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RESEARCH

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Efficacy of some washing solutions for removal of pesticide residues in lettuce

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Abstract

Background: When previous studies were examined, it was reported that a wide variety of pesticide residues were detected in lettuce (Bakırcı et al. in Food Chem 160:379–392, 2014; Balkan and Yılmaz in Food Chem 384:132516, 2022; Qin et al. in Food Res Int 72:161–167, 2015; Selim et al. in Res J Environ Sci 5:248–258, 2011). In addition, pesticide-contaminated lettuce poses a risk to consumers as it is a fresh food that is eaten raw. Therefore, pesticide removal processes must be applied before consumption. Some pesticide removal process, such as storage and heat treatment cannot be applied to lettuce because of unshelled, no long shelf life and consuming as fresh. Different practical methods are needed for the removal of pesticides process. The recommendations of suitable methods for cleaning salad materials in ready-to-eat sector, hotels, restaurants and homes are very important. It is important to reduce pesticide residues in vegetables and fruits that are consumed raw by washing them with non-toxic solutions. These approaches aim to protect public health. The study conducted in 2021 determined the effect of various washing treatments. For this purpose, before the washing trials, QuEChERS method was validated for determination of 7 pesticides in lettuce, by liquid chromatography–tandem mass spectrometry (LC–MS/MS). Lettuce plants were grown in the greenhouse for the study. Lettuces were brought to the laboratory 24 h after pesticides were applied in the greenhouse. The samples were kept in 2 L washing solution for pesticide analysis.

Results: The processing factor values of all washing applications were found below 1. This result shows that the residue level was reduced by washing solutions. Among the washing solution treatments, the rice vinegar washing solution was found to be most effective in reducing the pesticide residues which was due to the high degree in the pesticide degradation. Washing with filtered rice water also exhibited equivalent reduction capability similar to rice vinegar.

Conclusions: The effectiveness of washing solutions was different for boscalid, deltamethrin, fluopcolide, fluopyram, pyrimethanil, pyraclostrobin and sulfoxaflor. However, when the average removal of these pesticides was evaluated, the most effective solutions were rice vinegar, filtered rice water, carbonated water, NaCl + grape vinegar, hot tap water (40 °C), grape vinegar (6% acetic acid), grape vinegar + water, cold tap water (20 °C), lemon juice, baking soda water, grape vinegar (8% acetic acid), filtered mint water and grape vinegar (4% acetic acid), respectively. It was concluded that some of the solutions used in this study can significantly reduce exposure to pesticides for consumers.

Keywords: Food safety, LC–MS/MS, Method validation, Pesticide residue, Reduction, QuEChERS

1 Background

Lettuce, *Lactuca sativa* L. (Asteraceae), is one of the most important leafy vegetables. Lettuce is rich in minerals, such as vitamin A, C, calcium, iron, protein, and carbohydrates [34]. The world's largest lettuce producing countries are China, India, United States of America, Spain, Italy, and Turkey [17]. Lettuce is produced in

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open areas, greenhouses, and low plastic tunnels, especially in places where climatic conditions are suitable.

There are many pests causing damage on lettuce [9]. Pesticides are frequently used to prevent yield losses. Pesticide residues can have a negative impact on human health. These complications appear suddenly in the form of nausea and headaches. Chronically, it can cause neurotoxicity, cytogenetic damage, infertility, endocrine system problems [5], leukemia, non-Hodgkin lymphoma, brain, bone, breast, ovarian, ptate, testicular and liver cancers in the long-term [3, 21].

Due to these concerns, pesticide removal studies have been carried out in many parts of the world. In order to reduce the risk of pesticide residues, various methods, such as food processing, peeling, storage, washing with water and various solutions, use of heat treatments, ozone applications, fenton process, ultrasound application, electric current application and ultrasonic bath application methods were used and promising results were obtained [1, 10, 18, 25].

Lettuce is a fresh food that is eaten raw. Vitamins and minerals can enter the human body directly [20]. While it provides direct benefits to consumers, it poses a risk for consumers in terms of pesticides. Different pesticide residues were detected in lettuce [8, 13, 33].

Approaches which aimed at lowering values of MRL are important for safe food. Therefore, pesticide removal processes must be applied before consumption. Some pesticide removal process such as storage and heat treatment cannot be applied to lettuce because of unshelled, no long shelf life and consuming as fresh. Different practical methods are needed for the removal of pesticides process. The recommendations of suitable methods for cleaning salad materials in ready-to-eat sector, hotels and restaurants are very important.

