

**RESEARCH ARTICLE** 

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# Survey of Pesticides Residue Levels in Fresh Fruits and Vegetables across Southern **Jordanian Wholesale Markets**

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# ABSTRACT

**Article History** The use of pesticides enhances crop productivity and quality by controlling insect pests. Still, Article # 24-844 their excessive use negatively affects the environment, human health, groundwater quality, Received: 25-Sep-24 and reduces biodiversity. Pesticides, including imidacloprid can persist in the environment for Revised: 01-Nov-24 a long time, affecting air, water, and soil, consequently negatively impacting human health. Accepted: 19-Nov-24 This study evaluated imidacloprid residues in 390 samples of 13 types of fruits and vegetables, Online First: 21-Dec-24 sourced from 30 prominent wholesalers across southern Jordanian wholesale markets. Imidacloprid was selected due to its frequent use as a systemic insecticide in agricultural production, which increases the risks associated with residue accumulation. Residues were detected in 77.7% of samples, with concentrations ranging from below the detection threshold to 1.30mg.kg<sup>-1</sup>. The highest mean concentrations were observed in eggplant, apple, cauliflower, and cabbage (0.45, 0.41, 0.35, 0.30mg.kg<sup>-1</sup>, respectively), while apricots, potatoes, and grapes had the lowest concentrations. Imidacloprid was not detected in 32.3% of samples. Overall, 14.4% of samples exceeded the maximum residue limit (MRL) set by Codex, and 5.9% exceeded the Canadian PMRA standards. Furthermore, the results showed that eggplant and apple samples recorded for pesticide residues significantly exceeded Codex and PMRA MRLs. Despite some samples exceeding MRLs, the hazard index (HI) values for all samples were below unity (<1), indicating low immediate risk to consumer's health. These findings underscore the need for enhanced regulatory measures to mitigate potential health risks posed by pesticide residues in fresh produce.

Keywords: Imidacloprid, Insecticide residues, Maximum residue limit, Hazard index

# INTRODUCTION

The agricultural sector around the world faces many obstacles that threaten production, particularly in monoculture systems, such as abiotic and biotic factors, all of which declines productivity (Al-Sayaydeh et al. 2021; Samal et al., 2024; Zafar et al., 2024). These challenges often result in stunted plant growth, product damage, and additional complications (Bos & Parlevliet, 1995). Future agricultural models predict that vegetable farms and orchards will face increasing challenges due to pesticide contamination, which will negatively affect soil and water quality, and thus the health of plants and agricultural products (Linhart et al., 2021). As pest populations grow and species resistant to pesticides emerge, it is predicted that agricultural practices that rely more heavily on pesticides will be implemented (Parsa et al. 2014; Joshua, 2023). Changing environmental conditions creates more favorable conditions for notably insect and pests as well (Skendžić et al., 2021). Moreover, pest control methods in organic farming and biological control tend to be less effective under these conditions, further impacting production (Parsa et al., 2014). Therefore, developing safe pesticide usage schedules are crucial to ensure agricultural production that supports food security, especially in developing countries.

Insects are one of the largest groups in the animal kingdom, accounting for 75% of all animal species (van der Sluijs, 2020). Insects are distinguished by their exceptional behavior and their ability to reproduce and survive in striving environments, which explains their numbers that exceeds the numbers of other animals in the animal

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covers the bodies of most insects and forms an integrated system that gives them protection, adaptability, and success in surviving in the most challenging environments. Insects also have a variety of mouthparts to suit the type of food available, the most common being chewing and sucking, which contributes to competition in ecosystems (Adams et al., 2024; Tian et al., 2024). Most insects reproduce by laying eggs, quickly increasing their numbers and producing multiple generations yearly. For instance, a mature locust female can lay 100 eggs in a single pod. (Adams et al., 2024). In addition, insects cause direct and indirect damage to plants, for instance, locusts and many caterpillars larvae are so voracious that they can destroy the entire vegetation (Khairov et al., 2024; Kumar et al., 2024). On the other hand, some insects are considered vectors of dangerous viral diseases that affect plants, causing great losses in agricultural production as they lead to weakening plants and low productivity. The damage done to farmers due to viral diseases is irreparable. (Ansari et al., 2014; Adams et al., 2024).

Pesticides, particularly insecticides, represent the second-largest category of pesticide being used worldwide, following herbicides (Tian et al., 2024). Global reports indicate that pesticides consumption have reached 2.6 million tons annually, which raises concerns about their negative impacts on human health and the environment. (Guedes et al. 2024). In Jordan, the use of insecticides in agricultural fields is estimated to be 27% of the total 1,370 tons of pesticides applied (JDOS, 2017). One of the most widely used insecticides in the chloronicotinyl group is imidacloprid (C9H10CIN5O2) (Almeida Silva et al., 2024). Bayer Agro-product developed and commercialized it in the mid-nineties of the last century (Leverkusen, Germany) (Daraghmeh et al., 2007). Imidacloprid is a narrowspectrum systemic neonicotinoid primarily targeting piercing-sucking pests. It is commonly applied to cereals, corn, starchy crops, cotton, turf, vegetables and fruit orchards (Kagabu, 2011; Al-Hawadi et al., 2023). Insecticides containing imidacloprid as the active ingredient are marketed under many trade names, such as Alcador, Merit, Winner and Confidor (Alder et al., 2006; Henriques Martins et al., 2024). It degrades in soil within 60-90 days and in plants within 30 days or less to various derivatives (Akoijam & Singh, 2014; Yang et al., 2024). However, A previous study reported that imidacloprid concentrations did not decline after a long period in soil, with residues of the pesticide being found to be nearly constant after a year of application. Consequently, repeated use of these pesticides will pose a great danger to humans, as plants will absorb pesticides in larger quantities than expected from the soil, leading to their accumulation in edible parts (Cox, 2001). Moreover, due to its high-water solubility and low rate of degradation and dissipation, imidacloprid is a pesticide capable of reaching groundwater (Cox, 2001; Yang et al., 2024). An earlier investigation indicated that the solubility of imidacloprid was 500 mg/L, and this value varies depending on the concentration of the initial solution used and the type of surrounding soil (Zhou et al., 2006).

