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ORIGINAL ARTICLE

Evaluation of various soaking agents as a novel tool for pesticide residues mitigation from cauliflower (*Brassica oleracea* var. *botrytis*)

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Abstract The increasing use of pesticides for boosting the yield of agricultural crops also impart toxic residues which ultimately extend to numerous physiological disorders upon consumption. The present study was designed as an effort to assess the reduction potential of various chemical solutions and to minimize the pesticide residues in cauliflower (*Brassica oleracea* var. *botrytis*). The samples were soaked in various solutions along with tap water to mitigate pesticide residues. Afterwards, the extracted supernatant was passed through column containing anhydrous sodium sulfate trailed by activated carbon for clean-up. Eluents were first evaporated and then completely dried under gentle stream of Nitrogen. Finally, the residues were determined using gas chromatography equipped with electron capture detector (GC-ECD). Results revealed the highest reduction of endosulfan, bifenthrin and cypermethrin residues with acetic acid (10 %) was 1.133 ± 0.007 (41 %), 0.870 ± 0.022 (60 %) and 0.403 ± 0.003 (75 %), respectively among the tested solutions. However, simple tap water treatment also resulted in 0.990 ± 0.02 (12 %), 1.323 ± 0.015 (14 %) and 1.274 ± 0.002 (21 %) elimination of endosulfan, bifenthrin and cypermethrin residues, respectively. Moreover, among various solutions, acetic acid depicted maximum reduction potential followed by citric acid, hydrogen peroxide, sodium chloride and sodium carbonate solutions. The percent reduction by various solutions ranged from 12 to 41, 14 to 60 and 21 to 75 % for the elimination of endosulfan, bifenthrin and cypermethrin residues, respectively.

Keywords Contaminated cauliflower · Endosulfan residues · Bifenthrin and cypermethrin residues · Dipping solutions · GC-ECD

Introduction

Vegetables are edible portion of herbaceous plants and natural stashes of nutrients which contribute to nutritional security. They play a pivotal role in improving the human health by supplying minerals such as iron, calcium and magnesium, vitamins like A, B-complex and C, micronutrients, dietary fiber and antioxidants (Dalal et al. 2006; Supriya and Ram 2013). Furthermore, the use of vegetables maintains the body pH by adjusting the concentration of alkaline solutions and highly valued for certain nutrients which efficiently help in building and repairing mechanisms of the body (Marwat 2009; Bangash et al. 2011).

Cauliflower (*Brassica oleracea* var. *botrytis*) is a short duration annual vegetable crop which belongs to *Brassicaceae* family. It is an excellent source of vitamin C (ascorbic acid) and also provides omega-3 fatty acids, manganese, iron and fiber. Its consumption remarkably correlates in ameliorating the chances of colon, breast and prostate cancer in human due to the presence of indole-3-carbinol (Nachshon-Kedmi et al. 2003; Aggarwal and Ichikawa 2005; Lo-Scalzo et al. 2007). This vegetable is

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susceptible to attack of a number of devastating insect pests which reduce its production. Hence, the use of pesticides is inevitable for enhancement of crop production, fiber and forage. Pesticides comprise a variety of synthetic chemical substances used in agriculture to counter act the detrimental effects of insect pests attack on crops (Supriya and Ram 2013).

Organochlorine pesticides are extensively used by farmers because of their cost effectiveness and broad spectrum activity. However, due to highly toxic and persistent in nature, their residues may cause food toxicity and environmental pollution (Bempah et al. 2011). Endosulfan, an organochlorine was reported to be extremely toxic in suspected endocrine disrupters by US Environmental Protection Agency (Ahad et al. 2000; McKinlay et al. 2008; Rand et al. 2010). Its excessive applications has led to accumulation in water and soil which in turn, play a havoc role in damaging the environmental conditions that ultimately affect the human health (Bempah and Donkor 2011). Pyrethroids are another widely used class of insecticides with effective knock-down against a wide spectrum of insect pests. As these insecticides have a relatively shorter pre-harvest interval of 3–5 days, hence they are preferred choice to be used near harvesting of the crop. The characteristics like broad spectrum, relatively low application rates, less persistent nature and non-toxicity towards consumers have made the pyrethroids as environmentally safe and a suitable alternative to organochlorines (Sannino et al. 2003).

