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ORIGINAL ARTICLE

PESTICIDE RESIDUES IN STONE FRUITS FROM THE SOUTH-EASTERN REGION OF POLAND IN 2012 – 2014

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ABSTRACT

Background. Peaches, sour cherries, nectarines, apricots, plums and cherries are fruit commonly known as "stone fruit". Their nutritional properties namely, vitamins, minerals, fiber and numerous microelements, make them a very important component of human diet. As fruit trees can be attacked by numerous diseases and pests, chemical protection of these crops is used. Therefore, it is important that the relevant governmental agencies or institutions ensure correct application of pesticides

Objective. The aim of the study was to evaluate the occurrence of pesticide residues in stone fruits south-eastern region of Poland in 2012–2014 in order to provide data to estimate health risk to consumers.

Material and methods. Validated analytical methods based on liquid / liquid extraction coupled with gas chromatography with electron capture and nitrogen phosphorus detection (GC-ECD/NPD) and spectrophotometry (dithiocarbamates residues) were used for the analysis. 92 samples of stone fruits were tested for the presence of pesticide residues.

Results. 13 of all samples (14%) contained pesticide residues. 7 active substances were detected, including 5 fungicides: boscalide, bupirimate, difenoconazole, dithiocarbamates and captan, and 2 insecticides: cypermethrin and pirimicarb. In the analysed samples, the use of not recommended plant protection products in orchard crops were found. However, neither maximum residue levels (MRLs) recommended by the Regulation (EC) No 396/2005 were exceeded nor pesticides being unapproved by the Regulation (EC) No 1107/2009 detected in the analysed samples.

Conclusions. Lack of plant protection products for control specific diseases or pests in crops results in the use of formulations not recommended for use in certain orchard crops. On a basis of results reported in previous years it can be concluded that occurrence of pesticide residues in stone fruit samples dropped significantly.

Key words: *stone fruits, pesticides, pesticide residue analysis, maximum residue levels, MRLs*

STRESZCZENIE

Wprowadzenie. Brzoskwinie, wiśnie, nektarynki, morele, śliwy oraz czereśnie są to owoce powszechnie nazywane „pestkowe". Owoce te, ze względu na właściwości odżywcze tj.: witaminy, minerały, błonnik i liczne mikroelementy, pełnią bardzo ważną funkcję w diecie. Ponieważ drzewa owocowe mogą być atakowane przez liczne choroby i szkodniki stosuje się chemiczną ochronę tych upraw. Z tego powodu ważna jest kontrola prawidłowości stosowania pestycydów przez właściwe urzędy państwowe lub instytucje.

Cel badań. Celem pracy była ocena występowania pozostałości środków ochrony roślin w owocach pestkowych pochodzących z południowo-wschodniego regionu Polski w latach 2012–2014 i dostarczenie danych umożliwiających oszacowanie ryzyka zdrowotnego dla konsumentów.

Materiał i metoda. Do analizy zastosowano zwalidowane metody analityczne opierające się na ekstrakcji ciecz/ciecz w połączeniu z chromatografią gazową z detekcją wychwytu elektronów i azotowo-fosforową (GC-ECD/NPD) oraz spektrofotometryczne (pozostałości ditiokarbaminianów). Przebadano 92 próbki owoców pestkowych.

Wyniki. W 13 próbkach (14%) stwierdzono obecność pozostałości środków ochrony roślin. Wykryto 7 substancji czynnych takich jak boskalid, bupirymat, difenokonazol, ditiokarbaminiany oraz kaptan należące do grupy fungicydów, a także cypermetrynę i pirymikarb z grupy insektycydów. W analizowanych próbkach stwierdzono przypadki nieprawidłowości związanej z zastosowaniem preparatów niezalecanych do ochrony danej uprawy sadowniczej. Natomiast w żadnej próbce 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.

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Wnioski. Brak zarejestrowanych środków ochrony roślin do zwalczania chorób czy szkodników w uprawach jest przyczyną stosowania preparatów niezalecanych do niektórych upraw sadowniczych. Na podstawie wyników z lat ubiegłych można stwierdzić, że częstotliwość występowania pozostałości pestycydów w próbkach owoców pestkowych znacznie spada.

