MAKÜFEBED

ISSN Online: 1309-2243
http://dergipark.gov.tr/makufebed
https://doi.org/10.29048/makufebed.637515

Mehmet Akif Ersoy Üniversitesi Fen Bilimleri Enstitüsü Dergisi 10(2): 198-209 (2019)
The Journal of Graduate School of Natural and Applied Sciences of Mehmet Akif Ersoy University 10(2): 198-209 (2019)

Araştırma Makalesi / Research Paper

Good Agricultural Practices (GAPs) in 'Hicaznar' Pomegranate (*Punica granatum* L.) Cultivar

Nilda ERSOY

Akdeniz University, Vocational School of Technical Sciences, Antalya

Geliş Tarihi (Received): 24.10.2019, Kabul Tarihi (Accepted): 24.11.2019
Sorumlu Yazar (Corresponding author): nildaersoy@akdeniz.edu.tr
+90 242 3106748 *+90 242 3106717*

ABSTRACT

Good Agricultural Practices (GAPs) includes agricultural techniques which environmentally-conscious, is not harmful to human and animal health, target protection of natural resources, provide traceability and food security. With these kinds of production techniques, it is aimed at agricultural production which is socially viable, economically profitable and sustainable. In this experiment Hicaznar pomegranate cultivar grown by Good Agricultural Practices (GAPs) in the Serik District Belkis Locality of Antalya were investigated. In addition to irrigation water, soil analysis, analyzes of pesticide residues in fruits were carried out. According to the results of the analysis irrigation water and soil characteristics were found suitable. A total of 506 pesticide active substances were analyzed in LC-MS / MS and 113 pesticide active substances in GC-MS in fruit extracts. In this research carried out in 2016 and 2017, samples of both years were not found to be detectable to the tolerance values of Turkish Food Codex (TFC).

Keywords: Pomegranate, Good Agricultural Practices, pesticides, residue

Hicaznar Nar (*Punica granatum* L.) Çeşidinde İyi Tarım Uygulamaları (İTU)

ÖZ

İyi Tarım Uygulamaları (İTU) çevre bilincine sahip, insan ve hayvan sağlığına zararlı olmayan, doğal kaynakların korunmasını hedefleyen, izlenebilirlik ve gıda güvenliği sağlayan tarım tekniklerini kapsamaktadır. Bu tarz üretim teknikleri ile sosyal olarak yaşanabilir, ekonomik açıdan karlı ve sürdürülebilir bir tarımsal üretim amaçlanmaktadır. Bu araştırmada, Antalya'nın Serik ilçesi Belkıs Yöresi'nde İyi Tarım Uygulamaları çerçevesinde yetiştirilen Hicaznar nar çeşidi yer almaktadır. Sulama suyu ve toprak analizlerine ek olarak, nar meyvelerindeki pestisit kalıntı analizleri yapılmıştır. Analiz sonuçlarına göre sulama suyu ve toprak özellikleri uygun bulunmuştur. Meyve ekstraktlarında, toplam 506 pestisit etken maddesi LC-MS / MS ile 113 etken madde ise GC-MS'le analiz edilmiştir. 2016 ve 2017 yıllarında yapılan bu araştırmada, her iki yılın örneklerinin, Türk Gıda Kodeksinin (TFC) tolerans değerlerine göre pestisit içermediği tespit edilmiştir.

Anahtar Kelimeler: Nar, Iyi Tarim Uygulamalari, pestisit, kalıntı

INTRODUCTION

Pomegranate is widely recognised as a super food. Especially, pomegranate is one of the important fruit species in human nutrition and health due to it contains phenolic compounds, organic acids and other antioxidant compounds (Gündoğdu and Yılmaz, 2012). Nowadays its importance is increasing due to its rich nutritional value and its positive effects on human health (Aviram et al., 2000; Aviram and Dornfeld 2001; Rosenblat, et al., 2006; Basu and Penugonda, 2009; Utture et al., 2012). It is one of the world's most ancient foods, and it is a

Nilda ERSOY, https://orcid.org/0000-0001-9586-5450

symbol of fertility. Turkey is one of pomegranates homeland. Mainly in the Mediterranean, Aegean and Southeastern Anatolia Regions are produced as regular plantations. In other regions it is grown as border trees or single trees. Turkey pomegranate production is 537.847 tons in 2018 (TÜİK 2019). With this production value, it ranks 4th in the world after Iran, India and China. As a result, it is an exotic fruit which has an important place in fruit growing in our country.

Nowadays, there have been many problems threatening human health, this has increased the interest in food safety and has led to the development of good agricultural practices. The purposes of the GAPs are to make agricultural production that is not harmful to environment, human and animal health, to provide protection of natural resources, traceability and sustainability in agriculture and food security. GAP which is started by FAO is collected under 11 titles. These are soil, water, herbal and forage crops production, crop protection, animal production, animal welfare, harvest, cultivation in the farm and storage, energy and waste management, human welfare, health and security and wildlife and environment (Ersoy et al., 2017; Ersoy et al., 2018).

In Turkey, the procedures and principles of Good Agricultural Practices (GAPs) are defined by the regulation of GAPs published in the official gazette dated 07.12.2010 and no. 27778. Internationally recognized the criteria of GAPs are practiced within the frame of certification system and conformity criteria of GLOBALGAP, which is a special system implemented on an international scale. Nowadays, it is more important the security of food products than their prices for some consumers in the market of developed countries (Ersoy et al., 2017).

In this research, it is aimed to determine the presence of pesticide residues in fruit samples obtained from Hicaznar pomegranate cultivar in the Serik District Belkis Locality of Antalya with a certificate of good agricultural practices (GAPs).