In this study, cleaning solutions with different biochemical properties that were effective in previous studies on different pesticides and different plants were selected [2, 26, 28]. Thirteen solutions that have no negative effects on human health were used for the removal of pyraclostrobin, pyrimethanil, sulfoxaflor, deltamethrin, fluopyram, boscalid and fluopicolide, which are used extensively in lettuce against to pests.

2 Methods

2.1 Materials, reagents, and analytical standards

Pyraclostrobin, pyrimethanil, sulfoxaflor, deltamethrin, fluopyram, boscalid and fluopicolide, which are widely used in lettuce production in Turkey, were selected in this study. Certified pesticide references (Table 1) with purity between 99.0 and 99.66% were attained from Dr. Ehrenstorfer™ (Augsburg, Germany). Commercial pesticides were purchased from local market. HPLC grade acetonitrile, methanol and acetic acid were taken from Merck (Darmstadt, Germany). Commercial QuEChERS extraction salts packets (6g magnesium sulfate, anhydrous and 1.5g sodium acetate, anhydrous) and 15 mL tubes for dispersive solid-phase (dSPE) clean-up tubes containing 1200 mg of magnesium sulfate and 400 mg of primary secondary amine sorbent were obtained from Restek Corporation (Bellefonte, PA, United States).

2.2 Field trials, pesticide treatments and sampling

Field experiments were conducted in a greenhouse located in Tokat Province (Turkey) in 2021. Curly (*Lactuca sativa* var. crispa) lettuce variety was used. Lettuce seedlings were planted with 40 cm row spacings and 30 cm intra-row spacing. Plot length was 7 m and width were 4 m. The area of each plot was determined as 2 m^2 (2 × 1m) and each plot consisted of 24 plants. The study

Table 1 Some physicochemical properties and Maximum residue limits of the pesticides

Analyte	Purity of certified pesticide reference %	Commercial name	EU-MRL[a]	Group	Phytomobility	Solubility in water (mg L^{-1}) (20 °C)	$LogP_{O/W}$)[b] (pH 7, 20 °C)
Boscalid	99.02	Signum WG	50	Fungicide	Systemic	4.6	2.96
Pyraclostrobin	99.57		2			1.9	3.99
Pyrimethanil	99.57	Luna Tranquility SC 500	20	Fungicide	Non-systemic	110	2.84
Fluopyram	99.66		15			16	3.3
Fluopicolide	99.00	Infinito SC 687,5	9	Fungicide	Systemic	2.8	2.9
Deltamethrin	99.62	Demond EC 2.5	0.5	Insecticide	Non-systemic	0.0002	4.6
Sulfoxaflor	99.23	Breaker™ 240 SC	4	Insecticide	Systemic	568	0.802

[a] EU-MRL: European Union-Maximum Residue Limit (μg kg^{-1}) for lettuce [16]

[b] Octanol-water partition coefficient ($LogP_{O/W}$) of pesticides represents the ratio of the solubility of a compound in octanol (a non-polar solvent) to its water solubility (a polar solvent)" [27]

was carried out in the period of October 2020—January 2021. The lettuces were watered as needed and no fertilization was applied. Lettuces were sprayed with 2 insecticides (Demond EC 2.5 and Breaker 240 SC) and 3 fungicides (Signum WG, Infinito SC 687.5, Luna Tranquility) using knapsack sprayer. Detailed information about the pesticides is presented in Table 1. The recommended doses of pesticides were deltamethrin 100 ml/100 lt water, sulfoxaflor 15 ml/da, boscalid + pyraclostrobin 150 g/da and promocarb + fluopicolide 150 ml/100 lt water and pyrimethanil + fluopyram 100 ml/da. Lettuce was brought to the laboratory 24 hours after the pesticide application. Disposable latex gloves and polyethylene bags were used to prevent contamination during harvesting.

2.3 Washing treatments

All lettuce samples were divided into three analytical portions. The samples collected from 1 plot were analyzed without any washing process and were considered as the control group. Washing solutions were applied separately to samples the other 13 plots. The assayed washing solutions were the following: cold tap water (20 °C), hot tap water (40 °C), lemon juice, filtered mint water, filtered rice water, carbonated water, baking soda water, grape vinegar (4% acetic acid), grape vinegar (6% acetic acid), grape vinegar (8% acetic acid), rice vinegar, 50% grape vinegar + water and NaCl +grape vinegar). Lettuce samples were kept in 2 L washing solution for 10 minutes. Excess water of the washed samples was removed with a salad spinner and kept drying for 30 minutes at 25°C. Dried samples were used for pesticide analysis.