The extensive and injudicious use of endosulfan, bifenthrin and cypermethrin pesticides on vegetables is causing problem of residual toxicity (Ahmed et al. 2011). It has also been observed that pesticide residues cause chronic toxicity and many other ill health consequences upon consumption of chemically treated vegetables (Randhawa et al. 2014). Worldwide and particularly in developing countries vegetables are found to have endosulfan and pyrethroids residues above maximum residual limits (MRLs) because of their highly persistent behavior in natural environment (Kumari et al. 2004; Bai et al. 2006). In Pakistan, the situation of presence of pesticide residues in vegetable crops is more alarming as endosulfan, bifenthrin and cypermethrin residues have been reported in samples of brinjal, cauliflower, okra, spinach and arvi collected from different cities (Tahir et al. 2001; Randhawa et al. 2007). Resultantly, these residues cause harmful effects on human health, animals, birds and may initiate health disorders by disturbing metabolic activities (El-Morsi et al. 2012).

Hence, there is a need to curtail the level of pesticide residues in food commodities generally and in vegetables particularly. Washing with different organic acid solutions was found an effective approach in minimizing the pesticide residues in vegetables (Randhawa et al. 2014; Abdullah et al. 2016). Ahmed et al. (2011) and many other researchers have reported that pesticide residues can be mitigated by simple washing, hot water washing and blanching. Apart from these studies, limited data is available regarding the reduction potential of various acidic, neutral and alkaline solutions on lowering the level of pesticide residues in cauliflower. The current study has been designed to assess the influence of various soaking solutions i.e. acidic (acetic acid, citric acid and hydrogen peroxide), neutral (NaCl) and alkaline solutions (Na_2CO_3) along with tap water in minimizing the residual level of endosulfan, bifenthrin and cypermethrin from cauliflower. Moreover, the concentration of residues retained in soaking solutions was also determined.

Materials and methods

Procurement of raw materials

Cauliflower was harvested after a waiting period of 36 h from the supervised field by foliar spraying the insecticides with Knap-sack sprayer in the absence of air and precipitation. The pesticides (dissolved in water) along with applied doses used in this study were: endosulfan ($C_9H_6Cl_6O_3S$, 35EC 8 mL L^{-1}), bifenthrin ($C_{23}H_{22}ClF_3O_2$, 10EC 4.5 mL L^{-1}), and cypermethrin ($C_{22}H_{19}Cl_2NO_3$, 10 EC 5.5 mL L^{-1}). All the chemicals used in this study were purchased from Merck (Merck KGaA, Darmstadt, Germany). However, the pesticides standards were purchased from Dr. Ehrenstrofer GmbH (Augsburg, Germany).

Soaking of cauliflower in chemical solutions

The cauliflower samples were soaked in different acidic, neutral and alkaline solutions of different concentrations along with tap water for the comparison purpose (Table 1). The samples were soaked in chemical solutions for 10 min at 30 °C. Afterwards, the samples were chopped into small pieces (2 cm) on clean chopping boards with stainless steel knife and blended using household National blender (MJ176NR) to obtain homogeneous slurry.

Pesticide residues extraction

The pesticide residues from the samples were extracted following the method described earlier (Khan et al. 2009) with slight modifications. Briefly, homogenized slurry (50 g) was mixed with anhydrous sodium sulfate (20 g), sodium chloride (10 g) and ethyl acetate (70 mL) in 250 mL Erlenmeyer flask. Glass beads were also added in

Table 1 Treatments plan

Dipping solutions	Type of solution	Solution (g/100 mL)
T_0	Control without dipping	–
T_1	Tap water	–
T_2	Acetic acid	2
T_3	Acetic acid	4
T_4	Acetic acid	6
T_5	Acetic acid	8
T_6	Acetic acid	10
T_7	Citric acid	2
T_8	Citric acid	4
T_9	Citric acid	6
T_{10}	Citric acid	8
T_{11}	Citric acid	10
T_{12}	Hydrogen peroxide	2
T_{13}	Hydrogen peroxide	4
T_{14}	Hydrogen peroxide	6
T_{15}	Hydrogen peroxide	8
T_{16}	Hydrogen peroxide	10
T_{17}	Sodium chloride	4
T_{18}	Sodium chloride	7
T_{19}	Sodium chloride	10
T_{20}	Sodium carbonate	4
T_{21}	Sodium carbonate	7
T_{22}	Sodium carbonate	10

mixture to facilitate the extraction process. The mixture was shaken at 240 rpm for 1 h on a horizontal mechanical shaker. The resultant supernatant was allowed to stay for 30 min and collected in flask through filtration using Whatman filter paper.

