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MONITORING AND RISK ASSESSMENT OF PESTICIDE RESIDUES IN SOME LOCALLY FRUITS PRODUCED IN MINIA GOVERNORATE, MINIA, EGYPT

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ABSTRACT

The objective of this research was to monitor the presence of pesticide residues in grapes, oranges, and apples in the Minia Markets. 84 pesticides were investigated. The QuEChERS method was used for the extraction and purification, Residues were analyzed using GCMS/MS and LCMS/MS. The expected daily intake (EDI) and a hazard index were determined. The standard deviations were (1% to 7.5%, while the LOD varied from 0.0004 to 0.0231 ng/gm^{-1}. The LOQ (0.0012 to 0.0693 ng/gm^{-1}).

The results revealed that all samples were contaminated with residues of 35 different pesticides, out of which 31.48% exceeded the maximum residual limits (MRLs), while 64.8% contained concentrations below the MRLs. The most frequently detected pesticides were cypermethrin (found in 27 out of 54 samples), followed by carbendazim (20/54), profenofos (10/54), orthophenylphenol (OPP) (9/54), and Pascalid (9/54). Chloropyrifos, thiabendazole, and myclobutanil were detected in 6 out of 54 samples. On the other hand, lambda-cyhalothrin, thiophanate methylchlorobrofam, methoxybenzene, thiamethoxam, clothianidin, methoxyphenoside, hexethizox, malathion, and maloxone were the least frequently observed pesticides in the collected fruit samples.

During the winter of 2021 and summer of 2022, several pesticides exceeded the MRLs, including carbendazim, capatane, myclobutylene, thiophanate methyl, imidaclopride, dimethoate, omethoate, pyraclostrobin, imazalil, thiabendazole, propagrate, praclostrobin, and dimetomorph, while the rest of the pesticides remained within the MRLs. A risk ratio calculation indicated the presence of residues from propagret, carbendazim,

thiaclopride, OPP, thiophanate methyl, and dimethemorph, which pose a severe public health risk.

Key word: Pesticide residues, monitoring, QuEChERS, Risk ratio

1- INTRODUCTION

Pesticide residues in fruits are a major concern due to their potential impact effects on human health. Fruits grown in many parts of Egypt, and all world **(Omeiri and Khnayzer., 2022; Sahyoun et al., 2022; Sindhu and Manickavasagan, 2023; Stenrød et al., 2023)**. **Hussein et al., 2015; Abdallah et al., 2008)** including those from Minia Governorate, have been shown to contain detectable levels of pesticide residues. Recent studies have reported the presence of organochlorine, organophosphorus, and synthetic pyrethroid pesticide residues in some commonly consumed fruits in Egypt such as grapes, strawberries, and citrus fruits. **(Bashour and Nimah, 2012).**

The presence of pesticide residues is mainly attributed to the improper application of pesticides by farmers attempting to control pests and diseases in order to improve crop yield. However, lack of adherence to safe waiting periods before harvest and overuse of pesticides has led to contamination of fruits with these toxic chemical residues. **(Abou-Arab and Abou Donia, 2001; EFSA, 2013; Nasra et al., 2021).**

Continuous monitoring of pesticide residues is crucial to evaluate levels in fruits, estimate potential health risks to consumers, and promote safer and more judicious use of pesticides. Quantitative dietary risk assessment also helps estimate and manage risks associated with chronic exposure and highlight commodities that require more stringent regulation. **(Abdel-Sattar et al., 2010; Farag et al., 2011; Hussein et al., 2015; Ifdial et al., 2017; Ibrahim et al., 2022; Hussein et al., 2024).**

Several nations have implemented legislative regulations to regulate the residue of pesticides in food using maximum residue levels, or MRLs, to safeguard consumers' health (**FAO/WHO. Codex Alimentarius Commission—Procedural Manual; Wahab et al., 2022**) are typically designed to reduce consumer exposure to hazardous or needless pesticide intakes, guarantee the appropriate application of pesticides in accordance with authorization and registration (application rates and pre-harvest intervals (Darko and Akoto, 2008) and allow the free movement of pesticide-treated goods as long as they adhere to the established maximum residual contents (MRLs). When pesticide residues are found in amounts higher than the tolerance—a limit set especially for a given pesticide on a specific food item—violative residues are the result. The more prevalent version **(Katz and Winter, 2009; Sahyoun et al., 2022; Zhang et al., 2023).**

This study therefore aims to monitor and analyze pesticide residue levels in some major locally produced fruits including grapes, apple, and oranges from Minia Governorate. Multi-residue analytical methods using QuEChERS method of extraction and LC-MS/MS and GC-MS/MS apparatus will be utilized to detect a wide array of commonly used pesticides (48 pesticides). The quantified residues will be used to estimate potential health risks to offer recommendations to curb harmful levels and promote judicious pesticide application. Such periodic evaluation and dietary risk analysis would safeguard consumers from hazards of pesticide exposure through diet. The findings shall provide guidance for designing monitoring programs, modifying policies, and creating awareness among farmers and consumers regarding pesticide usage and risks from residues in commonly consumed fruits

2. MATERIALS AND METHODS

2.1. Reagents and chemicals

The active ingredient of the pesticide was sourced from Merck (Darmstadt, Germany), while anhydrous magnesium sulfate and sorbents (primary secondary amine; PSA particle size 40 µm) were acquired from Sigma-Aldrich (St. Louis, MO, USA). All the organic solvents used were of superior quality for high performance liquid chromatography (HPLC).

2.2. Sample Gathering and Preparation:

For each season (winter 2021, summer 2022), we collected three fruit sample packages, each weighing approximately 1 kg, from every grocery store in the three locations within Minia Governorate. The QuEChERS method, developed by (Gad Alla et al., 2013), was employed to extract 100 grams of the composite sample from each of the three one-kilogram duplicates that were generated. This approach is commonly employed in food safety assessments. The composite materials were homogenized and pulverized using a blender. Consequently, a 100g portion of the fruit sample, which had been thoroughly mixed, was measured using 100 milliliters of acetonitrile in a 200milliliter centrifuge tube. After time, 6 grams of magnesium sulfate (MgSO4) and 1.5 grams of sodium acetate were introduced into the samples contained in a centrifuge tube. Tube was homogenized by vortexing for one minute and then subjected to centrifugation at a speed of 3700 RPM for 5 minutes. The portion of the acetonitrile phase was moved into a tube containing 125 mg of (PSA) and 750 mg of MgSO4. The tube was agitated using a vortex mixer for 0.5 minutes and then subjected to centrifugation at a speed of 3700 revolutions per minute for 5 minutes. The entire sample was thereafter transferred to a sterile 15-mL tube and subjected to evaporation until complete desiccation using a moderate stream of nitrogen. The desiccated remnants were dissolved in 1 mL of acetonitrile for multiresidues determination using Liquid and gas chromatography-tandem mass spectrometry (GC-MS/MS) **(Gad Alla et al., 2013; Diop et al., 2016)**

2.3. Preparation of standards

Stock solutions (10 ng/ml): Each pesticide was prepared as a stock solution at a concentration of 10 ng/ml in 10-mL volumetric flasks using acetonitrile. These solutions were then kept at a temperature of -20 °C.

