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Tracing the efficiency of food processing steps on the pesticide residuals reduction rate in fruits and vegetables

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Abstract

Pesticides are widely used in food production to increase food security, although they can adversely affect consumers. Pesticide residues have been found in various fruits and vegetables, both raw and processed. Therefore, the study investigates the presence of pesticide residues such as systemic pesticides (Pyridaben and alpha-cypermethrin) and non-systemic pesticides (azoxystrobin and propamocarb hydrochloride) in commonly consumed fruits and vegetables, specifically okra, grapes, dates, and tomato. The researchers also evaluate the effectiveness of various processing methods in reducing pesticide residue levels, such as washing with water and other solutions such as acetic and citric acids (2%), peeling, blanching, alkaline and sulfuring treatment, drying and concentration at high temperature. Our finding showed that the processing method steps could reduce pesticide residuals with different reductions %, which are affected by the physical and chemical properties of pesticides, processing steps conditions and the nature of the fruit or vegetable. Even after some processing steps, samples still had residue levels exceeding the Maximum Residue Limits (MRLs), such as washing, peeling, juicing and alkaline or sulfating treatment steps. Indicating that consumers may still be exposed to unsafe levels of pesticides. Otherwise, at the end of processing methods such as blanching, drying and concentration, all final products become almost free of pesticide residuals. The study concludes that processing methods can reduce the levels of pesticide residues or eliminate them.

Keywords: Pesticide residuals, Blanching, Washing, peeling, Drying, Systemic, Non-systemic.

Introduction

Pesticides are pivotal in modern agriculture, protecting crops from pests, diseases, and weeds, increasing yield, and ensuring food security. However, the widespread use of these chemicals has raised significant concerns due to their potential impact on human health and the environment. Pesticides can leave residues on fruits and vegetables, which may pose health risks to consumers if the levels exceed the safety thresholds set by regulatory authorities (Tudi et al., 2021).

Several pesticides, such as chlorpyrifos, diazinon, and malathion, are known neurotoxins and have been associated with various adverse health effects, including developmental disorders, neurodegenerative diseases, and cancer. Furthermore, the simultaneous presence of multiple pesticide residues in food causes a phenomenon known as the "cocktail effect," which may lead to additive or synergistic toxicity, which is not currently considered in setting maximum residue limits (MRLs) (EFSA,2020). Detecting pesticide residues in fruit and vegetables worldwide is a significant public health concern, as these food items form a substantial part of the human diet, especially in populations that adhere to healthy eating guidelines. Therefore, it is crucial to monitor pesticide residues in fruits and vegetables and develop effective strategies for reducing consumer exposure (Gilden and Sattler 2020). The main objective of fruit and vegetable processing is to provide consumers with healthy, safe, nutritious, and acceptable food (Harris *et al*.,2023). Food processing refers to those methods or techniques applied to raw ingredients to transform them into a consumable form. Food processing also includes preparing basic raw materials such as washing, peeling, and trimming (Keikotlhaile *et al*., 2020).

A few studies focused on specific food processing techniques, such as canning, frying, juice, peeling, blanching, canning, concentrating, cutting and washing, and their effects on pesticide residues in plant products (Geetanjali and Naik 2019). Washing different fruits and vegetables using water and some detergents showed a decrease reached 0 - 90. (Wasilewski et *al* .2022). Moreover, the effects of juice extraction on fruits and vegetables depended on the preparation method before extraction (Holldna *et al*., 1994). For example, peeled cider contains less pesticide residue than whole cider (Kamonrat *et al* .,2022). At the same time, the blanching process efficiency in reducing pesticide residuals depends on the time, temperature, degree of humidity loss and whether the system is closed. The heat involved in cooking was said to increase waste degradation and volatility rates. The loss of stable compounds in heat was expected to be low or increased due to moisture loss during cooking. Fruits and vegetables were blanching (Hong *et al*., 2017). Studies showed a decrease in pesticides ranging from 0 to 100%.

Okra, grapes, dates, and tomatoes are crops widely consumed globally and are known to be susceptible to various pests, necessitating the use of pesticides. However, the levels of pesticide residues in these fruits and vegetables and the potential risks they pose to consumers are not fully understood (Ssemugabo et al .2020).