Słowa kluczowe: *owoce pestkowe, pestycydy, pozostałości środków ochrony roślin, analiza, najwyższe dopuszczalne pozostałości, NDP*

INTRODUCTION

Peaches, sour cherries, nectarines, apricots, plums and cherries are fruit commonly known as “stone fruit”. Their name comes from a very large and hard drupe containing seeds, resembling a stone. Stone fruit have a relatively short growing season, and provide fruit from June to September. Tasty stone fruit can be eaten fresh, but also offer a range of possibilities for processing to be used out of the season, including frozen fruit, juices, syrups, compotes, jams, dried fruit, and similar.

Their nutritional properties, due to presence of vitamins, minerals, fibre and numerous microelements, make them a very important component of human diet. Studies showed that stone fruit contain bioactive compounds that can possibly be used to combat obesity, diabetes and cardiovascular diseases [23].

As fruit trees can be attacked by numerous diseases and pests, chemical protection of these crops is used. Therefore, it is important that the relevant governmental agencies or institutions ensure correct application of pesticides. The levels of pesticide residues in foodstuffs are regulated so as to minimize the exposure of consumers to harmful or unnecessary intakes of pesticide [10]. The Regulation No. 396/2005 of the European Parliament and the Council establishes maximum residue levels (MRLs) for pesticides in foodstuffs and fodder [15].

The aim of this study was to evaluate the occurrence of pesticides in stone fruit from the southeast region of Poland in the years 2012–2014 in order to provide data to estimate health risk to consumers.

MATERIALS AND METHODS

In 2012–2014, the Pesticide Residue Analyses Laboratory analysed 92 samples of stone fruit (samples of: peaches – 19, cherries – 23, apricots – 16, plums – 7, and sour cherries – 27). Samples were obtained during an official inspection of pesticide residues conducted on behalf of the Polish Ministry of Agriculture and Rural Development, implemented in cooperation with the regional Inspectorates of Plant Health and Seed Inspection. Samples originated from production farms from: Podkarpackie, Lubelskie, Małopolskie and Świętokrzyskie Voivodeships. Products were sampled according to the Regulation of the Minister of Agriculture and Rural Development from 27 November 2013 on the sampling of plants, plant products or other objects to test for the presence of residues of plant protection products [16].

The programme of analyses covered determination of 206 active substances and their metabolites for commonly applied pesticides (Table 1).

Pesticide residues were analysed using analytical methods which were validated and accredited according to PN-EN ISO/IEC 17025 [13].

a) multiresidue gas chromatography method based on residues extraction with an organic solvent and further extract purification with column chromatography. Qualitative and quantitative residue analyses were performed on gas chromatographs Agilent 7890 and Agilent 6890, equipped with electron capture (ECD) and nitrogen-phosphorus (NPD) detectors. [7, 18, 19].
b) 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].

To ensure quality of performed tests and confirm its competencies, the laboratory regularly participates in the international proficiency tests organised by the European Union, e.g. EUPT-FV-13÷16/EURL (Spain), TestQual (Spain), and also in interlaboratory comparative research, in which it achieves satisfactory results for performed tests.

The test results were interpreted in accordance with the criteria included in the European Commission guidelines published in the Document SANCO/12571/2013 [3], as well as by comparison with the Maximum Residue Levels (MRLs) in force in EU countries [15] and regarding residues definition for MRLs [2]. Verification of correct application of pesticides was conducted on a basis of the current Register of Plant Protection Products Approved for Marketing and Application and “Labels-Instructions for use on plant protection products approved for marketing and application with a permit of the Ministry of Agriculture and Rural Development” [4, 17].

RESULTS AND DISCUSSION

92 samples of stone fruit were tested; pesticide residues were found in 13 samples (14%) including: 3

samples of apricots (19% of all tested samples of this crop), 3 samples of peaches (16%), 4 samples of sour cherries (20%) and 3 samples of cherries (13%).

