UNIVERSITY OF KRAGUJEVAC

FACULTY OF AGRONOMY IN ČAČAK

# SYMBIOTECH

## 4th INTERNATIONAL SYMPOSIUM ON BIOTECHNOLOGY

**12–13 March 2026**

**Faculty of Agronomy in Čačak, University of Kragujevac, Serbia**

# - PROCEEDINGS -

# 4th INTERNATIONAL SYMPOSIUM ON BIOTECHNOLOGY

XXXI Savetovanje o biotehnologiji sa međunarodnim učešćem

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**PREFACE**

CREATING FOOD FOR THE FUTURE – The agri-food sector identifies challenges and finds solutions through complex multidisciplinary research in the fields of biotechnical sciences, food and technological engineering. The Faculty of Agronomy in Čačak, in addition to educating students, has a strong scientific production, the results of which have been disseminated and shared through the traditional organization of the international scientific meeting – Symposium on Biotechnology (SYMBIOTECH) for thirty-one years.

At the 4th International Symposium on Biotechnology, a total of 104 papers were presented in the 6 sections: Field, Vegetable and Forage Crops, Pomology and Viticulture, Livestock Production, Plant Protection, Food Safety and the Environment, Food Technology and Nutritionism and Applied Chemistry.

We owe great gratitude to the Ministry of Science, Technological Development and Innovation of the Republic of Serbia and the City of Čačak for their traditional financial support and patronage of SYMBIOTECH26. We thank companies, entrepreneurs, stakeholders and all long-time friends of the Faculty of Agronomy for their material and organizational support.

In Čačak, March 2026

**Faculty of Agronomy in Čačak**
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**31st Symposium on Biotechnology with International Participation**
**12-13 March 2026**
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# DETERMINATION OF RESIDUE OF PESTICIDES IN RED PAPER FROM THE REGION OF STRUMICA, REPUBLIC OF NORTH MACEDONIA USING QuEChERS, GAS AND LIQUID CHROMATOGRAPHY

*Borjanka Veselinova[1,2], Sanja Kostadinović Veličkovska[1], Katarina Smilkov[2]*

**Abstract:** Determination of pesticides in red Ajvar pepper (*Capsicum annuum* cv. Kurtovska Kapija) was performed with the method of QuEChERS (EN 15662). A total of 40 samples of red pepper ajvarka (Kurtovska kapija) from the village of Dabile, Strumica region were analyzed. Identification and quantification of 397 pesticides were performed and imidacloprid was detected above the permitted concentrations (0.011 mg/kg) in samples treated by Nimazal before harvesting, while in the samples of Ajvar red pepper treated with a combination of Dikrsol 500 sp and Proteus, the same pesticide imidacloprid was detected at a concentration of 0.032 mg/kg sample. The permited MDK value for this pesticide is up to a maximum of 0.01 mg/kg.

**Keywords**: residues of pesticides, red paper, imidacloprid, QuEChERS, gas and liquid chromatography

## Introduction

Red pepper Kurtovska Kapija (known as ajvarka) is among the most common crops in Republic of North Macedonia. Pepper is an annual crop with an upright and weakly branched stem up to 1m high with hanging and flattened fruits. The fruits of Kurtovska Kapija have a thick and large pericarp. The length of the fruit is 12 -15 cm with a weight of 60 -70 g. At technological maturity it has a dark green color, while at biological maturity the fruit has an intense dark red color. The flesh is aromatic with a small amount of water, a thin peel that peels easily. As for ripening, this type belongs to the late varieties. It can be grown outdoors in warm places and under foil. Pepper can be used as

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a vegetable but also as a spice and medicine. It can be used fresh, canned, dried and ground.

The use of pesticides in the agricultural sector is widespread, mainly for the protection of plant harmful organism. Currently, however, pesticides are often overused, with more than 4.0 million tonnes of pesticides used worldwide each year to control a broad spectar of pests (Liu et al., 2021). Reports from the European Union (EU) indicate that around 333,000 tonnes of pesticides were used in 2021, with more than 400 new pesticides on the market (Eurostat, 2023). The use of these pesticides on crops makes it necessary to ensure that a minimum level find their way into foodstuffs. In order to prevent toxicity problems associated with the excessive use of pesticides, Maximum Residue Limits (MRLs) have been set by various regulations. In the EU, for example, the European Commission (EC) has set MRLs for various foodstuffs in the consolidated version of Regulation (EC) No 396/2005 (EC, 2005). This Regulation applies to both food and feed of plant and animal origin as well as to unprocessed spices such as pepper, but not to some processed foods such as flour or paprika. In these cases, manufacturers act differently depending on the type of product in order to ensure its safety.

The primary control of the production and use of plant protection preparations in agricultural production in the Republic of North Macedonia is carried out by the State Agricultural Inspectorate, and the control in trade is carried out by the food inspectors of the Food and Veterinary Agency. The country does not allow the import of products (fruits and vegetables) that contain plant protection preparations above the maximum permitted concentrations defined in the legal and by-laws that relate to the safety of this category of products. Unsafe shipments are not allowed to enter the country, i.e. they are returned to the exporting country, or are safely destroyed.

