfer to methyl blue, which was then analysed with the spectrometer Unicam Helios [1].

Analytical methods were verified in a validation process. Recovery assays of active substances were carried out on blank samples spiked with target compounds at two concentrations, in five replicates. The methods' trueness and precision parameters in terms of the average recovery and relative standard deviation were calculated and assessed according to the European Union guidelines [2, 3]. The linearity of the chromatographic and spectrophotometric responses were evaluated at five concentration levels. The measurement uncertainty of methods was estimated and was found to be compliant with European Union guidelines [2, 3]. Blank fortified samples were analysed within the framework of quality control/assurance, and methods' repeatability and reproducibility were also verified. Parameters of both methods were acceptable for standard tests of pesticide residues.

The laboratory regularly participates in proficiency tests organised by the European Union Reference Laboratories and by FAPAS, and confirms its analytical competencies for conducted tests.

The test results were interpreted in accordance with the criteria included in the European Commission guidelines published in the Document SANCO/12571/2013 (now superseded by Document SANTE 2015) [2, 3], as well as by comparison with the Maximum Residue Levels (MRLs) in force in EU countries [12]. Verification of proper application of pesticides was conducted on a basis of the current "Register of Plant Protection Products Approved for Marketing and Application" [14]. Non-compliances related to pesticide use were notified in the Rapid Alert System for Food and Feed (RASFF).

Table 1. Scope of analysis with levels of quantifications (mg/kg)