Restrictions on pesticide residues in agricultural products in wholesale markets in Jordan are crucial due to the widespread use by farmers in controlling insects that feed on various plant parts (Al-Hawadi et al., 2023). Furthermore, despite its effectiveness against many pests, it is a long-lasting systemic insecticide with recognized risks to humans, and studies have shown that many fresh edible agricultural products contain contaminated residues of imidacloprid. (Kagabu, 2011; Aralimarad et al., 2024). Therefore, recording high levels of imidacloprid concentrations in agricultural commodities requires monitoring and following up on these residues in freshrow products to preserve human health from the liability associated with consuming products contaminated with imidacloprid. (Philippe et al., 2021: Aralimarad et al., 2024). Consequently, international agencies, particularly FAO and WHO, have assessed pesticide residues, reevaluated the hazards of pesticide use, and developed guidelines and regulations to reduce them and thus determine safe thresholds of pesticide contaminants in agricultural products (Jallow et al., 2017; Algharibeh & AlFararjeh, 2019). Concerns have been raised about reports that high concentrations of imidacloprid are toxic to wild pollinators, including honey bees. It also has direct toxicity and health effects in humans, where it can cause chronic diseases when accumulated in food and water. (DiBartolomeis et al., 2019). Symptoms of oral exposure to the pesticide include headache, vomiting, and diarrhea. High doses may affect the nervous and immune systems and prolonged exposure can cause tremors and thyroid damage. (DiBartolomeis et al., 2019; Pang et al., 2020).

The need for further investigation across various agricultural commodities and locations in Jordan is heightened. A study by Al-Hawadi et al. (2023) tested 300 fresh products of fruits and vegetables in central parts of Jordan, revealing contamination in 40% of the samples, with the highest levels recorded in eggplant (0.40mg.kg<sup>-1</sup>) followed by apples (0.25mg.kg<sup>-1</sup>). Similarly, Algharibeh and AlFararjeh (2019) reported residue levels in hot peppers, sweet peppers, and zucchini that exceeded maximum residue limits (MRLs). In another study, 16 different plant products were analyzed and residues of 14 pesticides were detected at concentrations above the recommended limits. 19 samples were contaminated with imidacloprid, with the highest value recorded in cucumber at 1.2mg.kg<sup>-1</sup> (Philippe et al., 2021).

Although imidacloprid is used in Jordanian fields and farms against a wide range of insect pests, there is insufficient information about the extent of its residual effects in fruits and vegetables that are consumed fresh. Accordingly, this study aimed to identify and analyze the presence of imidacloprid residues in raw agricultural products to assess the potential risks to consumer health. Consequently, the study will help to accurately identify cases of imidacloprid contamination and detect products that contain concentrations exceeding safe limits, ultimately contributing to overcoming the challenge of managing unsafe agricultural practices, which will have positive consequences on food safety.

#### MATERIALS & METHODS

#### **Reagents and Standard**

Table 1 lists the main chemicals and solvents used to analyze imidacloprid residues in different vegetables and fruits in this study.

Table 1:	Chemicals	and	solutions	used	in	analy	sis
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Chemicals and reagents	Chemical	Source
	formula	
6-Chloronicotinic acid (99%)	$C_6H_4CIN_3O_2$	Sigma-Aldrich, Germany
Sodium hydroxide (97 %)	NaOH	Sigma-Aldrich, Germany
Anhydrous sodium sulfate (99 %)	Na₂SO₄	Sigma-Aldrich, Germany
Potassium permanganate (99 %)	KMnO₄	Sigma-Aldrich, Germany
Sodium-bisulfite	NaHSO₃	Sigma-Aldrich, Germany
t-butylmethyl ether (99 %)	$C_5H_{12}O$	Sigma-Aldrich, Germany
Amberlite XAD-4 (20–60mesh)	-	Sigma-Aldrich, Germany
N-methyl-N-(trimethylsilyl)	C6H12F3NOSi	Sigma-Aldrich, Germany
trifluoroacetamide		
Methanol (99.9 %)	CH₃OH	Sigma-Aldrich, Germany
acetonitrile (99.9 %)	C₂H₃N	Merck, Germany
imidacloprid Standard reference	-	Bayer, Germany
Nitrogen and Helium gases (99.99	N <sub>2</sub> , and He	LPG Filling Gas Station,
%)		Jordan
organic-free deionized water	DI. H₂O	Riedel de-Hean, Germany

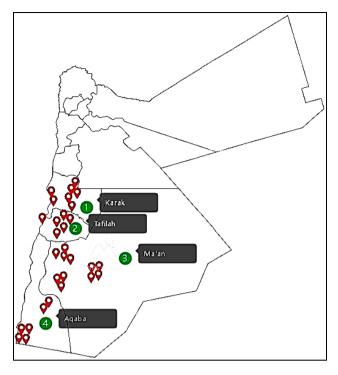
#### **Fresh Fruit and Vegetable Sampling**

The analysis of imidacloprid traces in staple crops such as apples, grapes, figs, watermelons, cantaloupes, tomatoes, cucumbers, potatoes, eggplants, zucchinis, cauliflowers, cabbages, and sweet peppers is of paramount importance. This ensures the safety of agricultural products and protects consumers from potential health risks associated with pesticide concentrations exceeding permissible levels while also meeting safety standards. To achieve this goal, 390 samples of fresh fruits and vegetables were randomly collected from 30 wholesale vegetable markets in four governorates in the south (Karak, Tafilah, Ma'an, and Agaba) during the period from July to October 2023, as the demographic distribution and food consumption patterns of the population of these different cities are similar Fig. 1. Samples were collected in polyethylene bags (1kg each) and transported to the laboratory for analysis in refrigerated boxes (4°C) to prevent deterioration and drying of the fruits.

# Sample Preparation and Analytical Method

Gas chromatography/mass spectrometry (GC/MS) was employed to detect imidacloprid. This approach is widely used for the identification and quantification of pesticides and is ideal for detecting volatile or semi-volatile substances that evaporate easily and have high sensitivity and selectivity for different compounds. (Radevski, 2024; Tufan-Cetin & Cetin, 2024).

As previously described, Subsamples of fruits and vegetables were prepared by cutting with a knife, homogenized, and weighed before immersing them in a solvent combination of methanol: water (75% v/v, 300mL) (Hu et al., 2019). Solvent extraction and phase separation were performed according to the method of Al-Hawadi et al. (2023), followed by centrifugation to obtain the extract, and ended with clean-up and filtration. After that, concentrated aliquot was used to increase the amount of pesticide residues to increase the detection sensitivity.



**Fig. 1:** Locations of vegetable and fruit markets from which samples of vegetables and fruits were collected in four governorates (Karak, Tafilah, Ma'an, and Aqaba).