2.4 Sample preparation, extraction, and clean-up

Extraction and clean-up were performed according to the procedures in the QuEChERS (Quick, Easy, Cheap, Effective, Rugged, Safe) (AOAC Official Method 2007.01) [22]. The steps of the procedure are shown in detail in Fig 1.

2.5 Instrumentation

Chromatographic separation was achieved with Shimadzu® Nexera X2. The UPLC separation was performed using a Inertsil (ODS IV) C18 column (2.1 mm × 150 mm, 3 µm particle size). The mobile phase A was a mixture of distilled water and 5 mM ammonium formate, and mobile phase B was a mixture of methanol and 5 mM ammonium formate. The flow rate, injection volume, oven temperature and autosampler temperature were 0.40 mL min^{-1},10 µL, 35 °C and 4 °C, respectively. For the mass spectrometric analysis of Shimadzu® LCMS-8050 was used. The electrospray ionization (ESI) interface was operated positive polarity and its parameters were as follows: capillary voltage 3 kV, extractor voltage 3V, heat block temperature 400 °C, desolvation line (DL) temperature 250 °C, Nitrogen (N_2) as nebulizer gas of 2.9 L min^{-1} and drying gas of 10 L min^{-1}. Collision-induced dissociation (CID) gas was argon (Ar, 99.999%) of 230 kpa with flow rate 0.15 mL min^{-1}. All parameters of instrument were controlled using LabSolution® software (5.97 version) [8].

2.6 Method validation

Analytical method was validated (with LOD, LOQ, linearity, recovery, precision (repeatability and within-laboratory reproducibility and measurement uncertainty), in accordance with the SANTE guidelines [15]. LOD and LOQ determination studies were performed at a single concentration (10 µg mL^{-1}) in 10 repetitions and the standard deviation (SD) and relative standard deviation (RSD%) values of each pesticide were calculated. Three times the calculated standard deviation values were determined as LOD and 10 times the LOQ value for each pesticide. The repeatibility study was performed at two different fortification levels (10 and 50 µg mL^{-1}) 5 times on the same day. The within-laboratory reproducibility study was carried out at two different fortification levels (10 and 50 µg mL^{-1}) at five different times [7]. The expanded measurement uncertainty (U') was calculated for all pesticides, according to Approach 2 (Estimating a generic measurement uncertainty using proficiency test data recommended by SANTE Guideline [8, 15].

2.7 Reduction ratio

After washing applications, pesticide residues were determined as mg kg^{-1}. Pesticide reduction rates were calculated according to Eq. (1) [1, 28].

$$\text{Reduction ratio} = \frac{(C_o - C_1)}{C_o} \times 100 \qquad (1)$$

C_o represents the concentration of pesticide residue in the untreated control lettuce sample, C_1 represents the concentration of residue in lettuce sample washed with various solutions.

2.8 Processing factor

The processing factor (Pf) of each washing application was calculated according to Eq. (2) [1]. If Pf < 1, it indicates a decrease in pesticide in a processed product, and Pf > 1 indicates an increase in pesticide residue in a processed product [12].

$$\text{Pf} = \frac{C_1}{C_o} \qquad (2)$$

2.9 Statistical analysis

Statistical analysis of the control group and the 13 cleaning solutions for seven pesticides was performed using

Fig. 1 AOAC Official Method 2007.01 sample preparation procedures

a one-way analysis of variance (ANOVA) and Tukey's post-test. A value of $p < 0.05$ was considered statistically significant. All values were expressed as mean standard deviation (SD). Statistical tests were performed using SPSS version 22.0. All treatments were replicated three times. Values are shown as means ± SD.

3 Results

3.1 Optimization LC–MS/MS parameters

Optimization of triple quadrupole mass spectrometry was analyzed via direct injection of 1000 μg kg^{-1} of each pesticide in the MS. The MS was initially run in full-scan mode for the selection of precursor (parent) ions. Then, after controlling the degradation of each precursor ion, two specific product (daughter) ions were selected for each pesticide and the collision energy (CE) voltages were optimized for each selected product ion [8]. Total ion chromatogram (TIC) and selected ion mass chromatograms for 7 pesticides are showed in Fig. 2.