Residues of the pesticide imidacloprid, which is banned in the EU, have been detected in cabbage from North Macedonia in the summer 2025 and 0.029 mg/kg of the substance was found in a sample of the vegetable (slobiden pecat.mk).

Long-term exposure to imidacloprid or improper handling can cause a number of adverse effects that can be divided into mild symptoms of poisoning at low exposure (GIT symptoms such as nausea, vomiting, stomach pain, hepatotoxicity, immune system disorders, neurological disorders headache, dizziness, drowsiness and disorientation), as well as serious life-threatening complications at high doses (difficulty breathing, hepatotoxicity, cardiovascular disorders, coma and death) (Mundhe et al., 2017).

This is the reson of examination of the level of imidacloprid in the most exported vegetable from the region of Strumica – red Ajvar pepper (*C. annuum* cv. Kurtovska Kapija).

Hence, the aim of this study was identification and quantification of 397 pesticides in 40 samples in red paper in red Ajvar pepper (*C. annuum* cv. Kurtovska Kapija) treated by Nimazal and Dikrsol 500 sp and Proteus before harvesting, with pervious extraction procedure by using the method of QuEChERS (EN 15662).

## Materials and methods

**Survey and sample collection.** The samples of Macedonian red Ajvar pepper (*C. annuum* cv. Kurtovska Kapija) were collected in September 2024 in the experimental fileds of village Dabilje (41° 26′ 35.74″ N, 22° 41′ 26.14″ E). Strumica, the south-east region of Republic of North Macedonia. A survey of treatment of red pepper by Nimazal and Dikrsol 500 sp was carried out in 18 cultivated fields at 9 areas of 4 provinces of Dabilje from July to September 20024 while red pepper treated by Proteus was carried out in 11 cultivated fields at 7 areas of 2 provinces of Dabilje. Forty samples of red pepper (*C. annuum* L. cv. Kurtovska Kapija) were collected from all experimental fileds and every sample was around 2 kg or approximately 20-25 freshly harvested red papers.

**Analysis of persticies**. For the analysis of pesticides in red Ajvar pepper (*C. annuum* cv. Kurtovska Kapija) the method of QuEChERS (EN 15662) was used, which involves extraction of pesticides with acetonitrile containing 1% formic acid and the use of NaCl and MgSO4 salts. Briefly, 10 g of chopped and blended pepper pulp was transferred to a Falcon tube and buffer solution. The mixture was vortexed, centrifuged and after 30 min. the salts were added. Then, 7 mL of the extract was transferred to another Falcon tube containing the salts for the purification step. After vortexing and centrifugation, 5 mL of supernatant was transferred to a mixture containing 50 μL of 5% formic acid (v/v) in acetonitrile, the solvent was dried under a stream of nitrogen, resuspended in 1 mL of methylethanol, and stored in a glass tube in a freezer until the time of analysis by liquid and gas chromatography. A total of 40 samples of red pepper Ajvarka (Kurtovska kapija) from the village of Dabile, Strumica region, were analyzed by liquid and gas chromatography with mass spectrometry. A total of 397 pesticides were identified and quantified.

The identification of pesticides in this research was done by gas and liquid chromatography with triple quadrupole detector MS/MS. For quantification of pesticides, pure standards of pesticides were used and calibration with an external standard was used. For analysis of pesticides by liquid chromatography-mass spectrometry, the following conditions were used: HPLC column: C18 (10 cm x 3.0 mm, 2.7μm), precolumn C18 (0.5 cm x 3.0 mm, 2.7μm). Liquid chromatography conditions were: Solvent composition: A: 1mmol/l ammonium formate with 0.1% formic acid in water;: B:Methanol Flow rate: 500 μL/min; injection volume 20 μL; Temperature: 40 Cº. MS/MS fragmentation conditions were: IS 5500V, Temperature $400^0$, GS1 35 psi, GC2 35 psi, Gas chromatography conditions, Column DB 5 MS crosslinked, 30 m x 0.25 mm x 0.25 μm, 5 % Ph Me Silicon, Carrier gas Helium, Flow rate 2 ml/min, GC temperature program 2 min at 40°C then with a step of 30 °C/min to 220 °C with a step of 5 °C/min to 260 °C then with a step of 20 °C/min to 280 °C (15 min), Injection volume 3 μl.

## Results and discussion

The part of the results from analyses of pesticides in of red pepper Ajvarka (Kurtovska Kapija) from the village of Dabile, Strumica region are presented on Fig 1. All analyzed pesticides such 2-Chloraniline, 2-Phenyl Phenol, 3-Chloraniline, 4-Chloraniline, Aldrin, Alpha, Endosulfan, Alphamethrin, Aminocarb, Benfluralin, Beta-Endosulfan, Cyfluthrin, Beta, BHC, Bifenazate, Biphenyl, Bromocyclen, Bromopropylate, Captafol, Captan, Carbophenothion, Chlorbenside, Chlordane, Chlorothalonil, Chlozolinate, Cycloate, Cypermethrin, Dazomet, DDD-4,4 and other pesticides presented on Fig. 1, were detected bellow limit of permitted concentrations. Only imidacloprid was detected above the permitted concentrations (0.011 mg/kg) in samples treated by Nimazal before harvesting, while in the samples of Ajvar red pepper treated with a a combination of Dikrsol 500 sp and Proteus, the same pesticide imidacloprid was detected at a concentration of 0.032 mg/kg sample. The permited MDK value for this pesticide is up to a maximum of 0.01 mg/kg.