MATERIAL AND METHODS

The experiment was carried out in a pomegranate garden which is "Hicaznar" cultivar established in the Serik District Belkis Locality of Antalya, Turkey. The experiment was set up to have 3 replications and 15 trees in each replicate, according to the design of randomized block design. Necessary samples were taken at the harvest time from the trees included in the experiment and analyzes were carried out. The plant protection program applied to pomegranate trees was given in Table 1 and the reports of soil and water analysis of the trial site were also given in Tables 2- 4.

Table 1. Chemical control program of applied in pomegranate garden

Application dates	Trade name and Active substance	Application reason	Application dosage	Standby time	Application method
15.07.2016	HARBOR 150 g/l Propiconazole +150 g/l Difenoconazole	Brown spot disease (*Alternaria alternate (Fr.)* Keissl.)	50 ml/100 lt	7 days	Atomiser
15.07.2017	HARBOR 150 g/l Propiconazole +150 g/l Difenoconazole	Brown spot disease (*Alternaria alternate (Fr.)* Keissl.)	50 ml/100 lt	7 days	Atomiser
17.09.2016	CALYPSO OD, Thiacloprid	Pomegranate aphid (*Aphis punicae* Passerini)	40 ml/100lt	7 days	Atomiser
17.09.2017	CALYPSO OD, Thiacloprid	Pomegranate aphid (*Aphis punicae* Passerini)	40 ml/100lt	7 days	Atomiser
17.09.2016	DELPHIN, Bacillus thuringiensis 3200 BTU	Carob moth (*Ectomyelois ceratoniae* Zell.)	100 gr/100 lt	0 days	Atomiser
17.09.2017	DELPHIN, Bacillus thuringiensis 3200 BTU	Carob moth (*Ectomyelois ceratoniae* Zell.)	100 gr/100 lt	0 days	Atomiser

Table 2. Soil analysis report of pomegranate garden

Analysis Parameters	Unit	Methods	Results	Assessment
Ph	-	Richards (1954), Tüzüner (1990)	6,88	Neutral
Lime	(%)	Çağlar (1949), Tüzüner (1990)	25,08	Too high limy
Salt (conductivity)	(%)	Richards (1954), Tüzüner (1990)	0,046	Salt free
Saturation	(%)	Richards (1954), Tüzüner (1990)	83,00	Clayey
*Organic Substance	(%)	Richards (1954)	5,81	Highy
P (useful for plant)	(kg P_2O_5/decar)	Tüzüner (1990)	61,83	Too much
K (useful for plant)	(kg P_2O_5/decar)	Tüzüner (1990)	75,52	Much

Table 3. Chemical analysis results of irrigation water used for pomegranate garden

Analysis Parameters	Unit	Method/Instrument	Results (2017)	Results (2016)
pH	-	Richards 1954	7,58	
EC	dS/cm	Richards 1954	0,04	Less salty
CO_3^{2-}	me/l	Richards 1954	0,20	
HCO_3^-	me/l	Richards 1954	4,80	
Cl^-	me/l	Richards 1954	0,70	Good
SO_4^{2-}	me/l	Eltan 1998	0,20	
Na^+	me/l	TS 4530/ICP OES	1,62	
K^+	me/l	TS 4530/ICP OES	0,08	
Ca^{2+}	me/l	TS 2879/ICP OES	2,56	
Mg^{2+}	me/l	TS 2879/ICP OES	3,18	
B	ppm	Richards 1954	0,35	Class 1
Sodium Adsorption Ratio (SAR) me/L	me/l	TS 7739/Calculation	0,96	Low sodium
Residual Sodium Carbonate (RSC) me/L	me/l	TS 7739/Calculation	-0,74	Used for irrigation
Water Class			T1A1	T1A1

Table 4. Microbiological analysis results of irrigation water used for mandarin garden

Analysis	Result/Unit	Analysis Method
*Intestinal Enterococci	Not detected kob/100 ml	ISO 7899-2
*Escherichla coli (E.coli)	Not detected kob/100 ml	TS EN ISO 9308-1
*Coliform Bacteria	Not detected kob/100 ml	TS EN ISO 9308-1

In the experiment, pesticides given in Tables 5 and 6 are searched in the examples of pomegranate fruit, which are the materials. All extraction studies and residue analysis of the examples made in Proanaliz Food Control Laboratory.

Table 5. Active substances examined in pomegranate fruit examples on GC-MSD device

No	Analit	Detection Limit µg/kg	No	Analit	Detection Limit µg/kg
1	1-Naphthyl acetamide(0.010)	0.010	254	Fosthiazate	0.010
2	2,4-D (0.010)	0.010	255	Fuberidazole	0.010
3	3,4,5 Trimethacarb (0.010)	0.010	256	Furalaxyl	0.010
4	Abamectin (0.010)	0.010	257	Furathiocarb	0.010
5	Acephate (0.010)	0.010	258	Halfenprox	0.010
6	Acequinocyl (0.010)	0.010	259	Halosulfuron Methyl	0.010
7	Acetamiprid (0.010)	0.010	260	Haloxyfop-2-Ethoxy-Ethyl	0.010
8	Acetochlor (0.010)	0.010	261	Heptanafos	0.010