2.4. Percentage of recovery

In order to prevent pesticide contamination, the samples were subjected to extraction using acetonitrile and subsequently utilized as blank samples for the spiking investigations. The collected samples were enriched with varying quantities of pesticides, extracted using the identical method, and assessed by GC-MS/MS. The recovery percentages were calculated according to **(Charan et al., 2010**)

$$(\text{Concentration as ppm } =) \left(\frac{\text{Area of sample peak}}{\text{Area of std peak}} \times \frac{\text{Final volume}}{\text{gm of sample}} \times \frac{\mu\text{l of sampling}}{\mu\text{l of std injected}}\right) \times (con.of\ stand)$$

Recovery % = ((1-(CB- CA)/CB)) ×100

where

CA= concentration after treatment

CB= concentration before treatment

2.5. Operational solutions: In accordance with the recommendations of the European Commission **(Ahmed et al., 2022**), calibration curves were established for every pesticide. In order to get concentrations ranging from 0.01 to 10 mg/L1, calibration standards were created by incorporating multi-residue working solutions into acetonitrile extracts of fruit blanks. The standard is stored at a temperature of -20 °C until it is required. These standards were subsequently employed for the determination of limits of detection (LOD), limits of quantification (LOQ), the recovery %. A calibration curve was generated by plotting the peak area and concentration using an Excel application. The regression analysis using Excel program was used to calculate the standard deviation and slope of the curve for all samples.

LOD was calculated as follows:

LOD = 3.3 * SE / b and LOQ = 10* SE/ b **(Dolan, 2009**)

Where SE= standard error of calibration curve

b = slope of calibration curve

The limits of detection varied from 0.0004 ng/gm-1 to 0.0231 ng/gm-1. The limit of quantification was found to be in the range of 0.0012 ng/gm-1 to 0.0693 ng/gm-1.

R^2 value greater than 0.99 was obtained.

The recovery rates for most pesticides in various fruits were 94.09 – 106.79 % with relative standard deviations 1 -7.5 %.

2.6. Mass spectrometer configuration

The interface heater was maintained at a temperature of 550 °C using an ion-spray (IS) voltage of 5500. Prior to evaluating each set of samples, the mass spectrometer underwent complete automatic tuning. To identify the most prevalent mass to charge ratio (m/z) ion (Q1), a thorough analysis of the mass spectra of all pesticides was conducted using continuous infusion of each pesticide in the positive ionization mode of ESI. Since the product mass spectra were obtained through continuous infusion of each analyte, the Q1 value, which represents the protonated precursor ion, remained consistent. Subsequently, MRM analysis was conducted on the chemical's most prevalent product ion. At least two highly concentrated product ions were separated. One ion was utilized for quantification, while the other was employed for confirmation, following the three main criteria outlined for mass spectrometry investigations of pesticides (**Gad Alla et al., 2013; Jallow et al., 2017; Ibrahim et al., 2020; Sindhu and Manickavasagan, 2023; Zhang et al., 2023)**. An analysis was conducted in the selected ion monitoring (SIM) mode, utilizing one target ion and two qualifier ions. The identification of pesticides was based on their retention durations, as well as the presence of certain target and qualifier ions. The quantification was determined by calculating the ratio of the peak area of the target ion to that of the internal standards.

2.7. The temperature program used was as follows:

The temperature starts at 50˚ C and is held for 1 minute. It then increases at a pace of 20˚ C per minute until it reaches 180˚ C. From there, it increases at a rate of 10˚ C per minute until it reaches 190˚ C. The temperature then increases at a rate of 3˚ C per minute until it reaches 240˚ C. Finally, it increases at a rate of 10˚ C per minute until it reaches 300˚ C, and remains at this temperature for 5 minutes. The injection port temperature was set to 220˚ C, and a volume of 1 ml was injected. The ionization source was maintained at a temperature of 230˚ C. Identification was based on the consideration of both the main ions (m/z) and retention durations.

2.8. Statistical analysis:

The data analysis was conducted using the Costat program. The pesticide data from various fruit samples were subjected to analysis using a two-way ANOVA and LSD (least significant difference) test at a significance threshold of $p < 0.05$. A three-way analysis of variance was undertaken to compare the effects of seasons, crops, and localities on the pesticides.

3.RESULTS AND DISCUSSION

3.1. pesticide residues in fruits collected from Minia Governorate markets during winter 2021 and summer 2022.

After analyzing 54 fruit samples residues were detected in all samples (100 %) of the samples. The limit of detection (LOD) varied from 0.0004 ng/gm-1 to 0.0231 ng/gm-1. The limit of quantification (LOQ) was found to be in the range of 0.0012 ng/gm-1 to 0.0693 ng/gm-1. Furthermore, an R^2 value was greater than 0.99.

3.1.1. Residues of pesticides in fruits collected from El- Minia city markets.

Data of winter season 2021, showed that samples of grape vine were contaminated with carbendazim, with concentration more than MRLs and dimethoate, omethoate, thiaclopride, myclobutanil with concentration ranged between 0.005 to 0.01µg/g-1 pesticides and exceeded MRL's. While in orange captan (0.236µg/g-1) and chloropham with concentration (0.015µg/g-1) and both exceeded MRL's samples were contaminated with tubacanzole, acetampride, methoxybenzenone and chlorantraniliprole with concentrations less than MRL's and ranged from 0.01 to 0.0.089µg/g-1). Samples of apple only cypermethrin was detected with concentrations less than MRL. (0.012µg/g-1) and OPP with concentration more than MRL's (0.249µg/g-1) Table 1.

Results of summer 2022 showed that samples of grape were contaminated with cyfaluthrin (006µg/g-1) and pyraclostrobin (0.061) and both less than MRL's and frequented two times from 3 samples while orange in Minia Center was contaminated with propagrite and imazalil and thiabendazole with concentraions (0.048, 0.115 and 0.021 µg/g-1) and more than MRL's and frequented 1, 2 and one time from 9 samples respectively and contaminated with cypermethrin, carbendazime, acetampride and OPP with concentrations less than MRL's. Apple in Minia Center markets were contaminated with carbendazime with concentrations (0.1µg/g-1) and more than MRL's values and contaminated with cypermethrin, acetampride and imidaclopride with concentations less than MRL's. Table 1.