The precise and accurate results from the residues of pesticides in red paprika strongly depends of crucial factors which affect the methodology of examination. For instance, sample size of peppers can vary significantly and can be challenging to accurately sample the fruit in a way that represents the overall population. Contamination is a crucial factor to avoid the contamination of the sample during the preparation process, as this can affect the accuracy of the results. Pesticide distribution may not be uniformly distributed on the surface of the peppers, making it difficult to sample the fruit accurately. Extraction efficiency which can impact the accuracy of the results, and some pesticides may be more difficult to extract than others. Finally, matrix effects or more precisely, the presence of other compounds in the peppers (such as proteins, carbohydrates, and lipids) can interfere with the analysis process and impact the accuracy of the results (Shim et al., 2023).

According to the findings of Veiga-del-Baño et al. (2023) the results showed that the uncertainty associated with subsampling is greater than the uncertainty associated with sampling when the product is sufficiently homogeneous, as in the case of spices. The minimum sample size required for pesticide analysis using the QuEChERS method on this type of product is 5 g, but it has been shown that a better sample size is between 20 and 30 g found in this study (Veiga-del-Baño et al., 2023). Furthermore, residue levels detected, especially for chlorpyrifos in the study of Mörtl et al., 2018 correlated well with pesticide treatments at increasing dosages applied.

Generally speaking, residues of chlorpyrifos found even in samples treated with low dose verify that the ban on the use of this active ingredient in red pepper crops was reasonable to avoid risks of contamination. Residues along the technical line reflect changes in water content and highlight the impact of the concentration of pollutants from the crop to the final product (Mörtl et al., 2018).

**Fig. 1.** Results from the determination of 397 residues of pesticides in red Ajvar paprika (Capsicum annuum cv. Kurtovska Kapija) from the region of Strumica, Republic of North Macedonia.

***Name of analyzed pesticide which is made by Quechers TS EN 15662 method with GC/MS***

1-)*2,4-5 T(0.010) , 2-)*2 Chloraniline(0.010) , 3-)*2-Phenyl Phenol(0.010) , 4-)*3-Chloraniline(0.010) , 5-)*4-Chloraniline(0.010) , 6-)*Aldrin(0.010) , 7-)*Alpha Endosulfan(0.010) , 8)*Alphamethrin(0.010) , 9-)*Aminocarb(0.010) , 10-)*Benfluralin(0.010) , 11-)*Beta-Endosulfan(0.010) , 12-)*Cyfluthrin,Beta(0.010) , 13-)*BHC(0.010) , 14-)*Bifenazate(0.010) , 15-)*Biphenyl(0.010) , 16-)*Bromocyclen(0.010) , 17-)*Bromopropylate(0.010) , 18-)*Captafol(0.010) , 19-)*Captan(0.010) , 20-)*Carbofuran 3 Hydroxy(0.010) , 21-)*Carbophenothion(0.010) , 22-)*Chlorbenside(0.010) , 23-)*Chlordane Cis Alpha(0.010) , 24-)*Chlordane Trans Gamma(0.010) , 25-)*Chlorfenapyr(0.010) , 26-)*Chlorfenson(0.010) , 27-)*Chlorobenzilate(0.010) , 28-)*Chloroneb(0.010) , 29-)*Chlorothalonil(0.010) , 30-)*Chlozolinate(0.010) , 31-)*Cycloate(0.010) , 32-)*Cypermethrin(0.010) , 33-)*Dazomet(0.010) , 34-)*DDD-2,4(0.010) , 35-)*DDD-4,4(0.010) , 36-)*DDE-2,4(0.010) , 37-)*DDE-4,4(0.010) , 38-)*DDT-2,4(0.010) , 39-)*DDT-4,4(0.010) , 40-)*Deltamethrin(0.010) , 41-)*Dicamba(0.010) , 42-)*Dichlobenil(0.010) , 43-)*4.4-Dichlorobenzophenone(0.010) , 44-)*Dicofol(0.010) , 45-)*Dieldrin(0.010) , 46-)*Diethatyl Ethyl(0.010) , 47-)*Dinobuton(0.010) , 48-)*Dinoseb Acetate(0.010) , 49-)*Dioxathion(0.010) , 50-)*Diphenylamine(0.010) , 51-)*Diphenylmercury(0.010) , 52-)*Endosulfansulfate(0.010) , 53-)*Endrin(0.010) , 54-)*Esfenvalarate&*Fenvelarate (0.010) , 55-)*Ethalfluralin(0.010) , 56-)*Fenchlorphos(0.010) , 57-)*Fenson(0.010) , 59-)*Fluchloralin(0.010) , 60-)*Flurprimidol(0.010) , 61-)*Flutriafol(0.010) , 62-)*Folpet(0.010) , 63-)*Formothion(0.010) , 64-)*Haloxyfop R Methyl(0.010) , 65-)*HCH Delta(0.010) , 66-)*HCH Alpha(0.010) , 67-)*HCH Beta(0.010) , 68-)*HCH Gamma(0.010) , 69-)*Heptachlor(0.010) , 70-)*Heptachlor Endo Epoxide Cis İsomer(0.010) , 71-)*Heptachlor Endo Epoxide Trans İsomer(0.010) , 72-)*Hexachlorobenzene(0.010) , 73-)*Iodofenphos(0.010) , 74-)*Isodrin(0.010) , 75-)*Isofenphos(0.010) , 76-)*Cyhalothrin,Lambda-(0.010) , 77-)*Leptophos(0.010) , 78-)*Mefenpyr Diethyl(0.010) , 79-)*Methoxychlor(0.010) , 80-)*Mirex(0.010) , 81-)*Nitrapyrin(0.010) , 82-)*Nitrofen(0.010) , 83-)*Nitral-Isopropyl(0.010) , 84-)*Pentachloroaniline(0.010) , 85-)*Permethrin(0.010) , 86-)*Perthane(0.010) , 87-)*Phenmedipham(0.010) , 88-)*Procymidone(0.010) , 89-)*Profluralin(0.010) , 90-)*Propamocarb , 91-)*Quinomethionat(0.010) , 92-)*Quintozene(0.010) , 93-)*S Metolachlor(0.010) , 94-)*Fluvalinate,tau-(0.010) , 95-)*Tecnazene(0.010) , 96-)*Tefluthrin(0.010) , 97-)*Terbacil(0.010) , 98-)*Tetrachlorvinphos(0.010) , 99-)*Tetradifon(0.010) , 100-)*Tetrasul(0.010) , 101-)*Thiometon(0.010) , 102-)*Tolylfluanid(0.010) , 103-)*Transfluthrin(0.010) , 104-)*Tributyl Phosphate(0.010) , 105-)*Trifluralin(0.010) , 106-)*Vinclozolin(0.010)

