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Determination of Pesticide Residues in Sour Cherry used in the Fruit Juice Production in Tokat provinces

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ABSTRACT

Sour cherry (*Prunus cerasus* L.) (Rosaceae) is a spring fruits. It is not preferred to be consumed as fresh because it is sour, but it is extremely beneficial for human health. In addition to fresh consumption, it is used in the production of fruit juice, syrup, jam, marmalade, cake and ice cream in the food industry. Sour cherry is grown widely in Tokat and 80-85% of the grown cherries are sold to juice factories. Producers mostly adopt chemical control against pests. In this respect, monitoring pesticide residues on sour cherry is extremely important. This study was carried out to determine the pesticide residue levels in samples taken from sour cherry production areas in Tokat province in 2020. The residue analyses were performed by using QuEChERS method and LC-MS / MS (Liquid Chromatography / Tandem Mass Spectrometer). According to the results, the pesticide residue levels were found below the maximum residue limits (MRL) given in Turkish Food Codex (TFC).

Türk Tarım – Gıda Bilim ve Teknoloji Dergisi, 8(sp1): 106-110, 2020

Tokat ve Çevresinde Meyve Suyu İmalatında Kullanılan Vişnelerdeki Pestisit Kalıntılarının Belirlenmesi

MAKALE BİLGİSİ

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ÖZ

Vişne (*Prunus cerasus* L.) (Rosaceae) bahar meyvelerinden olup, ekşi olduğu için taze olarak tüketilmesi zor fakat sağlığımız için son derece faydalı bir meyvedir. Bu yüzden taze tüketimi yanında meyve suyu olarak, gıda endüstrisinde şurup, reçel, marmelat, pasta ve dondurma üretiminde kullanılmakta bunlara ilaveten kurutularak da tüketilebilmektedir. Tokatta yaygın olarak vişne yetiştiriciliği yapılmakta olup üretilen vişnenin %80-85'i fabrikalara satılmaktadır. Üreticiler vişnedeki zararlı, hastalık ve yabancı otlara karşı çoğunlukla kimyasal mücadeleyi benimsemektedirler. Bu nedenle vişnede pestisit kalıntılarının takibi önemlidir. Bu çalışma, 2020 yılında Tokat ilinde üretimi yapılan vişnelerden alınan örneklerdeki pestisit kalıntı düzeylerinin belirlenmesi amacıyla gerçekleştirilmiştir. Alınan örneklerin kalıntı analizleri QuEChERS metodu kullanılarak LC-MS/MS (Sıvı Kromatografi/Tandem Kütle Spektrometresi) cihazında yapılmıştır. Araştırma sonuçlarına göre, Tokatta vişne üretimi yapılan lokasyonlardan alınan örnekler değerlendirilmiş, pestisit kalıntı düzeyleri Türk Gıda Kodeksi (TGK) Maksimum Kalıntı Limitleri yönetmeliğinde belirtilen değerlerinin altında bulunmuştur.

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Introduction

The homeland of sour cherry is probably a region between the Caspian Sea and the North Anatolian mountains. It has been reported that it was first cultivated in Anatolia and transferred to Greece (Özbek, 1978). According to 2016 data, the sour cherry production in world is 13.8 million tons on an area of 214396 hectares and 192 thousand tons on an area of 22324 hectares in Turkey. Russia is the biggest sour cherry producer in the World. Poland follows Russia with 14.14% production and Turkey with 13.97% of production, respectively. According to 2018 data, Afyonkarahisar ranks first in cherry production with 47,485 tons. Konya is second sour cherry producer with 30,451 tons and Kütahya is third with 26,051 tons. Tokat ranks 7th with 5922 tons of cherry production (Anonymous, 2020). 80-85% of sour cherries grown in Tokat are sold to factories. Chemical control is carried out commonly against sour cherry pests, which are intense from time to time in region. These chemicals are found in food in traces and have a negative effect if their content exceeds the maximum residue level. Food safety is of a considerable importance to consumers, food industry and economy (Jevšnik et al., 2008). In sour cherry, it seems that research on this problem is extremely limited. Słowik-Borowiec et al. (2015) in their study founded pesticide residues in 4 out of 27 sour cherry samples in Poland between 2012 and 2014. In a 2-year study conducted in Poland, fifty-nine percent of 71 sour cherry samples determined pesticide residues (Nowacka and Holodynska-Kulas, 2020). In a study conducted in Turkey in 2010, 4,0 µg/kg Acetamiprid in one of 3 sour cherry samples, in another sample, Chlorpyrifos (forbidden to use) pesticide residue at the level of 5,0 µg/kg was found (Ersoy et al., 2011b). In addition, Özgün et al. (1997), in a study on peach and apricot nectar and cherry and apple juices, found chlorinated hydrocarbon insecticide residues in 26 of 203 samples.