Column clean-up of filtered extract

Pesticide residues analysis requires high sensitivity as these are present in traces. For this valid reason, column clean-up was carried out to remove interfering substances in extract to get precise measurement of residues. Therefore, extracts having pesticide residues were cleaned up using column chromatographic technique (Kumari 2008). Chromatographic columns supported by glass wool were used for clean-up. Florisil and charcoal were activated at 200 °C for 24 h before filling of the column. Activated florisil and charcoal were mixed in 5:1 (w/w) ratio. A thin layer of anhydrous sodium sulphate was placed on glass wool. The activated mixture (9 g) of florisil and charcoal was placed on sodium sulphate layer. Activated mixture was covered with thin layer of anhydrous sodium sulphate and glass wool, respectively. Two washings were given to the prepared column containing adsorbents with acetone (HPLC grade) before using it for clean-up purpose.

After column preparation, the sample extract was loaded and extract was eluted using 50 mL of ethyl acetate and n-hexane mixture (3:7 v/v). Subsequently, 10 mL of acetone was used for washing of the column and eluted samples were received in 150 mL round bottom flask. Elute was then concentrated in rotary evaporator at 40 °C till 1–1.5 mL. The concentrated elute was transferred to new 1.5 mL clean vials and placed under the gentle stream of Nitrogen until complete drying.

Extraction and determination of residues from dipping solutions

After applying the treatments, various dipping solutions were filtered using Whatman filter paper and analyzed by following the method (Asi 2003). For this purpose, SEP-PAK C_{18} cartridges were used for extraction and concentration of multi-residues of pesticides. The cartridges were conditioned with 5 mL of hexane, acetone, methanol and double distilled water in sequence, then collected filtered water was passed under pressure at 5 mL per min flow rate. After passing 200 mL of filtered water the cartridge was dried for 2 h at maximum pressure. The targeted compounds were eluted with 2 mL of acetonitrile, ethyl acetate and methanol. Eluted samples were transferred to vials and dried to 0.5–1 mL using gentle stream of Nitrogen.

Quantification of pesticides residues through gas chromatography

Cauliflower and dipping water samples were analyzed using gas chromatography (GC) equipped with electron capture detector (ECD). For the determination of organochlorine and pyrethroid residues, the re-dissolved samples were subjected to the GC-ECD analysis by following the guidelines (Kumari et al. 2002; Sannino et al. 2003). The inlet temperature was 280 °C, detector temperature was 300 °C, the filtered prepared sample injection volume was 2 µL and Nitrogen gas was used as the carrier. The total flow rate was 60 mL per min, while the column flow rate was 2 mL per min.

Statistical analysis

The data obtained after three independent replicates were analyzed through completely randomized design using standard statistical procedures (Statistix 8.1). Moreover, Analysis of Variance (ANOVA) was performed to determine the level of significance while least significant difference (LSD) test was used to compare the means of treatments (Steel et al. 1997). The percent residual reduction of pesticide residues was calculated as follows: the % residual reduction rate = $(R_0 - R_1/R_0) \times 100$. Where

R_0 = the amount of pesticide residues present in control sample, R_1 = the amount of residues present in samples after soaking treatments.

Results and discussion

Endosulfan residues retained in chemically washed cauliflower and dipping solutions

The residues of endosulfan detected in cauliflower samples ranges between 0.661 and 0.979 mg kg^{-1} which exceeded the level of maximum residual limits (MRLs) i.e. 0.05 mg kg^{-1} as established by the European Union, Council Directive. However, in control cauliflower samples the amount of endosulfan residues were 1.133 mg kg^{-1} (Table 2). While, the residues retained in dipping solutions were in the range of 0.138–1.133 mg kg^{-1} and in tap water soaked samples was 0.473 mg kg^{-1}. Among the acid washing treatments, residues detected in dipping solutions were in the range of 0.199–1.133 mg kg^{-1} (Table 2). Generally, by increasing the concentration of acids; the endosulfan residues retained in water increased significantly. Maximum residues were found in acetic acid (T_6) and minimum residues of endosulfan in hydrogen peroxide (T_{12}). Likewise in salt solutions, sodium chloride (T_{19}) revealed 0.335 mg kg^{-1} while sodium carbonate (T_{20}) removed 0.138 mg kg^{-1} endosulfan residues from soaked cauliflower, respectively (Tables 1, 2).