Insecticides, their isomers and metabolites	acetamiprid (0.05), acrinathrin (0.01), aldrin (0.01), alpha-cypermethrin (0.01), azinophos-ethyl (0.01), azinophos-methyl (0.05), beta-cyfluthrin (0.01), bifenthrin (0.01), bromophos-ethyl (0.01), bromophos-methyl (0.01), bromopropylate (0.01), buprofezin (0.01), cadusafos (0.01), carbaryl (0.02), carbofuran (0.02), carbosulfan (0.01), chlorantraniliprole (0.01), chlorfenvinphos (0.01), chlorpyrifos (0.01), chlorpyrifos-methyl (0.01), cyfluthrin (0.01), cypermethrin (0.01), p,p'-DDD (0.01), p,p'-DDE (0.01), o,p'-DDT (0.01), p,p'-DDT (0.01), deltamethrin (0.02), diazinon (0.01), dichlorvos (0.01), dicofol (0.01), dieldrin (0.006), dimethoate (0.02), endosulfan alfa (0.01), endosulfan beta (0.01), endosulfan sulphate (0.01), endrin (0.01), esfenvalerate (0.01), ethion (0.01), ethoprophos (0.01), EPN (0.01), etoxazole (0.02), fenamiphos (0.02), fenazaquin (0.01), fenchlorphos (0.01), fenitrothion (0.01), fenoxycarb (0.05), fenpropathrin (0.01), fenthion (0.01), fenvalerate (0.01), fipronil (0.005), flonicamid (0.01), flubendiamide (0.01), formothion (0.01), HCB (0.01), α-HCH (0.01), β-HCH (0.01), γ-HCH (lindane) (0.01), heptachlor (0.01), heptachlor-endo-epoxide (0.003), heptachlor-exo-epoxide (0.001), heptenophos (0.01), hexythiazox (0.01), indoxacarb (0.02), isocarbophos (0.01), isofenphos (0.01), isofenphos-methyl (0.01), isoprocarb (0.01), lambda-cyhalothrin (0.01), lufenuron (0.02), malathion (0.01), mecarbam (0.01), methacrifos (0.01), methidathion (0.01), methoxychlor (0.01), mevinphos (0.01), parathion-ethyl (0.01), parathion-methyl (0.01), permethrin (0.02), phenthoate (0.01), phosalone (0.01), phosmet (0.01), pirimicarb (0.01), pirimiphos-ethyl (0.01), pirimiphos-methyl (0.01), profenofos (0.01), propoxur (0.05), prothiofos (0.01), pyrethrins (0.1), pyridaben (0.02), pyriproxyfen (0.02), quinalphos (0.01), spirodiclofen (0.02), spiromesifen (0.02), spirotetramat (0.1), tau-fluvalinate (0.01), tebufenozide (0.05), tebufenpyrad (0.01), teflubenzuron (0.01), tefluthrin (0.01), tetrachlorvinphos (0.01), tetradifon (0.01), tetramethrin (0.01), thiacloprid (0.02), triazophos (0.01), triflumuron (0.05), zeta-cypermethrin (0.01)
Fungicides	azaconazole (0.01), azoxystrobin (0.01), benalaxyl (0.05), benthiavalicarb-isopropyl (0.01), bitertanol (0.05), bixafen (0.01), boscalid (0.01), bromuconazole (0.01), bupirimate (0.01), captafol (0.02), captan (0.02), carbendazim* (0.05), chlorothalonil (0.01), chlozolinate (0.01), cyflufenamid (0.02), cymoxanil (0.05), cyproconazole (0.01), cyprodinil (0.01), dichlofluanid (0.01), dicloran (0.01), diethofencarb (0.05), difenoconazole (0.01), dimethomorph (0.01), dimoxystrobin (0.01), diniconazole (0.01), diphenylamine (0.05), dithiocarbamates* (mancozeb, maneb metiram propineb, thiram, zineb, ziram) (0.05), epoxiconazole (0.01), etaconazole (0.01), fenamidone (0.02), fenarimol (0.01), famoxadone (0.02), fenbuconazole (0.02), fenhexamid (0.05), fenpropidin (0.01), fenpropimorph (0.02), fludioxonil (0.01), fluquinconazole (0.01), flusilazole (0.01), fluopicolide (0.01), flutolanil (0.02), flutriafol (0.02), folpet (0.01), fuberidazole (0.05), hexaconazole (0.01), imazalil (0.02), imibenconazole (0.01), iprodione (0.02), iprovalicarb (0.04), isoprothiolane (0.01), krezoxim-methyl (0.01), mepanipyrim (0.01), metalaxyl (0.01), metalaxyl-M (0.05), metconazole (0.02), metrafenone (0.01), myclobutanil (0.01), oxadixyl (0.01), penconazole (0.01), pencycuron (0.05), picoxystrobin (0.01), prochloraz (0.01), procymidone (0.01), propiconazole (0.01), prothioconazole-desthio (0.02), proquinazid (0.02), pyraclostrobin (0.02), pyrazophos (0.01), pyrimethanil (0.01), quinoxyfen (0.01), quintozene (0.01), spiroxamine (0.05), tebuconazole (0.02), tecnazene (0.01), tetraconazole (0.01), thiabendazole (0.05), tolclofos-methyl (0.01), tolylfluanid (0.01), triadimefon (0.01), triadimenol (0.01), trifloxystrobin (0.01), triflumizole (0.1), triticonazole (0.01), vinclozolin (0.01), zoxamide (0.01)
Herbicides	acetochlor (0.01), atrazine (0.01), bromacil (0.01), carfentrazone-ethyl (0.01), chloridazon (0.05), chlorotoluron (0.05), chlorpropham (0.01), clomazone (0.01), cyanazine (0.01), cyprazine (0.01), diflufenican (0.01), dimethachlor (0.02), diuron (0.01), fenoxaprop-P (0.1), fluazifop-P (0.05), flufenacet (0.02), flumioxazine (0.02), flurochloridone (0.01), flurtamone (0.02), haloxyfop-2-etotyl (0.05), haloxyfop-methyl (0.05), isoproturon (0.05), lenacil (0.05), linuron (0.05), metamitron (0.1), metobromuron (0.01), metolachlor (0.02), metribuzin (0.01), metazachlor (0.01), monolinuron (0.05), napropamide (0.05), nitrofen (0.01), oxyfluorfen (0.01), pendimethalin (0.02), pethoxamid (0.01), procyazine (0.01), prometryn (0.01), propachlor (0.01), propaquizafop (0.05), propazine (0.01), propham (0.02), propyzamide (0.01), prosulfocarb (0.01), quinoclamine (0.01), simazine (0.01), S-metolachlor (0.02), terbuthylazine (0.02), terbutryn (0.01), trifluralin (0.01)
Growth retardant	paclobutrazol (0.01)
Plant activator	acibenzolar-S-methyl (0.01)
Acaricide	fenpyroximate (0.05)

* determinated as CS_2 residues

RESULTS AND DISCUSSION

In 2012–2015, 138 samples of fruiting vegetables in total (85 samples of tomatoes, 37 samples of cucumbers and 16 samples of pepper) from primary production were tested. The test results were interpreted in accordance with criteria adapted in Europe included in the European Commission recommendations and published in the document SANCO/12571/2013 (now superseded by Document SANTE 2015) [2, 3], as well as on a basis of a MRLs list for pesticides in force in Poland [12].