In this study, it was aimed to determine the pesticide residue levels in sour cherries used in fruit juice production in Tokat province.

Materials and Methods

Reagents and Chemicals

Pesticide reference standards were supplied Dr. Ehrenstorfer Laboratories GmbH (Bgm.-Schlosser-STr. 6A, Augsburg, Germany). Acetonitrile (MeCN), methanol (MeOH), Magnesium sulfate anhydrous (MgSO4), sodium acetate (NaOAc) and acetic acid (AcOH) were taken from Merck (Darmstadt, Germany). Primary-secondary amine (PSA) was taken from Supelco Analytical (595 N Harrison Rd, Bellefonte, PA, USA)

Sampling

The materials used in this study were obtained from sour cherry production areas in Kazova, Almus and Niksar. 10 samples were randomly selected, samples were taken at a minimum of 1 kg each for analysis (EC, 2002) and brought to the laboratory in cool, dark conditions within 24 hours. The samples were analyzed without waiting.

Extraction and Clean Up Procedure

The official QuEChERS AOAC Method 2007.01 was used for extraction and clean up procedures, (Lehotay, 2007). Samples are homogenized and made uniform. The samples were weighed about 15 grams in a 50 ml clean tube.15 mL of acetonitrile (MeCN) including %1 acetic acid was added to the sample weighed in a clean tube of 50 ml. Samples shake vigorously by hand for 1 minute. The steps in Figure 1 were followed for the next process. Each analytical portion was analysed in triplicates (3 GC vials) with LC-MS/MS.

LC-MS/MS Analyses

The residue analyses of the collected samples were performed in TOGÜ Scientific and Technological Research Application and Research Centre using LC-MS / MS. LC analysis was carried out using chromatography system (Shimadzu, Kyoto, Japan) equipped with degasser (DGU-20A3R), pump (LC-30AD), auto sampler (SIL-20A) and column furnace (CTO-10AS VP). MS / MS analysis was carried out using LCMS-8050 triple-quadrupole tandem mass spectrometer (Shimadzu, Kyoto, Japan). The parameters of the device are given in Table 1.

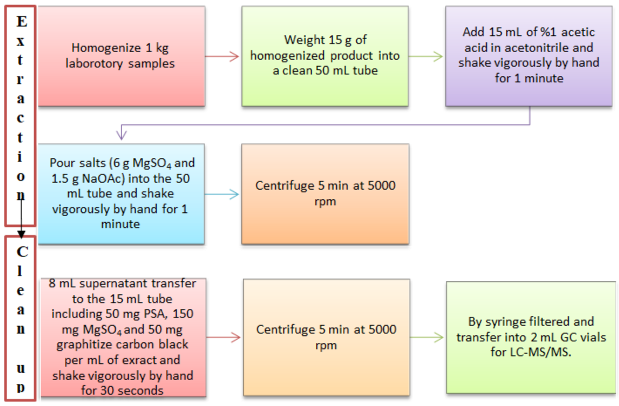

Figure 1. Analytical steps of the QuEChERS-AOAC Official Method 2007.01

Table 1. LC-MS/MS parameters

Mobile Phase A	Distilled water + 5 mmol ammonium formate		
Mobile Phase B	Methanol + 5 mmol ammonium formate		
Mobile Phase Flow	0,4 mL/min		
Column	C18 Inertsil ODS-4; 3µm; 2,1×150mm		
Gradient	Time (min)	%A	%B
	0-3	95	5
	3,01-6	40	60
	6,01-7	30	70
	7,01-8,50	20	80
	8,51-15	95	5
Column oven temp.	35°C		
Injection Volume	10 µl		
MS Gas Temperature	300°C		
MS Gas Flow	10 L/min		
Nebulizer Pressure	270 kPa		
Room Temperature	20°C		