Among the acid washing treatments, percentage reduction of endosulfan residues was in the range of 17–41 %. Acetic acid (10 %) showed the maximum percent reduction (41 %) while hydrogen peroxide (2 %) caused minimum reduction of endosulfan residues (17 %). In case of salt solutions, the percent reduction of residues was in the range of 15–29 %. Sodium chloride (10 %) revealed maximum reduction (29 %) and sodium carbonate (4 %) showed minimum reduction of 13 % (Table 2).

There were significant differences in reduction potential of endosulfan residues among various washing solutions due to their different concentrations. Therefore, to ensure food safety during processing operations particularly washing solutions and cooking lead to high level of residues reduction (Kaushik et al. 2009). The results of present study are consistent with earlier findings on reduction of pesticide residues in vegetables by household processes like cooking, boiling and washing (Kumari 2008). The results were further reinforced; a study reported 15–30 %

Table 2 Endosulfan residues present in treated cauliflower and dipping solutions (mg kg^{-1}) along with reduction

Type of solution	Solution (g/100 mL)	Cauliflower	Dipping solutions	Reduction (%)
Controlled	–	1.133 ± 0.015^{a}	–	–
Tap water washed	–	0.990 ± 0.02^{b}	0.473 ± 0.020^{b}	12
Acetic acid	2	0.912 ± 0.002^{ef}	0.217 ± 0.013^{cde}	19
	4	0.846 ± 0.004^{i}	$0.286 \pm 0.016^{b–e}$	25
	6	0.802 ± 0.003^{jkl}	$0.326 \pm 0.012^{b–e}$	29
	8	0.754 ± 0.013^{m}	0.471 ± 0.017^{a}	33
	10	0.661 ± 0.027^{n}	1.133 ± 0.007^{b}	41
Citric acid	2	0.929 ± 0.054^{de}	0.202 ± 0.059^{cde}	18
	4	0.888 ± 0.052^{gh}	0.226 ± 0.005^{cde}	21
	6	0.866 ± 0.036^{hi}	0.255 ± 0.004^{cde}	23
	8	0.818 ± 0.016^{jk}	$0.314 \pm 0.031^{b–e}$	27
	10	0.781 ± 0.003^{l}	0.344 ± 0.005^{bcd}	31
Hydrogen peroxide	2	0.930 ± 0.051^{cde}	0.199 ± 0.010^{cde}	17
	4	0.891 ± 0.002^{fg}	0.239 ± 0.015^{cde}	21
	6	0.857 ± 0.028^{i}	0.273 ± 0.016^{cde}	24
	8	0.821 ± 0.004^{j}	$0.310 \pm 0.013^{b–e}$	27
	10	0.784 ± 0.015^{l}	0.346 ± 0.002^{bc}	32
Sodium chloride	4	0.953 ± 0.045^{c}	$0.178 \pm 0.012^{c–f}$	15
	7	0.880 ± 0.001^{gh}	0.248 ± 0.019^{cde}	22
	10	0.797 ± 0.015^{kl}	0.335 ± 0.018^{bcd}	29
Sodium carbonate	4	0.979 ± 0.003^{b}	0.138 ± 0.018^{def}	13
	7	0.949 ± 0.005^{cd}	$0.149 \pm 0.020^{c–f}$	16
	10	0.928 ± 0.021^{de}	0.182 ± 0.049^{ef}	18

Data are mean ± SD (n = 3)

Means in column with different superscript letters differed significantly according to LSD test ($p < 0.05$)

dislodging of endosulfan residues through washing from winter and summer vegetables (Randhawa et al. 2007). Results are further strengthened by the added support with studies who reported that chemical washing treatments and food processing significantly reduced pesticide residues from spinach and food crops (Ahmed et al. 2011; Abdullah et al. 2016).