3.1.2. Residues of pesticides in fruits from Samalout markets during winter 2021 and summer 2022-

Data of winter season 2021, showed that samples of grape were contaminated with carbendazim (11.2µg/g-1), omethoate (0.89 µg/g-1) and lambada cyhalothrin 0.144 µg/g-1) with concentrations more than MRL's values. While carbendazim, myclobutanil, thiamethoxam, clothianidin, tetraconazole, thiophanate-methyl, cypermethrin, dimethoate, imidaclopride, propecanazole, tetraconazole and hexythiazox were detected with concentrations ranged between 0.005 to 2.4 µg/g-1 pesticides and not exceeded MRL's. While in orange samples dimethoate, (0.011µg/g-1) was detected and was more than MRL's while Ortho-Phenyl Phenol (OPP), omethoate, Imazalil, profenofos, and thiapendazole were detected with concentrations less than MRLs as shown in Table 2.

Results of summer 2022 showed that samples of grape were of samples of Summer 2022 from Samalot center were contaminated with boscalid, chlorofenpyr and myclobutanil with concentrations less than MRLs. ranged from 0.003 to 0.01µg/g-1. While orange samples from Samalot center markets at summer 2022 season were contaminated with cyfluthrin, cyhalothrin, cypermethrin, myclobutanil and OPP with concentrations less than MRL's values and only Imazalil which detected with values more than MRL's values (0.064µg/g-1) as shown in Table 2 .and Fig 2 . Results of summer 2022 indicated that profenofos, propargrite carbendizim were detected with concentrations (0.08, 0.19 and 0.025 µg/g-1) and their residual values were exceeded MRLs values.

3.1.3. Residues of pesticides in fruits from Abu Qurqas markets during winter 2021 and summer 2022:

Results of samples collected during winter 2021 indicated that grape samples from markets of Abu Qurqas are highly contaminated with dimethoate, omethoate, carbendazim, OPP, and thiophanate-methyl with concentrations exceeded MRL's values and myclobutanil, lambda - Cyhalothrin, Cypermethrin, Propiconazo, imidacloprid, boscalid, pyraclostrobin, tetraconazole, hexythiazox, malathion, malaoxon, myclobutanil with concentrations less than MRLs values (0.005 – 0.02 ug/g). While orange of winter 2021 results showed that Ortho-Phenyl Phenol (OPP) were detected with concentration more than MRL's (1.065 $\mu g/g^{-1}$) and imazalil (0.802$\mu g/g^{-1}$) and thiabendazol (0.155$\mu g/g^{-1}$) and less than MRL's values.

Also, samples of apple collected from Abu Qurqas during winter 2021 were contaminated with Captan, Tebuconazole, and Chlorpropham with concentrations ranged between (0.005 – 0.015 $\mu g/g^{-1}$). Results of analysis of samples collected during Summer 2022 from markets of Abu Qurqas were less contaminated than Minia and Samalot markets. Where as shown in Table 3

Boscalid was detected with concentrations more than MRL's (0.03µg/g-1). And metaferenon with concentration (0.003 µg/g^{-1}) and less than MRL's. IN orange samples cyfluthrin, cypermethrin and imazalil were detected with concentrations 0.01, 0.16 and 0.126µg/g-1) and not exceeded MRL's values. Bifenthrin, chloropyrifos, cyfalothrin and azoxystrobin with concentration (0.005, 0.007,0.01 and 0.005 µg/g^{-1} respectively) and their concentrations less than MRL. Analysis of variance indicated that Minia markets are the most markets contaminated followed by Samalut. Grape samples are the most crop contaminated.

A number of investigations have been carried out in Egypt to ascertain the presence of pesticide residues in fruits. The findings we obtained align with the conclusions documented by **((Ibrahim et al., 2020; Ibrahim et al., 2022)**). Nevertheless, our findings diverged from theirs regarding chlorpyrifos and propargite, as they neglected to include the other substances in their investigation, but we did. Regarding apple samples, our findings diverged from those of (**(Ibrahim et al., 2022))** in terms of imidacloprid and chlorpyrifos. While they did not include these substances in their survey, we detected their presence. The results obtained for the samples of tomatoes, apples, and grapes were consistent with the findings reported by **((Hamilton and Crossley, 2004; Latif et al., 2011; Mohamed et al., 2014; Nishant and Upadhyay, 2016; Ifdial et al., 2017; Jallow et al., 2017; Mebrouki et al., 2021).** In the same way, the grape samples we tested also showed the presence of the analytes dimethoate and profenofos, which was previously reported **(Hamilton and Crossley, 2004; Latif et al., 2011; Jallow et al., 2017).** However**,** our results did not align with their findings regarding other pesticides such as carbendazim, chlorpyrifos, omethoate, and thiophanate methyl. The survey conducted by **(Wahab et al., 2022)** did not include the reporting of these pesticides.

Certain agricultural products, such as grape, may contain elevated amounts of pesticide residues as a result of intense pest and disease infestations, necessitating repeated applications of pesticides. Pesticides are commonly administered directly to the consumable portion of the crop in close proximity to the time of harvest in order to guarantee plant safeguarding. In addition, certain fruit farms employ pesticides with a frequency ranging from biweekly to weekly. In order to safeguard public health from the adverse effects of pesticide residues, it is crucial to use pest management techniques that guarantee pesticide concentrations in commercially available fruits remain below (MRLs). (**(Hussein et al., 2015; European Food Safety Authority (EFSA). (2021). The 2020 European Union report on pesticide residues in food. EFSA Journal, 2021; Hussein et al., 2024)**

3.2. Dietary intake of tested pesticide residues through fruits from area of Minia.

The obtained data was utilized to assess the potential health hazard linked to the exposure of these pesticide residues. The collected results were utilized to compute the expected daily intake (EDI), The health risk indices of the residues were calculated based on the data collected and food consumption.

The food consumption rate in Minia Governorate was assessed by conducting a survey of 600 individuals in the regions of Minia, Samalout, and Abu Qurqas each (Table 4).

The findings highlight the presence of pesticides in fruits collected from markets of Minia Governorate. It is necessary to regularly monitor these contaminants in food products to prevent, and reduce pollution, as well as to minimize health hazards. Additionally, it is worth noting that in certain instances, pesticide levels exceeded the acceptable daily intake (ADI). The presence of carbendazim, thioclopride, OPP, imazalil, acetamiprid, pyraclostrobin, and dimethomorph in grape, apple, and orange samples from Minia, Samalout, and Abuqurqas poses a significant danger. The hazard indices varied between 0% and 1272% for the pesticides were determined. Therefore, the eating of these fruits over one's lifetime does not present a health danger for the inhabitants of Minia Governorate, as several of the residue indices were below 100%. Nevertheless, the current investigation reveals a significant prevalence of chemical residues (mostly insecticides and fungicides) in fruits. Attention should be given to the fact that the examined fruits are mostly utilized in their raw form, without undergoing any cooking treatment, and are commonly used for making fresh juices. Furthermore, individuals who consume fruit may potentially be exposed to many pesticides simultaneously. In conclusion, the previous discussion indicates that the consumers in Minia Governorate are only exposed to lesser levels of pesticides, which have the potential to lead to chronic illnesses. The concentration of the different pesticides varied, ranging from below to over the Maximum Residue degree (MRL). Therefore, continuing consumption of fruits with a moderate degree of contamination might lead to the accumulation of these pesticides in the body of the consumer. In the long run, this can have catastrophic consequences for the human population.