Studies' primary focus has predominantly been the development of effective methods for reducing pesticide residue on fruits and vegetables. Most participants utilized washing procedures, including tap water and reported favorable results with the washing process. Therefore, this is the first study that aims to trace the presence and stability of systemic and non-systemic pesticide residues affected by different steps of different food processing methods such as washing, peeling, blanching, drying, concentration and treatments with alkaline and sodium meta-bisulfate in various commonly consumed fruit and vegetables (okra, grapes, dates, and tomatoes) or their final products.

Materials and methods

Chemical and reagents

The organic solvent acetonitrile HPLC grade, citric acid, acetic acid, sodium hypochlorite, magnesium sulfate, sodium metabi-sulfate, and sodium hydroxide were purchased from Sigma Aldrich Chemical Co. St. Louis, MO, USA. The technical standards of the pesticides used were Promide EC and Extra 250 SC sprayed on grapes purchased from the Advanced Company for Agricultural Pesticide Industry in Jordan. Flash pesticide 10% EC purchased from the company Tagros Chemicals India. Rival 722 SL was purchased from AGRIA/Bulgaria (Figure 1).

Sampling Collection

The procedure for sample collection has been meticulously devised to ensure an inclusive representation of pesticide residues in four types of fruit and vegetables (Table 1) that are treated with different pesticides: Okra, Gold Coast variety (Promite EC, Pyridaben 150 gm W/V), grapes, Thompson Seedless variety (Amistar 250 SC, Azoxystrobin 250g/l), dates, Al-Barahi variety (The Flash 10 EC, Alpha-cypermethrin 10%), and tomatoes, Father-1006 variety (The fungicide RIVAL 722 SL, Propamocarb hydrochloride 722 g/l) as fresh or after processing. Pesticides have been applied to these fruit and vegetables as follows:

Table (1). Physical and chemical properties of pesticides.

Parameter	Pyridaben	Azoxystrobin	Alpha-cypermethrin	Propamocarb hydrochloride
Chemical formula	$C_{19}H_{25}CIN_2OS$	$C_{22}H_{17}N_3O_5$	$C_{22}H_{19}Cl_2NO_3$	$C_9H_{21}ClN_2O_2$
90]\f’*gPesticide type	Insecticide, Acaricide	Fungicide	Insecticide	Fungicide
Mode of action	Non-systemic	Systemic	Non-systemic	Synthetic
Safety periods (days)	14	14	21	7
Solubility in water at 20 °C mg/L	0.022	6.7	0.004	1005000
Solubility in organic solvents at 20 °C mg/L	333000	57	596000	40
Melting point (°C)	109.4	116	82.1	64.2
Boiling point (°C)	Decomposes before boiling	360	Decomposes before boiling	Decomposes before boiling

Degradation point(°C)	200	345	248	150
Aqueous hydrolysis DT_{50} (days) at 20 °C and pH 7	Stable at 5 to 9 pH at 25 °C	Not pH sensitive, hydrolytically stable	Sensitive to pH: stable at pH 4 to pH 9 & 25 °C	Stable at pH 4 to pH 9, at 25 and 50 °C

Data were mentioned, according to (Lewis *et al.,* 2016).

Samples (ranging between 20 to 30 kg for each type) were collected according to the method of Eman, (2018) from distinct agricultural regions in Baghdad and Salah al-Din governorates farms, Iraq, during the summer of 2022. Samples of commonly cultivated varieties were obtained for each of the four crops. The selection of these varieties was predicated on their widespread cultivation and susceptibility to pests, which typically necessitates the application of pesticides. The acquisition procedure involved sampling at different intervals post-pesticide application. Initial samples were procured before any pesticide treatment, serving as a baseline. Subsequent samples were obtained after the safety period of post-pesticide spraying treatments, which ranged between 2 weeks for okra and grapes, 3 weeks for dates and 7 days for tomatoes. In every instance, the procedure for sample collection was executed adhering to rigorous protocols to avert contamination and guarantee the integrity of the samples. The gathered samples were then readied for the ensuing stages of pesticide residue analysis and evaluation of processing methodologies.