7 active substances were found in analysed fruit, including boscalide, bupirimate, difenoconazole, dithiocarbamates and captan from the fungicides group, and cypermethrin and pirimicarb from the insecticides group. The substances detected most frequently were dithiocarbamates and captan (1.7% and 1.3% of all tested samples, respectively), and those found less frequently included cypermethrin, difenoconazole, and pirimicarb (0.7% each), as well as boscalid and bupirimate (0.3% each).

In 5 analysed samples residues of active substances not recommended for protection of a relevant orchard crop were detected; and these were captan and bupirimate found in peaches, and dithiocarbamates and captan in apricots. Fungicide formulations, such as: Captan 80 WG and Kaptan Plus 71.5 WP, Captan Suspension 50 WP, Flint Plus 64 WG, Merpan 480 SC, Merpan 80 WG, and Malvin 80 WG, with captan as an active substance, are used solely for protection of cherry, apple, and pear trees against scab (*Venturia inaequalis*; *Venturia pirina*), or bitter rot (*Glomerella cingulata* (Stonem) Spauld. Et Schrenk., ST. Kon. *Colletotrichum gloeosporioides* (Penzing)). The situation is similar for dithiocarbamate formulations (Aplosar 80 WG, Pomarsol Forte 80 WG, Sadoplon 75 WP, Thiram Granuflo 80 WG, Aplosar 80 WG), registered for protection of apple, peach, and pear trees against scab, grey mould (*Botrytis cinerea* Pers), or peach leaf curl. The formulation Nimrod 250 EC, with bupirimate as an active substance, is registered solely for protection of apple trees against powdery mildew caused by fungi (*Podosphaera leucotricha* Ell et Ev./ Salm). Therefore, we have a situation in which used formulations were legally purchased but did not have the Ministry of Agriculture and Rural Development approval for use in a specific crop.

The results obtained in this study were compared with MRLs in force within EU countries [15]. In no sample pesticide residue levels exceeded the MRL or any substances were found the use of which as plant protection products is forbidden [14].

Table 1. Active substance, scope of analysis with limits of quantifications (mg/kg)

Insecticides	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), 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 SO_2 (0.01), endrin (0.01), esfenvalerate (0.01), ethion (0.01), ethoprophos (0.01), EPN (0.01), 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), 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), 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), 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), tau-fluvalinate (0.01), tebufenpyrad (0.01), teflubenzuron (0.01), tefluthrin (0.01), tetrachlorvinphos (0.01), tetradifon (0.01), triazophos (0.01), zeta-cypermethrin (0.01)
Fungicides	azaconazole (0.01), azoxystrobin (0.01), benalaxyl (0.05), bitertanol (0.05), boscalid (0.01), bromuconazole (0.01), bupirimate (0.01), captafol (0.02), captan (0.02), chlorothalonil (0.01), chlozolinate (0.01), cyproconazole (0.01), cyprodinil (0.02), dichlofluanid (0.01), dicloran (0.01), 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), 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), 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 destio (0.02), pyrazophos (0.01), pyrimethanil (0.01), quinoxyfen (0.01), quintozene (0.01), tebuconazole (0.02), tecnazene (0.01), tetraconazole (0.01), tolclofos-methyl (0.01), tolylfluanid (0.01), triadimefon (0.01), triadimenol (0.01), trifloxystrobin (0.01), triflumizole (0.1), vinclozolin (0.01), zoxamide (0.01)
Herbicides	acetochlor (0.01), atrazine (0.01), bromacil (0.01), 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), flufenacet (0.02), flurochloridone (0.01),), flurtamone (0.02), 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), prometryn (0.01), propachlor (0.01), propaquizafop (0.05), propazine (0.01), propham (0.02), propyzamide (0.01), simazine (0.01), terbuthylazine (0.02), terbutryn (0.01), trifluralin (0.01)
Growth retardant	paclobutrazol (0.01)

Table 2. Pesticide residues in tested stone fruits (2012–2014)