Results and Discussion

Calibration curves of pesticide standards in representative sample matrix were linear over the range of 5–200 µg L^{-1}, with r^2, ($r^2 \geq 0.99$). For linearity, it is important that the correlation coefficient is greater than 0.99 (Tiryaki et al., 2008). This result shows that the method was linear with a specified concentration ranges. The studies were performed in 10 replicates at a single concentration (10 µg L^{-1}) and the standard deviation (SD) and relative standard deviation (RSD%) values of each pesticide were calculated. The LOD value was determined as 3 times the calculated standard deviation values for each pesticides. LOQ value was calculated as 10 times the standard deviation values calculated for each pesticides (SANTE, 2019) (Table 2a, b).

Table 2a. 259 pesticide included in the LC-MS/MS method and limit of quantitation

No	Pesticide	LOQ (µg/kg)	No	Pesticide	LOQ (µg/kg)	No	Pesticide	LOQ (µg/kg)
1	2.4-D	6.54	88	EPTC	23.06	175	Oxadixyl	6.44
2	Abamectin	5.65	89	Ethiofencarb	3.03	176	Oxamyl	1.74
3	Acephate	3.88	90	Ethion	6.23	177	Oxycarboxin	3.08
4	Acequinocyl	9.26	91	Ethirimol	3.70	178	Oxydemeton-methyl	7.08
5	Acetamiprid	6.66	92	Etofenprox	21.43	179	Paclobutrazol	5.58
6	Acetochlor	15.09	93	Etoxazole	3.50	180	Paraoxon-ethyl	6.48
7	Acrinathrin	13.46	94	Famaxadone	15.55	181	Paraoxon-methyl	13.01
8	Alachlor	7.04	95	Fenamidone	5.08	182	Penconazole	5.46
9	Aldicarb	15.56	96	Fenamiphos	4.86	183	Pencycuron	8.80
10	Aldicarb-sulfone	2.26	97	Fenamiphos-sulfone	5.11	184	Pendimenthalin	3.23
11	Aldicarb-sulfoxide	9.57	98	Fenamiphos-sulfoxide	3.87	185	Permethrin	15.84
12	Ametoctradin	3.93	99	Fenarimol	16.07	186	Phenmedipham	7.60
13	Amitraz	11.01	100	Fenazaquin	2.68	187	Phenthoate	5.83
14	Atrazine	6.59	101	Fenbuconazole	5.07	188	Phorate	7.05
15	Azinphos-ethyl	10.04	102	Fenbutatin oxide	3.80	189	Phorate-sulfone	15.00
16	Azinphos-methyl	6.71	103	Fenhexamide	9.11	190	Phorate-sulfoxide	3.13
17	Azoxystrobin	14.86	104	Fenoxycarb	8.24	191	Phosalone	4.83
18	Benalaxyl	3.62	105	Fenoxyprob -ethyl	13.54	192	Phosmet	6.09
19	Benfuracarb	10.82	106	Fenpropathrin	10.78	193	Phosphamidon	7.09
20	Benomyl	4.87	107	Fenproxymate	2.10	194	Pirimicarb-Desmethyl	3.81
21	Bensulfuron-methyl	3.19	108	Fenthion	9.90	195	Primicarb	5.54