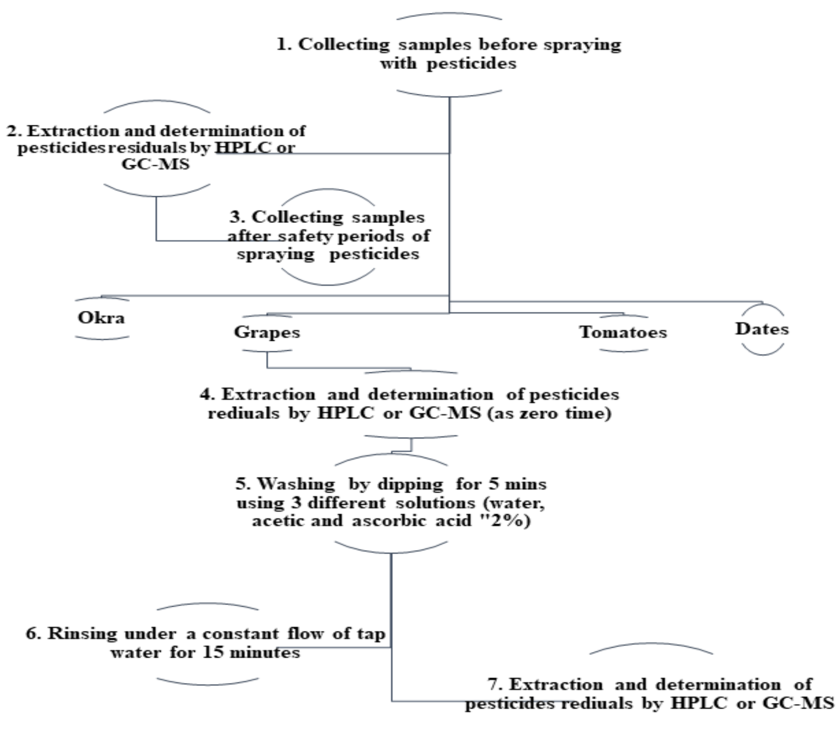

Figure (1): The study plan's flow chart begins with collecting samples till the washing treatments.

Preparation of washing solutions

Three different types of washing solutions were used to treat fruit and vegetables. The solutions comprised chlorinated filtered water using sodium hypochlorite, 2 % acetic acid (AA) and 2 % citric acid (CA). The chlorinated filtered water was used as diluents for all the washing solutions. The fruit or vegetables were washed by dipping them into a washing solution for 2 min (1kg fruit for 3L of washing solution), then rinsing them under a constant tap water flow for 15 min.

Processing of fruit and vegetables

The study evaluated the impact of various steps of common processing methods on the levels of pesticide residues in the sampled fruit and vegetables, such as okra, grapes, dates, and tomatoes. These methods encompass freezing, drying and concentration at high temperatures, as shown in Figures 1 and 2.

The processing methods were utilized as follows: the fresh okra was processed according to (Falade and Omojola 2010) as follows; sorted, washed, drained, and blanched in hot water for three minutes at 97°C (20:1 water to vegetable ratio, no agitation). Fresh okra pods were then cooled with cool water and packaged. The fresh grape was dried using conventional drying according to (Pahlavanzadeh *et al*., 2001) with some modifications, as shown in Figure 2, after alkaline treatment at 50°C for 36 h. Also, according to (Song *et al*., 2011), solar drying was used at 35°C for 60 h. Tomato sauce and dates molasses were prepared according to (Karaman and Kayacier 2011). reaching 25% and 70% TSS, respectively.