Imidacloprid residues (Particularly, 6-chloropicolyl moiety) were obtained in 1mL of metabolite extract and oxidized to 6-chloronicotinic acid as per the steps mentioned by Placke and Weber (1993). The imidacloprid residues (Particularly, 6-chlorobic group) were isolated in 1mL of the metabolite concentrate and oxidized to 6-chloronicotinic acid following Placke & Weber (1993) procedure. After that, 100mL of water was used to dilute the concentrate. followed by the addition of an oxidizing agent incorporating 32% NaOH and KMnO<sub>4</sub>. The solution was refluxed and stirred for 5 minutes, then 50mL of water was added to the samples. The samples were cooled for 10 minutes to 15°C with continuous agitation in an ice bath.

50mL 10% sulfuric acid and 3g of solid sodium bisulfite were added under cooling and continuous stirring conditions to keep the pH around  $\leq$ 1. Additional sulfuric acid was added as needed. To obtain the final extract 150mL of t-butyl ether was used, and was dried in an evaporator after filtration of the organic phase applying 30g of anhydrous sodium sulfate, after which the extract was dissolved in 2mL of acetonitrile 250µL aliquot of the solution to derivatize to trimethylsilyl ester of 6-chloronicotinic acid by mixing it vigorously with N-methyl trimethyl silyl trifluoroacetamide (MSTFA). Then, 1µL of the aliquot was injected into the GC/MS instrument in non-splitting mode.

Standard solutions of 6-chloronicotinic acid and their aliquots were created and injected as described by Al-Hawadi et al., (2023). The analytical method was validated according to Al-Hawadi et al. (2023), with findings that align with those stated by Placke and Weber (1993). The standard method detection limit (MDL) Was revealed to be in the range of 0.015 and 0.030mg.kg<sup>-1</sup>, as determined through various dilution levels (using the same method as

described above, with the triple standard deviation of the blank sample generated). Additionally, spike recovery was observed to be within the 74–106% range.

To detect imidacloprid residues in the samples selected for the study, an Agilent 6890 Series II GC was used, supplied with an auto-sampler injector (7683 Series) and a mass-selective quadrupole detector, along with a DB-5 capillary column. Helium gas (99.999%) was used as the carrier gas, at a flowing rate of 1mL.min<sup>-1</sup>. Samples of 1 $\mu$ L volume were prepared for sample injection and analyzed in the splitless mode at 260°C. The oven temperature cycles were performed as described by Al-Hawadi et al. (2023). For the estimation of 6-chloronicotinic acid trimethylsilyl ester, the monitored ion was set to 214, and the qualifying ion to 170.

#### **Estimation of Residue Levels Hazard Index**

Risk assessment comprises monitoring exposure to pesticides in edible goods and comparing samples to estimated amounts of those pesticides permitted for daily content in consumed foods. This method is important in determining the amount of plant products that a human can consume without endangering their health referred to as the acceptable daily intake (ADI), Which was agreed upon as 60µg.kg<sup>-1</sup> bw<sup>-1</sup> day<sup>-1</sup> for imidacloprid according to Codex (2005). To determine the concentrations of imidacloprid residues in the studied samples, the arithmetic mean of all results was employed. In addition, according to the principles of the relevant international institutions, the estimated daily intake (EDI) of imidacloprid residues was determined using the following equation: (WHO, 1997; Nazeehet al., 2024):

$$EDI = \frac{\Sigma C \times F}{D \times W}$$

Where C Stands for the Imidacloprid mean level in each commodity ( $\mu$ g kg<sup>-1</sup>), A expresses the average annual consumption of each product for each individual (kg), D represents the number of days in a year (365) and W Signifies the average body mass used according to international guidelines, which is 60kg.

A hazard index (HI) was calculated to obtain a longterm assessment of the risks associated with imidacloprid residues as described previously (Al-Hawadi et al., 2023):

$$H = \frac{EDI}{ADI} \times 100\%$$

EDI expresses Estimated Daily Intake and ADI represents Acceptable Daily Intake.

#### **RESULTS AND DISCUSSION**

#### **Imidacloprid Residues in Raw Produce**

The concentrations of imidacloprid residues in the analyzed fruits and vegetables are presented in Table 2. The detection frequencies showed wide variation among the 390 tested samples, ranging from 0–100 %, throughout the 30 analyzed samples for each commodity. Imidacloprid concentrations in the studied raw product samples ranged from the Lower Limit of Quantification to 1.30mg.kg<sup>-1</sup>.

The highest mean concentrations were in eggplant, apples, cauliflower, and cabbage (0.045, 0.41, 0.35, and

0.30mg.kg<sup>-1</sup>, respectively). In contrast, the lowest values were found in apricots, potatoes, and grapes (0.04, 0.06, and 0.10mg.kg<sup>-1</sup>, respectively). Although imidacloprid residues were detected in most of the samples, they were not detected in several samples, particularly in potatoes (60%), apricots (50%), and grapes (40%) of the analyzed products.

Table 2:	Mean residues of imidacloprid, range values, and percentage of				
samples detected in 13 raw fruit and vegetable products					

Fruit/vegetable	Mean±SD	Range	Samples with	% of samples
	(mg⁻¹)	(Min-Max)	detectable	with detectable
			residues	residues
Eggplant	0.45±0.32	0.04-1.30	30	100.0
Apple	0.41±0.25	0.00-0.83	24	80.0
Grape	0.10±0.10	0.00-0.32	18	60.0
Apricot	0.04±0.06	0.00-0.23	15	50.0
Watermelon	0.17±0.17	0.00-0.56	20	66.7
Muskmelon	0.19±0.16	0.00-0.61	24	80.0
Tomato	0.19±0.20	0.00-0.63	20	66.7
Pepper	0.15±0.13	0.00-0.42	21	70.0
Potato	0.06±0.10	0.00-0.34	12	40.0
Cucumber	0.24±0.15	0.05-0.62	30	100.0
Zucchini	0.20±0.15	0.00-0.57	27	90.0
Cauliflower	0.35±0.15	0.10-0.62	30	100.0
Cabbage	0.30±0.06	0.16-0.42	30	100.0
		Total	301	77.2

LOD of imidacloprid (0.03mg.kg<sup>-1</sup>). Values (mean+SD) are of n=30/fruit. Total samples=390.