CONCLUSION

Our study yields crucial data regarding the presence of pesticides in some fresh fruits obtained from markets in Minia governorate. While the pesticide levels in the samples were noteworthy, those that over the allowed limits (MRL) did not pose any health hazards to consumers. Nevertheless, it is imperative to consistently check pesticide residues by frequently collecting random samples for examination. Furthermore, it is imperative to implement an extension program aimed at enhancing farmers' understanding of the proper utilization and administration of pesticides, as well as the significance of strict adherence to safety measures.

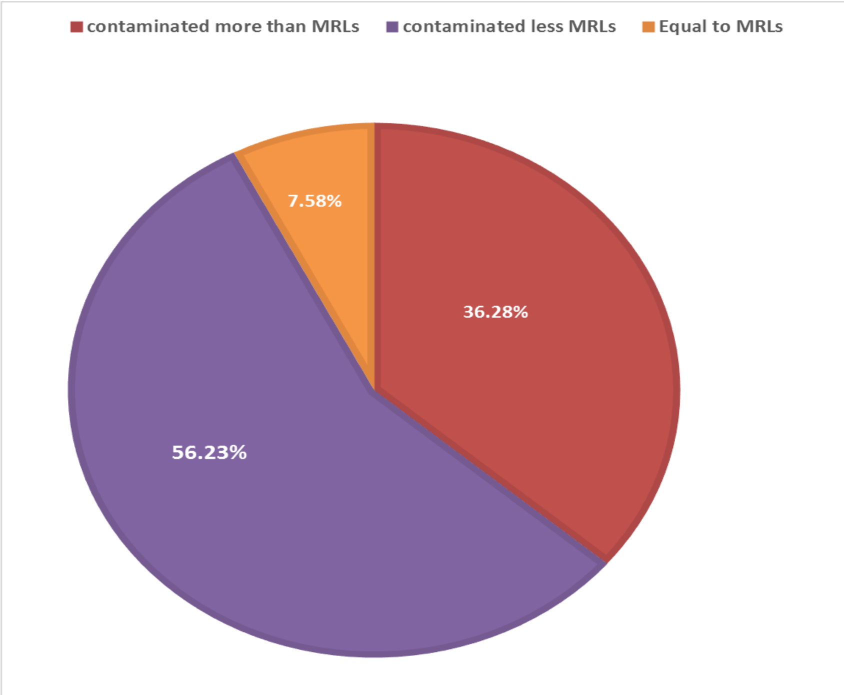

Fig. 1: percentage of contaminated, that samples exceeded MRL's, less than MRL's and equal MRL's in fruit samples collected from Minia Governorate Markets during Winter 2021and summer 2022.

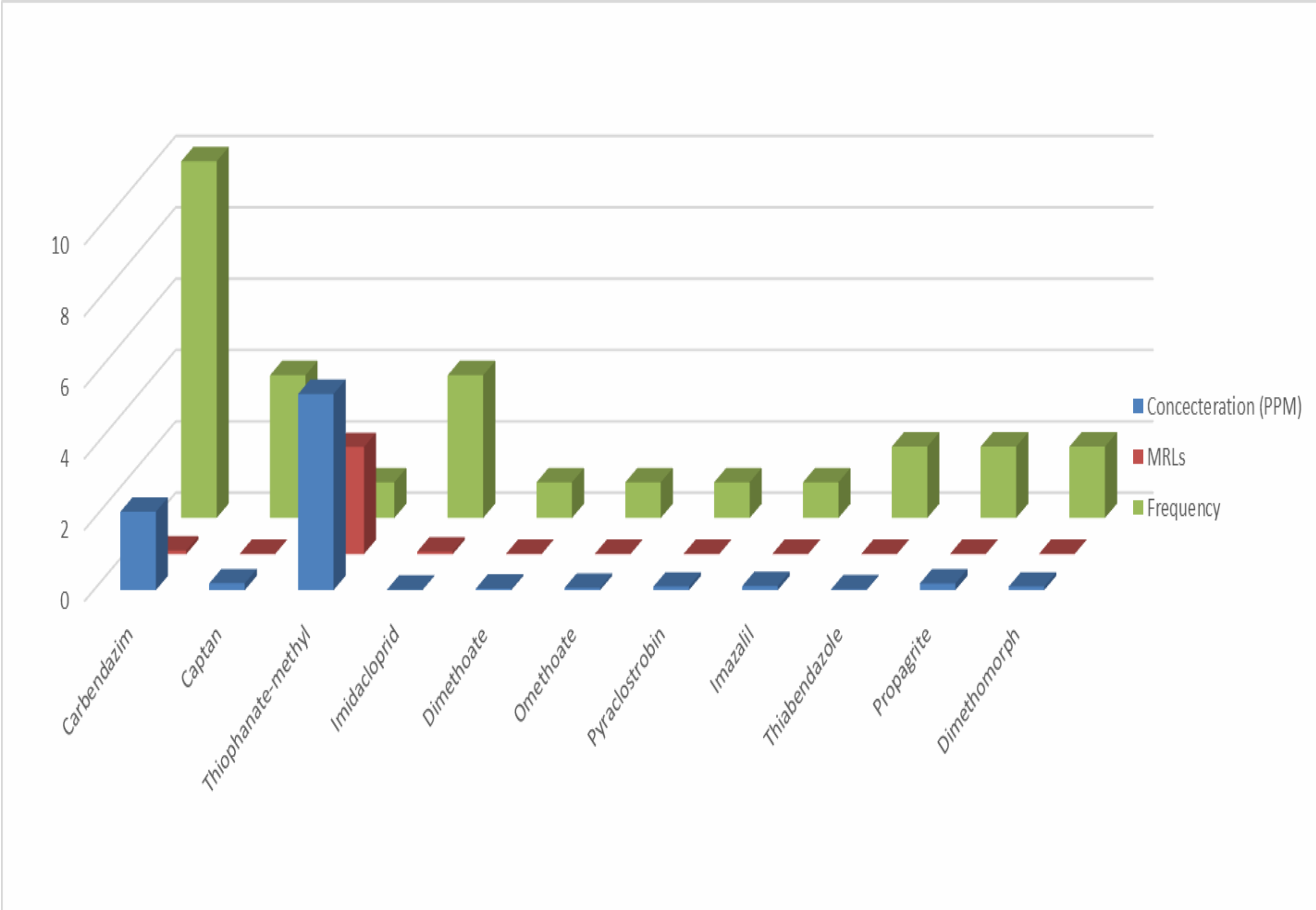

Fig.2: Residues of pesticides that exceeded MRLs, (PPM), (MRLs and their Frequency) in different fruit crops Minia Granorate During summer 2021 and winter 2022.