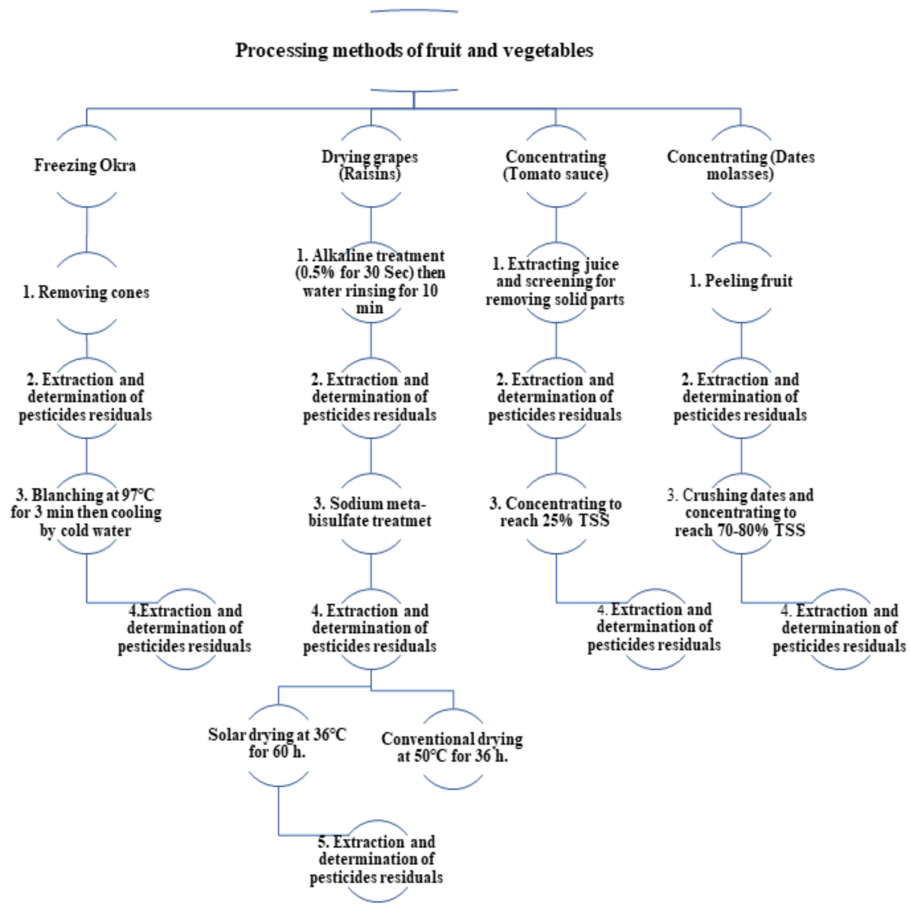

Figure (2). Flow chart of fruit and vegetable processing methods steps.

Pesticide Residuals Analysis

The analysis of pesticide residues in the collected samples was performed using a multi-residue method based on QuEChERS (Eman,2018). The pesticide extraction began with homogenizing the samples 50 g of each sample in the blender with 100 ml of acetonitrile (ACN). The mixture was mixed for 5 minutes at 3000 rpm and left for one minute to allow deposition. After vigorous shaking and centrifugation, the supernatant was collected and cleaned using Primary Secondary Amine (PSA) and Graphitized Carbon Black (GCB) to remove unwanted matrix components. The cleaned extract was analyzed by HPLC (SYKAM HPLC model, Germany) or GC-MS analysis (a Shimaduzu GC-2010 model, Japan). The GC-MS analyses were performed using an Agilent 7890A GC system coupled with an Agilent 5975C inert XL EI/CI MSD and the mobile phase was acetonitrile: D.W (80:20 V/V), the column used was C18-OSD (25cm * 4.6mm). The flow rate was 1 ml/min, and the detector used was UV-254nm for HPLC respectively.

The identification and quantification of the pesticide residues were achieved by comparing the retention times and mass spectra of the peaks in the sample chromatograms with those of the reference standards. The limit of detection (LOD) and quantification (LOQ) for each pesticide were determined based on the signal-to-noise ratio of 3:1 and 10:1, respectively.

Results and discussion

Effect of washing treatments on pesticide residuals

Table 2 and Figure 3 show the analysis of GC-Mass and HPLC, revealing the erosion of pesticides sprayed on okra, grape, dates and tomato samples due to the impact of washing treatments performed on samples using water only or with 2% concentrated citric and citric acid solutions. According to (Bonnechère *et al*., 2012), washing can help minimize pesticide residues loosely adhered to commercial surfaces. Pesticide molecules, ambient factors (such as the pH of the washing solution), kinds, and portions of the commodities all affect where pesticide residues are found on product surfaces (Balkan and Yılmaz 2022). Therefore, the obtained results showed that the treatment with CA effectively reduced pesticide residuals, followed by AA, then water. The reduction % in okra reached 85.90 % in the CA treatment and 83.25%, respectively, and 81.97% in AA and water treatments. The same trend was shown in all treated fruit and vegetables, which reached 52.35, 40.65 and 24.88% in treated grapes, 61.56, 39.80 and 10% in treated dates and 90.48, 61.54 and 9.25% in tomatoes.