22	Bentazone	13.52	109	Fenthion-sulfone	13.35	196	Primiphos -ethyl	6.84
23	Bifenazate	7.16	110	Fenthion-sulfoxide	4.57	197	Primiphos -methyl	7.76
24	Bitertanol	12.88	111	Fipronil	5.66	198	Prochloraz	10.26
25	Boscalid	9.90	112	Fipronil-sulfone	9.01	199	Profenefos	8.12
26	Bromuconazole	9.60	113	Fluazifop-p-butyl	7.14	200	Profoxydim-lithium	8.43
27	Buprimate	9.56	114	Fluazinam	20.45	201	Promecarb	5.16
28	Buprofezin	5.82	115	Flubendiamide	18.58	202	Prometryn	5.01
29	Butralin	4.44	116	Fludioxinil	12.63	203	Propaquizafob	11.10
30	Butylate	6.62	117	Flufenoxuron	3.94	204	Propargite	3.38
31	Cadusafos	18.08	118	Fluopicolide	7.31	205	Propazine	3.45
32	Carbaryl	6.19	119	Fluopyram	3.38	206	Propiconazole	18.68
33	Carbendazim	5.19	120	Fluquinconazole	5.93	207	Propoxur	5.07
34	Carbofuran	5.52	121	Flurochloridone	17.38	208	Propyzamide	5.40
35	Carbofuran-3-hydroxy	7.18	122	Fluroxypyr	10.07	209	Prothiophos	6.34
36	Carbosulfan	3.46	123	Flusilazole	13.80	210	Pymetrozine	4.73
37	Carboxin	4.35	124	Flutriafol	7.16	211	Pyraclostrobin	5.43
38	Carfentrazone-ethyl	4.14	125	Forchlorfenuron	3.27	212	Pyrazophos	4.21
39	Chlorantraniliprole	13.00	126	Formetanete hydrochloride	7.67	213	Pyridaben	6.28
40	Chlorbufam	26.30	127	Fosthiazate	3.40	214	Pyridaphenthion	3.58
41	Chlorfenvinhos	9.31	128	Furathiocarb	3.83	215	Pyridate	3.66
42	Chlorfluazuron	18.78	129	Haloxyfop-R-methyl	8.99	216	Pyrimethanil	10.03
43	Chloridazon	4.07	130	Hexaconazole	7.81	217	Pyriproxyfen	5.30
44	Chlorsulfuron	8.14	131	Hexaflumuron	9.29	218	Quinalphos	9.62
45	Clethodim	10.05	132	Hexythiazox	8.61	219	Quizalofop-ethyl	10.00
46	Clodinofop-propargyl	8.20	133	Imazalil sulfate	12.23	220	Rimsulfuron	6.57
47	Clofentezine	10.81	134	Imazapyr	3.30	221	Sethoxydim	6.33
48	Clothianidine	11.22	135	Imidacloprid	5.42	222	Simazine	7.24
49	Cyantraniliprole	14.15	136	Indoxacarb	11.87	223	Spinosyn A	8.24
50	Cyazofamid	13.39	137	Iodosulfuron-methyl-sodium	4.29	224	Spinosyn D	10.25
51	Cycloate	11.66	138	Ioxynil	11.69	225	Spirodiclofen	11.49
52	Cycloxydim	9.18	139	Isocarbofos	13.93	226	Spiromesifen	12.46
53	Cyflufenamid	6.58	140	Kresoxim Methyl	8.08	227	Spiroxamine	16.87
54	Cyhalothrin	25.63	141	Lenacil	9.14	228	Sulfoxaflor	8.41
55	Cymoxanil	4.37	142	Linuron	11.46	229	Tebuconazole	7.62
56	Cypermethrin	18.08	143	Lufenuron	5.42	230	Tebufenozide	17.03