9	Acibenzolar-S-Methyl (0.010)	0.010	262	Hexaconazole	0.010
10	Aclonifen (0.010)	0.010	263	Hexaflumuron	0.010
11	Acrinathrin (0.010)	0.010	264	Hexazinone	0.010
12	Alachlor	0.010	265	Hexythiazox	0.010
13	Aldicarb	0.010	266	Imazalil	0.010
14	Aldicarb-Sulfone	0.010	267	Imazamox	0.010
15	Aldicarb-Sulfoxide	0.010	268	Imazapic	0.010
16	Allethrin	0.010	269	Imazapyr	0.010
17	Ametoctradin	0.010	270	Imazaquin	0.010
18	Ametryn	0.010	271	Imazethapyr	0.010
19	Amidosulfuron	0.010	272	Imazosulfuron	0.010
20	Amisulbrom	0.010	273	Imibenconazole	0.010
21	Amitraz	0.010	274	Imidachloprid	0.010
22	Amitrole	0.010	275	Indoxacarb Sum	0.010
23	Anilazine	0.010	276	Iodosulfuron methyl sodium	0.010
24	Anilofos	0.010	277	Ioxynil	0.010
25	Aramite	0.010	278	Ipconazole	0.010
26	Asulam	0.010	279	Iprobenfos	0.010
27	Atrazine	0.010	280	Iprodione	0.010
28	Azaconazole	0.010	281	Iprovalicarb	0.010
29	Azamethiphos	0.010	282	Isazofos	0.010
30	Azimsulfuron	0.010	283	Isocarbofos	0.010
31	Azinphos-Ethyl	0.010	284	Isoprocarb	0.010
32	Azinphos-Methyl	0.010	285	Isoproturon	0.010
33	Aziprotryne	0.010	286	Isoxaben	0.010
34	Azocyclotin	0.010	287	Isoxadifen Ethyl	0.010
35	Azoxystrobin	0.010	288	Isoxaflutole	0.010
36	Barban	0.010	289	Isoxathion	0.010
37	Beflubutamid	0.010	290	Kinetin	0.010
38	Benalaxyl	0.010	291	Kresoxim-methyl	0.010
39	Bendiocarb	0.010	292	Lenacil	0.010
40	Benfuracarb	0.010	293	Linuron	0.010
41	Benomyl	0.010	294	Lufenuron	0.010
42	Bensulfuron methyl	0.010	295	Malaoxon	0.010
43	Bentazone	0.010	296	Malathion	0.010
44	Benthiovalicarb Isopropyl	0.010	297	Maleic Hydrazide	0.010
45	Benzoximate	0.010	298	Mandipropamide	0.010
46	Bifenox	0.010	299	MCPA	0.010
47	Bifentrin	0.010	300	Mecarbam	0.010
48	Binapacryl	0.010	301	Mecoprop (MCPP)	0.010
49	Bioresmethrin	0.010	302	Mecoprop-P (MCPP-P)	0.010
50	Bispyribac	0.010	303	Mepanipyrim	0.010
51	Bitertanol	0.010	304	Mephosfolan	0.010
52	Boscalid	0.010	305	Mepronil	0.010
53	Bromacil	0.010	306	Meptyldinocap	0.010
54	Bromophos methyl	0.010	307	Mesosulfuron Methyl	0.010
55	Bromophos-Ethyl	0.010	308	Mesotrione	0.010
56	Bromoxynl	0.010	309	Metaflumizone	0.010
57	Bromuconazole	0.010	310	Metalaxyl	0.010
58	Bupirimate	0.010	311	Metalaxyl M	0.010
59	Buprofezine	0.010	312	Metamitron	0.010
60	Butafenacil	0.010	313	Metazachlor	0.010
61	Butocarboxim	0.010	314	Metconazole	0.010
62	Butocarboxim-sulfone	0.010	315	Methabenzthiazuron	0.010
63	Butocarboxim-sulfoxide	0.010	316	Methacrifos	0.010
64	Butoxycarboxim	0.010	317	Methamidophos	0.010

65	Butralin	0.010	318	Methidathion	0.010
66	Buturon	0.010	319	Methiocarb	0.010
67	Butylate	0.010	320	Methiocarb sulfone	0.010
68	Cadusafos	0.010	321	Methiocarb sulfoxide	0.010
69	Campheclor	0.010	322	Methiocarb Sum	0.010
70	Campheclor-methyl	0.010	323	Methomyl	0.010
71	Campheclor-oxon	0.010	324	Methomyl oxime	0.010
72	Campheclor-oxon-sulfone	0.010	325	Methomyl Sulfone	0.010
73	Campheclor-oxon-sulfoxide	0.010	326	Methoxyfenozide	0.010
74	Campheclor-sulfone	0.010	327	Metobromuron	0.010
75	Campheclor-sulfoxide	0.010	328	Metolachlor	0.010
76	Carbaryl	0.010	329	Metolcarb	0.010
77	Carbendazim	0.010	330	Metosulam	0.010
78	Carbofuran	0.010	331	Metoxuron	0.010
79	Carbosulfan	0.010	332	Metribuzin	0.010
80	Carboxin	0.010	333	Metrofenone	0.010
81	Carfentrazone-Ethyl	0.010	334	Metsulfuron-Methyl	0.010
82	Chlorantraniliprole	0.010	335	Mevinphos	0.010
83	Chlorbromuron	0.010	336	Milbemectin A3	0.010
84	Chlorbufam	0.010	337	Milbemectin A4	0.010
85	Chlorfenvinphos	0.010	338	Molinate	0.010
86	Chlorfluazuron	0.010	339	Monocrotophos	0.010
87	Chloridazon	0.010	340	Monolinuron	0.010
88	Chlormequat chloride	0.010	341	Monuron	0.010
89	Chlorotoluron	0.010	342	Myclobutanil	0.010
90	Chloroxuron	0.010	343	Naled (Dibrom)	0.010
91	Chlorpropham	0.010	344	Naphthalene Acetamide (NAD)	0.010
92	Chlorpyrifos (0.04)	0.004	345	Napropamide	0.010
93	Chlorpyrifos-Methyl	0.010	346	Napthol-1	0.010
94	Chlorsulfuron	0.010	347	Neburon	0.010
95	Chlortal-dimethyl	0.010	348	Nicosulfuron	0.010
96	Chlorthiamid	0.010	349	Nitenpyram	0.010
97	Chromafenozide	0.010	350	Norfluazuron	0.010
98	Cinidon-ethyl	0.010	351	Novaluron	0.010
99	Clethodim	0.010	352	Nuarimol	0.010
100	Clethodim Iminsulfone	0.010	353	Ofurace	0.010
101	Clethodim Iminsulfoxide	0.010	354	Omethoate	0.010
102	Clethodim Sulfoxide	0.010	355	Orthosulfamuron	0.010
103	Climbazole	0.010	356	Oxadiazon	0.010
104	Clodinafop-proparpyl ester	0.010	357	Oxadixyl	0.010
105	Clofentezine	0.010	358	Oxamyl	0.010
106	Clomazone	0.010	359	Oxasulfuron	0.010
107	Cloquintocet-methylhexyl es-ter	0.010	360	Oxycarboxin	0.010
108	Clothianidin	0.010	361	Oxyfluorfen	0.010
109	Coumaphos	0.010	362	Paclobutrazol	0.010
110	Crimidine	0.010	363	Paraoxon Ethyl	0.010
111	Cyanazine	0.010	364	Paraoxon Methyl	0.010
112	Cyanofenphos	0.010	365	Parathion-Ethyl	0.010
113	Cyazofamid	0.010	366	Parathion-Methyl (0.002)	0.002
114	Cyclanilide	0.010	367	Pebulate	0.010
115	Cycloloxydim	0.010	368	Penconazole(0.005)	0.005
116	Cyflufenamid	0.010	369	Pencycuron	0.010
117	Cyhalofop	0.010	370	Pendimethalin	0.010
118	Cyhalofop butyl	0.010	371	Penoxsulam	0.010
119	Cyhalofop diacid	0.010	372	Pethoxamid	0.010