3.2 Method validation

The analytical method has been validated to determine residues of boscalid, pyraclostrobin, pyrimethanil, fluopyram, fluopicolide, deltamethrin and sulfoxaflor in lettuce, by QuEChERS and LC-MS/MS. The matrix-matched calibration curve of nine active substances were prepared at the concentration of 5, 10, 25, 50, 100 and 200 μg L^{-1}. Each calibration point was obtained by 3

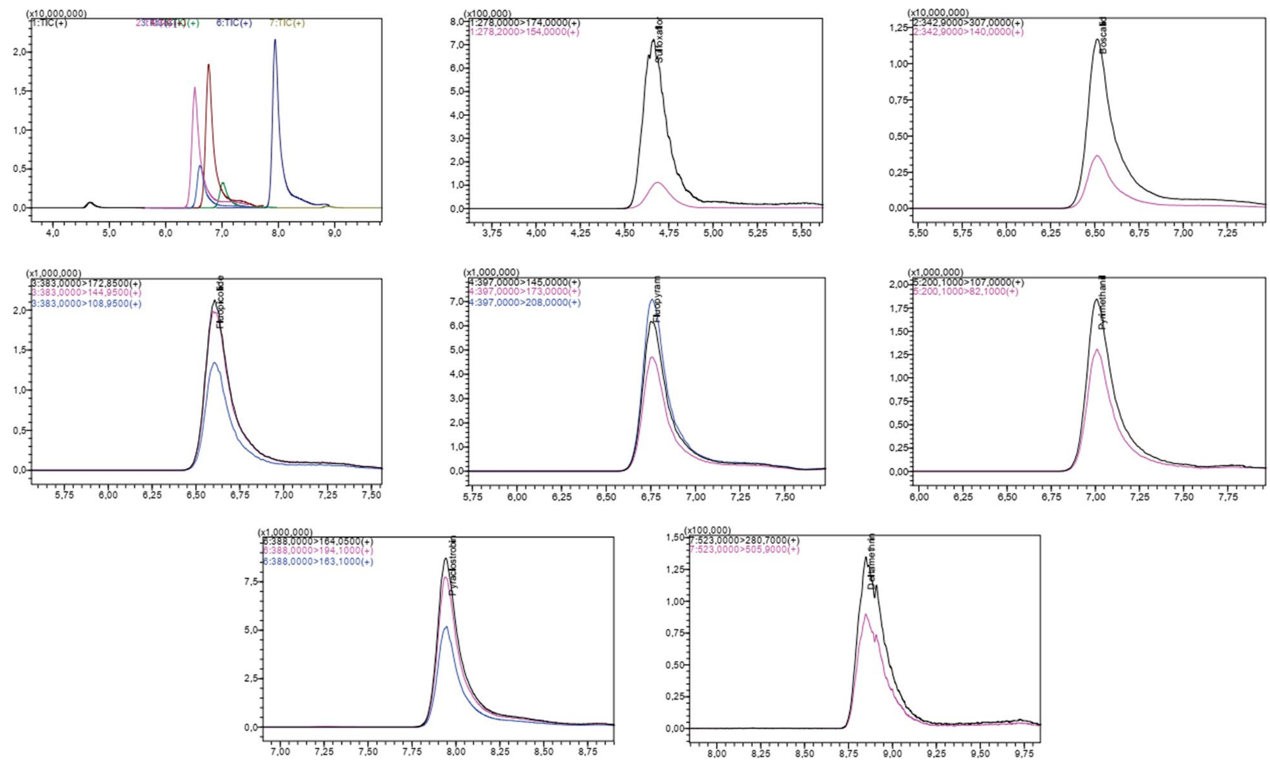

Fig. 2 Total ion chromatogram (TIC) and selected ion mass chromatograms for 7 pesticides

repeated injections. The method showed suitable specificity, linearity ($R^2 > 0.990$) and LOD/LOQ for 7 pesticide active ingredients in lettuce, with satisfactory precision (RSD < 20%) and trueness values (75.99–119.91%). The expanded measurement uncertainties were for all the pesticides between 17.97 and 48.50% (Table 2) [14].