***Name of analyzed pesticide which is made by Quechers TS EN 15662 method with LC-MS/MS***

1-)*2,4D-Acid(0.010) , 2-)*3,4,5 Trimethacarb(0.010) , 3-)*Abamectin(0.010) , 4-)*Acephate(0.010) , 5-)*Acetamiprid(0.010) , 6-)*Acetochlor(0.010) , 7-)*Acibenzolar-S-Methyl(0.010) , 8-)*Aclonifen(0.010) , 9-)*Acrinatrin(0.010) , 10-)*Alachlor(0.010) , 11-)*Aldicarb(0.010) , 12-)*Aldicarb Sulfone(0.010) , 13-)*Aldicarb Sulfoxide(0.010) , 14-)*Ametryn(0.010) , 15-)*Amitraz(0.010) , 16-)*Amitrole(0.010) , 17-)*Anilazine(0.010) , 18-)*Aramite(0.010) , 19-)*Atrazine(0.010) , 20-)*Azimsulfuron(0.010) , 21-)*Azinphos Ethyl(0.010) , 22-)*Azinphos Methyl(0.010) , 23-)*Azaconazole(0.010) , 24-)*Azocyclotin(0.010) , 25-)*Azoxystrobin(0.010) , 26-)*Barban(0.010) , 27-)*Benalaxyl(0.010) , 28-)*Bendiocarb(0.010) , 29-)*Benfuracarb(0.010) , 30-)*Benomyl(0.010) , 31-)*Bensulfuron-Methyl(0.010) , 32-)*Bentazone(0.010) , 33-)*Bifentrin(0.010) , 34-)*Binapacryl(0.010) , 35-)*Bioresmethrin(0.010) , 36-)*Bitertanol(0.010) , 37-)*Boscalid(0.010) , 38-)*Bromacil(0.010) , 39-)*Bromophos-Ethyl(0.010) , 40-)*Bromophos-Methyl(0.010) , 41-)*Bromoxyil(0.010) , 42-)*Bromuconazole(0.010) , 43-)*Bupirimate(0.010) , 44-)*Buprofezin(0.010) , 45-)*Butafenacil(0.010) , 46-)*Butocarboxim(0.010) , 47-)*Butocarboxim-Sulfone(0.010) , 48-)*Butocarboxim-Sulfoxide(0.010) , 49-)*Butoxycarboxim(0.010) , 50-)*Butralin(0.010) , 51-)*Buturon(0.010) , 52-)*Cadusafos(0.010) 60-)*Carbaryl(0.010) , 61-)*Carbendazim(0.010) , 62-)*Carbofuran(0.010) , 63-)*Carbosulfan(0.010) , 64-)*Carboxin(0.010) , 65-)*Carfentrazone Ethyl(0.010) , 66-)*Chlorbromuron(0.010) , 67-)*Chlorbufam(0.010) , 68-)*Chlorfenvinphos(0.010) , 69-)*Chlorfluazuron(0.010) , 70-)*Chloridazon(0.010) , 71-)*Chlormequat-Chloride(0.010) , 72-)*Chlorotoluron(0.010) , 73-)*Chloroxuron(0.010) , 74-)*Chlorpropham(0.010) , 75-)*Chlorpyrifos(0.010) , 76-)*Chlorpyrifos-Methyl(0.010) , 77-)*Chlorsulfuron(0.010) , 78-)*Chlortal-Dimethyl(0.010) , 79-)*Chlorantraniliprole(0.010) , 80-)*Cinidon-Ethyl(0.010) , 81-)*Clethodim(0.010) , 82-)*Clethodim-Iminsulfone(0.010) , 83-)*Clethodim-Iminsulfoxide(0.010) , 84-)*Clethodim-Sulfoxide(0.010) , 85-)*Climbazole(0.010) , 86-)*Clodinafop-Propargyl-Ester(0.010) , 87-)*Clofentezine(0.010) , 88-)*Clomazone(0.010) , 89-)*Cloquintocet-Mexyl-Ester(0.010) , 90-)*Clothianidin(0.010) , 91-)*Coumaphos(0.010) , 92-)*Crimidine(0.010) , 93-)*Cyanazine(0.010) , 94-)*Cyanofenphos(0.010) , 95-)*Cyazofamid(0.010) , 96-)*Cycloxydim(0.010) , 97-)*Cyhalofop(0.010) , 98-)*Cyhalofop-Butyl(0.010) , 99-)*Cyhalofop Diacid(0.010) , 100-)*Cyhexatin(0.010) , 101-)*Cymoxanil(0.010) , 102-)*Cyproconazole(0.010) , 