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PESTICIDE RESIDUES IN FRUIT AND VEGETABLE CROPS FROM THE CENTRAL AND EASTERN REGION OF POLAND

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ABSTRACT

Background. Fruit and vegetables have health and nutritional value, but can also be a source of toxic contaminants such as pesticide residues.

Objective. The aim of this study was to evaluate presence of pesticide residues in fruit and vegetable crops from the central and eastern region of Poland in order to check the compliance of these products with the maximum residue levels' (MRLs) requirements set in legal EU regulation for products present in the market.

Material and methods. Samples of fruit and vegetables were obtained from production farms as a part of an official premarket monitoring of pesticide residues conducted on behalf of the Ministry of Agriculture and Rural Development, implemented in cooperation with the regional Inspectorates of Plant Health and Seed Inspection. The tests covered determination of 207 pesticides.

Results. In 2014, a total of 317 samples of fruit and vegetables, collected from the central and eastern region of Poland, were analysed for the presence of pesticide residues. Pesticide residues were detected in 89 (28.1%) analysed samples: in 65 (38.2%) samples of fruit, and in 24 (16.3%) samples of vegetables. MRLs were exceeded in 2 samples (0.6%). Most often, the pesticide residues were found in gooseberry (100% of gooseberry samples) and apple (71.4%).

Conclusions. Monitoring of pesticide residues in the agricultural crops at premarket stage should be continued to prevent market from the penetration by products with non-acceptable residues of pesticides and to protect consumers against excessive exposure to pesticide residues.

Key words: *pesticide residues, fruit, vegetables, agricultural crops, multiresidue method*

STRESZCZENIE

Wprowadzenie. Owoce i warzywa mają wartości zdrowotne i odżywcze ale mogą być również źródłem toksycznych zanieczyszczeń takich jak pozostałości pestycydów.

Cel badań. Celem badań była ocena występowania pozostałości pestycydów w owocach i warzywach z terenu centralnej i wschodniej Polski w odniesieniu do najwyższych dopuszczalnych poziomów pozostałości (NDP) określonych w rozporządzeniu UE dla produktów obecnych na rynku.

Materiał i metody. Próbki owoców i warzyw zostały pobrane z gospodarstw produkcyjnych w ramach urzędowej kontroli pozostałości pestycydów w produkcji pierwotnej, prowadzonej na zlecenie Ministerstwa Rolnictwa i Rozwoju Wsi, realizowanej we współpracy z wojewódzkimi Inspektoratami Inspekcji Ochrony Roślin i Nasiennictwa. Program kontroli obejmował oznaczenie 207 pestycydów.

Wyniki. W 2014 roku wykonano analizy 317 próbek owoców i warzyw na obecność pozostałości pestycydów. Pozostałości tych substancji wykryto w 89 próbkach (28,1%): w 65 próbkach (38,2%) owoców i w 24 próbkach (16,3%) warzyw. Przekroczenia NDP stwierdzono w 2 próbkach (0,6%). Uprawami, w których najczęściej stwierdzano pozostałości były agrest (100% próbek agrestu) i jabłka (71,4%).

Wnioski. Monitoring pozostałości pestycydów w produktach rolnych powinien być kontynuowany, aby zapobiec wprowadzaniu na rynek produktów zawierających niedozwolone pestycydy oraz w celu ochrony konsumentów przed narażeniem na pozostałości pestycydów w spożywanych produktach.

Słowa kluczowe: *pozostałości pestycydów, owoce, warzywa, uprawy rolne, metoda wielopozostałościowa*

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INTRODUCTION

One of the most important methods for protecting plants and plant products against harmful organisms, including weeds, and for improving agricultural production is the use of plant protection products. However, plant protection products can also have adverse effects on plant production. Their use may involve risks and hazards for humans, animals and the environment, especially, when placed on the market without having been officially tested and authorised, and used incorrectly [3].

Fruit and vegetables are subjected many applications of plant protection products, and then they are often eaten fresh and unprocessed. As consumption of fruit and vegetables constitutes over 30% of consumers' diet [22], they are the main source of pesticide residues for human.

In Poland, the regulatory compliance in the use of plant protection products is supervised by the Inspectorates of Plant Health and Seed Inspection on behalf of the Ministry of Agriculture and Rural Development. As part of this supervision, monitoring of pesticide residues in agricultural crops is conducted every year, over the course of which analyses are conducted, inter alia, by the Laboratory of Pesticide Residue Analysis in Rzeszow.