Bifenthrin residues retained in chemically washed cauliflower and dipping solutions

Bifenthrin residues in soaked cauliflower samples ranged from 0.625 to 1.530 mg kg^{-1}, while in dipping solutions were from 0.197 to 0.870 mg kg^{-1} (Table 3). However, residue of Bifenthrin in tap water soaked cauliflower was 0.193 mg kg^{-1}. Amongst the acid dipping solutions, residues detected were in the range of 0.19–0.87 mg kg^{-1}. Acetic acid (10 %) revealed maximum while hydrogen peroxide (2 %) possessed minimum potential in residues reduction. In case of salt solutions, bifenthrin residues were in the range of 0.319–0.801 mg kg^{-1} (Table 3).

The present study results established that tap water treatment caused 14 % bifenthrin residues reduction while washing cauliflower with different acetic acid concentration resulted 21, 35, 41, 49 and 60 % reduction in treatments T_2, T_3, T_4, T_5 and T_6, respectively. Correspondingly, citric acid concentrations resulted 17, 30, 35, 46 and 51 % reduction in T_7, T_8, T_9, T_{10} and T_{11} treatments. Likewise, reduction by hydrogen peroxide in different treatments T_{12}, T_{13}, T_{14}, T_{15} and T_{16} were as 15, 29, 35, 44 and 49 %. Furthermore, sodium chloride treatments T_{17}, T_{18} and T_{19} caused 25, 43, 59 % and sodium carbonate different concentration T_{20}, T_{21} andT_{22} caused 21, 38, and 41 % reduction respectively (Tables 1, 3).

The study drew up that by increasing chemical concentration in solutions; the reduction rate of residues also increases. Furthermore, soaking solutions showed capability to retain and extract residues which indicated their dissolution capacity, more solubility lead to higher recovery of residues present in samples under investigations. Acetic acid solutions were found most effective in diminishing the residues among the soaking solutions used. Conclusively, acetic acid solutions performed better than citric acid, neutral and alkaline solutions. However, sodium chloride working solutions removed more pesticide residues as compared to sodium carbonate solutions (Table 3).

Table 3 Bifenthrin residues present in treated cauliflower and dipping solutions (mg kg^{-1}) along with reduction

Type of solution	Solution (g/100 mL)	Cauliflower	Dipping solutions	Reduction (%)
Controlled	–	1.530 ± 0.020^{a}	–	–
Tap water washed	–	1.323 ± 0.015^{b}	0.193 ± 0.035^{l}	14
Acetic acid	2	1.207 ± 0.025^{d}	0.303 ± 0.020^{j}	21
	4	0.995 ± 0.002^{g}	0.533 ± 0.016^{g}	35
	6	0.903 ± 0.028^{h}	0.630 ± 0.010^{f}	41
	8	0.793 ± 0.014^{k}	0.716 ± 0.005^{cd}	49
	10	0.625 ± 0.005^{m}	0.870 ± 0.022^{a}	60
Citric acid	2	1.267 ± 0.051^{c}	0.237 ± 0.153^{k}	17
	4	1.067 ± 0.017^{f}	0.440 ± 0.040^{h}	30
	6	0.994 ± 0.002^{g}	0.533 ± 0.022^{g}	35
	8	0.828 ± 0.032^{j}	0.693 ± 0.008^{de}	46
	10	0.749 ± 0.002^{l}	0.778 ± 0.021^{b}	51
Hydrogen peroxide	2	1.307 ± 0.030^{b}	0.197 ± 0.041^{kl}	15
	4	1.086 ± 0.025^{f}	0.442 ± 0.022^{h}	29
	6	0.994 ± 0.015^{g}	0.534 ± 0.022^{g}	35
	8	0.856 ± 0.003^{i}	0.670 ± 0.023^{e}	44
	10	0.780 ± 0.002^{k}	0.738 ± 0.027^{c}	49
Sodium chloride	4	1.140 ± 0.021^{d}	0.366 ± 0.016^{j}	25
	7	0.872 ± 0.002^{g}	0.655 ± 0.025^{g}	43
	10	0.627 ± 0.036^{k}	0.801 ± 0.026^{c}	59
Sodium carbonate	4	1.208 ± 0.026^{e}	0.319 ± 0.045^{i}	21
	7	0.994 ± 0.002^{i}	0.533 ± 0.023^{ef}	38
	10	0.781 ± 0.003^{n}	0.737 ± 0.022^{a}	49

Data are mean ± SD (n = 3)

Means in column with different superscript letters differed significantly according to LSD test ($p < 0.05$)

The prime mechanism involved in toxic residues dissipation was dissolution of pesticide residues from cauliflower samples in soaking solutions. Hence, reduction indicates dissolution capacity of working solutions while concentration of soaking solutions presented linear correlation in the dissolution of residues which ultimately enhanced the reduction rate of toxic residues (Kin and Huat 2010).