103-)*Cyprodinil(0.010) , 104-)*Cyromazine(0.010) , 105-)*Daminozide(0.010) , 106-)*Demeton O+S (0.010) , 107-)*Demeton-S-Methyl(0.010) , 108-)*Demeton-S-Methylsulfone(0.010) , 109-)*Demeton-S-Methylsulfoxide(0.010) , 110-)*Desmedipham(0.010) , 111-)*Desmetryn(0.010) , 112-)*Diafenthiuron(0.010) , 113-)*Dialifos(0.010) , 114-)*Di-Allate(0.010) , 115-)*Diazinon(0.010) , 116-)*Dichlofenthion(0.010) , 117-)*Dichlofluanid(0.010) , 118-)*Dichloprop(0.010) , 119-)*Dichlorvos(DDVP)(0.010) , 120-)*Diclobutrazol(0.010) , 121-)*Diclofop-Methyl(0.010) , 122-)*Dicloran(0.010) , 123-)*Dicrotophos(0.010) , 124-)*Diethofencarb(0.010) , 125-)*Difenoconazole (0.010) , 126-)*Diflubenzuron(0.010) , 127-)*Diflufenican(0.010) , 128-)*Dimethenamid(0.010) , 129-)*Dimethoate(0.010) , 130-)*Dimethomorph(0.010) , 131-)*Dimoxystrobin(0.010) , 132-)*Diniconazole(0.010) , 133-)*Dinitramine(0.010) , 134-)*Dinocap(0.010) , 135-)*Dinoseb(0.010) , 136-)*Dinoterb(0.010) , 137-)*Diphenamid(0.010) , 138-)*Dipropetryn(0.010) , 139-)*Disulfoton(0.010) , 140-)*Ditalimfos(0.010) , 141-)*Dithianon(0.010) , 142-)*Diuron(0.010) , 143-)*DNOC(0.010) , 144-)*Dodine(0.010) , 145-)*E Fenpyroxymate(0.010) , 146-)*Epichlorohydrin(0.010) , 147-)*EPN(0.010) , 148-)*Epoxiconazole(0.010) , 149-)*EPTC(0.010) , 150-)*Etaconazole(0.010) , 151-)*Ethiofencarb(0.010) , 152-)*Ethiofencarb-Sulfone(0.010) , 153-)*Ethiofencarb-Sulfoxide(0.010) , 154-)*Ethion(0.010) , 155-)*Ethirimol(0.010) , 156-)*Ethofenprox(0.010) , 157-)*Ethofumesate(0.010) , 158-)*Ethoprophos(0.010) , 159-)*Ethoxyquin(0.010) , 160-)*Ethylene Thiourea(0.010) , 161-)*Etoxazole(0.010) , 162-)*Etridiazole(0.010) , 163-)*Etrimfos(0.010) , 164-)*Famoxadone(0.010) , 165-)*Famphur(0.010) , 166-)*Fenamidone(0.010) , 167-)*Fenamiphos(0.010) , 168-)*Fenarimol(0.010) , 169-)*Fenazaquin(0.010) , 170-)*Fenbuconazole(0.010) , 171-)*Fenbutatin-Oxide(0.010) , 172-)*Fenhexamid(0.010) , 173-)*Fenitrothion(0.010) , 174-)*Fenobucarb(0.010) , 175-)*Fenoxaprop-P-Ethyl(0.010) , 176-)*Fenoxycarb(0.010) , 177-)*Fenpiclonil(0.010) , 178-)*Fenpropathrin(0.010) , 179-)*Fenproprimorph(0.010) , 180-)*Fensulfothion(0.010) , 181-)*Fenthion(0.010) , 182-)*Fenthion-Sulfone(0.010) , 183-)*Fenthion-Sulfoxide(0.010) , 184-)*Fentin-Acetate(0.010) , 185-)*Fipronil(0.010) , 186-)*Fluazifop-P-Butyl(0.010) , 187-)*Fluazinam(0.010) 188-)*Flubenzimine(0.010) , 189-)*Flucythrinate(0.010) , 190-)*Fludioxonil(0.010) , 191-)*Flufenacet(0.010) , 192-)*Flufenoxuron(0.010) , 193-)*Flumioxazin(0.010) , 194-)*Fluopicolide(0.010) , 195-)*Fluopyram(0.010) , 196-)*Flurochloridone(0.010) , 197-)*Fluquinconazole(0.010) , 198-)*Fluroxypyr(0.010) , 