Table (1): Residues of different pesticides (PPM) in different fruit crops in Minia city during winter 2021 and summer 2022 seasons with their MRLs.

Detected Pesticides	Minia Winnter 2021				Minia Summer 2022				MRL		
	Frequency	Grape	Orange	Apple	Frequency	Grape	Orange	Apple	Grape	Orange	Apple
		PPM	PPM	PPM		PPM	PPM	PPM	PPM	PPM	PPM
Carbendazim	2	2.2	ND	ND	2	ND	0.04	0.1	0.1	0.01	0.01
Chlorpyrifos	2	ND	ND	0.01					0.01		0.01
Dimethoate	1	0.005	ND	ND					0.01		
Omethoate	1	0.005	ND	ND					0.01		
Thiacloprid	2	0.084	ND	ND					0.01		
Myclobutanil	2	0.01	ND	ND					1.5		
Cypermethrin	2	ND	ND	0.012	1	ND	0.01	ND		0.2	1
Captan	1	ND	0.236	ND					0.03	0.03	10
Tebuconazole	1	ND	0.089	ND	1	ND	0.021	ND	0.5	0.9	0.3
Chlorpropham	1	ND	0.015	ND					0.01	0.01	0.01
Acetamiprid	1	ND	0.014	ND	1	ND	0.035	0.03	0.5	0.9	0.4
Methoxybenzenes	1	ND	0.011	ND					2	1	2
Chlorantraniliprole	1	ND	0.01	ND					1	0.7	0.5
Ortho-Phenyl Phenol (OPP)	1	ND	ND	0.249	2	ND	0.21	ND	0.1	0.01	0.01
Boscalid					2	0.19	ND	ND	5		
Cyfluthrin					1	0.006	ND	ND	0.3		
Cypermethrin					2	ND	0.13	0.02		0.5	1
Imazalil					2	ND	0.115	ND		0.01	
Propagrite					1	ND	0.048	ND		4	
Pyraclostrobin					2	0.061	ND	ND	0.3		
Imidacloprid					2	ND	ND	0.03			0.5
Clothianidin					1	ND	0.02	ND		0.06	

Table (2): Residues of different pesticides (PPM) in different fruit crops in samalout city during winter 2021 and summer 2022 seasons with their MRLs.

Detected Pesticides	Samalout Winnter 2021				Samalout Summer 2022				MRL		
	Frequency	Grape	Orange	Apple	Frequency	Grape	Orange	Apple	Grape	Orange	Apple
		PPM	PPM	PPM		PPM	PPM	PPM	PPM	PPM	PPM
Carbendazim	**2**	**11.2**	**ND**	**ND**	**2**	**ND**	**ND**	**0.025**	**0.1**		**0.2**
Chlorpyrifos	**2**	**ND**	**0.01**	**ND**	_-					**0.01**	
Myclobutanil	**1**	**0.13**	**ND**	**ND**	**1**	**0.01**	**ND**	**ND**	**1.5**		
Thiamethoxam	**1**	**0.018**	**ND**	**ND**	-_	**ND**	**ND**	**ND**	**0.4**		
Clothianidin	**1**	**0.01**	**ND**	**ND**	-_	**ND**	**ND**	**ND**	**0.7**		
Tetraconazole	**1**	**0.022**	**ND**	**ND**	-_	**ND**	**ND**	**ND**	**0.5**		
Thiophanate-methyl	**1**	**5.5**	**ND**	**ND**	-_	**ND**	**ND**	**ND**	**3**		
Lambda-Cyhalothrin	**1**	**0.144**	**ND**	**ND**	-_	**ND**	**ND**	**ND**	**0.08**		
Cypermethrin	**2**	**0.042**	**0.021**	**ND**	**2**	**ND**	**0.27**	**ND**	**0.5**	**2**	
Dimethoate	**1**	**0.089**	**0.011**	**ND**	-_	**ND**	**ND**	**ND**	**0.01**	**0.01**	
Omethoate	**1**	**0.094**	**0.01**	**ND**	-_	**ND**	**ND**	**ND**	**0.01**	**0.01**	
Propiconazole	**1**	**0.005**	**ND**	**ND**	-_	**ND**	**ND**	**ND**	**0.01**		
Imidacloprid	**2**	**0.012**	**ND**	**ND**	-_	**ND**	**ND**	**ND**	**0.7**		
Boscalid	**2**	**2.4**	**ND**	**ND**	**2**	**0.02**	**ND**	**ND**	**5**		
Pyraclostrobin	**1**	**0.1**	**ND**	**ND**	-_	**ND**	**ND**	**ND**	**0.3**		
Tetraconazole	**1**	**0.082**	**ND**	**ND**	-_	**ND**	**ND**	**ND**	**0.5**		
Hexythiazox	**1**	**0.01**	**ND**	**ND**	-_	**ND**	**ND**	**ND**	**1**		
Captan	**1**	**ND**	**ND**	**0.191**	-_	**ND**	**ND**	**ND**			**10**
Tebuconazole	**1**	**ND**	**ND**	**0.071**	-_	**ND**	**ND**	**ND**			**0.3**
Chlorpropham	**1**	**ND**	**ND**	**0.013**	-_	**ND**	**ND**	**ND**			**0.01**
Acetamiprid	**1**	**ND**	**ND**	**0.017**	-_	**ND**	**ND**	**ND**			**0.4**
Methoxyfenozide	**1**	**ND**	**ND**	**0.005**	-_						**0.01**
Chlorantraniliprole	**1**	**ND**	**ND**	**0.01**	-_						**0.5**
Ortho-Phenyl Phenol (OPP)	**1**	**ND**	**0.673**	**0.191**	**2**	**ND**	**0.21**	**ND**	**5**	**0.01**	
Imazalil	**2**	**ND**	**0.409**	**ND**	**2**	**ND**	**0.064**	**ND**		**4**	
Profenofos	**2**	**ND**	**0.01**	**ND**	**2**	**ND**	**ND**	**0.08**	**0.01**	**0.01**	**0.01**
Thiabendazole	**2**	**ND**	**0.053**	**ND**		**ND**	**ND**			**11**	
Chlorfenpyr					**2**	**0.0003**	**ND**	**ND**	**0.01**		
Cyfluthrin					**1**	**ND**	**0.01**	**ND**		**0.3**	
Cyhalothrin					**2**	**ND**	**0.02**	**ND**		**0.2**	
Propagrite					**1**	**ND**	**ND**	**0.19**			**0.01**
Pyriproxyfen					**1**	**ND**	**ND**	**0.01**			**0.2**
Dimethomorph					**1**	**ND**	**ND**	**0.01**			**0.01**