3.3 Effects of washing on the removal of pesticide residues

The effectiveness of washing solutions was different for boscalid, deltamethrin, fluopcolide, fluopyram, pyrimethanil, pyraclostrobin and sulfoxaflor. All solutions were effective in reducing residues in the lettuce, as all PF values were less than 1. The results after application with 13 different solutions, processing factor, and reduction ratio are shown in Table 3. Statistical analysis results of residue levels in lettuce after removal are given in Table 4.

4 Discussion

Cold and hot tap water were highly effective in removing sulfoxaflor (96.6%, 96.2%), pyraclostrobin (80.5%, 94.4%), fluopyram (78.5%, 78%), and boscalid (77.4%, 78.4%) residues, but less effective in removing pyrimethanil (55.5%, 41.1%), deltamethrin (54.5%, 39.3%) and fluopicolide (29.2%, 59.4%) residues ($p < 0.05$). The fluopicolite residue decreased with the increase in water temperature, while the deltamethrin and pyrimethanil residues increased with the decrease in water temperature. López-Fernández et al. [24] applied low and high doses of mancozeb to 2 different types of lettuce (curly, straight) and washed them with water at two different temperatures (15 °C and 25 °C). They determined that the residue removal efficiency of mancozeb decreased with the increase in temperature in the curly lettuce they applied low dose. Rasolonjatova et al. [30] washed tomatoes with 25 °C and 40 °C water to remove methomyl and acetamiprid residues. They reported that the reduction rate of both pesticides was the same statistically.

Grape vinegar (4% a.a), Salt + vinegar, Grape vinegar+ water, grape vinegar (6% a.a), and grape vinegar (4% a.a), were highly effective in removing sulfoxaflor (92.4% to 98.1%), pyraclostrobin (78.4% to 89.8%), fluopyram (65.1% to 80.3%), and boscalid (58.6% to 75.2%) residues, but less effective in removing fluopicolide (30.5% to 53.6%), pyrimethanil (26.8% to 68%), and deltamethrin (37.8% to 66%) residues ($p < 0.05$). The highest removal was determined for sulfoxaflor, and the lowest removal was determined in pyrimethanil with grape vinegar+water application. The highest average removal rate was 70.1% in salty vinegar solution and the lowest average removal was 61% in grape vinegar (4% a.a) solution. Acoglu and Omeroglu [1] applied grape vinegar and 50% grape vinegar for the removal of abamectin,

Table 2 Method validation parameters

Analyte	Linear regression equation	LOD (μg kg^{-1})	LOQ (μg kg^{-1})	R^2	Repeatibility ($n=10$)				Reproducibility ($n=10$)				U′. %
					10 μg kg^{-1}		50 μg kg^{-1}		10 μg kg^{-1}		50 μg kg^{-1}		
					Recovery (%)	RSD (%)	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)	
Sulfoxaflor	Y = (34962.2) X + (− 23619.5)	1.49	4.96	0.998	104.10	8.35	101.52	4.14	106.27	5.76	106.79	3.70	17.97
Boscalid	Y = (144108) X + (1.06333e + 008)	2.15	7.17	0.990	90.43	3.29	109.56	3.68	75.99	17.37	119.91	3.87	48.50
Fluopicolide	Y = (102904)X + (− 125512)	1.07	3.55	0.999	81.23	3.10	105.22	3.06	80.33	7.90	113.12	3.56	33.79
Fluopyram	Y = (309627)X + (460338)	1.64	5.47	0.998	90.34	3.80	106.31	2.61	98.96	3.01	114.00	3.17	19.93
Pyrimethanil	Y = (102797)X + (− 124541)	1.96	6.54	0.998	110.07	10.86	103.46	4.01	113.77	6.34	108.64	3.50	25.35
Pyraclostrobin	Y = (112831) X + (2.75553e + 007)	1.94	6.48	0.999	119.26	4.93	118.46	4.03	110.09	9.68	117.14	5.90	39.23
Deltamethrin	Y = (7050.47) X + (− 4870.01)	2.54	8.45	0.999	98.25	12.23	94.56	6.15	115.04	11.88	116.04	7.81	35.04

Table 3 Pesticide residues, reduction rates and their PFs achieved with the present applications