199-)*Flusilazole(0.010) , 200-)*Flutolanil(0.010) , 201-)*Fonofos(0.010) , 202-)*Foramsulfuron(0.010) , 203-)*Formetanate Hydrochloride(0.010) , 204-)*Formetanate(0.010) , 205-)*Fosthiazate(0.010) , 206-)*Fuberidazole(0.010) , 207-)*Furalaxyl(0.010) , 208-)*Furathiocarb(0.010) , 209-)*Halfenprox(0.010) , 210-)*Haloxyfop 2 Ethoxyethyl(0.010) , 211-)*Heptenophos(0.010) , 212-)*Hexaconazole(0.010) , 213-*Hexaflumuron(0.010) , 214-)*Hexazinone(0.010) , 215-)*Hexythiazox(0.010) , 216-)*Imazalil(0.010) , 217-)*Imazaquin(0.010) , 218-)*Imazethapyr(0.010) , 219-)*Imidacloprid(0.010) , 220-)*Indoxacarb Sum(0.010) , 221-)*Iodosulfuron Methyl Sodium(0.010) , 222-)*Ioxynil(0.010) , 223-)*Iprodione(0.010) , 224-)*Iprovalicarb(0.010) , 225-)*Isazofos(0.010) , 226-)*Isocarbophos(0.010) , 227-)*Isoprocarb(0.010) , 228-)*Isoproturon(0.010) , 229-)*Isoxaben(0.010) , 230-)*Isoxaflutole(0.010) , 231-)*Isoxathion(0.010) , 232-)*Kresoxim-Methyl(0.010) , 233-)*Lenacil(0.010) , 234-)*Linuron(0.010) , 235-)*Lufenuron(0.010) , 236-)*Malaoxon(0.010) , 237-)*Malathion(0.010) , 238-)*Maleic Hyrdazide(0.010) , 239-)*Mandipropamid(0.010) , 240-)*MCPA(0.010) , 241-)*Mecarbam(0.010) , 242-)*Mepronil(0.010) , 243-)*Mesotrione(0.010) , 244-)*Metobromuron(0.010) , 245-)*Metalaxyl&*Metalaxyl M (0.010) , 247-)*Metazachlor(0.010) , 248-)*Metconazole(0.010) , 249-)*Methabenzthiazuron(0.010) , 250-)*Methacrifos(0.010) , 251-)*Methamidophos(0.010) , 252-)*Metamitron(0.010) , 253-)*Methidathion(0.010) , 254-)*Methiocarb(0.010) , 255-)*Methiocarb Sulfone(0.010) , 256-)*Methiocarb Sulfoxide(0.010) , 257-)*Methiocarb Sum(0.010) , 258-)*Methomyl(0.010) , 259-)*Methomyl-Oxime(0.010) , 260-)*Methomyl-Sulfone(0.010) , 261-)*Metolachlor(0.010) , 262-)*Metolcarb(0.010) , 263-)*Metosulam(0.010) 264-)*Metoxuron(0.010) , 265-)*Metribuzin(0.010) , 266-)*Metrofenone(0.010) , 267-)*Metsulfuron-Methyl(0.010) , 268-)*Mevinphos(0.010) , 269-)*Molinate(0.010) , 270-)*Monocrotophos(0.010) , 271-)*Monolinuron(0.010) , 272-)*Monuron(0.010) , 273-)*Myclobutanil(0.010) , 274-)*Nabam(0.010) , 275-)*Naphthol,1-(0.010) , 276-)*Napropamide(0.010) , 277-)*Neburon(0.010) , 278-)*Nicosulfuron(0.010) , 279-)*Nitenpyram(0.010) , 280-)*Novaluron(0.010) , 281-)*Nuarimol(0.010) , 282-)*Ofurace(0.010) , 283-)*Omethoate(0.010) , 284-)*Oxadiazon(0.010) , 285-)*Oxadixyl(0.010) , 286-)*Oxamyl(0.010) , 287-)*Oxyfluorfen(0.010) , 288-)*Paclobutrazol(0.010) , 289-)*Paraoxon-Ethyl(0.010) , 290-)*Paraoxon-Methyl(0.010) , 291-)*Parathion Ethyl(0.010) , 292-)*Parathion Methyl(0.010) , 293-)*Pebulate(0.010) , 294-)*Penconazole(0.010) , 295-)*Pencycuron(0.010) , 296-)*Pendimethalin(0.010) , 297-)*Phenthoate(0.010)