This variation in imidacloprid values between samples of the same product or between different fruits and vegetables may be explained by differing agricultural practices, including insecticide application methods or the accumulation of imidacloprid in the soil over consecutive seasons (Al-Hawadi et al., 2023; Wang et al., 2023). Previously, studies have reported high concentrations of imidacloprid residues in several agricultural products such as eggplant, watermelon, apple, and tomato, which are consistent with the results of the current study (Algharibeh & AlFararjeh 2019; Philippe et al., 2021). On the contrary, a recent study on apples showed that the use of safe pesticide applications, including imidacloprid, ensured that harmful residues remained below regulatory limits, underscoring the importance of adopting best practices to mitigate health risks and maintain environmental safety (Wang et al., 2023). In Jordan, some farmers prefer to use imidacloprid repeatedly during the same agricultural season to control many pests such as the flat-headed codling moth and the whitefly to ensure the quality of agricultural products. This explains the presence of high concentrations in some products while they are not detected in other products of the same type (Algharibeh & AlFararjeh 2019; Al-Hawadi et al., 2023). Furthermore, it has been reported that imidacloprid has a degradation period that varies from one crop to another depending on the concentration at the time of application and requires 10 to 30 days after application to be within the safe range (Wang et al., 2023; Khalil et al., 2024). On the other hand, imidacloprid residues were found to have a greater environmental impact on soil and were reported to be more stable in soil than agricultural products, with a halflife range from 40-174 days (Işıldak et al., 2024; Thekkumpurath et al., 2024).

# Imidacloprid Maximum Residual Limits in Fruits and Vegetables

Rising temperatures associated with climate change, coupled with intensive agriculture, have disrupted ecosystems and made environmental conditions more conducive to pest infestation. Although irrigation and fertilization are essential to boost productivity, overirrigation can increase plants' susceptibility to pests and diseases (Chen & Liu, 2023). As a result, there has been an increased focus on the use of pesticides to control the huge numbers of harmful endemic pests, as a result, excessive use has played a fundamental role in environmental pollution and the subsequent serious health risks to humans (Islam et al., 2017). In the 1960s, the Food and Agriculture Organization (FAO) and the World Health Organization (WHO) jointly established the Codex Alimentarius Commission to formulate standards governing maximum residue limits for food products at the global level. (Codex, 2005; Kabbashi, 2024). In addition to the efforts of international institutions, some countries have taken up the same mission such as the Canadian Pest Control Regulatory Agency (PMRA) implements these standards (2005). This study shows Imidacloprid maximum residue limits in 13 different fruits and vegetables crops following Codex (2005) and Canadian Pest Management Regulatory Agency (PMRA, 2005) Table 3.

**Table 3:** Number of samples with levels exceeding the maximum residue

 limit for imidacloprid in 13 different fruit and vegetable products

Fruit/vegetables	Codex	Samples with	Canadian	Samples with
	MRL	residue < Codex	MRL	residue <
		MRL		Canadian MRL
Eggplant	0.2	7	1.0	28
Apple	0.5	19	0.6	22
Grape	1.0	30	1.5	30
Apricot	0.5	30	3.0	30
Watermelon	0.2	17	0.5	29
Muskmelon	1.0	30	0.5	28
Tomato	0.5	26	1.0	30
Bell Pepper	1.0	30	1.0	30
Potato	0.5	30	0.4	30
Cucumber	1.0	30	0.5	28
Zucchini	0.7	30	1.0	30
Cauliflower	0.5	25	0.5	25
Cabbage	0.5	30	0.3	17

\* MRL (Maximum Residue Limits) in mg.kg<sup>-1</sup> according to Codex standards (2005) and Canadian regulations.

Of the 390 samples analyzed, 56 (14.4%) exceeded the Codex maximum residue limit for imidacloprid, while the number of samples exceeding the PMRA maximum residue limit was less, with 23 (5.9%) (Table 3). As shown in Table 3, the fruit and vegetable samples showed a significant variation in imidacloprid residue concentrations, with the highest concentrations recorded in eggplant, apple, cauliflower, and watermelon (1.20, 0.76, 0.62 and 0.61 mg.kg<sup>-1</sup>, respectively). Based on the Codex and Parma standards, the imidacloprid concentration in eggplant was significantly above the permissible limits. In contrast, the concentrations in grapes, apricots, potatoes, zucchini, and sweet pepper were around or below the recommended limits, meaning that they were below the hazardous levels. In the case of watermelon, cucumber, and cabbage, all samples were within the safe levels according to the Codex; however, 13 cabbage samples exceeded the maximum residue limits set by the Canadian Pest Control Regulatory Agency (PMRA).

Few studies have been conducted on the contamination of fresh fruits and vegetables with imidacloprid in the Middle East, despite its widespread use in many countries in the region it has not been included in concrete monitoring programs to determine its impact on life on Earth (Gaber & Ali, 2023; Rasool et al., 2024). A previous study of 200 samples of fruits and vegetables analyzed for residues of various pesticides showed that 25-53% of samples were identified as contaminated with at least one pesticide, and imidacloprid is among the common pesticides in many of these crops (Contreras & Garrido, 2000). In a comparison of the results of the current study with previous research, pesticide residues are substantially present in agricultural products, which rings the alarm bell for increasing awareness in understanding maximum residue limits (MRLs) and their role in determining pesticide levels that products (Keikotlhaile & Spanoghe, 2011; Sun et al., 2023).

The interest of organizations in adopting maximum residue limits for pesticides in food indicates their importance in preserving human health by ensuring their safety for consumers and reducing them to a minimum (Rosa et al., 2024). However, the results of many studies on pesticide residues in some products, such as imidacloprid, have shown high concentrations of residues. For example, some studies have recorded worrying results for eggplant and apples, which recorded concentrations higher than those recommended by Codex and PMRA standards (Al-Hawadi et al., 2023).

The presence of residues above the maximum residue limits in several samples suggests possible overuse or improper application of imidacloprid, which necessitates stricter control of agricultural practices among some farmers. Furthermore, excessive use of imidacloprid, like other pesticides, can lead to pest tolerance and will make it difficult to control some insect pests in the future. (Damalas & Eleftherohorinos, 2011; Alvarez et al., 2024).

# **Risk Estimation for Imidacloprid Intake**

Monitoring pesticide residues in food requires adherence to approved hazardous levels to protect consumer health (Aralimarad et al., 2024). Therefore, the potential serious impact of exposure to imidacloprid residues on human health was evaluated, as presented in Table 4. Acceptable Daily Intakes (ADIs) established by the FAO/WHO were compared to the Estimated Daily Intake (EDI) according to the Jordanian Department of Statistics to estimate the harmful effects of imidacloprid residues resulting from the consumption of various fruits and vegetables.