Treatment	Sulfoxaflor			Fluopcolide			Fluopyram			Pyrimethanil			Pyraclostrobin			Deltamethrin			Boscalid		
	Mean residue (μg kg^{-1}) (± SD)	PF	Reduction (%)	Mean residue (μg kg^{-1}) (± SD)	PF	Reduction (%)	Mean residue (μg kg^{-1}) (± SD)	PF	Reduction (%)	Mean residue (μg kg^{-1}) (± SD)	PF	Reduction (%)	Mean residue (μg kg^{-1}) (± SD)	PF	Reduction (%)	Mean residue (μg kg^{-1}) (± SD)	PF	Reduction (%)	Mean residue (μg kg^{-1}) (± SD)	PF	Reduction (%)
Unwashed	6229 ± 101			1893 ± 29			1206 ± 54			2909 ± 24			2379 ± 21			545 ± 54			7118 ± 98		
Cold tap water (20 °C)	212 ± 9	0.03	96.59	1341 ± 25	0.70	29.18	259 ± 3	0.21	78.49	1293 ± 18	0.44	55.53	462 ± 3	0.19	80.50	248 ± 11	0.45	54.48	1610 ± 64	0.22	77.38
Hot tap water (40 °C)	237 ± 2	0.03	96.20	769 ± 23	0.40	59.39	266 ± 7	0.22	77.96	1714 ± 49	0.58	41.06	133 ± 5	0.05	94.40	332 ± 5	0.60	39.26	1535 ± 72	0.21	78.44
Grape vinegar (4% a.acid)	474 ± 8	0.07	92.38	1286 ± 40	0.67	32.06	421 ± 3	0.34	65.12	2150 ± 23	0.73	26.08	465 ± 2	0.19	80.40	234 ± 5	0.42	57.14	1743 ± 48	0.24	73.53
NaCl + grape vinegar	240 ± 4	0.03	96.14	975 ± 21	0.51	48.51	238 ± 8	0.19	80.25	1052 ± 17	0.36	63.83	242 ± 5	0.10	89.79	339 ± 11	0.62	37.83	1833 ± 45	0.25	74.25
Grape vinegar + water	119 ± 3	0.01	98.08	1120 ± 59	0.59	40.82	252 ± 5	0.20	79.10	930 ± 22	0.31	68.01	512 ± 10	0.21	78.37	227 ± 5	0.41	58.45	2946 ± 35	0.41	58.62
Rice vinegar	145 ± 3	0.02	97.66	630 ± 25	0.33	66.71	239 ± 6	0.19	80.16	866 ± 7	0.29	70.23	291 ± 18	0.12	87.73	236 ± 2	0.43	56.74	1768 ± 33	0.24	75.17
Filtered mint water	411 ± 5	0.06	93.40	1271 ± 78	0.67	32.87	302 ± 9	0.25	74.96	1616 ± 85	0.55	44.44	262 ± 16	0.11	88.95	385 ± 19	0.70	29.40	1272 ± 27	0.17	82.13
Filtered rice water	470 ± 10	0.07	92.45	445 ± 23	0.23	76.49	234 ± 3	0.19	80.63	744 ± 35	0.25	74.42	395 ± 1	0.16	83.31	414 ± 21	0.76	24.07	1321 ± 28	0.18	81.45
Carbonated water	266 ± 12	0.04	95.72	536 ± 16	0.28	71.66	399 ± 17	0.33	66.94	2004 ± 43	0.68	31.10	108 ± 5	0.04	95.43	257 ± 3	0.42	52.85	910 ± 39	0.12	87.22
Baking soda water	312 ± 21	0.05	94.99	734 ± 49	0.38	61.24	427 ± 34	0.35	64.61	1284 ± 71	0.44	55.87	511 ± 4	0.21	78.44	420 ± 1	0.77	22.98	697 ± 21	0.09	90.21
Grape vinegar (6% a.acid)	305 ± 6	0.04	95.09	878 ± 14	0.46	53.62	327 ± 6	0.27	72.90	1767 ± 52	0.60	39.23	385 ± 3	0.16	83.77	186 ± 3	0.34	66.00	1772 ± 44	0.24	75.12
Grape vinegar (8% a.acid)	152 ± 2	0.02	97.55	1315 ± 23	0.69	30.53	264 ± 2	0.21	78.06	1129 ± 33	0.38	61.19	305 ± 9	0.12	87.10	318 ± 1	0.58	41.70	1983 ± 88	0.27	72.16
Lemon juice	139 ± 9	0.02	97.77	731 ± 56	0.38	61.38	290 ± 16	0.24	75.97	1311 ± 68	0.45	54.91	102 ± 3	0.04	95.67	481 ± 34	0.88	11.88	2060 ± 76	0.28	71.07