***Name of analyzed pesticide which is made by Quechers TS EN 15662 method with LC-MS/MS***

298-)*Phorate(0.010) , 299-)*Phosalone(0.010) , 300-)*Phosmet(0.010) , 301-)*Phosmet Oxon(0.010) , 302-)*Phosphamidon(0.010) , 303-)*Phoxim(0.010) , 304-)*Picolinafen(0.010) , 305-)*Picoxystrobin(0.010) , 306-)*Pirimicarb(0.010) , 307-)*Pirimiphos Ethyl(0.010) , 308-)*Pirimiphos Methyl(0.010) , 309-)*Prochloraz(0.010) , 310-)*Profenofos(0.010) , 311-)*Profoxydim(0.010) , 312-)*Profoxidim-Lithium(0.010) , 313-)*Prohexadione Calcium(0.010) , 314-)*Promecarb(0.010) , 315-)*Prometryn(0.010) , 316-)*Propachlor(0.010) , 317-)*Propanil(0.010) , 318-)*Propaquizafop(0.010) , 319-)*Propargite(0.010) , 320-)*Propazine(0.010) , 321-)*Propham(0.010) , 322-)*Propiconazole(0.010) , 323-)*Propoxur(0.010) , 324-)*Propoxycarbazone Sodium(0.010) , 325-)*Propyzamide(0.010) , 326-)*Prosulfocarb(0.010) , 327-)*Prosulfuron(0.010) , 328-)*Prothiofos(0.010) , 329-)*Pymetrozine(0.010) , 330-)*Pyraclostrobin(0.010) , 331-)*Pyraflufen(0.010) , 332-)*Pyraflufen Ethyl(0.010) , 333-)*Pyrazophos(0.010) , 334-)*Pyrethrins(0.010) , 335-)*Pyridaben(0.010) , 336-)*Pyridalyl(0.010) , 337-)*Pyridaphenthion(0.010) , 338-)*Pyridate(0.010) , 339-)*Pyrifenox(0.010) , 340-)*Pyrimethanil(0.010) , 341-)*Pyriproxyfen(0.010) , 342-)*Quinalphos(0.010) , 343-)*Quinoxyfen(0.010) , 344-)*Quizalofop Ethyl(0.010) , 345-)*Resmethrin(0.010) , 346-)*Rimsulfuron(0.010) , 347-)*Sethoxydim(0.010) , 348-)*Simazine(0.010) , 349-)*Spinosad(0.010) ,

350-)*Spirodiclofen(0.010) , 351-)*Spiroxamine(0.010) , 352-)*Sulfosulfuron(0.010) , 353-)*Sulfotep(0.010) , 354-)*Sulprofos(0.010) , 355-)*Tebuconazole(0.010) , 356-)*Tebufenozide(0.010) , 357-)*Tebufenpyrad(0.010) , 358-)*Teflubenzuron(0.010) , 360-)*Tepraloxydim(0.010) , 361-)*Terbumeton(0.010) , 362-)*Terbuthylazine(0.010) , 363-)*Terbutryn(0.010) , 364-)*Tetraconazole(0.010) , 365-)*Tetramethrin(0.010) , 366-)*Thiabendazole(0.010) , 367-)*Thiacloprid(0.010) , 368-)*Thiamethoxam(0.010) , 369-)*Thifensulfuron Methyl(0.010) , 370-)*Thiobencarb(0.010) , 371-)*Thiodicarb (0.010) , 372-)*Thiofanox (0.010) , 373-)*Thiofanox Sulfone(0.010) , 374-)*Thiofanox Sulfoxide(0.010) , 375-)*Thiophanate Methyl(0.010) , 376-)*Tolclofos Methyl(0.010) , 377-)*Tralkoxydim(0.010) , 378-)*Triadimefon(0.010) , 379-)*Triadimenol(0.010) , 380-)*Tri-Allate(0.010) , 381-)*Triasulfuron(0.010) , 382-)*Triazophos(0.010) , 383-)*Tribenuron Methyl(0.010) , 384-)*Trichlorfon(0.010) , 385-)*Trichloronat(0.010) , 386-)*Tricyclazole(0.010) , 387-)*Tridemorph(0.010) , 388-)*Triethyl Phosphate(0.010) , 389-)*Trifloxystrobin(0.010) , 390-)*Triflumizole(0.010) , 391-)*Triflumuron(0.010) 392-)*Triforine(0.010) , 393-)*Triphenylphosphate(0.010) , 394-)*Triticonazole(0.010) , 395-)*Vamidothion(0.010) , 396-)*Zoxamide(0.010) , 397-)*Terbufos(0.010)

According to working group of Park et al. (2024), the top 10 pesticides with the highest number of detected cases were clothianidin, imidacloprid, thiamethoxam, chlorpyrifos, thiacloprid, metalaxyl, myclobutanil, azoxystrobin, carbendazim, and cyhalothrin, and their detection rates varied from 10.52% to 28.66%. Clothianidin is pesticide is effective against various insect species, such as Hemiptera, Coleoptera, Thysanoptera, Lepidoptera, and Diptera. It is also easily absorbed by plants, but it is safe for crops and exhibits low toxicity in mammals, birds, and aquatic species. Additionally, long-term control effects can be achieved even at low doses of clothianidin owing to its high insecticidal activity and chemical stability (Uneme et al., 2006).