120	Cyhexatin	0.010	373	Phenmedipham	0.010
121	Cymoxanil	0.010	374	Phenothrin	0.010
122	Cyproconazole	0.010	375	Phentoate	0.010
123	Cyprodinil	0.010	376	Phorate	0.010
124	Cyromazine	0.010	377	Phorate Sulfone	0.010
125	Daminozide	0.010	378	Phosalone	0.010
126	Demeton(O+S)	0.010	379	Phosmet	0.010
127	Demeton-S-Methyl	0.010	380	Phosmet oxon	0.010
128	Demeton-S-Methyl-Sulfone	0.010	381	Phosphamidon	0.010
129	Demeton-S-Methyl-Sulfoxide	0.010	382	Phoxim	0.010
130	Desmedipham	0.010	383	Picolinafen	0.010
131	Desmetryn	0.010	384	Picoxystrobin	0.010
132	Diafenthiuron	0.010	385	Pinoxaden	0.010
133	Dialifos	0.010	386	Pirimicarb	0.010
134	Di-Allate	0.010	387	Pirimicarb Desmethyl	0.010
135	Diazinon	0.010	388	Pirimicarb Desmethyl Formamido	0.010
136	Dichlofenthion	0.010	389	Pirimiphos-Ethyl	0.010
137	Dichlofluanid	0.010	390	Pirimiphos-Methyl	0.004
138	Dichlorprop	0.010	391	Prochloraz	0.010
139	Dichlorvos (DDVP)	0.010	392	Profenofos	0.010
140	Diclobutrazol	0.010	393	Profoxydim	0.010
141	Diclofop-Methyl	0.010	394	Profoxydim lithium	0.010
142	Dicloran	0.010	395	Prohexadione calcium	0.010
143	Dicrotophos	0.010	396	Promecarb	0.010
144	Diethofencarb	0.010	397	Promethryn	0.010
145	Difenoconazole	0.010	398	Propachlor	0.010
146	Diflubenzuron	0.010	399	Propanil	0.010
147	Diflufenican	0.010	400	Propaquizafop	0.010
148	Dimefox	0.010	401	Propargite	0.010
149	Dimethachlor	0.010	402	Propazine	0.010
150	Dimethenamid	0.010	403	Propetamphos	0.010
151	Dimethoate	0.010	404	Propham	0.010
152	Dimethomorph	0.010	405	Propiconazole	0.010
153	Dimetilan	0.010	406	Propisochlor	0.010
154	Dimoxystrobin	0.010	407	Propoxur	0.010
155	Diniconazole	0.010	408	Propoxycarbazone sodium	0.010
156	Dinitramine	0.010	409	Propyzamide	0.010
157	Dinocap	0.010	410	Proquinazid	0.010
158	Dinoseb	0.010	411	Prosulfocarb	0.010
159	Dinoterb	0.010	412	Prosulfuron	0.010
160	Dioxacarb	0.010	413	Prothioconazole	0.010
161	Diphenamid	0.010	414	Prothiophos	0.010
162	Dipropetryn	0.010	415	Pymetrozine	0.010
163	Disulfoton	0.010	416	Pyraclostrobin	0.010
164	Disulfoton Sulfone	0.010	417	Pyraflufen	0.010
165	Disulfoton Sulfoxide	0.010	418	Pyraflufen ethyl	0.010
166	Ditalimfos	0.010	419	Pyrasulfotole	0.010
167	Dithianon	0.010	420	Pyrazophos	0.010
168	Diuron	0.010	421	Pyrethrins	0.010
169	DNOC	0.010	422	Pyridaben	0.010
170	Dodine	0.010	423	Pyridaly	0.010
171	E-Fenpyroxymate	0.010	424	Pyridaphenthion	0.010
172	Emamectin Benzoate	0.010	425	Pyridate	0.010
173	Epichlorohydrin	0.010	426	Pyrifenox	0.010
174	EPN	0.010	427	Pyrimethanil	0.010
175	Epoxiconazole	0.010	428	Pyriproxyfen	0.010