Table 4 Significant differences in pesticide residues (μg kg^{-1}) of the washing applications

Treatment	Sulfoxaflor	Fluopcolide	Fluopyram	Pyrimethanil	Pyraclostrobin	Deltamethrin	Boscalid
Unwashed	6229^{a}	1893^{a}	1206^{a}	2909^{a}	2379^{a}	545^{a}	7118^{a}
Cold tap water (20 °C)	212^{e,f}	1341^{b}	259^{f,g}	1293^{d}	462^{b}	248^{f,g}	1610^{i}
Hot tap water (40 °C)	237^{e,f}	769^{f,g}	266^{f,g}	1714^{c}	133^{f}	332^{c,d}	1535^{i}
Grape vinegar (4% a.acid)	474^{b}	1286^{b,c}	421^{b,c}	2150^{b}	465^{b}	234^{f,g}	1743^{h}
NaCl + grape vinegar	240^{e,f}	975^{d,e}	238^{f,g}	1052^{e,f}	242^{e}	339^{c,d}	1833^{e}
Grape vinegar + water	119^{g,h}	1120^{c,d}	252^{f,g}	930^{f,g}	512^{b}	227^{f,g}	2946^{b}
Rice vinegar	145^{g}	630^{g,h,i}	239^{f,g}	866^{g,h}	291^{d,e}	236^{f,g}	1768^{g}
Filtered mint water	411^{b,c}	1271^{b,c}	302^{e,f}	1616^{c}	262^{d,e}	385^{b,c}	1272^{k}
Filtered rice water	470^{b}	445^{i}	234^{f,g}	744^{h,i}	395^{c}	414^{a,b}	1321^{j}
Carbonated water	266^{d,e}	536^{h,i}	399^{c,d}	2004^{b}	108^{f}	257^{e,f}	910^{l}
Baking soda water	312^{c,d}	734^{f,g,h}	427^{b}	1284^{d,e}	511^{b}	420^{a,b}	697^{m}
Grape vinegar (6% a.acid)	305^{c,d}	878^{e,f}	327^{d,e}	1767^{c}	385^{c}	186^{g,h}	1772^{f}
Grape vinegar (8% a.acid)	152^{g}	1315^{b,c}	264^{f,g}	1129^{d,e}	305^{d}	318^{d,e}	1983^{d}
Lemon juice	139^{g}	731^{f,g,h}	290^{f,g}	1311^{d}	102^{f}	481^{a}	2060^{c}

Different letters in a column indicating a statistically significant difference ($p < 0.05$)

buprofezin, etoxazole, imazalil sulfate and thiophanate-methyl active ingredients, and they found the average removal rate on all active ingredients as 46.7% and 51.8%, respectively. In our study, 50% grape vinegar had a removal rate of 68.8% and was more effective than grape vinegar (61%).

Rice vinegar has been very effective in removing all pesticides. Application of filtered rice water yielded effective results in all pesticides except deltamethrin. The average removal of rice vinegar and filtered rice water on all active ingredients was determined as 76.3% and 73.3%, respectively ($p < 0.05$). Rasolonjatova [28] reported that rice water removed acetamiprid 85.3% and methomyl 57.1% from tomatoes. Adachi and Okano [2] reported that rice bran removes chlorothalonil and tetradifon active ingredients at a rate of 85.2 and 93.2, respectively. Chowdhury et al. [11] reported that rice bran reduced cypermethrin by 97.6% after 10 minutes of application. Rice water and rice bran have an absorbent effect on many pesticides. Rice water offers an important solution for pesticide removal because it is both natural and easily available. However, Zhang et al. [35] reported that rice water was more ineffective than tap water on dimethoate, dicofol, and cyhalothrin residues.

Lemon juice was the most ineffective solution in removing deltamethrin residue (11.9%, $p < 0.05$). In addition, it has shown effective results in the removal of sulfoxaflor (97.8%), pyraclostrobin (95.7%), fluopyram (76%), boscalid (71.7%), fluopicolide (61.4%), and pyrimethanil (54.9%) ($p < 0.05$). Rasolonjatova et al. [30] recorded that lemon juice removed acetamiprid by 67% and methomyl by 57.3%.