Cypermethrin residues in chemically washed cauliflower and dipping solutions

Cypermethrin residues in soaked cauliflower samples were ranged from 0.403 to 1.610 mg kg^{-1}, while in dipping solutions ranged from 0.432 to 1.204 mg kg^{-1} (Table 4). However, residue of cypermethrin present in tap water soaking solution was 0.334 mg kg^{-1}. Amongst the acid washing treatments, residues detected were in the range of 0.507–1.204 mg kg^{-1}. Maximum residues retained in T_6 (acetic acid 10 %) and T_{12} (hydrogen peroxide 2 %) caused residues reduction in minimum amount. In case of salt solutions, cypermethrin residues were present in the range of 0.606–0.995 mg kg^{-1}. T_{19} (sodium chloride 10 %) expressed maximum and T_{20} (sodium carbonate 4 %) showed minimum reduction potential in elimination of residues (Tables 1, 4).

Cypermethrin residues in raw untreated cauliflower sample were 1.610 mg kg^{-1}. Amongst the acidic treatments, percent reduction of residues was 27–75 %. The maximum percent reduction of residues was observed for T_6 i.e. 75 % while T_{12} showed minimum reduction (27 %). Among salt solutions, T_{19} caused maximum reduction (62 %) while T_{20} observed minimum reduction 38 % in treated samples (Tables 1, 4). Conclusively; among the treatments acetic acid showed maximum percent reduction potential of cypermethrin followed by citric acid, hydrogen peroxide, sodium chloride and sodium carbonate, respectively.

Generally, pyrthroids residues were present in higher levels than organochlorine in cauliflower samples. The data in Tables 2, 3 and 4 showed the percent rate of pesticides removal from contaminated cauliflower after soaking in acidic, neutral and alkaline solutions. The results indicated that soaking solutions have an efficient role in the elimination of toxic residues. The pyrethroids residues were more rapidly degraded than organochlorine. There was creeping increase in the reduction percentage due to the

Table 4 Cypermethrin residues present in treated cauliflower and dipping solutions (mg kg^{-1}) along with residual reduction

Type of solution	Solution (g/100 mL)	Cauliflower	Dipping solutions	Reduction (%)
Controlled	–	1.610 ± 0.020^{a}	–	–
Tap water washed	–	1.274 ± 0.002^{b}	0.334 ± 0.021^{l}	21
Acetic acid	2	1.087 ± 0.015^{e}	0.507 ± 0.015^{ijk}	33
	4	0.902 ± 0.002^{i}	0.705 ± 0.023^{fg}	44
	6	0.790 ± 0.004^{k}	0.818 ± 0.024^{ef}	51
	8	0.613 ± 0.041^{o}	1.194 ± 0.0117^{a}	62
	10	0.403 ± 0.003^{q}	1.204 ± 0.031^{a}	75
Citric acid	2	1.117 ± 0.031^{d}	0.457 ± 0.031^{jkl}	31
	4	0.965 ± 0.003^{h}	0.641 ± 0.017^{gh}	40
	6	0.805 ± 0.002^{k}	0.802 ± 0.017^{ef}	50
	8	0.663 ± 0.025^{n}	0.944 ± 0.003^{bcd}	59
	10	0.577 ± 0.003^{p}	1.020 ± 0.027^{b}	65
Hydrogen peroxide	2	1.175 ± 0.003^{c}	0.432 ± 0.016^{kl}	27
	4	1.046 ± 0.002^{f}	0.560 ± 0.022^{hij}	35
	6	0.824 ± 0.003^{j}	0.782 ± 0.021^{ef}	49
	8	0.743 ± 0.004^{l}	0.864 ± 0.021^{de}	54
	10	0.613 ± 0.002^{o}	0.999 ± 0.009^{bc}	62
Sodium chloride	4	0.982 ± 0.003^{h}	0.625 ± 0.024^{ghi}	39
	7	0.723 ± 0.031^{m}	0.883 ± 0.023^{cde}	55
	10	0.612 ± 0.035^{o}	0.995 ± 0.016^{bc}	62
Sodium carbonate	4	1.001 ± 0.007^{g}	0.606 ± 0.014^{ghi}	38
	7	0.824 ± 0.003^{j}	0.783 ± 0.021^{ef}	49
	10	0.725 ± 0.004^{m}	0.882 ± 0.024^{cde}	55