176	EPTC	0.010	429	Quaizalofop_P_Ethyl	0.010
177	Etaconazole	0.010	430	Quinalphos	0.010
178	Ethametsulfuron Methyl	0.010	431	Quinclorac	0.010
179	Ethiofencarb	0.010	432	Quinmerac	0.010
180	Ethiofencarb-sulfone	0.010	433	Quinoxyfen	0.010
181	Ethiofencarb-sulfoxide	0.010	434	Resmethrin	0.010
182	Ethion	0.010	435	Rimsulfuron	0.010
183	Ethiprole	0.010	436	Rotenone	0.010
184	Ethirimol	0.010	437	Sethoxydim	0.010
185	Ethofenprox	0.010	438	Silthiofam	0.010
186	Ethofumesate	0.010	439	Simazine	0.010
187	Ethoprophos	0.010	440	Spinetoram	0.010
188	Ethoxyquin	0.010	441	Spinosad	0.010
189	Ethoxysulfuron	0.010	442	Spirodiclofen	0.010
190	Ethylene thiourea	0.010	443	Spiromesifen	0.010
191	Etoxazole	0.010	444	Spirotetramat	0.010
192	Etridiazole	0.010	445	Spirotetramat-Enol	0.010
193	Etrimfos	0.010	446	Spirotetramat-Enol-Glucoside	0.010
194	Famoxadone	0.010	447	Spirotetramat-Ketohydroxy	0.010
195	Famphur	0.010	448	Spirotetramat-Monohydroxy	0.010
196	Fenamidone	0.010	449	Spiroxamine	0.010
197	Fenamiphos	0.010	450	Sulcotrione	0.010
198	Fenarimol	0.010	451	Sulfosulfuron	0.010
199	Fenazaquin	0.010	452	Sulfotep	0.010
200	Fenbuconazole	0.010	453	Sulprofos	0.010
201	Fenbutatin oxide	0.010	454	Tebuconazole	0.010
202	Fenhexamid	0.010	455	Tebufenozide	0.010
203	Fenitrothion	0.010	456	Tebufenpyrad	0.010
204	Fenobucarb	0.010	457	Tebupirimfos	0.010
205	Fenoxyaprop-P-ethyl	0.010	458	Teflubenzuron	0.010
206	Fenoxycarb	0.010	459	Tembotrione	0.010
207	Fenpiclonil	0.010	460	Temephos	0.010
208	Fenpropathrin	0.010	461	TEPP(O.O-TEPP)	0.010
209	Fenpropidin	0.010	462	Tepraloxydim	0.010
210	Fenpropimorph	0.010	463	Terbufos	0.010
211	Fensulfothion	0.010	464	Terbumeton	0.010
212	Fenthion	0.010	465	Terbuthylazine	0.010
213	Fenthion Oxon	0.010	466	Terbutryn	0.010
214	Fenthion Oxon Sulfone	0.010	467	Tetramethrin	0.010
215	Fenthion Oxon Sulfoxide	0.010	468	Tetraconazole	0.010
216	Fenthion-Sulfone	0.010	469	Thiabendazole	0.010
217	Fenthion-Sulfoxide	0.010	470	Thiacloprid	0.010
218	Fentin acetate	0.010	471	Thiamethoxam	0.010
219	Fentin Hydroxide	0.010	472	Thidiazuron	0.010
220	Fipronil	0.010	473	Thifensulfuron-methyl	0.010
221	Flamprop-M-Isopropyl	0.010	474	Thiobencarb	0.010
222	Flazasulfuron	0.010	475	Thiodicarb	0.010
223	Flonicamid	0.010	476	Thiofanox	0.010
224	Florasulam	0.010	477	Thiofanox Sulfone	0.010
225	Fluazifop-p-butyl	0.010	478	Thiofanox Sulfoxide	0.010
226	Fluazinam	0.010	479	Thiophanate-methyl	0.010
227	Flubendiamide	0.010	480	Tolclofos-Methyl	0.010
228	Flubenzimine	0.010	481	Tolfenpyrad	0.010
229	Flucycloxuron	0.010	482	Topramezone	0.010
230	Flucythrinate	0.010	483	Tralkoxydim	0.010
231	Fludioxonil	0.010	484	Triadimefon	0.010

232	Flufenacet	0.010	485	Triadimenol	0.010
233	Flufenoxuron	0.010	486	Tri-allate	0.010
234	Flumioxazine	0.010	487	Triasulfuron	0.010
235	Fluometuron	0.010	488	Triazophos	0.010
236	Fluopicolide	0.010	489	Tribenuron-Methyl	0.010
237	Fluopyram	0.010	490	Trichlorfon	0.010
238	Fluorochloridone	0.010	491	Trichloronat	0.010
239	Fluoroglycofen Ethyl	0.010	492	Triclopyr	0.010
240	Fluoxastrobin	0.010	493	Tricyclazole	0.010
241	Flupyrsulfuron Methyl	0.010	494	Tridemorph	0.010
242	Fluquinconazole	0.010	495	Triethyl Phosphate	0.010
243	Fluroxypyr	0.010	496	Trifloxystrobin	0.010
244	Flurtamone	0.010	497	Triflumizole	0.010
245	Flusilazole	0.010	498	Triflumuron	0.010
246	Flutolanil	0.010	499	Triflusulfuron Methyl	0.010
247	Fluxapyroxad	0.010	500	Triforine	0.010
248	Fomesafen	0.010	501	Trinexapac Ethyl	0.010
249	Fonofos	0.010	502	Triticonazole	0.010
250	Foramsulfuron	0.010	503	Tritosulfuron	0.010
251	Forchlorfenuron	0.010	504	Uniconazole	0.010
252	Formetanate	0.010	505	Vamidothion	0.010
253	Formetanate hydrochloride	0.010	506	Zoxamide	0.010