The aim of this study was to evaluate presence of pesticide residues in fruit and vegetable crops from the central and eastern region of Poland in order to check the compliance of these products with the maximum residue levels' (MRLs) requirements set in legal EU regulation for products present in the market [2].

MATERIAL AND METHODS

Samples of fruit and vegetable crops were taken from production farms as a part of an official premarket

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), 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), 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), carbendazim* (0.05), chlorothalonil (0.01), chlozolinate (0.01), cyproconazole (0.01), cyprodinil (0.01), 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-desthio (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)

* – analysed only in apple

monitoring of pesticide residues conducted on behalf of the Ministry of Agriculture and Rural Development, implemented in cooperation with the regional Inspectorates of Plant Health and Seed Inspection.

The analyses covered the determination of 207 pesticides (Table 1). Accredited methods according to ISO/IEC 17025:2005 [13] were used to determine the presence of pesticide residues. The multiresidue analytical method was based on the extraction of residues with acetone and dichloromethane and further purification of the extract using a florisil column [8, 16]. Quantification of residues was carried out with Agilent 6890 and Agilent 7890 gas chromatographs, each equipped with ECD and NPD detectors. Along with the multiresidue method, spectrophotometric determination of dithiocarbamate residues, expressed in milligrams of CS_2 per kilogram, and thin layer chromatographic determination of benzimidazoles, expressed as carbendazim residues, were carried out [1, 10]. Test results were confirmed in compliance with European Commission guidelines [4]. Laboratory competencies were verified in proficiency tests organized by the European Union reference laboratories.

The obtained results were compared with the MRLs in force in both Poland and the European Union [2, 6]. According to the guidelines in the SANCO document for samples under official control, MRL violations were determined for pesticide levels exceeding the MRL; with the method uncertainty (50%) considered [4]. Moreover, it was verified, if detected pesticides could be used to protect particular crops [5, 15].

RESULTS

In 2014, a total of 317 samples: 170 samples of fruit and 147 samples of vegetables, collected from the central and eastern region of Poland, were analysed for the presence of pesticide residues. Pesticide residues were detected in 89 (28.1%) analysed samples: in 65 (38.2%) samples of fruit and in 24 (16.3%) samples of vegetables (Figure 1 and 2). Violations of MRLs were found in 2 samples of raspberry (0.6%) and they concerned: flutriafol, penconazole and spirodiclofen residues (Table 2).

Among fruit samples the pesticide residues were most often found in: gooseberry (100% of the gooseberry samples), apple (71.4%), blueberry (66.7%), currant (60.0%), and raspberry (55.1%), while among vegetable samples: tomato (50.0%), broccoli (50.0%), parsley root (21.4%), cucumber (16.7%), and Peking cabbage (11.8%) (Figure 3 and 4).