The results indicate that imidacloprid concentrations in fruit and vegetable samples were below the ADIs, demonstrating that exposure levels remain within safe limits for humans (Table 4). Eggplant and apple samples showed the highest exposure values (448 and 409 µg.kg<sup>-1</sup>, respectively), followed by cauliflower, cabbage, cucumber, and zucchini (350, 297, 241, and 200 µg.kg<sup>-1</sup>, respectively). Muskmelon, tomato, watermelon, bell pepper, and grape recorded lower levels (191, 185, 171, 146, and 101 µg.kg<sup>-1</sup>, respectively). Notably, potatoes and apricots had exposure

 Table 4: Mean imidacloprid exposure values, annual fruit intake (F), estimated daily intakes (EDI), and hazard index (HI) of imidacloprid in different analyzed samples

Fruit/vegetables	Mean exposure	F (kg person	EDI (µg.kg <sup>-1</sup>	Hazard
	values (µg.kg⁻¹)	<sup>-1</sup> yr <sup>-1</sup>	bw day⁻¹)	index (%)
Eggplant	448	6.58	0.135	0.157
Apple	409	7.37	0.138	0.161
Grape	101	2.87	0.013	0.015
Apricot	41	1.7	0.003	0.004
Watermelon	171	7.94	0.062	0.072
Muskmelon	191	2.44	0.021	0.025
Tomato	185	28.69	0.242	0.283
Bell Pepper	146	3.20	0.021	0.025
Potato	64	18.24	0.053	0.062
Cucumber	241	14.42	0.159	0.185
Zucchini	200	4.64	0.042	0.049
Cauliflower	350	4.26	0.068	0.079
Cabbage	297	1.73	0.023	0.027

\*F = Average annual per capita consumption of a comodity; EDI (µg.kg<sup>-1</sup> bw daily) Based on (Jordanian Department of Statistics; ADI (60µg.kg<sup>-1</sup> body weight day-1) As stated by Codex Alimentarius Commission (FAO/WHO, 2004).

levels below 65  $\mu$ g.kg<sup>-1</sup>. Without exception, imidacloprid residues were lower than the ADI based on the Codex Alimentarius Commission (FAO/WHO, 2004).

To enhance the assessment of the safety of the analyzed samples, the Hazard Index (HI) was calculated. The (HI) analysis of pesticide residues highlights the potential health risks associated with long-term consumption (Aralimarad et al., 2024). HI values ranged from 0.004 to 0.283% (Table 3). Tomatoes are the most commonly used vegetable in Jordanian cuisine, with an EDI of 0.242µg.kg<sup>-1</sup> bw day<sup>-1</sup>, which is reflected in its HI value of 0.283, considerably higher than those of other fruits and vegetables. Similarly, cucumber, apple, and eggplant are frequently used in Jordanian meals and showed high levels of EDIs (>0.150µg.kg<sup>-1</sup> bw day<sup>-1</sup>) and equivalent HI values (>0.135%). Although the present results do not indicate a human health risk associated with the concentrations of imidacloprid residues in the studied products, the high HI values suggest caution, as risks may arise from long-term use or larger quantities (Bonnechère et al., 2012; Li et al., 2024). Previous studies have shown that high Hazard Index (HI) values of pesticides in fresh agricultural products may pose cumulative risks when consumed regularly, and these findings underscore the need for continuous monitoring and regulation to ensure that pesticide residue levels remain within safe consumption limits (Trenteseaux et al., 2024; Zondo & Mahlambi, 2024). Pan et al. (2024) highlighted the growing need for further research into Hazard Index (HI) values of pesticides, which could inform more effective strategies for managing pesticide contaminants in fresh agricultural products.

Middle Eastern cuisine heavily relies on fresh fruits and vegetables, making these products indispensable in daily consumption. This study seeks to raise awareness of the potential risks posed by elevated imidacloprid residues in these agricultural products. Consequently, it has become essential to guide farmers toward correcting improper agricultural practices to produce safe food (Al-Hawadi et al., 2023). However, the lack of accurate practical information on the optimal safe harvesting periods after application of the pesticide raises questions about its effect on residue levels. (Darko & Akoto, 2008; Wang et al., 2023). The limitations of intensive imidacloprid pesticide use emphasize the need for more comprehensive and accurate research to obtain a more effective and representative assessment of its residues in vegetables and fruits.

# Conclusion

The present study aimed to evaluate the presence of imidacloprid residues in selected samples of raw fruits and vegetables collected from the southern part of Jordan. The results showed that most values were within safe limits, although some significantly exceeded them. The results showed that 77.7% of them were contaminated with residues with varying levels from 0.0-0.120µg.kg<sup>-1</sup> and several samples exceeded the maximum residue limit. It is of great importance to monitor the concentrations of this pesticide since many of these products are consumed fresh. The potential risks associated with the consumption of fruits and vegetables that have been repeatedly found to be contaminated with this pesticide raise concerns about cumulative effects as these agricultural products are almost always present in diets.

This calls for more stringent regulatory rules to establish reliable and sustainable technologies to predict potential risks of imidacloprid residues in agricultural commodities; the importance of taking care of fruit and vegetable preparation before consumption and adopting proper cleaning, peeling, and, boiling practices before consumption should be highlighted. Additionally, monitoring of imidacloprid residues in soil and water should be done in addition to agricultural products. Furthermore, farmers should be encouraged and guided to implement good agricultural practices (GAP) in the use of pesticides to mitigate potential risks to human health.