Table 6. Active substances examined in pomegranate fruit examples on GC-MSD device

No	Analit	Detection Limit µg/kg	No	Analit	Detection Limit µg/kg
1	2,4-5T	0.020	58	Endosulfan, Beta	0.002
2	2-Chloranilline	0.015	59	Endrin	0.015
3	2-Phenyl phenol	0.015	60	Ethalfluralin	0.015
4	3-Chloranilline	0.015	61	Fenchlorphos	0.015
5	4.4 Dichlorobenzophenone	0.020	62	Fenson	0.015
6	4-Chloranilline	0.015	63	Fenvelarate & Esfenvelarate	0.010
7	Aldrin (HHDN)	0.015	64	Fluchloralin	0.015
8	Alpha cypermethrin	0.005	65	Fluotrimazole	0.015
9	Aminocarp	0.015	66	Flurprimidol	0.015
10	Benfluralin	0.015	67	Flutriafol	0.015
11	BHC	0.015	68	Fluvalnate, tau	0.010
12	Bifenazate	0.015	69	Folpet	0.015
13	Biphenyl	0.015	70	Formothion	0.015
14	Bromocyclen	0.015	71	Haloxyfop R Methyl	0.015
15	Bromopropylate	0.010	72	HCL Alpha	0.020
16	Captafol	0.015	73	HCL Beta	0.020
17	Captan	0.010	74	HCL Delta	0.020
18	Carbofuran-3 hydroxy	0.010	75	HCL Gamma	0.020
19	Carbophenothion	0.015	76	Heptachlor	0.015
20	Chlorbenside	0.015	77	Heptachlor EE Cis Isomer	0.015
21	Chlordane-Cis Alpha	0.015	78	Heptachlor EE Trans Isomer	0.015
22	Chlordane Trans Gamma	0.015	79	Hexachlorobenzene	0.020
23	Chlordecone	0.015	80	Iodofenphos	0.015
24	Chlorfenapyr	0.015	81	Isodrin	0.015
25	Chlorfenson	0.020	82	Isofenphos	0.015
26	Chlorobenzilate	0.020	83	Lactofen	0.015
27	Chloroneb	0.015	84	Leptophos	0.015
28	Chlorothalonil	0.020	85	Mefenpyr Diethyl	0.015
29	Chlorthion	0.015	86	Methoprene	0.015

30	Chlozolinate	0.015	87	Methoxychlor	0.020
31	Cyanaphos	0.015	88	Mirex	0.015
32	Cycloate	0.020	89	Nitrothal-isopropyl	0.020
33	Cyfluthrin	0.015	90	Nitrapyrin	0.015
34	Cyflutrin-beta	0.015	91	Nitrofen	0.020
35	Cyhalothrin, Lambda	0.010	92	Oxadargyl	0.015
36	Cypermethrin	0.010	93	Pentachloroaniline	0.015
37	Dazomet	0.020	94	Permethrin	0.010
38	DDD-2.4'	0.020	95	Perhane	0.015
39	DDD-4.4'	0.020	96	Procymidone	0.020
40	DDE-2.4'	0.020	97	Profuralin	0.015
41	DDE-4.4'	0.020	98	Propamocarb	0.015
42	DDT-2.4'	0.020	99	Qunomethionate	0.005
43	DDT-4.4'	0.020	100	Quintozene	0.015
44	Deltamethrin	0.010	101	S-Metolachlor	0.015
45	Dicamba	0.015	102	Tecnazene	0.020
46	Dichlobenil	0.015	103	Tefluthrin	0.015
47	Dicofol	0.010	104	Terbacil	0.015
48	Dieldrin	0.015	105	Tetrachlovinphos	0.015
49	Diethatyl Ethyl	0.015	106	Tetradifon	0.015
50	Dimethypin	0.015	107	Tetrasul	0.020
51	Dinobuton	0.015	108	Thiometon	0.015
52	Dinoseb Asetate	0.015	109	Tolyfuanid	0.020
53	Dioxathion	0.015	110	Transfuthrin	0.015
54	Diphenylamine	0.020	111	Tributtyl Phosphate	0.015
55	Dihenylmercury	0.015	112	Trifuralin	0.010
56	Endosulfan-sulfate	0.002	113	Vinclozolin	0.020
57	Endosulfan, Alpha	0.002			

All the solvents and chemicals (water, acetonitrile, methanol, formic acid, acetic acid and ammonium formate) used as mobile phases in example extractions are chosen in accordance to a profound quality. Pesticide standards are prepared at least a 90% rate of purity. Extractions and clearance of the examples are generalized in accordance with AOAC (International Official Methods of Analysis) methods (Lehotay, 2007).

Examples’ Preparation for Analysis

15 g examples were homogenized in a mechanical shredder. Other similars of the same example were put into same processes separately. Example amounts that put into extraction were taken from these homogenised examples after weighing.

Extraction of Examples

Whole examples were homogenised with steel blenders by shredding and 5 g of analyse examples from the main example were weighed and mixed with 10ml’s of water and 15ml’s of acetonitrile with 1% acetic acid and strongly shaked for 1 minute. Afterwards, 6 g of waterless magnesium sulfate ($MgSO_4$) and 1.5 g of Sodium Acetate ($C_2H_3NaO_2.3H_2O$) is added into falcon tubes and after being shaked for 1 minute, centrifugated for 5 minutes at 4000 rpm rate. As the next step, 8 ml of examples from the previous examples’ high phases were collected for the cleaning process and transported into 15 ml falcon tubes and mixed with 1.2 g of waterless $MgSO_4$ and 0.4 g of PSA and centrifuged for 5 minutes at 4000 rpm rate, once again. Later, the high phase was transported into viales and kept in a freezer until the device evaluations. As the last injections into LC-MS/MS and GC-MS/MS devices were conducted and residue rates were determined. Chromatographical conditions of LC-MS/MS and GC-MS/MS devices are explained on Table 7 and Table 8 in detail.