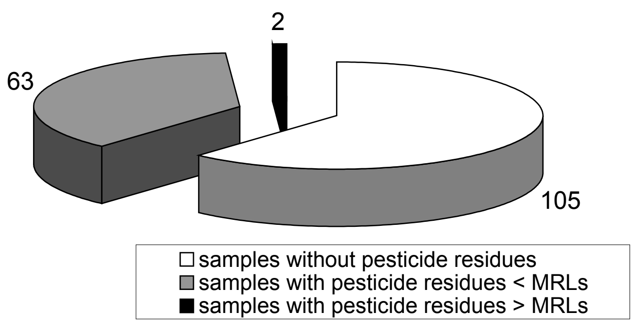

Figure 1. Occurrence of pesticide residues in fruits

Figure 2. Occurrence of pesticide residues in vegetables

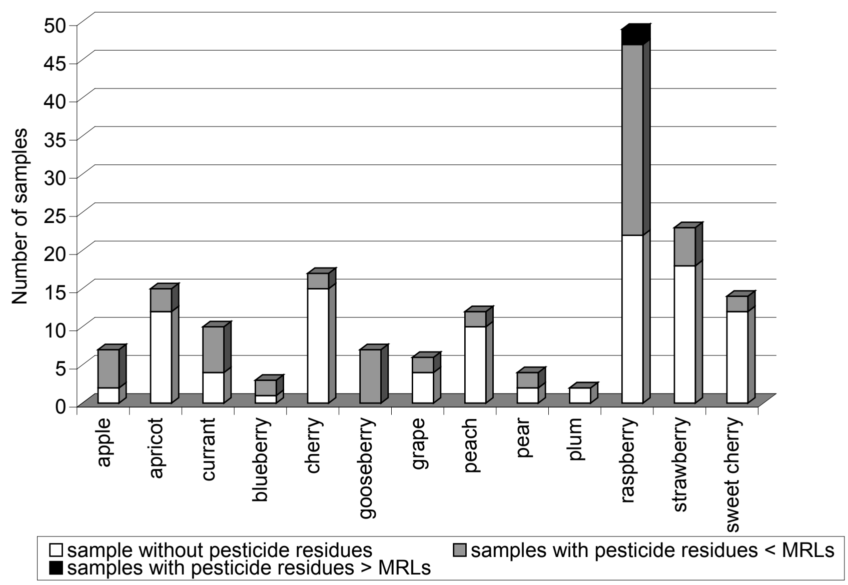

Figure 3. Occurrence of pesticide residues in particular groups of fruit

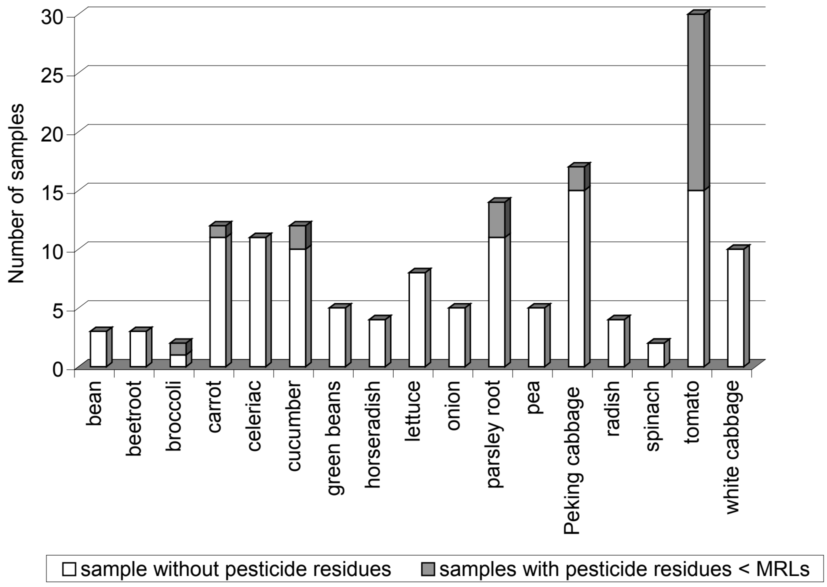

Figure 4. Occurrence of pesticide residues in particular groups of vegetables

Table 2. Pesticide residues detected in analysed samples

Crop	No. of analysed samples	No. of samples with residues	Active substance (category)	No. of positive samples	Range min–max or value [mg/kg]	Mean ± SD [mg/kg]	MRL [mg/kg]
Fruits							
Apple	7	5	boscalid (F)	1	0.01	–	2
			captan (F)	3	0.02–0.18	0.11±0.08	3
			difenoconazole (F)	1	0.01	–	0.5
			dithiocarbamates (F)	1	0.13	–	5
			spirodiclofen (I)	1	0.04	–	0.8
Apricot	15	3	captan[a] (F)	2	0.03–0.04	0.04±0.01	4
			dithiocarbamates[a] (F)	1	0.35	–	2
Currant	10	6	deltamethrin (I)	2	0.03–0.25	0.14±0.16	0.5
			difenoconazole (F)	4	0.02–0.09	0.06±0.03	0.2
			dithiocarbamates (F)	2	0.15–0.64	0.40±0.35	5
			lambda-cyhalothrin (I)	1	0.03	–	0.2
			trifloxystrobin (F)	2	0.02–0.06	0.04±0.03	1
Blueberry	3	2	boscalid (F)	2	0.01–0.10	0.06±0.06	10
			iprodione (F)	1	0.18	–	10
Cherry	17	2	cypermethrin (I)	1	0.08	–	2
			difenoconazole (F)	1	0.01	–	0.3
			dithiocarbamates (F)	1	0.29	–	2
Gooseberry	7	7	boscalid[a] (F)	1	0.06	–	10
			bupirimate (F)	1	0.02	–	5
			chlorpyrifos[a] (I)	1	0.01	–	1
			chlorpyrifos-methyl[a] (I)	1	0.01	–	0.05
			cyprodinil[a] (F)	2	0.02–0.10	0.06±0.06	5
			difenoconazole (F)	6	0.01–0.07	0.04±0.02	0.1
			dithiocarbamates (F)	3	0.15–0.28	0.19±0.08	5
			fludioxonil[a] (F)	1	0.07	–	2
			myclobutanil[a] (F)	1	0.18	–	1
			trifloxystrobin (F)	4	0.03–0.10	0.06±0.03	1
Grape	6	2	cyprodinil (F)	1	0.09	–	5
			dithiocarbamates (F)	1	0.15	–	5
			fludioxonil (F)	1	0.03	–	5
			iprodione (F)	1	0.26	–	10
Peach	12	2	bupirimate[a] (F)	1	0.01	–	0.2
			captan[a] (F)	1	0.05	–	4
Pear	4	2	captan (F)	2	0.02	–	3
			cypermethrin (I)	1	0.03	–	1
			dithiocarbamates (F)	1	0.07	–	5
Plum	2	0	–	–	–	–	–