Of 242 analysed compounds, residues of 17 active substances were found: 15 fungicides and 2 insecticides (Table 2).

Plant protection agent residues were found in 47 samples, representing 34% of all tested samples.

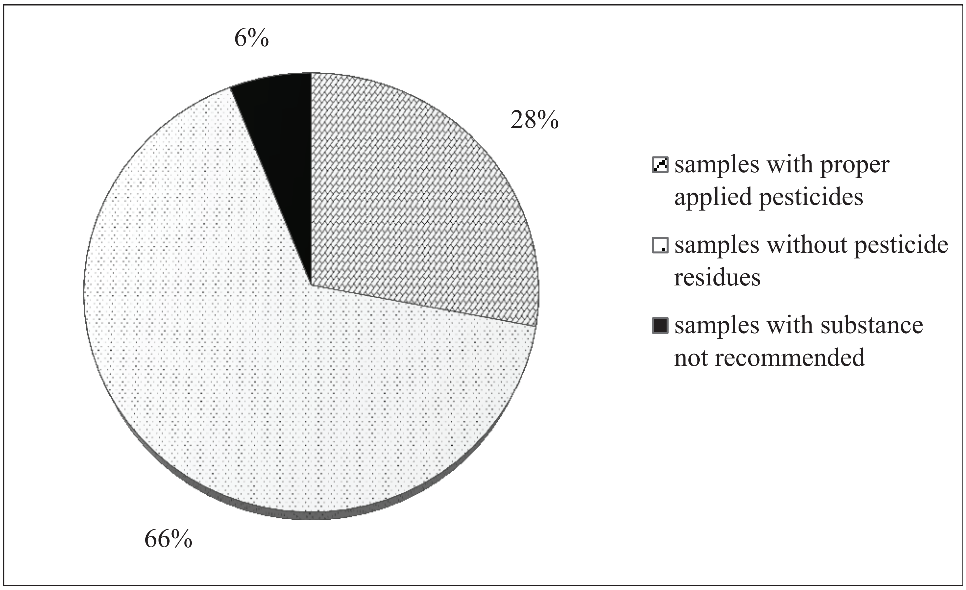

Figure 1. Pesticide residues in fruiting vegetables (2012–2015)

Pesticide residues were most commonly found in tomato – 46% of samples with residues, followed by pepper (25%), while cucumber was a plant in which residues were least common – 12% of samples with residues.

Quantified residue levels were generally low, near or equal to respective quantification limits of analytical methods used.

Of active substances which residues were found in samples of fruiting vegetables, most commonly found included azoxystrobin (as many as 38% of tested samples), boscalid (28%) and chlorothalonil (21%), belonging to fungicides.

Multiple residues were found in analysed vegetables, residues of one compound were found in 26 samples, while residues of two, three and four active substances were found in 13, 5 and 2 samples, respectively. In one sample, residues of as many as five active substances were found.

Non-compliances related to use of plant protection products not authorized for protection of given crops were found in the analysed samples. Azoxystrobin and chlorpyrifos were found in cucumber; chlorpyrifos, as well as fluopicolide and esfenvalerate were found in tomatoes; while boscalid, pyrimethanil and iprodione were found in pepper. Non-compliances related to use of plant protection products containing active substances not approved in the UE were found in practically all crops indicating insufficient number of available agents for their protection. A problem of an insufficient number of plant protection agents for minor uses, including vegetable crops, emerged with withdrawal of many formulations from the market, following the EU review of active substances. It aimed at leaving in the market only those substances that are safe to human health and to the environment. One of the ways to limit use of unapproved pesticides is continuous extending of marketing approvals to small-scale crops [10] and dissemination of information concerning changes in pesticide applications.

No substance was found in any sample which use for plant protection is forbidden [11], in any sample pesticide residues above MRLs were also found. According to other authors in Poland in 2011-2015 the most frequently notified products due to exceeding the MRL were: black currants, tea, Chinese cabbage, lettuce and carrots, but these are marginal percent of all tested samples of food of plant origin [18].

Analysis results and MRL values are listed in Table 2.