Table 7. Chromatographic Working Conditions of LC-MS/MS

LC-MS/MS	Agilent 6420			
Mobile Phase A	5 mM Amonium Formate&Water + Acetonitrile			
Mobile Phase A	Pure methanol			
Column	Poroshell 120 SB-C18 (3.0 x 100 mm 2.7 Micron)			
Injection Volume	10 µl			
Flow Rate	0.6 ml/min			
MS Gas Temperature	300°C			
Sheat Gas Temperature	350°C			
The Column Oven	35°C			
Pump Gradient Program	Time	Mobile phase A %	Mobile phase B %	Flow rate ml/min
	0:00	80	20	0.6
	0:00	80	20	0.6
	0:20	80	20	0.6
	1:50	30	70	0.6
	6:00	5	95	0.6
	7:50	5	95	0.6
	7:60	80	20	0.6
	10:00	80	20	0.6

Table 8. Chromatographic Working Conditions of GC/MS

GC-MS		Agilent 5975	
Carrier gases		Helium	
Column		HP-5MS 30 m × 250 µm × 250 µm × 0.25 µm	
Injection Volume		5 µl	
Flow Rate		2.4 ml/min	
Duration of Injection		18.5 min	
MS Gas Temperature		300°C	
Sheat Gas Temperature		350°C	
The Column Oven		35°C	
Inlet temperature program			
Start	Rate of increase (°C/min)	Temperature (°C)	Retention Time (RT) (min)
1	0	55	0.21
2	600	325	18.5
The Column Oven temperature program			
Start	Rate of increase (°C/min)	Temperature (°C)	Retention Time (RT) (min)
1	0	50	0
2	50	150	0
3	20	230	1
4	8	290	3
5	0	290	18.5

RESULTS AND DISCUSSION

Residue quantities obtained from the research were evaluated according to Turkish Food Codex (TFC) Regulation on Maximum Residue Limits of Pesticides (Turkish Official Gazette No 21.01.2011-27822; Notification No: 2011/2). The TFC residue limits of each pesticide sample are indicated separately in the tables presented.

In residue limits determined by using high-precision analytical instruments such as GC-MS and LC-MS/MS, in pomegranate fruit samples analyses of total 506 pesticide active ingredients were made in LC-MS/MS instrument and 113 pesticide active ingredients in GC-MS instrument. In this research carried out between 2016 and 2017, detectable levels of the residues were not found in the samples of these two years.

Mohapatra (2014) researched on the residue dynamics of chlorpyrifos and cypermethrin in/on pomegranate and soil was carried out by conducting supervised field trials as per good agricultural practices. The limit of quantification (LOQ) of chlorpyrifos and cypermethrin were 0.01 and 0.05 mg kg−1, respectively. Residues of the insecticides remained on the fruit surface and movement to the edible part (aril) was not observed.

Mohapatra et al. (2019) expressed that neonicotinoid insecticides such as imidacloprid, indoxacarb and thiamethoxam are widely used for control of a large number of insect pests of pomegranate crop. The researchers evaluated the residual levels of these pesticides in pomegranate for 2 years. They found that the maximum residue levels of imidacloprid on pomegranate was less than its MRL of 1 mg/kg, so the pre-harvest interval (PHI) required was 1 day only. For indoxacarb, 31–42 days PHI was needed for the residues to reduce to its MRL of 0.02 mg/kg. The PHI of thiamethoxam was 46–77 days, the time required for its residues to reduce to its MRL of 0.01 mg/kg. Licensed pesticides may be used in good agricultural practices but they should be administered at the right time and dose.

Shafi et al. (2014) conducted research on the identification of residues of some pesticides in fruits collected from various markets in Lahore. They collected apple, banana, guava, melon, orange, papaya, pomegranate and strawberry fruit samples from various markets. Pesticide residues in pomegranate fruit to be mg/kg were 0.129 for Cypermethrin, 0.105 for Bifenthrin, 0.052 for Carbofuran, 0.045 for λ-Cyhalothrin and 0.035 for Chlorpyrifos. Another researcher, Kolekar et al. (2011), collected 179 exportable pomegranate fruit samples during 2007 to 2009 from various districts in Maharashtra and Karnataka States of India too. All samples were monitored for 86 pesticide residues which included pesticides from diversified chemical classes including organochlorine, organophosphates, carbamates, synthetic pyrethroids, neonicotinoids, triazines, triazoles, natural product derivatives and others using established and validated methods. Overall 35.55% of the total analyzed samples contained no detectable residues, 44.44% samples contained detectable residues while 20.0% samples contained residues that exceeded the maximum residue limits (MRLs) set by the European Union. The majority of the samples contained pesticide residues from the organochlorine, organophosphates, neonicotinoids and triazole group of pesticides. The results of the current study showed that no restricted or banned pesticides such as DDT, HCH and their isomers were found in any of the samples analyzed. The violative percentage for the samples analyzed during 2007-2008 was 3.08% which increased to 29.56% in the year 2008-2009 indicating increased use of pesticides to control pests and diseases on pomegranate. The violative percentage increased dramatically due to the presence of fungicide residues in 61.11% samples of the non-conformed samples. Bakırcı et al. (2014) investigated pesticide residues in fruits and vegetables from the Aegean region of Turkey and they found that all pomegranate, cauliflower and cabbage samples were pesticides-free. In a similar study, we conducted previously, no pesticide residues were found in 5 pomegranate fruit samples collected from Konya region (Ersoy et al., 2011).