Moreover, the CA treatment greatly affected systemic (Pyridaben and Alpha-cypermethrin and non-systemic pesticide residuals (Azoxystrobin and Propamocarb hydrochloride) compared to other treatments. It may be due to the difference in the pH of CA and AA because the pH of CA reached 2.1. However, the pH of AA was 3.4 and 7 for water, which significantly affects the solubility and decomposition of pesticide residuals and increases its reduction % at low pH. Otherwise, their stability on slight acidic and alkaline pH, as presented in Table 1 (Sunil and Singh 2020). Therefore, (Balkan and Yılmaz 2022) stated that soaking potatoes in acidic solutions rather than other washing solutions was more successful at removing organochlorine pesticides. Contrarily, (Ochir *et al.,* 2021) found that acetic acid was the most efficient reagent for eliminating pesticide residues from the samples, with a 44–70% clearance rate. In general, pesticides can be removed from plants more efficiently through soaking with acid, alkali, and/or salt than just water. Pesticides can react with acid or alkali to generate adducts or go through hydrolysis, but the composition of these metabolite(s) has not been explored simultaneously. As a result, it is advised that future research in this field focus on both the synthesis of metabolites and the decrease of pesticide residues.

Our results are in accordance with (Polat and Tiryaki 2020) and (Heshmati *et al*., 2020), who indicated that non-systemic pesticides were more efficiently removed or reduced through washing processes. That may be because the surface pesticide residues can be too easily dismissed with washing treatments for non-systemic residuals but not systemic (Polat and Tiryaki, 2020). Contact pesticides are typically applied on commodity surfaces; they do not enter the fruit pod, making them simple to remove through washing procedures. Systemic pesticides, on the other hand, seep into the fruit pods. Leaves absorb sprays of pesticides and stems before being transported through the vascular system to various plant parts. Because of this, removing systemic pesticides through washing methods is extremely challenging, if not impossible (Polat and Tiryaki, 2020).Otherwise, Soaking and rinsing are roughly comparable; however, soaking has a longer contact duration and no mechanical action to remove pesticide residues (Chung, 2017).

(Osman *et al*., 2014) deduced from their investigation the relationship between the amount of pesticide eliminated by soaking and its water solubility and K_{ow} (Octanol–water partition coefficient). Therefore, our study followed washing treatment with rinsing in running water for 15 min to provide additional benefits in reducing risks. Boulaid *et al*. (2005) revealed that removing pesticide residues in tomatoes was more effective after soaking in 20 g kg^{-1} citric acid or at 4% vinegar for 10 min, then rinsing under a constant tap water flow.

Table (2). The efficiency of washing treatment in reducing pesticide residuals in fruit and vegetables.

Samples	Pesticide	Pesticide residuals (ppm)			
		Initial residue	**T1**	**T2**	**T3**
Okra	Pyridaben	52.85	9.52	7.45	8.85
Grapes	Azoxystrobin	41.36	24.88	19.71	29.37
Dates	Alpha-cypermethrin	14.70	10.00	5.65	8.85
Tomato	Propamocarb hydrochloride	13.65	9.25	1.30	5.25

T1: samples washed with tap water, T2: samples washed with 2% citric acid, and T3: samples washed with 2% acetic acid.