Carbonated water reduced pyrimethanil residue by 31.1%, while other pesticides reduced between 52.9% and 95.7% ($p < 0.05$). Baking soda was the most ineffective solution at removing fluopyram (64.6%) and pyraclostrobin residue (55.9%) when compared to other solutions ($p < 0.05$). Baking soda removed lowest reduction (23%) of deltamethrin after lemon juice. Rasolonjatova et al. [30] reported that water with baking soda removed 54.3% of acetamiprid and 68.5% of methomyl. Zhang et al. [35] noted that the baking soda water and salt mixture removed the dimethoate, dicofol, and cyhalothrin residues from Chinese cabbage by 32.5%, 26.9%, and 44.4%, respectively. Satpathy et al. [31] reported that 0.1% sodium bicarbonate solution removed the residues of malathion, formation, parathion, methyl parathion and chlorpyriphos except fenitrothion. Heshmati et al. [19] reported that water with baking soda removed penconazole, hexaconazole, diazinon, ethion and phosalone residues by 94.5%, 93.7%, 95.4%, 71.6% and 63.1%, respectively in grapes. Liang et al. [23] noted that baking soda water (5%) removed dichlorvos, fenitrothion and chlorpyrifos residues by 98.8%, 66.7% and 85.2%, respectively in cucumbers. Andrade et al. [4] recorded that baking soda water (5%) removed acetamiprid (67.4%), azoxystrobin (46.1%), diflubenzuron (34.6%), dimethoate (61.8%), fipronil (2.32%), imidacloprid (They found that 62.1%, procymidone (14.6%) and thiamethoxam (72.1%) residues in tomato. Baking soda water had different effects on the reduction of the same active ingredient on different vegetables. In addition, pesticides which have high water solubility coefficients generally provide better removal.

Filtered mint water provided effective results in the removal of sulfoxaflor (93.4%), pyraclostrobin (89%), boscalid (82.1%), and fluopyram (75%) while it has no effect on other pesticide reduction ($p < 0.05$). Filtered mint water has the lowest effect (63.7%) on all pesticides after grape vinegar (4% a.a) (61%). Sulfoxaflor residue (95.7%) was highly removed in all wash solutions, while deltamethrin residue (42.5%) was found to be lower in all washing solutions on average.

5 Conclusions

The effects of nontoxic solutions that are easily accessible at home were studied removing boscalid, deltamethrin, fluopyram, fluopicolide, pyraclostrobin, pyrimethanil and sulfoxaflor pesticide residues in lettuce. Pf values of all washing applications were found below 1. This result shows that the residue level was reduced by washing solutions.

Sulfoxaflor, which is highly soluble pesticides and lower octanol-water partition coefficient was easily removed by washing treatments. On the other hand, deltamethrin, which has lower soluble and lower octanol-water partition coefficient, exhibited low washing efficiency. The effectiveness of washing processes may depend not only on the active content of the solution, the behavior of the pesticide and its physicochemical properties, but also on the temperature of the solution, the surface area the pesticides come into contact with the fruit or vegetable, the duration of contact, and the way the pesticide is used.

We conclude that some solutions used in this study could significantly reduce the exposure to pesticides for consumers. Wash solutions which readily available at home can be used to effectively remove pesticide residues. On the other hand, studies should be carried out with highly effective solutions to reduce different types of pesticide residues.

Abbreviations

NaCl: Sodium chloride; (N_2): Nitrogen; QuEChERS: QuEChERS stands for Quick, Easy, Cheap, Effective, Rugged, Safe and is the acronym for a highly beneficial analytical approach that vastly simplifies the analysis of multiple pesticide residues in fruit, vegetables, cereals and processed products thereof; dSPE: Dispersive solid-phase; RSD: Relative standard deviation; MRL: Maximum residue limits; LOD: Limit of detection; LOQ: Limit of quantification; LC–MS/MS: Liquid chromatography–tandem mass spectrometry; CE: Collision energy; ESI: Electrospray ionization; DL: Desolvation line; CID: Collision-induced dissociation; TIC: Total ion chromatogram; U′: Measurement uncertainties; Pf: Processing factor.

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Author contributions

TB made significant contributions to the design and execution of the study. ÖY contributed to conduction of research. TB and ÖY performed the experiments. TB and ÖY calculated, analyzed and interpreted data. TB attended in designing and writing the manuscript. TB gave final confirmation for the submission of revised version. All authors read, approved final manuscript.

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Availability of data and materials

Data available on request from the authors.

Code availability

Not applicable.

Declarations

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Not applicable.

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The authors declare that they have no competing interests.

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