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# REFERENCES

- Adams, I., Yang, G., Asad, M., & Tariq, M. (2024). An Overview of Reproduction in Insects. Asian Journal of Advances in Agricultural Research, 24(7), 133-147. https://doi.org/10.9734/ajaar/2024/v24i7529
- Akoijam, R., & Singh, B. (2014). Metabolic degradation of imidacloprid in paddy field soil. *Environmental Monitoring and Assessment, 186*, 5977– 5984. <u>https://doi.org/10.1007/s10661-014-3833-3</u>
- Alder, L., Greulich, K., Kempe, G., & Vieth, B. (2006). Residue analysis of 500 high priority pesticides: better by GC–MS or LC–MS/MS? Mass Spectrometry Reviews, 25(6), 838-865. <u>https://doi.org/10.1002/mas.</u> 20091
- Algharibeh, G.R., & AlFararjeh, M.S. (2019). Pesticide residues in fruits and vegetables in Jordan using liquid chromatography/tandem mass spectrometry. *Food Additives & Contaminants: Part B*, 12(1), 65-73. <u>https://doi.org/10.1080/19393210.2018.1548505</u>
- Al-Hawadi, J.S., Al-Sayaydeh, R.S., Al-Rawashdeh, Z.B., & Ayad, J.Y. (2023). Monitoring of imidacloprid residues in fresh fruits and vegetables from the central parts of Jordan. *Heliyon*, 9(11), e22136. <u>https://doi.org/10.1016/j.heliyon.2023.e22136</u>
- Almeida Silva, M., Viegas, S., Curwin, B., Marder, M.E., & Schlüter, U. (2024). Exposure Science and Occupational Health: Insights from ISES 2022. Frontiers in Public Health, 12, 1515173. Doi: 10.3389/fpubh.2024.1515173
- Al-Sayaydeh, R., Al-Habahbeh, K., Akkeh, Z., & Albdaiwi, R.N. (2021). In silico gene expression analysis of the stress-related NAC-a gene subfamily to dissect their role in abiotic stress tolerance in bread wheat (Triticum aestivum L.). Jordan Journal of Agricultural Sciences, 17(3), 341-354. <u>https://doi.org/10.35516/jjas.v17i3.90</u>
- Alvarez, D.D.L., Hayashida, R., Cavallaro, M.C., Santos, D.M., Santos, L.M., Müller, C., Watanabe, L.F.M., Bello, V.H., Krause-Sakate, R., Hoback, W.W., & Oliveira, R.C.D. (2024). Susceptibility of Bemisia tabaci Gennadius (Hemiptera: Aleyrodidae) Mediterranean Populations Found in São Paulo, Brazil to 11 Insecticides and Characterization of Their Endosymbionts. *Insects*, *15*(9), 670. <u>https://doi.org/10.3390/ insects15090670</u>
- Ansari, M., Moraiet, M., & Ahmad, S. (2014). Insecticides: Impact on the Environment and Human Health. In: Malik, A., Grohmann, E., Akhtar, R. (eds) Environmental Deterioration and Human Health, pp: 99-123. Springer, Dordrecht. <u>https://doi.org/10.1007/978-94-007-7890-0\_6</u>
- Aralimarad, P., Bedar, J., Mahato, S., Hanchinal, S., Naik, N., Shwetha, U., Pavankumar, K., Naveenkumar, P., Gavai, A., Rathod, R.S. & Patil, M. (2024). Simultaneous determination of multi-class pesticide residues in solanaceous vegetables and selected fruits using LC-MS/MS and potential impact on consumer health with measurement uncertainty. *Researchsquare*. <u>https://doi.org/10.21203/rs.3.rs-5199827/v1</u>
- Bos, L., & Parlevliet, J.E. (1995). Concepts and terminology on plant/pest relationships: toward consensus in plant pathology and crop protection. *Annual review of Phytopathology*, 33(1), 69-102. <u>https://doi.org/10.1146/annurev.py.33.090195.000441</u>
- Bonnechère, A., Hanot, V., Jolie, R., Hendrickx, M., Bragard, C., Bedoret, T., & Van Loco, J. (2012). Effect of household and industrial processing on levels of five pesticide residues and two degradation products in spinach. *Food Control*, 25(1), 397-406. <u>https://doi.org/10.1016/j. foodcont.2011.11.010</u>
- Chen, M., & Liu, X.D. (2023). Estimating insect pest density using the physiological index of crop leaf. *Frontiers in Plant Science*, 14, 1152698. <u>https://doi.org/10.3389/fpls.2023.1152698</u>
- Codex (2005). Codex Alimentarius Commission: Codex Alimentarius, Codex Alimentarius: pesticide residues in food, maximum residue limits; extraneous maximum residue limits. Date accessed on May 2019: <u>http://www.fao.org/fao-who-codexalimentarius/standards/pestres/en</u>
- Contreras, M., & Garrido, J. (2000). Determination of imidacloprid and benzimidazole residues in fruits and vegetables by liquid chromatography–mass spectrometry after ethyl acetate multiresidue extraction. *Journal of AOAC International, 83*, 748–755. https://doi.org/10.1093/jaoac/83.3.748

Cox, C. (2001). Imidacloprid. Journal of Pesticide Reforms, 21, 15-21.

- Damalas, C.A., & Eleftherohorinos, I.G. (2011). Pesticide exposure, safety issues and risk assessment indicators, *International Journal of Environmental Research and Public Health*, 8(5) 1402–1419, <u>https://doi.org/10.3390/ijerph8051402</u>
- Daraghmeh, A., Shraim, A., & Abulhaj, S. (2007). Imidacloprid residues in fruits, vegetables and water samples from Palestine. *Environmental Geochemistry and Health, 29*, 45–50. <u>https://doi.org/10.1007/s10653-006-9060-2</u>

Darko, G., & Akoto, O. (2008). Dietary intake of organophosphorus pesticide

residues through vegetables from Kumasi, Ghana, Food and Chemical Toxicology, 46, 3703–3706, https://doi.org/10.1016/j.fct.2008.09.049