Table (3): Residues of different pesticides (PPM) in different fruit crops Abo Qurakas city during winter 2021 and summer 2022 seasons with their MRLs

Detected Pesticides	Abo Qurakas Winnter 2021				Abo Qurakas Summer 2022				MRL		
	Frequency	Grape	Orange	Apple	Frequency	Grape	Orange	Apple	Grape	Orange	Apple
		PPM	PPM	PPM		PPM	PPM	PPM	PPM	PPM	PPM
Carbendazim	**2**	**9.8**	**ND**	**ND**	**1**	**ND**	**ND**	**0.005**	**0.3**		**0.01**
Myclobutanil	**2**	**0.091**	**ND**	**ND**	**—**	**ND**	**ND**	**ND**	**1.5**		
Thiophanate-methyl	**1**	**2.9**	**ND**	**ND**	**—**	**ND**	**ND**	**ND**	**0.1**		
Lambda-Cyhalothrin	**1**	**0.108**	**0.065**	**ND**	**—**	**ND**	**ND**	**ND**	**0.08**	**0.2**	
Cypermethrin	**2**	**0.016**	**ND**	**ND**	**2**	**ND**	**0.16**	**ND**	**0.5**	**2**	
Dimethoate	**1**	**0.042**	**ND**	**ND**	**—**	**ND**	**ND**	**ND**	**0.01**		
Omethoate	**1**	**0.068**	**ND**	**ND**	**—**	**ND**	**ND**	**ND**	**0.01**		
Propiconazol	**1**	**0.005**	**ND**	**ND**	**—**	**ND**	**ND**	**ND**	**0.01**		
Imidacloprid	**2**	**0.033**	**ND**	**ND**	**—**	**ND**	**ND**	**ND**	**5**		
Boscalid	**2**	**0.95**	**ND**	**ND**	**2**	**0.32**	**ND**	**ND**	**2**		
Pyraclostrobin	**2**	**0.068**	**ND**	**ND**	**—**	**ND**	**ND**	**ND**	**0.3**		
Tetraconazole	**1**	**0.1**	**ND**	**ND**	**—**	**ND**	**ND**	**ND**	**0.5**		
Hexythiazox	**1**	**0.005**	**ND**	**ND**	**—**	**ND**	**ND**	**ND**	**1**		
Malathion	**1**	**0.02**	**ND**	**ND**	**—**	**ND**	**ND**	**ND**	**0.02**		
Malaoxon	**1**	**0.005**	**ND**	**ND**	**—**	**ND**	**ND**	**ND**	**0.02**		**2**
Captan	**1**	**ND**	**ND**	**0.005**	**—**	**ND**	**ND**	**ND**			**10**
Tebuconazole	**1**	**ND**	**ND**	**0.01**	**—**	**ND**	**ND**	**ND**			**0.3**
Chlorpropham	**1**	**ND**	**ND**	**0.015**	**—**	**ND**	**ND**	**ND**			**0.01**
Ortho-Phenyl Phenol (OPP)	**1**	**ND**	**1.065**	**ND**	**2**	**ND**	**0.25**	**ND**		**0.01**	
Imazalil	**2**	**ND**	**0.802**	**ND**	**2**	**ND**	**0.126**	**ND**		**4**	
Thiabendazole	**2**	**ND**	**0.155**	**ND**	**2**	**ND**	**0.033**	**ND**		**7**	
Carbendazim	**2**	**9.8**	**ND**	**ND**	**—**	**ND**	**ND**	**ND**	**0.3**		
Myclobutanil	**1**	**0.091**	**ND**	**ND**	**—**	**ND**	**ND**	**ND**	**1.5**		
Thiophanate-methyl	**1**	**2.9**	**ND**	**ND**	**—**	**ND**	**ND**	**ND**	**0.1**		
Bifenthrin	**-_**	**ND**	**ND**	**ND**	**1**	**ND**	**ND**	**0.005**			**0.01**
chlorpyrifos	**-_**	**ND**	**ND**	**ND**	**2**	**ND**	**ND**	**0.007**			**0.01**
Cyfluthrin	**-_**	**ND**	**ND**	**ND**	**1**	**ND**	**ND**	**0.01**			**0.2**
Cyhalothrin	**-_**	**ND**	**ND**	**ND**	**1**	**ND**	**0.01**	**ND**		**0.2**	
Metrafenone	**-_**	**ND**	**ND**	**ND**	**1**	**0.003**	**ND**	**ND**	**7**		
profenofos	**-_**	**ND**	**ND**	**ND**	**2**	**ND**	**ND**	**0.02**			**0.01**
Pyraclostrobin	**-_**	**ND**	**ND**	**ND**	**1**	**0.1**	**ND**	**ND**	**0.01**		
Dimethomorph	**-_**	**ND**	**ND**	**ND**	**1**	**0.1**	**ND**	**ND**	**0.01**		

Table (4): Risk assessment of pesticide residues in fruit crops at Minia Governorate During summer 2021 and winter 2022 with their Hazard index.