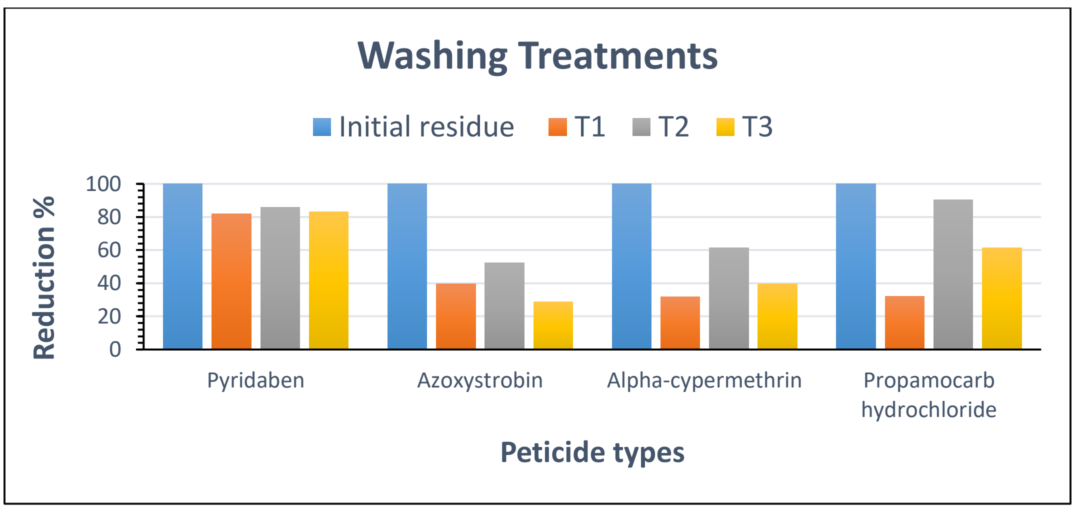

Figure (3). Pesticide residuals reduction % affected by washing treatments of fruit and vegetables. T1: samples washed with tap water, T2: samples washed with 2% citric acid, and T3: samples washed with 2% acetic acid.

Effect of processing method on pesticide residuals in fruit and vegetables:

Peeling

When processing numerous fruits and vegetables, peeling comes first. It is a helpful technique for removing some fruits and vegetables' peels or outer layers and lowering pesticide residues (Chung *et al*., 2018). In the scope of this study, it was concluded that the peeling step increases the reduction rate of pesticide residuals, as shown in Table 3. The pesticide residual values in all crops after washing treatments in okra were recorded at 9.52, 7.45 and 8.85 with the following washing treatment: T1: water, T2: CA and T3: AA. Then, after peeling (removing cones of fruit), the pesticide residuals of washed fruit were recorded 8.07, 5.98 and 7.72 inT1, T2 and T3, respectively. A reduction rate reached 15.23%, 19.73 and 12.77%. The same trend was shown with dates treated with non-systemic pesticides.

Meanwhile, the reduction rate in tomato fruit treated with systemic pesticides after peeling and screening was higher than the non-systemic and reached 54, 65 and 60 %. That may be because the bulk of pesticides and fungicides on crops directly move or penetrate the cuticle very little. As a result, these materials' remnants are restricted to the exterior surfaces, where peeling, hulling, or trimming procedures can remove them (Ahmed *et al*., 2011). When fruits or vegetables are peeled, even pesticides that have gotten into the cuticles of the produce are removed (Abou-Arab, 1999). The obtained results followed (Boulaid *et al*., 2005 and Kwon *et al*., 2015), who reported that when the skin of tomatoes was peeled, over 60% of the pesticide residues were removed.

Table (3). The efficiency of processing methods and their steps in reducing pesticide residuals of washed fruits and vegetables.

Processing method steps	**Pesticide residuals (ppm)**		
Okra	**Pyridaben**		
	T1	T2	T3
Step 1: Removing cones	8.07	5.98	7.72
Step 2: Blanching and cooling	ND	ND	ND
Raisins	**Azoxystrobin**		
	T1	T2	T3
Step 1: Alkaline treatment	20.54	11.96	17.44
Step 2: Sodium meta-bisulfate treatment	15.95	8.26	15.97
Step 3: Drying	ND	ND	ND
Dates molasses	**Alpha-cypermethrin**		
	T1	T2	T3
Step 1: Peeling	8.50	4.50	7.67
Step 2: Concentrating at high temperature	ND	ND	ND
Tomato sauce	**Propamocarb hydrochloride**		
	T1	T2	
Step 1: Juice extraction and screening	4.24	0.53	2.10
Step 2: Concentrating at high temperature	ND	ND	ND

T1: Samples washed with tap water, T2: Samples washed with 2% citric acid and T3:Samples washed with 2% acetic acid. **ND:** Means not detectable.

Moreover, all washed and peeled fruits and vegetables in our study still had a higher MRL of pesticide residuals, according to (FAO and WHO 2023), which was limited to 0.1, 1.02, 0.67 and 1.00 ppm in okra, grapes, dates and tomato.