The second most detected pesticide was thiamethoxam, which was detected in 277 out of 967 inspections, giving a detection rate of 28.65%. According to the Son et al., 2017, Thiamethoxam is one of the most widely used insecticides, given that it is less toxic to humans but exhibits high activity against pests and insects. However, several countries in Europe placed a ban on its use between 2013 and 2015 owing to concerns regarding its association with the occurrence of honeybee colony collapse disorder (Son et al., 2017).

The only residue of pesticides in Kurtovska Kapija red paper which was ubove the limit of permitted concentrations in our study was Imidacloprid. Imidacloprid, with 277 detections out of 1014 inspections, showed the third-highest detection rate (27.32%) among the top 10 most frequently detected pesticides. Imidacloprid is a systemic pesticide with a long-life span of up to 190 days, a half-life by photolysis of 39 days, and an indoor half-life of up to 997 days (Ihm et al., 2004).

Imidacloprid is the well-known and widely used belongs to the group of the neonicotinoid insecticides. It is a broad-spectrum neonicotinoid insecticide, with excellent systemic and contact activity that supports its use on many food crops, turf, and ornamentals and flea control (Sheets et al., 2010). They are designed to act on nicotinic receptors to control insect pests and shows low toxicity to vertebrate species. By oral administration, imidacloprid is rapidly absorbed, metabolized in the liver, and excreted, primarily via the urine (Nugnes et al., 2023). There are two major routes of metabolism in mammals. The first involves oxidative cleavage to imidazolidine and 6-chloronicotinic acid, with the imidazolidine moiety excreted via the urine (Mourikes et al., 2023). The second substantive route in the biotransformation of imidacloprid involves the hydroxylation of the intact molecule in the imidazolidine ring, followed by the elimination of water and the formation of an unsaturated metabolite. Imidacloprid is absorbed and widely distributed to organs within 1 h following oral administration to rats (Zhang et al., 2018). Acute toxicity is characterized by nicotinic signs at relatively high levels of exposure. Due to its high insecticidal potency and relatively low mammalian toxicity, imidacloprid has a very high margin of safety. Imidacloprid is not mutagenic or carcinogenic, it is not a primary embryotoxicant, is not teratogenic, and has no effect on reproduction or development (Liu et al., 2022; Wang et al., 2023b). Imidacloprid is widely used in tree injection, soil injection, and pesticide-coated seed treatment. Imidacloprid applies to the central nervous system, while the prenatal and postpartum exposure can bring about neurobehavioral defects (Guo et al., 2021). According to the newly method developed by the working group of Liang et al.(2023), FRET technology between two carbon based QDs was studied, known as GQDs and CQDs (Luo et al., 2022). After optimization, the LOD of this biosensor was 0.823 nM and the linearity was maintained from 5 to 4000 nM (Liang et al., 2023). This method was applied to detect the imidacloprid residues in banana, apple, cabbage, cucumber samples, with a satisfactory recovery rate of 93.0%–105.6% and RSDs of lower than 4%. FRET based on GO and UCNPs was also built for the rapid analysis of imidacloprid (Guo et al., 2021). Under optimum conditions, this FRET immunoassay showed a wide range of 0.08–50 ng/mL for the detection of imidacloprid. The average recoveries in samples of Chinese cabbage, cucumber, honey, spiked water, and tea were 76.8%–101.8% with RSDs lower than 14.3%.

Chlorpyrifos, the pesticide with the fourth-highest detection rate (20.92%), was detected in 204 out of 975 inspections. This pesticide is a widely used insecticide in agriculture and households, and it is known for its low solubility in water and high soil adsorption coefficient. It also exhibits poor biodegradability and possesses a strong affinity for soil particles, leading to a prolonged persistence of 60–120 days in soil. This characteristic raises environmental pollution concerns, including soil, river, and groundwater contamination (Kim et al., 2007).

Metalaxyl, which was detected in 123 out of 936 inspections, showed the fifth-highest detection rate among the top 10 pesticides (13.14%). Metalaxyl is a stable compound under a wide range of pH conditions, temperatures, and light intensities. Owing to these characteristics as well as its wide-spectrum activity, it is used on a broad range of crops and in several countries with temperate, subtropical, and tropical climates (Adolphe et al., 2005).

## Conclusion

The results presented in this study are the first published results for the evaluation of the residues of presticides in the most famous red pepper Kurtovska Kapija (known as ajvarka) fom the region of Strumica, Republic of North Macedonia. Identification and quantification of 397 pesticides were performed and imidacloprid was detected above the permitted concentrations (0.011 mg/kg) in samples treated by Nimazal before harvesting, while in the samples of Ajvar red pepper treated with a a combination of Dikrsol 500 sp and Proteus, the same pesticide imidacloprid was detected at a concentration of 0.032 mg/kg sample. All other examined residues of pesticides were in the concentrations bellow limit of permission and we can conlude that vetetable and especially red pepper Kurtovska Kapija is safe for consumption without any hamrfil effect on human health.

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