Concerning results obtained in previous years in fruiting vegetable samples from the region of south-eastern Poland it can be said that the percentage of detected residues decreased slightly (by ca. 10%) versus years 2010 and 2011 [16, 19]. The obtained results are consistent with monitoring data covering the whole territory of Poland [9, 10, 21] – the comparable level of residues (17%–46%) and the same active substances were detected in the same species of tested fruiting vegetables. The most commonly found substances were fungicides, azoxystrobin, boscalid and chlorothalonil, used to fight mildew, grey mould, alternariosis, white mould or potato blight [14].

Referring to the results obtained from the monitoring encompassing all groups of fruits and vegetables, conducted in previous years (2004 – 2007) [6] and from the central and eastern region of Poland (2014) [20] it can be concluded that in vegetables, especially in fruiting vegetables, pesticide residues were less frequently indicates than in the fruit.

Results of the studies published by The European Food Safety Authority (EFSA) for 2012 and 2013 and concerning monitoring tests for pesticide residues in food products in the European Union Member States were as follows: 0.9%–1.4% of fruiting vegetable samples exceeded MRLs, 22%–27% contained residues of multiple pesticides (more than one substance in a sample), and 49%–53% of samples did not contain pesticide residues [4, 5]. In Chinese crops overall percentage of samples with pesticide residues was 31%, including 25% and 29% of green peppers and cucumbers, respectively [22]. Nevertheless, more than half (59%) of the cucumbers and tomatoes from the Almaty region of Kazakhstan contained pesticides, while 28% contained pesticide residues above MRLs [8].

When obtained test results are compared to this data, it can be assumed that Polish plant production contained less pesticide residues (66% of residue-free samples) versus EU states and other countries, the percentage of samples with multiple residues (15%) was also lower.

Table 2. Occurrence of pesticide residues in fruiting vegetables (2012–2015)

Fruiting vegetables	Number of samples	Active substance		Samples with residues	Values of found residues [mg/kg]	MRLs [mg/kg]
Greenhouse cucumber	16	azoxystrobin *	F	2	0.02; 0.03	1.0
Field cucumber	21	chlorpyrifos *	I	1	0.03	0.05
		chlorothalonil	F	1	0.01	5.0
		fluopicolide	F	1	0.02	0.5
Greenhouse sweet pepper	15	azoxystrobin	F	1	0.02	1.0
		iprodione *	F	1	0.13	4.0
		pyrimethanil *	F	1	0.02	0.7
Field sweet pepper	1	boscalid *	F	1	0.01	3.0
Greenhouse tomato	82	azoxystrobin	F	15	0.01; 0.01; 0.01; 0.01; 0.01; 0.02; 0.02; 0.02; 0.02; 0.02; 0.03; 0.06; 0.07; 0.09; 0.14	3.0
		boscalid	F	12	0.02; 0.02; 0.03; 0.03; 0.03; 0.04; 0.05; 0.07; 0.08; 0.13; 0.13; 0.21	3.0
		bupirimate	F	1	0.29	2.0
		chlorpyrifos *	I	1	0.11	0.5
		chlorothalonil	F	9	0.01; 0.01; 0.03; 0.03; 0.03; 0.05; 0.07; 0.11; 0.81	6.0
		cyprodinil	F	8	0.01; 0.03; 0.03; 0.03; 0.03; 0.04; 0.04; 0.28	1.5
		difenoconazol	F	2	0.01; 0.02	2.0
		dimethomorph	F	1	0.05	1.0
		dithiocarbamates	F	3	0.07; 0.10; 0.21	3.0
		esfenvalerate *	I	1	0.04	0.1
		famoxadone	F	6	0.02; 0.03; 0.04; 0.05; 0.05; 0.07	2.0
		fenamidone	F	1	0.04	1.0
		fludioxonil	F	8	0.01; 0.01; 0.02; 0.02; 0.03; 0.03; 0.04; 0.04	3.0
		fluopicolide *	F	2	0.02; 0.02	1.0
		iprodione	F	3	0.07; 0.10; 0.98	5.0
		metalaxyl	F	1	0.03	0.2
Field tomato	3	–		–	–	–

MRLs – maximum residues level
* application of the substance not recommended for that crop
– no pesticide residues were found
F –fungicides
I - insecticides

CONCLUSIONS

Agricultural crop monitoring shows to what extent such products are contaminated with pesticide residues ensuring protection of consumers' health and verifying the proper regulatory compliance in the use of the plant protection products. Therefore, these surveys should be continued, and, moreover, this monitoring should be continuously expanded with new active substances.

Conflict of interest
The authors declare no conflict of interest.

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