Raspberry	49	27	boscalid (F)	14	0.02–0.39	0.16±0.16	10
			captan[a] (F)	1	0.05	–	10
			chlorpyrifos[a] (I)	1	0.04	–	0.5
			cyprodinil (F)	10	0.01–0.85	0.18±0.25	10
			fludioxonil (F)	7	0.01–0.16	0.07±0.06	5
			flutriafol[a, b] (F)	1	0.14	–	0.05
			iprodione (F)	1	0.29	–	10
			penconazole[a, b] (F)	1	0.35	–	0.05
			pyrimethanil (F)	17	0.01–0.79	0.20±0.20	10
			spirodiclofen[a, b] (I)	2	0.07–0.08	0.08±0.01	0.02
Strawberry	24	5	azoxystrobin (F)	1	0.04	–	10
			boscalid (F)	4	0.02–0.06	0.03±0.02	10
			chlorpyrifos[a] (I)	1	0.01	–	0.2
			cyprodinil (F)	3	0.02–0.18	0.07±0.09	5
			difenoconazole (F)	1	0.04	–	0.4
			fludioxonil (F)	2	0.03–0.10	0.07±0.05	4
			folpet[a] (F)	2	0.02–0.07	0.05±0.04	3
			pyrimethanil (F)	1	0.01	–	5
Sweet cherry	14	2	boscalid (F)	1	0.08	–	4
			cypermethrin (I)	1	0.04	–	2
			dithiocarbamates (F)	1	0.05	–	2
Vegetables							
Bean	3	0	–	–	–	–	–
Beetroot	3	0	–	–	–	–	–
Broccoli	2	1	chlorpyrifos (I)	1	0.06	–	0.05
Carrot	12	1	boscalid (F)	1	0.01	–	2
			cyprodinil (F)	1	0.02	–	2
			fludioxonil (F)	1	0.01	–	1
			iprodione (F)	1	0.07	–	0.5
Celeriac	11	0	–	–	–	–	–
Cucumber	12	2	azoxystrobin (F)	1	0.03	–	1
			fluopicolide (F)	1	0.02	–	0.5
Green beans	5	0	–	–	–	–	–
Horseradish	4	0	–	–	–	–	–
Lettuce	8	0	–	–	–	–	–
Onion	5	0	–	–	–	–	–
Parsley root	14	3	bupirimate (F)	1	0.02	–	0.05
			linuron (H)	1	0.05	–	0.2
			tebuconazole[a] (F)	1	0.02	–	0.4
Pea	5	0	–	–	–	–	–
Peking cabbage	17	2	azoxystrobin (F)	1	0.20	–	5
			bifenthrin[c] (I)	1	0.05	–	0.05
			cypermethrin (I)	1	0.02	–	1
			indoxacarb[a] (I)	1	0.02	–	3
			iprodione (F)	1	0.06	–	5
			lambda-cyhalothrin (I)	1	0.04	–	1
Radish	4	0	–	–	–	–	–
Spinach	2	0	–	–	–	–	–
Tomato	30	15	azoxystrobin (F)	5	0.01–0.09	0.04±0.04	3
			boscalid (F)	4	0.05–0.13	0.10±0.04	3
			chlorothalonil (F)	2	0.03	–	2
			cyprodinil (F)	6	0.03–0.28	0.08±0.10	1
			dimethomorph (F)	1	0.05	–	1
			dithiocarbamates (F)	3	0.07–0.21	0.13±0.07	3
			esfenvalerate[a] (I)	1	0.04	–	0.1
			fludioxonil (F)	6	0.01–0.04	0.03±0.01	0.9
			fluopicolide[a] (F)	1	0.02	–	1
			famoxadone (F)	3	0.02–0.05	0.04±0.02	1
			iprodione (F)	1	0.07	–	5
			metalaxyl (F)	1	0.03	–	0.2
White cabbage	10	0	–	–	–	–	–