Crop	Number of analysed samples	Active substance	Samples with residues		Values of found residues [mg/kg]	MRLs [mg/kg]
			number	[%]		
Apricot	16	Captan *	2	12	0.03; 0.04	4.0
		Dithiocarbamates *	1	6	0.35	2.0
Peach	19	Bupirimate *	1	5	0.01	0.2
		Captan *	1	5	0.05	4.0
		Difenoconazole	1	5	0.04	0.5
		Dithiocarbamates	1	5	0.35	2.0
Plum	7	–	–	–	–	–
Sour cherry	23	Boscalide	1	4	0.08	4.0
		Cypermethrin	1	4	0.04	2.0
		Dithiocarbamates	1	4	0.05	2.0
		Pirimicarb	1	4	0.02	5.0
Sweet cherry	27	Captan	1	4	0.04	5.0
		Cypermethrin	1	4	0.08	2.0
		Difenoconazole	1	4	0.01	0.3
		Dithiocarbamates	2	7	0.06; 0.29	2.0
		Pirimicarb	1	4	0.06	5.0

MRLs - maximum residues levels
* application of the substance not recommended for that crop
– no pesticide residues were found

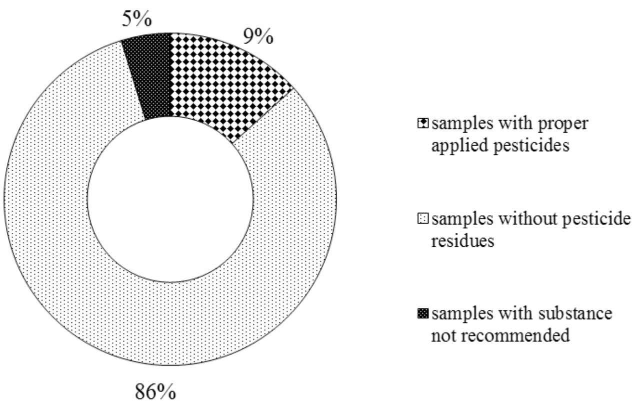

Figure 1. Occurrence of pesticide residues in tested stone fruits (2012–2014).

Analysis results and MRL values are shown in Table 2 and Figure 1.

An analysis of pesticide residues performed in 2009 and 2010 [20, 21] indicates that the residues found in stone fruit dropped significantly (2009 – 36%, 2010 – 27%) in years 2012–2014. Our results show clearly that the residue profiles were comparable; usually residues of the same active substances in the same species of fruit were found. However, use of formulations not recommended for protection of certain crops increased.

When comparing to the results obtained in 2010–2012 for all tested groups of fruit from the south-eastern region of Poland, the percentage of samples with residues amounted to 51%, and there were also samples with MRL exceeded (2%) [22], therefore, it can be concluded that stone fruit do not represent a group of fruit most exposed to pesticide contamination.

Results of studies published by other authors, including those concerning samples from monitoring covering whole Poland [11, 12] and from the north-eastern region of Poland [8] indicate that active substances found in stone fruit were the same, except for tebuconazole. The residues of pesticides found in these studies are considerably lower than those reported in berries, harvested in the same region of Poland [9].

As the European Food Safety Authority (EFSA) states in its report summing up the results of monitoring activities focusing on pesticide residues in foodstuffs, conducted in years 2010 and 2011 in 27 EU Member States, in stone fruit about 2% of samples had MRLs exceeded, 39–41% contained multiple pesticide residues (more than one substance in the sample), and 33–36% did not contain any pesticide residues [5, 6]. According to information provided in the report, for example, 79 different pesticides were found in peaches in 2010, with tebuconazole (20%) found most often, followed by dithiocarbamates (19%), and iprodione (16%). 17 substances were found to be at levels exceeding MRLs; majority of these samples came from Spain, Turkey, and Malta. Captan showed the highest rate of samples exceeding the MRL [5]. Compared with these results coming from European countries, stone fruits from the region of southeast Poland contain much less pesticide residues.

CONCLUSIONS

1. Plant protection products residues were found in 14% of tested samples. Residues of dithiocarbamates and captan were found most commonly.
2. Lack of plant protection products for control specific diseases or pests in crops results in use of formulations not recommended for use in certain orchard crops.

3. On a basis of results of previous years it can be said that occurrence of pesticide residues in stone fruit samples dropped significantly.

Conflict of interest

The authors declare no conflict of interest.

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