Data are mean ± SD (n = 3)

Means in column with different superscript letters differed significantly according to LSD test ($p < 0.05$)

increase in concentration of different washing reagents, being more efficient than tap water.

In this study, soaking solutions showed capability to retain residues which indicated their dissolution capacity, more solubility lead to higher recovery of residues present in samples under investigations. Conclusively, present study results had inferred that tap water alone or in combination with different acidic, alkaline or neutral solutions was indispensable to mitigate toxic residues. Furthermore, study established momentous reduction of pesticide residues by increasing chemical concentrations in soaking solutions. Amongst all the soaking solutions, acetic acid solution 10 % was most effective in dislodging pesticide residues. The results of the study find added support with some earlier findings where water rinsing, household processing and chemical washing treatments efficiently reduced toxic residues from vegetables which includes asparagus, capsicum, tomato, okra, beans and eggplant (Chavarri et al. 2005; Randhawa et al. 2007; Kaushik et al. 2009). Moreover, a study on vegetables documented 20–89 % organophosphorous residues reduction through different chemical washings (Satpathy et al. 2011). Similarly, rinsing practice efficiently removed endosulfan and permethrin insecticides residues from food crops under investigations (Krol et al. 2000).

Conclusion

To diminish the risks associated with injudicious use of pesticides, soaking the cauliflower in various washing solutions such as acetic acid, citric acid and hydrogen peroxide could be an efficient strategy in minimizing the pesticide residues. However, dipping in the brine solution followed by tap water washing was more effective to mitigate residues as compared to the usual practice at home i.e. tap water washing. Among the selected pesticides, pyrthroids residues were eliminated more by acidic, neutral and alkaline solutions than that of organochlorine residues. Amongst the various soaking solutions, acidic solutions were more effective in the elimination of pesticide residues than alkaline and neutral solutions. In conclusion, acetic acid 10 % was the most effective solution that showed significant elimination of these toxic pesticide residues.

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References

Abdullah Randhawa MA, Akhtar S, Mansoor-ul-Hassan Asghar A, Sohaib M, Aadil RM, Jahangir MA (2016) Assessment of different washing treatments to mitigate imidacloprid and acetamaprid residues in spinach. J Sci Food Agric. doi:10.1002/jsfa.7563

Aggarwal BB, Ichikawa H (2005) Molecular targets and anticancer potential of indole-3-carbinol and its derivatives. Cell Cycle 4:1201–1215

Ahad K, Anwar T, Ahmad I, Mohammad A, Tahir S, Aziz S, Baloch U (2000) Determination of insecticide residues in groundwater of Mardan Division, NWFP, Pakistan: a case study. Wat Pret 26:409–412

Ahmed A, Randhawa MA, Yusuf MJ, Khalid N (2011) Effect of processing on pesticide residues in food crops: a review. J Agric Res 49:379–390

Asi MR (2003) Solid-phase extraction and chromatographic determination of pesticides in food and water samples. University of the Punjab, Lahore

Bai Y, Zhou L, Wang J (2006) Organophosphorus pesticide residues in market foods in Shaanxi area, China. Food Chem 98:240–242

Bangash JA, Arif M, Khan M, Khan F, Hussain I (2011) Proximate composition, minerals and vitamins content of selected vegetables grown in Peshawar. J Chem Soc Pak 33:118–122

Bempah CK, Donkor AK (2011) Pesticide residues in fruits at the market level in Accra Metropolis, Ghana, a preliminary study. Environ Monit Assess 175:551–561

Bempah CK, Donkor AK, Yeboah PO, Dubey B, Osei-Fosu P (2011) A preliminary assessment of consumer's exposure to organochlorine pesticides in fruits and vegetables and the potential health risk in Accra Metropolis, Ghana. Food Chem 128:1058–1065