SD – Standard Deviation

I – Insecticide, F – Fungicide, H – Herbicide
[a] Pesticide not recommended for application
[b] Pesticide residue above MRL
[c] Pesticide not approved in Poland

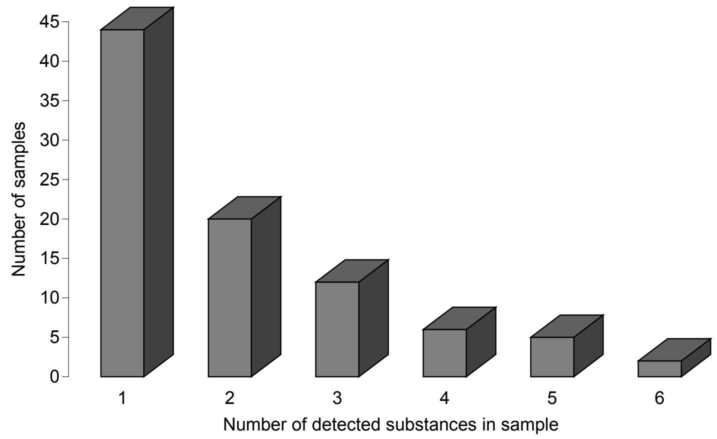

Figure 5. Single and multiple pesticide residues detected in samples

Of 207 analysed substances, 31 were found: 21 fungicides, 9 insecticides, and 1 herbicide.

The most commonly determined fungicides included: boscalid (8.8%), cyprodinil (7.3%), pyrimethanil (5.7%) and fludioxonil (5.7%), while the most often found insecticides were chlorpyrifos (1.3%) and cypermethrin (1.3%) (Table 2). Analyses of samples also showed presence of substances (boscalid, bupirimate, captan, chlorpyrifos, chlorpyrifos-methyl, cyprodinil, dithiocarbamates, esfenvalerate, fludioxonil, fluopicolide, flutriafol, folpet, indoxacarb, myclobutanil, penconazole, spirodiclofen and tebuconazole) not recommended for a given crop in 17 (5.4%) samples (Table 2). These substances can be used in Poland but to protect others crops. Bifenthrin, pesticide not approved in Poland, was detected in one sample of Peking cabbage.

Half of the samples with residues contained multiple residues, at least 2 substances with up to 6 in two samples (Figure 5). Those multiple residues were found most frequently in: gooseberry, raspberry and tomato.

DISCUSSION

Results of this study were interpreted in relation to MRLs requirements set in legal EU regulation for products present in the market [2]. This study has both a preventive nature, preventing the appearance on the market of products posing a risk to the consumer do not meet the requirements of relevant legislation, as well as to verify whether the producers properly used the principles of Good Agriculture Practices (GAP).

Fruit and vegetables are food products in which pesticide residues are frequently found [7, 17,18]. According to the most recent data from 2012 and 2013, pesticide residues were present from 30.1% [12] to 74.4% [9] of Polish fruit, and in 17.5% [12] to 37.4% [9] of Polish vegetables. Results of our studies conducted in 2014 and in previous years (2010–2012) [21] are within this range.

In comparison to other European country, Polish crops contain pesticide residues at similar level. Violation of MRLs are on the level 1.6–1.9% for EU-coordinated programmes and 2.5–2.8% in national programmes of European Union countries [17, 18], while in Polish crops they range from 0.4% [12] to 5.4% [9]. Results of our studies concerning exceedance of MRLs are also within this range.

Other authors also detected fungicides as the most common residue, followed by insecticide and herbicide residues [11, 14]. The kinds of detected substances are also very similar [11, 14].

In recent years, an increase in the number of samples with non-recommended substances is observed, due to reductions in the number of approved plant protection products and changes in the scope of application [19, 20].

CONCLUSIONS

1. The detectable pesticide residues have been found in 100% of gooseberry and over 70% of apple samples.
2. In many agricultural products the residues of more than one pesticide are present.
3. Monitoring of pesticide residues in the agricultural crops at premarket stage should be continued to prevent market from the penetration by products with non-acceptable residues of pesticides and to protect consumers against excessive exposure to pesticide residues.

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Conflict of interest

The authors declare no conflict of interest.

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