- DiBartolomeis, M., Kegley, S., Mineau, P., Radford, R., & Klein, K. (2019). An assessment of acute insecticide toxicity loading (AITL) of chemical pesticides used on agricultural land in the United States. *PloS one*, *14*(8), e0220029. https://doi.org/10.1371/journal.pone.0220029
- Food and Agriculture Organization/World Health Organization (FAO/WHO) (2004). Food Standards Programme. Codex Alimentarius Commission, Twenty-seventh Session, Geneva, Switzerland, 2004, 28 Junee03 July 2004.
- Gaber, A.S., & Ali, M.A. (2024). Spatial and Temporal Dynamics of Aonidiella aurantii in Orange Orchards: Implications for Control in the Subtropical Region. *Egyptian Academic Journal of Biological Sciences*. *A, Entomology*, *17*(3), 83-99. <u>https://dx.doi.org/10.21608/eajbsa.2024</u>. <u>376320</u>
- Henriques Martins, C.A., Azpiazu, C., Bosch, J., Burgio, G., Dindo, M.L., Francati, S., & Sgolastra, F. (2024). Different Sensitivity of Flower-Visiting Diptera to a Neonicotinoid Insecticide: Expanding the Base for a Multiple-Species Risk Assessment Approach. *Insects*, 15(5), 317. <u>https://doi.org/10.3390/insects15050317</u>
- Hu, K., Liu, J., Li, B., Liu, L., Gharibzahedi, S.M.T., Su, Y., Jiang, Y., Tan, J., Wang, Y., & Guo, Y. (2019). Global research trends in food safety in agriculture and industry from 1991 to 2018: A data-driven analysis. *Trends in Food Science & Technology*, 85, 262-276. <u>https://doi.org/10. 1016/j.tifs.2019.01.011</u>
- Işıldak, S., Tekin, N., Karatay, S. E., & Dönmez, G. (2024). A study on bioremoval of a neonicotinoid insecticide, imidacloprid, by a newly isolated Acremonium sclerotigenum. *Biologia*, 79(8), 2621-2628. <u>https://doi.org/10.1007/s11756-024-01750-1</u>
- Islam, M.N., Bint-E-Naser, S.F., & Khan, M.S. (2017). Pesticide Food Laws and Regulations. In: Khan, M., Rahman, M. (eds) Pesticide Residue in Foods. pp. 37-51. Springer, Cham. <u>https://doi.org/10.1007/978-3-319-52683-6\_3</u>
- Jallow, M.F., Awadh, D.G., Albaho, M.S., Devi, V.Y., & Ahmad, N. (2017). Monitoring of pesticide residues in commonly used fruits and vegetables in Kuwait. *International Journal of Environmental Research* and Public Health, 14(8), 833. <u>https://doi.org/10.3390/ijerph14080833</u>
- Jordanian Department of Statistics (JDOS) (Household Expenditures & Income Survey), Average of Annual Household Individuals Consumption from Food Commodities, 2017. <u>http://dosweb.dos.gov.jo/ar/economic/expenditures-income/expend\_tables/</u>.
- Joshua, B. (2023). Current Knowledge on Biotic Stresses affecting Legumes: Perspectives in Cowpea and Soybean. In Phetole Mangena, Sifau A. Adejumo (Eds.), Advances in Legume Research: Physiological Responses and Genetic Improvement for Stress Resistance, 2nd Volume, (pp. 14-30), Bentham Science Publishers <u>https://doi.org/10.</u> 2174/97898151653191230201
- Kabbashi, E. B. (2024). Major Contaminants of Peanut and Its Products and their Methods of Management. In: Marc, R. A., Mureşan, C. C., & Postolache, A. N. (eds) Nut Consumption and its Usefulness in the Modern World. IntechOpen, London. <u>https://doi.org/10.5772/ intechopen.1004630</u>
- Keikotlhaile, B. M., & Spanoghe, P. (2011). In: Stoytcheva, M. (Ed.) Pesticide residues in fruits and vegetables. *Pesticides—formulations, effects, fate.* pp. 243-252, BoD–Books on Demand, London.
- Kagabu, S. (2011). Discovery of imidacloprid and further developments from strategic molecular designs. *Journal of Agricultural and Food Chemistry*, 59(7), 2887-2896. <u>https://doi.org/10.1021/jf101824y</u>
- Khairov, K.S., Lazutkaite, E., & Latchininsky, A.V. (2024). Distribution, Population Dynamics, and Management of Moroccan Locust Dociostaurus maroccanus (Thunberg, 1815) (Orthoptera, Acrididae) in Tajikistan. *Insects*, 15(9), 684. https://doi.org/10.3390/insects15090684
- Khalil, R. E., Shaheen, F. A., Hady, A. E., & Saleh, A. A. (2024). Quantitative Analysis of Some Insecticide Residues Using Quechers Methodology on Pepper (Capsicum annuum) Fruits Under Greenhouse Conditions. Journal of Plant Protection and Pathology, 15(9), 257-265. https://dx.doi.org/10.21608/jppp.2024.300634.1244
- Kumar, R., Sharma, S.K., Goswami, T.N., & Ahmed, N. (2024). Morphometric Studies of Tobacco Caterpillar, [Spodoptera litura (Fabricius)] on Different Host Plants. *International Journal of Plant & Soil Science*, 36(5), 922-928. <u>https://doi.org/10.9734/ijpss/2024/v36i54587</u>
- Li, X., Yu, S., Huang, K., Zhu, W., Ye, G., Qi, J., Hu, Y., Chen, X., Wang, Z., Maimaiti, S., & Lu, S. (2024). Neonicotinoid residues in fruits and vegetables in Shenzhen: Assessing human exposure and health risks. *Chemosphere*, 364, 143267. <u>https://doi.org/10.1016/j.chemosphere.</u> 2024.143267
- Linhart, C., Panzacchi, S., Belpoggi, F., Clausing, P., Zaller, J. G., & Hertoge, K. (2021). Year-round pesticide contamination of public sites near

intensively managed agricultural areas in South Tyrol. *Environmental Sciences Europe*, *33*, 1-12. <u>https://doi.org/10.1186/s12302-020-00446-</u>

- Nazeeh, N., Radwan, E.H., Mosalam Hosin, E., & Khalifa, H. (2024). A comparative study of the effect of pesticides used in agriculture on catfish in some Nile Delta governorates (Damietta branch and Rasheed branch). *Biological and Biomedical Journal*, 2(2), 168-180. https://dx.doi.org/10.21608/bbj.2024.311031.1035
- Pan, W., Chen, Z., Wang, X., Wang, F., Liu, J., & Li, L. (2024). Occurrence, dissipation and processing factors of multi-pesticides in goji berry. *Journal of Hazardous Materials*, 473, 134696. <u>https://doi.org/10.1016/j.jhazmat.2024.134696</u>
- Pang, S., Lin, Z., Zhang, Y., Zhang, W., Alansary, N., Mishra, S., Bhatt, P., & Chen, S. (2020). Insights into the toxicity and degradation mechanisms of imidacloprid via physicochemical and microbial approaches. *Toxics*, 8(3), 65. https://doi.org/10.3390/toxics8030065.
- Parsa, S., Morse, S., Bonifacio, A., Chancellor, T. C., Condori, B., Crespo-Pérez, V., Hobbs, S.L., Kroschel, J., Ba, M.N., Rebaudo, F. & Dangles, O. (2014). Obstacles to integrated pest management adoption in developing countries. *Proceedings of the National Academy of Sciences*, 111(10), 3889-3894. <u>https://doi.org/10.1073/pnas.1312693111</u>
- Philippe, V., Neveen, A., Marwa, A., & Basel, A.Y.A. (2021). Occurrence of pesticide residues in fruits and vegetables for the Eastern Mediterranean Region and potential impact on public health. *Food Control*, 119, 107457. <u>https://doi.org/10.1016/j.foodcont.2020.107457</u>
- Placke, F. & Weber, E. (1993) Method of determining imidacloprid residues in plant materials, Pflanzenschutz-Nachrichten Bayer, 46(2), 109–182.
- PMRA (Pest Management Regulatory Agency, Canada), Maximum residue limits for pesticides, schedule No.1367 (imidacloprid). http://www.pmra-arla.gc.ca/english/pdf/mrl/part2/1367-imidaclopride.pdf, 2005. (Accessed 10 August 2016).
- Radevski, N. (2024). Systematic assessment and profiling of volatile organic chemicals in commercial shipping containers based on new material physical sorption sampling technology (Doctoral dissertation, Murdoch University).
- Rasool, K.G., Husain, M., Alwaneen, W.S., Sutanto, K.D., Omer, A.O., Tufail, M., & Aldawood, A.S. (2024). Assessing the toxicity of six insecticides on larvae of red palm weevil under laboratory condition. *Journal of King Saud University-Science*, 36, 103268. <u>https://doi.org/10.1016/j. jksus.2024.103268</u>
- Rosa, M.P.L., Gotardo, A.T., Maramarque, A.R.O., Kindlein, G., Tomaszewski, C.A., Hillesheim, D.R., da Silva, G.F., Barnet, L.S., Rau, R.B., Castilhos, T.D.S. & Górniak, S.L. (2024). The effect of breed on ivermectin residues in the edible tissues of cattle and the estimated withdrawal period. *Food Additives & Contaminants: Part A.* <u>https://doi.org/10.</u> 1080/19440049.2024.2423199
- Samal, I., Bhoi, T.K., Mahanta, D.K., Komal, J., & Pradhan, A.K. (2024). Sustainable Plant Protection Measures in Regenerative Farming. In: Kumar, S., Meena, R.S., Sheoran, P., Jhariya, M.K. (eds) Regenerative Agriculture for Sustainable Food Systems. Springer, Singapore. pp. 387-421. <u>https://doi.org/10.1007/978-981-97-6691-8\_12</u>
- Guedes, V.S., Morato, B.N., Couto, C., da Silva Pinto, J.D., Meireles, C.R.V., de Oliveira Correia, R., & Almeida, M.L.B. (2024). Environmentally Friendly Biocomposites for The Adsorption of Mancozeb Pesticide from Water. *Revista de Gestão Social e Ambiental*, *18*(10), 1-13.
- Skendžić, S., Zovko, M., Živković, I.P., Lešić, V., & Lemić, D. (2021). The impact of climate change on agricultural insect pests. *Insects*, 12(5), 440. <u>https://doi.org/10.3390/insects12050440</u>
- Sun, Y.X., Su, H.Y., Liang, C.W., Yang, H.X., Zhou, J., Zhu, W.B., & Song, X.