Pesticide	PPM	Crops	Season	Centers	ADI (mg/kg body/weight/day)	Average consumption	EDI (mg/kg body/weight/day)	Risk ratio=) EDI / ADI)	Results
Carbendazim	2.2	grapes	winter	Minia	0.1	35.22	3.033	303%	danger
Chlorpyrifos	0.01	Apple	winter	Minia	0.01	36.04	0.014	14%	safe
Dimethoate	0.005	grapes	winter	Minia	0.01	35.22	0.007	7%	safe
Omethoate	0.005	grapes	winter	Minia	0.01	35.22	0.007	7%	safe
Thiacloprid	0.084	grapes	winter	Minia	0.01	35.22	0.116	116%	danger
Myclobutanil	0.01	grapes	winter	Minia	1.5	35.22	0.014	0%	safe
Cypermethrin	0.012	Apple	winter	Minia	0.012	36.04	0.017	14%	safe
Captan	0.236	Orange	winter	Minia	0.03	28.13	0.260	87%	safe
Tebuconazole	0.089	Orange	winter	Minia	0.9	28.13	0.098	1%	safe
Chlorpropham	0.015	Orange	winter	Minia	0.01	28.13	0.017	17%	safe
Acetamiprid	0.014	Orange	winter	Minia	0.9	28.13	0.015	0%	safe
Methoxybenzenes	0.011	Orange	winter	Minia	1	28.13	0.012	0%	safe
Chlorantraniliprole	0.01	Orange	winter	Minia	0.7	28.13	0.011	0%	safe
Ortho-Phenyl Phenol) OPP(	0.249	Apple	winter	Minia	0.01	36.04	0.351	351%	danger
Imazalil	0.047	Apple	winter	Minia	0.01	36.04	0.066	66%	safe
Carbendazim	11.2	grapes	winter	Samalout	0.1	26.21	11.488	1149%	danger
Chlorpyrifos	0.01	Orange	winter	Samalout	0.01	22.85	0.009	9%	safe
Myclobutanil	0.13	grapes	winter	Samalout	1.5	26.21	0.133	1%	safe
Thiamethoxam	0.018	grapes	winter	Samalout	0.4	26.21	0.018	0%	safe
Clothianidin	0.01	grapes	winter	Samalout	0.7	26.21	0.010	0%	safe
Tetraconazole	0.022	grapes	winter	Samalout	0.5	26.21	0.023	0%	safe
Thiophanate-methyl	5.5	grapes	winter	Samalout	3	26.21	5.642	19%	safe
Lambda-Cyhalothrin	0.144	grapes	winter	Samalout	0.08	26.21	0.148	18%	safe
Cypermethrin	0.021	Orange	winter	Samalout	2	22.85	0.019	0%	safe
Dimethoate	0.011	Orange	winter	Samalout	0.01	22.85	0.010	10%	safe
Omethoate	0.01	Orange	winter	Samalout	0.01	22.85	0.009	9%	safe
Cypermethrin	0.042	grapes	winter	Samalout	0.5	26.21	0.043	1%	safe
Dimethoate	0.089	grapes	winter	Samalout	0.01	26.21	0.091	91%	safe
Omethoate	0.094	grapes	winter	Samalout	0.01	26.21	0.096	96%	safe
Propiconazole	0.005	grapes	winter	Samalout	0.01	26.21	0.005	5%	safe
Imidacloprid	0.012	grapes	winter	Samalout	0.7	26.21	0.012	0%	safe
Boscalid	2.4	grapes	winter	Samalout	5	26.21	2.462	5%	safe
Pyraclostrobin	0.1	grapes	winter	Samalout	0.3	26.21	0.103	3%	safe
Tetraconazole	0.082	grapes	winter	Samalout	0.5	26.21	0.084	2%	safe
Hexythiazox	0.01	grapes	winter	Samalout	1	26.21	0.010	0%	safe
Captan	0.191	Apple	winter	Samalout	10	25.5	0.191	0%	safe
Tebuconazole	0.071	Apple	winter	Samalout	0.3	25.5	0.071	2%	safe
Chlorpropham	0.013	Apple	winter	Samalout	0.01	25.5	0.013	13%	safe
Acetamiprid	0.017	Apple	winter	Samalout	0.4	25.5	0.017	0%	safe
Methoxyfenozide	0.005	Apple	winter	Samalout	0.01	25.5	0.005	5%	safe
Chlorantraniliprole	0.01	Apple	winter	Samalout	0.5	25.5	0.010	0%	safe
Ortho-Phenyl Phenol) OPP(	0.673	Orange	winter	Samalout	0.01	22.85	0.602	602%	danger
Imazalil	0.409	Orange	winter	Samalout	0.3	22.85	0.366	12%	safe
Profenofos	0.01	Orange	winter	Samalout	0.01	22.85	0.009	9%	safe
Thiabendazole	0.053	Orange	winter	Samalout	0.01	22.85	0.047	47%	safe
Carbendazim	9.8	grapes	winter	Abo Quarkas	0.3	28.23	10.828	361%	danger
Myclobutanil	0.091	grapes	winter	Abo Quarkas	1.5	28.23	0.101	1%	safe

Table (4): Cont.