Blanching

Blanching refers to rapidly immersing vegetables in boiling water for a brief period, typically one to two minutes. The vegetables are then promptly transferred to a bowl of ice water, commonly known as an ice bath (Chung,2017). The results presented in Table 3 demonstrate that the process of blanched okra led to a significant decrease, nearly eliminating (~100%) of pyridaben pesticide residues. That may be due to the pyridaben pesticide decomposite before boiling point degree, as shown in Table 1. According to various studies, it has been found that the blanching process is a more effective method for removing pesticide residues compared to washing with tap water. In cauliflower, blanching was able to eliminate 92% of pesticide residues, while in okra, it was able to eliminate 75% of pesticide residues. Additionally, boiling was also found to be a relatively effective method for pesticide residue removal (Kumari, 2008; Sheikh *et al*., 2012).

Similar to washing, the temperature of the water is an essential factor in the removal of pesticides. While blanching, toxic metabolites can form. However, it is necessary to note that these metabolites are typically soluble in water, meaning some may still be present in vegetables. Placing vegetables in cold water can remove metabolites before cooking. Blanching is considered the most effective household processing method for pesticide removal (Chung, 2017).

Drying and its pre-treatment

Fruits and vegetables are usually subjected to physical or chemical pretreatment before drying to shorten the drying time, reduce energy consumption and preserve the quality of final products (Yu *et al*., 2017). Drying grapes presents challenges due to a wax outer peel layer, which functions as a barrier hindering moisture transfer over the membrane. Hence, (Esmaiili *et al*., 2007) comprehensively reviewed several drying processes and pretreatment techniques. Chemical pretreatment procedures are commonly employed to facilitate the wax layer's dissolution and enhance the drying pace. Chemical pretreatment involves immersing grapes in an alkaline solution such as sodium hydroxide (NaOH) for several minutes. This method effectively dissolves the wax, decreasing the resistance to water passage across the peel (Deng *et al*., 2019).

Otherwise, sulfur has been widely used in the food industry to reduce darkening during drying and prevent quality loss during the process and storage of foods (Miranda *et al*., 2009).

The results presented in Table 3 demonstrate that the grapes' drying pretreatments could decrease the pesticide residue reduction rate, ranging from 7.44 to 40.62% in alkaline treatment and from 8.18 to 30.94 % in sulfuring treatment. Moreover, the drying process of pretreated grapes led to a significant decrease in pesticide residuals, nearly eliminating (~100%) of Amistar 250 SC. Pesticide residuals decreased during all drying processes. These results are supported by a previous study by Özbey *et al*. (2017).

Juice extraction and concentration process

Results in Table 3 indicated the ability of juice extraction to reduce Propamocarb hydrochloride pesticide residual. That refers to the nature of the pesticide: systemic, which means it could penetrate the fruit and juice extraction process, remove peels and seeds by screening, and reduce the percentage of pesticide residuals. Moreover, concentrating at high temperatures almost eliminates (~100%) pesticide residuals. The processes that generally occur in pesticides during concentration are volatilization, hydrolysis and thermal breakdown (Balinova *et al*., 2006; Stepan *et al*., 2005). These results may be influenced by the physicochemical properties of the pesticides, as presented in Table 1, which showed that the Propamocarb hydrochloride decomposed before boiling point and degraded completely at 150°C. Abou-Arab (1999) found that open-systems home canning reduced pesticide residue levels of organochlorine pesticide by heat destroying.

Conclusion

These findings underscore pesticide residues in fruits and vegetables, particularly in okra, grapes, dates, and tomatoes. This concern has far-reaching implications for both human health and the environment. Our research has shown that these commonly consumed fruits and vegetables contain pesticide residues, exceeding the Maximum Residue Limits (MRLs) set by (FAO/WHO 2023). The presence of these pesticides in our food supply calls for urgent action and stricter regulation of pesticide use in agriculture. Furthermore, provides valuable insights into the effectiveness of various processing methods steps like washing, peeling, and blanching in reducing pesticide residues. The processing steps significantly reduced pesticide residual levels. Moreover, after washing, all samples still had residue levels exceeding the MRLs, Suggesting that consumers may still be exposed to unsafe levels of pesticides. Otherwise, the processing method eliminates them in the final products (frozen okra, Raisins, dates molasses and tomato sauce) depending on the pesticides' physical and chemical properties.

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Conflicts of Interest

The authors declare no conflict of interest.

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