Table 2b. 259 pesticide included in the LC-MS/MS method and limit of quantitation

No	Pesticide	LOQ (µg/kg)	No	Pesticide	LOQ (µg/kg)	No	Pesticide	LOQ (µg/kg)
57	Cyproconazole	20.30	144	Malaoxon	3.04	231	Tebufenpyrad	10.85
58	Cyprodinil	11.92	145	Malathion	4.62	232	Teflubenzuron	14.60
59	Dazomet	5.79	146	Mandipropamid	5.60	233	Tepraloxydim	7.07
60	Deltamethrin	13.44	147	MCPA	3.66	234	Terbutryn	4.39
61	Demeton-s-methyl	22.79	148	Mecarbam	6.03	235	Terbutylazine	18.75
62	Demeton-S-methyl-sulfone	3.02	149	Mepanipyrim	25.22	236	Tetraconazole	5.56
63	Desmedipham	4.21	150	Mepanipyrim-hyroxypropyl	4.51	237	Tetramethrin	5.55
64	Diafenthiuran	5.14	151	Metaflumizone	13.32	238	Thiabendazole	5.22
65	Diazinon	5.70	152	Metalaxyl M	4.32	239	Thiacloprid	3.77
66	Dichlofluanid	16.19	153	Metamitron	9.83	240	Thiamethoxam	3.30
67	Dichlorfos	6.21	154	Methacrifos	19.96	241	Thifensulfuron-methyl	3.82
68	Diclofop -methyl	14.78	155	Methamidophos	11.69	242	Thiobencarb	8.22
69	Dicrotophos	3.36	156	Methidathion	5.18	243	Thiodicarb	4.19
70	Diethofencarb	4.68	157	Methiocarb	5.08	244	Thiophanate-methyl	3.22
71	Difenacozole	6.16	158	Methiocarb-sulfone	4.76	245	Tolclofos-methyl	15.39
72	Diflubenzuran	9.65	159	Methiocarb-sulfoxide	4.22	246	Tolfenpyrad	9.14
73	Dimethenamid	4.11	160	Methomyl	3.96	247	Tolyfluanid	10
74	Dimethoate	5.45	161	Methoxyfenozide	17.45	248	Tralkoxydim	5.42
75	Dimethomorph	15.97	162	Metolachlor-S	5.38	249	Triadimefon	6.18
76	Diniconazole	5.29	163	Metosulam	5.09	250	Triadimenol	20.52
77	Dinocap	18.88	164	Metrafenone	6.66	251	Tri-allate	5.70
78	Dioxacarb	4.31	165	Metribuzin	8.96	252	Triasulfuron	6.05
79	Diphenamid	7.29	166	Mevinphos	10.22	253	Triazophos	2.16
80	Diphenylamine	23.14	167	Molinate	15.43	254	Tribenuron methyl	4.40
81	Diuron	8.64	168	Monocrotophos	5.41	255	Trichlorfon	6.32
82	DMF	4.06	169	Monolinuron	5.83	256	Trifloxystrobin	4.35
83	Dodine	8.95	170	Myclobutanil	5.40	257	Triflumizole	4.44
84	Emamectin	5.29	171	Nicosulfuron	4.38	258	Triflumuron	9.08
85	Emamectin benzoat	17.10	172	Novaluron	15.73	259	Triticonazole	4.06
86	EPN	6.21	173	Nuarimol	7.58			
87	Epoxiconazole	10.54	174	Omethoate	6.96			

The pesticide residues determined in the study were evaluated according to the "Turkish Food Codex (TFC) Communiqué on Maximum Residue Limits of Pesticides Allowed in Foodstuffs". The pesticide residue level in each sample was the average of 3 replicates. It was determined that the pesticide residue levels in 10 sour cherry samples taken from different producers were lower than the LOQ values and no pesticide residues were found.

The researches on pesticide residues in fruits is very limited in Turkey. In the studies carried out in Turkey, the residual values were above Turkish Food Codex limits, while in some studies it was below the acceptable values. Ay et al, (2003; 2007); Ersoy et al. (2011a); Bakırcı et al. (2014); Dinçay and Civelek (2017); Dinçay et al. (2017) and Yakar (2018); found residues above the tolerance values allowed in the Turkish Food Codex. Tunur (2009); Sungur and Tunur (2012); Nalcı et al. (2018) and Tiryaki and Özel (2019); determined the residue levels below the MRLs in the Turkish Food Codex. There is only 1 study about the residue in sour cherry. Ersoy et al. (2011b), carried out a study on residue in sour cherries. As a result of the study, 4,0 µg/kg Acetamiprid in one of 3 cherry samples, in another sample, Chlorpyrifos (forbidden to use) pesticide residue at the level of 5,0 µg/kg was found. The values are below the tolerance values permitted in the Turkish Food Codex.

There are not many studies about pesticide residues in Turkey. It is obvious that such studies should be done. It is known that consumers are increasingly conscious and selective about this issue. At this point, our producers are required to implement plant protection measures by paying attention to these sensitivities of consumers. In addition, in recent years, some of agricultural products have been rejected at customs due to the residual problems. This situation creates negativities for Turkey in terms of international trade. It is very important to carry out such studies frequently in order to eliminate these problems and to increase the awareness of the producers on the subject.

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