(2023). Resistance of Musca domestica to five insecticides in Dezhou city in 2022. *Chinese Journal of Hygienic Insecticides & Equipments*, 29(3), 213-215. https://doi.org/10.19821/j.1671-2781.2023.03.006

- Thekkumpurath, A.S., Ghotgalkar, P., Ekatpure, S., Bhanbhane, V., Pardeshi, A., & Deore, P. (2024). Assessment of degradation mechanism of imidacloprid residues in grape rhizosphere soil by UHPLC-Orbitrap<sup>™</sup>-MS and its residual impact on soil enzyme activity. *Environmental Science and Pollution Research*, 31, 3763–3774. <u>https://doi.org/10.1007</u> /s11356-023-31285-y
- Tian, Y., Yan, X., & Sun, K. (2024). Dual effects of additional food supply and threshold control on the dynamics of a Leslie–Gower model with pest herd behavior. *Chaos, Solitons & Fractals*, 185, 115163. <u>https://doi.org/10.1016/j.chaos.2024.115163</u>
- Trenteseaux, C., Fontaine, K., Chatzidimitriou, E., Bouscaillou, W., Mienné, A., & Sarda, X. (2024). Cumulative dietary risk assessment for French consumers exposed to succinate dehydrogenase inhibitor pesticides. *Food and Chemical Toxicology*, 191, 114890. <u>https://doi.org/10.1016/j. fct.2024.114890</u>
- Tufan-Cetin, O., & Cetin, H. (2024). Insecticidal potential of Cedrus libani tar in eco-friendly control of cat flea, Ctenocephalides felis, from different populations in Türkiye. *Heliyon.* 10, 21, e39958. <u>https://doi.org/10. 1016/j.heliyon.2024.e39958</u>
- van der Sluijs, J.P. (2020). Insect decline, an emerging global environmental risk. Current Opinion in Environmental Sustainability, 46, 39-42. https://doi.org/10.1016/j.cosust.2020.08.012
- Wang, Z., Huang, W., Liu, Z., Zeng, J., He, Z., & Shu, L. (2023). The neonicotinoid insecticide imidacloprid has unexpected effects on the growth and development of soil amoebae. *Science of The Total Environment*, 869, 161884. <u>https://doi.org/10.1016/j.scitotenv.2023. 161884</u>
- WHO (World Health Organization), Guidelines for predicting dietary intake of pesticide residues (revised), Global Environment monitoring System- Food Contamination and Assessment Programme (GEMS/Food) in, WHO/FSF/FOS/97.7, collaboration with Codex Committee on Pesticide Residues, 1997, pp: 40–60. <u>https://iris.who.int/bitstream/handle/10665/63787/WHO\_FSF\_FOS\_97.</u> <u>7.pdf?sequence=1&isAllowed=y</u>
- Yang, D., An, Z., Huo, J., Chen, L., Dong, H., Duan, W., Zheng, Y., Wang, M., He, M., Gao, S. & Zhang, J. (2024). Ultrastable Cobalt-Based Chainmail Catalyst for Degradation of Emerging Contaminants in Water. *Applied Catalysis B: Environment and Energy*, 363, 124768. <u>https://doi.org/10. 1016/j.apcatb.2024.124768</u>
- Zafar, M.M., Razzaq, A., Chattha, W.S., Ali, A., Parvaiz, A., Amin, J., Saleem, H., Shoukat, A., Elhindi, K. M., Shakeel, A., Ercisli, S., Qiao, F., & Jiang, X. (2024). Investigation of salt tolerance in cotton germplasm by analyzing agro-physiological traits and ERF genes expression. *Scientific Reports*, 14(1), 11809. <u>https://doi.org/10.1038/s41598-024-60778-0</u>
- Zhou, Q., Ding, Y. & Xiao, J. (2006). Sensitive determination of thiamethoxam, imidacloprid and acetamiprid in environmental water samples with solid-phase extraction packed with multiwalled carbon nanotubes prior to high-performance liquid chromatography. *Analytical and Bioanalytical Chemistry*, 385, 1520–1525. <u>https://doi.org/10.1007/s00216-006-0554-7</u>
- Zondo, S., & Mahlambi, P. (2024). Comparison of soxhlet and microwave-assisted extractions efficiency for the determination of herbicides in soil and maize crop: Cumulative and health risks assessment. eFood, 5(4), e177. <u>https://doi.org/10.1002/efd2.177</u>