Chavarri MJ, Herrera A, Arino A (2005) The decrease in pesticides in fruit and vegetables during commercial processing. Int J Food Sci Technol 40:205–211

Dalal M, Dani RG, Kumar PA (2006) Current trends in the genetic engineering of vegetable crops. Sci Hortic 107:215–225

El-Morsi D, Rahman R, Abou-Arab A (2012) Pesticides residues in Egyptian diabetic children: a preliminary study. J Clin Toxicol 2:2161-0495

Kaushik G, Satya S, Naik SN (2009) Food processing a tool to pesticide residue dissipation—A review. Food Res Int 42:26–40

Khan BA, Farid A, Asi MR, Shah H, Badshah AK (2009) Determination of residues of trichlorfon and dimethoate on guava using HPLC. Food Chem 114:286–288

Kin CM, Huat TG (2010) Headspace solid-phase microextraction for the evaluation of pesticide residue contents in cucumber and strawberry after washing treatment. Food Chem 123:760–764

Krol WJ, Arsenault TL, Pylypiw HM, Incorvia MJ (2000) Reduction of pesticide residues on produce by rinsing. J Agric Food Chem 48:4666–4670

Kumari B (2008) Effects of household processing on reduction of pesticide residues in vegetables. ARPN J Agric Biol Sci 3:46–51

Kumari B, Madan V, Kumar R, Kathpal T (2002) Monitoring of seasonal vegetables for pesticide residues. Environ Monit Assess 74:263–270

Kumari B, Madan V, Singh J, Singh S, Kathpal T (2004) Monitoring of pesticidal contamination of farmgate vegetables from Hisar. Environ Monit Assess 90:65–71

Lo-Scalzo R, Bianchi G, Genna A, Summa C (2007) Antioxidant properties and lipidic profile as quality indexes of cauliflower (*Brassica oleracea* L. var. botrytis) in relation to harvest time. Food Chem 100:1019–1025

Marwat SK (2009) Vegetables mentioned in the Holy Qura'n and Ahadith and their ethnomedicinal studies in Dera Ismail Khan, NWFP, Pakistan. Pak J Nutr 8:530–538

Mckinlay R, Plant J, Bell J, Voulvoulis N (2008) Endocrine disrupting pesticides: implications for risk assessment. Environ Int 34:168–183

Nachshon-Kedmi M, Yannai S, Haj A, Fares FA (2003) Indole-3-carbinol and 3,3′-diindolylmethane induce apoptosis in human prostate cancer cells. Food Chem Toxicol 41:745–752

Rand GM, Carriger JF, Gardinali PR, Castro J (2010) Endosulfan and its metabolite, endosulfan sulfate, in freshwater ecosystems of South Florida: a probabilistic aquatic ecological risk assessment. Ecotoxicology 19:879–900

Randhawa MA, Anjum FM, Asi MR, Butt MS, Ahmed A, Randhawa MS (2007) Removal of endosulfan residues from vegetables by household processing. J Sci Ind Res 66:849–852

Randhawa MA, Anjum FM, Asi MR, Ahmed A, Nawaz H (2014) Field incurred endosulfan residues in fresh and processed vegetables and dietary intake assessment. Int J Food Prop 17:1109–1115

Sannino A, Bandini M, Bolzoni L (2003) Determination of pyrethroid pesticide residues in processed fruits and vegetables by gas chromatography with electron capture and mass spectrometric detection. J AOAC Int 86:101–108

Satpathy G, Tyagi YK, Gupta RK (2011) Removal of organophosphorus (OP) pesticide residues from vegetables using washing solutions and boiling. J Agric Sci 4:69

Steel RGD, Torrie JH, Dickey D (1997) Principles and procedures of statistics: a biometrical approach. McGraw Hill Book Co, New York

Supriya U, Ram D (2013) Comparative profile of adoption of integrated pest management (IPM) on cabbage and cauliflower growers. Res J Agric Sci 4:640–643

Tahir S, Anwar T, Ahmad I, Aziz S, Mohammad A, Ahad K (2001) Determination of pesticide residues in fruits and vegetables in Islamabad market. J Environ Biol 22:71–74