Utture et al. (2012) expressed that buprofezin, dimethoate and imidacloprid could be used safely. Our research results showed that thiacloprid, propiconazole and difenoconazole could not effect fruits negatively and we did not find any pesticide residues (see Table 1).

As it can be understood from the above mentioned research examples, some negative situations, which may pose a risk to human health, may be encountered. Residue analysis have an important place in Good Agricultural Practices (GAPs). It depends on having made residue analysis in accredited laboratories and not including any residues to get the certificate of GAPs for a product. It can be said that sensitivity to world food security and the importance of good agricultural production techniques is growing gradually. This situation shows rapid progress of especially export oriented agricultural production activities with regard to the GAPs of Turkey in recent years. It is considered useful to improve a national strategy to expand this production technique.

REFERENCES

Aviram, M., Dornfeld, L. (2001). Pomegranate juice consumption inhibits serumangiotensin converting enzyme activity and reduces systolic blood pressure.Atherosclerosis, 158(1), 195–198.

Aviram, M., Dornfeld, L., Rosenblat, M., Volkova, N., Kaplan, M., Coleman, R., (2000). Pomegranate juice consumption reduces oxidative stress, atherogenicmodifications to LDL, and platelet aggregation: Studies in humans and inatherosclerotic apolipoprotein E-deficient mice.The American Journal of Clinical Nutrition, 71(5), 1062–1076.

Bakırcı, G.T., Acay, D.B.Y., Bakırcı, F., Otles, S. (2014). Pesticide residues in fruits and vegetables from the Aegean region, Turkey. Food Chemistry, 160: 379-392, DOI: 10.1016/j.foodchem.2014.02.051.

Basu, A., Penugonda, K. (2009). Pomegranate juice: A heart-healthy fruit juice.Nutrition Reviews, 67, 49–56.

Çağlar, K. Ö. (1949). Toprak Bilgisi A. Ü. Ziraat Yayınları, No; 10. Ankara.

Eltan, E. (1998). Köy Hizmetleri Gn. Müdürlüğü İçme ve Sulama Suyu Analiz Yöntemleri Yayın No: 19

Ersoy, N., Tatlı, Ö., Özcan, S., Evcil, E., Coşkun, L.Ş., Erdoğan, E. (2011). Bazı Tropikal ve Suptropikal Meyve Türlerinde Pestisit Kalıntıları. Selçuk Tarım ve Gıda Bilimleri Dergisi, 25 (2):81-88.

Ersoy, N., Yılmaz, S., Gümüş, E. (2017). The Importance of Good Agricultural Practices in EU Membership Process, Mediterranean Agricultural Sciences, vol.11, no.1, pp.1-16, 2017.

Ersoy, N., Tekinarslan, O., Akcay Özgür, E., Göktaş, U. (2018). Determination of Pesticide Residues in Apricot (Prunus armeniaca L.) Grown at Good Agricultural Practices (GAPs) by LC-MS/MS and GC-MS. Erwerbs-Obstbau 60(1–3), DOI: 10.1007/s10341-018-0383-9

Kolekar, V.S., Wakure, D.D., Raut, P.N., Utture, S.C. (2011). Monitoring of Pesticide Residues in Exportable Pomegranate Fruits. Acta Hortic. 890, 547-555, DOI: 10.17660/ActaHortic.2011.890.77

Lehotay, S. J. (2007). Determination of pesticide residues in foods by acetonitrile extraction and partitioning with magnesium sulfate: collaborative study. Journal of AOAC International, 2007 Mar-Apr; 90(2):485-520.

Mohapatra, S. (2014). Residue dynamics of chlorpyrifos and cypermethrin in/on pomegranate (*Punica granatum* L.) fruits and soil. International Journal of Environmental Analytical Chemistry 94(14-15):1394-1406

Mohapatra, S., Siddamallaiah, L., Yogendraiah, N., Udupi, V.R., Raj, D.P., Gadigeppa, S. (2019). Dissipation of neonicotinoid insecticides imidacloprid, indoxacarb and thiamethoxam on pomegranate (Punica granatum L.), Ecotoxicology and Environmental Safety 171:130-137.

Gündoğdu, M., Yılmaz, H. (2012). Organic acid, phenolic profile and antioxidant capacities of pomegranate (Punica granatum L.) cultivars and selected genotypes, 143, 38-42.

Richards, L.A. (1954). Diagnosis and Improvement of Saline Alkali Soils, Agriculture, 160, Handbook 60. US Department of Agriculture, Washington DC.

Rosenblat, M., Hayek, T., Aviram, M. (2006). Anti-oxidative effects of pomegranatejuice (PJ) consumption by diabetic patients on serum and on macrophages.Atherosclerosis, 187(2), 363–371.

Shafi, M., Imran, M., Sarwar, M., Kalsoom, S., Mujahid, H. (2014). A Study of Pesticide Residues in Different Fruits Collected from Different Fruit Markets of Lahore, Punjab. Journal of Agroalimentary Processes and Technologies, 20(4), 298-303.

TÜİK, (2019). https://biruni.tuik.gov.tr/medas/?kn=92&locale=tr

Tüzüner, A. (1990). Toprak ve su analiz laboratuvarları el kitabı. T.C. Tarım ve Köy İşleri Bakanlığı, Köy Hizmetleri Genel Müdürlüğü, s: 61-73, Ankara.

Utture, S.C., Banerjee, K., Kolekar, S.S., Dasgupta, S., Oulkar, D.P., Patil, S.H., Wagh, S.S., Adsule, P.G., Anuse, M.A. (2012). Food safety evaluation of buprofezin, dimethoate and imidacloprid residuesin pomegranate. Food Chemistry, 131:787–795.