Pesticide	PPM	Crops	Season	Centers	ADI (mg/kg body/weight/day)	Average consumption	EDI (mg/kg body/weight/day)	RIZK) EDI / ADI)	Results
Thiophanate-methyl	2.9	grapes	winter	Abo Quarkas	0.1	28.23	3.204	320%	danger
Lambda-Cyhalothrin	0.108	grapes	winter	Abo Quarkas	0.08	28.23	0.119	15%	safe
Cypermethrin	0.016	grapes	winter	Abo Quarkas	0.5	28.23	0.018	0%	safe
Dimethoate	0.042	grapes	winter	Abo Quarkas	0.01	28.23	0.046	46%	safe
Omethoate	0.068	grapes	winter	Abo Quarkas	0.01	28.23	0.075	75%	safe
Propiconazol	0.005	grapes	winter	Abo Quarkas	0.01	28.23	0.006	6%	safe
Imidacloprid	0.033	grapes	winter	Abo Quarkas	5	28.23	0.036	0%	safe
Boscalid	0.95	grapes	winter	Abo Quarkas	5	28.23	1.050	2%	safe
Pyraclostrobin	0.068	grapes	winter	Abo Quarkas	0.3	28.23	0.075	3%	safe
Tetraconazole	0.1	grapes	winter	Abo Quarkas	0.5	28.23	0.110	2%	safe
Hexythiazox	0.005	grapes	winter	Abo Quarkas	1	28.23	0.006	0%	safe
Malathion	0.02	grapes	winter	Abo Quarkas	0.02	28.23	0.022	11%	safe
Malaoxon	0.005	grapes	winter	Abo Quarkas	0.02	28.23	0.006	3%	safe
Captan	0.03	Apple	winter	Abo Quarkas	2	18.60	0.022	0%	safe
Tebuconazole	0.005	Apple	winter	Abo Quarkas	10	18.60	0.004	0%	safe
Chlorpropham	0.01	Apple	winter	Abo Quarkas	0.3	18.60	0.007	0%	safe
(OPP) Ortho-Phenyl Phenol	1.065	Orange	winter	Abo Quarkas	0.01	30.51	1.272	1272%	danger
Imazalil	0.802	Orange	winter	Abo Quarkas	4	30.51	0.958	2%	safe
Boscalid	0.19	grapes	summer	Minia	5	35.22	0.262	1%	safe
cyfluthrin	0.006	grapes	summer	Minia	0.3	35.22	0.008	0%	safe
Cyhalothrin	0.01	Orange	summer	Minia	0.2	30.51	0.012	1%	safe
Cypermethrin	0.13	Orange	summer	Minia	0.5	30.51	0.155	3%	safe
Imazalil	0.115	Orange	summer	Minia	0.01	30.51	0.137	137%	danger
(OPP) Ortho-Phenyl Phenol	0.21	Orange	summer	Minia	0.01	30.51	0.251	251%	danger
Propagrite	0.048	Orange	summer	Minia	4	30.51	0.057	0%	safe
Pyraclostrobin	0.061	grapes	summer	Minia	0.3	35.22	0.084	3%	safe
Acetamiprid	8.155	Apple	summer	Minia	0.8	36.04	11.503	144%	danger
Acetamiprid	0.03	Orange	summer	Minia	0.9	30.51	0.036	0%	safe
Imidacclorid	0.03	Apple	summer	Minia	0.5	36.04	0.042	1%	safe
Cabendazim	0.04	Orange	summer	Minia	0.01	30.51	0.048	48%	safe
Clothianidin	0.02	Orange	summer	Minia	0.06	30.51	0.024	4%	safe
Thiabendazole	0.021	Orange	summer	Minia	0.01	30.51	0.025	25%	safe
Boscalid	0.02	grapes	summer	Samalout	5	26.21	0.021	0%	safe
Chlorfenpyr	0.0003	grapes	summer	Samalout	0.01	26.21	0.000	0%	safe
cyfluthrin	0.01	Orange	summer	Samalout	0.3	22.85	0.009	0%	safe
Cyhalothrin	0.02	Orange	summer	Samalout	0.2	22.85	0.018	1%	safe
Cypermethrin	0.27	Orange	summer	Samalout	0.5	22.85	0.241	5%	safe
Imazalil	0.064	Orange	summer	Samalout	0.01	22.85	0.057	57%	safe
Myclobutanil	0.1	grapes	summer	Samalout	1.5	26.21	0.103	1%	safe
(OPP) Ortho-Phenyl Phenol	0.21	Orange	summer	Samalout	0.01	22.85	0.188	188%	danger
profenofos	0.08	Apple	summer	Samalout	0.01	25.49	0.080	80%	safe
Propagrite	0.19	Apple	summer	Samalout	0.01	25.49	0.190	190%	danger
Pyriproxyfen	0.01	Apple	summer	Samalout	0.2	25.49	0.010	0%	safe
Cabendazim	0.025	Apple	summer	Samalout	0.2	25.49	0.025	1%	safe
Dimethomorph	0.01	Apple	summer	Samalout	0.01	25.49	0.010	10%	safe
Bifenthrin	0.005	Apple	summer	Abo Quarkas	0.1	18.60	0.004	0%	safe
Boscalid	0.32	grapes	summer	Abo Quarkas	2	28.23	0.354	2%	safe
chlorpyrifos	0.007	Apple	summer	Abo Quarkas	0.01	18.60	0.005	5%	safe
cyfluthrin	0.01	Apple	summer	Abo Quarkas	0.2	18.60	0.007	0%	safe
Cyhalothrin	0.01	Orange	summer	Abo Quarkas	0.2	30.51	0.012	1%	safe
Cypermethrin	0.16	Orange	summer	Abo Quarkas	2	30.51	0.191	1%	safe
Imazalil	0.126	Orange	summer	Abo Quarkas	4	30.51	0.150	0%	safe
Metrafenone	0.003	grapes	summer	Abo Quarkas	7	28.23	0.003	0%	safe
(OPP) Ortho-Phenyl Phenol	0.25	Orange	summer	Abo Quarkas	0.01	30.51	0.299	299%	danger
profenofos	0.02	Apple	summer	Abo Quarkas	0.01	18.60	0.015	15%	safe
Pyraclostrobin	0.1	grapes	summer	Abo Quarkas	0.01	28.23	0.110	110%	danger
Dimethomorph	0.1	grapes	summer	Abo Quarkas	0.01	28.23	0.110	110%	danger
Azoxystrobin	0.033	Orange	summer	Abo Quarkas	7	30.51	0.039	0%	safe

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الملخص العربي

رصد وتقييم مخاطر متبقيات المبيدات في بعض الفواكه المنتجة محليا بمحافظة المنيا، المنيا، مصر

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يهدف البحث لرصد متبقيات 84 مبيد تتبع لمجموعات كيميائية مختلفة في 54 عينة من العنب والبرتقال والتفاح تم جمعها من أسواق ثلاث مراكز من محافظة المنيا (المنيا- سمالوط وابوقرقاص)، أُستخدمت طريقة كاتشرز QuEChERS لاستخلاص العينات وتنقيتها، وقُدّرت متبقيات المبيدات باستخدام الكروماتوغرافيا الغازية والسائلة ذو مطياف الكتلة، كما قدر متوسط الاستهلاك اليومي من كل نوع من الفاكهة لحساب معدل الاخذ اليومي المتوقع من كل مبيد في كل محصول في كل مركز وذلك لحساب معدل الخطورة علي الصحة العامة.

أظهرت النتائج كفاءة الطريقة المتبعة للاستخلاص حيث كانت نسبة الاسترجاع من (94.09-106.76)%، والانحراف المعياري النسبي (1-7.5)%، بينما حد الكشف (0.0004-0.0231) ميكروجرام /جم، وحد التقدير الكمي (0.0012-0.0693) ميكروجرام /جم ، وسجل معامل التحديد R^2 قيمة أكبر من 0.99، وتبين أن كل العينات كانت ملوثة بمتبقيات 35 مبيد وتجاوزت كمية المبيدات الحدود القصوى المسموحة في 31.48% من عدد العينات الكلي، وكانت 64.8% ملوثة بتركيزات أقل من الحد الأقصى للمتبقيات، وكانت المبيدات الأكثر رصدا وتكرارا السايبرمثرين، التي تم تكراره في 27 عينة من 54 عينة، والكاربيندازيم 54/20، والبروفينوفوس 54/10، وأورثو فينيل فينول (OPP) 54//9 و باسكاليد 54/9 يليه كلوروبيريفوس وثيابندازول، وميكلوبوتانيل لكل منهما تكرر في 6عينات من 54. وكانت المبيدات الحشرية الأقل شيوعًا هي لامبادا سيهالوثرين، وثيوفانات ميثيل كلوربروفام، وميثوكسي بنزين، وميكلوبوتانيل، وثياميثوكسام، وكلوثيانيدين، وميثوكسي فينوزيد، وهيكسيثيزوكس، وملاثيون، وملاكسون، في عينات الفاكهة التي تم فحصها من أسواق مختلفة في ثلاثة مواقع مختلفة في شتاء 2021 وصيف 2022. 31.48% من عينات الفاكهة تجاوزت الحدود القصوى للمتبقيات وهذه المبيدات هي كاربيندازيم، كاباتان، ميكلوبوتانيل، ثيوفانات ميثيل، إيميداكلوبرايد، دايميثوات، أوميثوات، بيراكلوستروبين، إيمازاليل، ثيابندازول بروباجريت، براكلوستروبين وديميتومورف، بينما لم تتجاوز المبيدات الأخرى الحدود القصوى للمتبقيات. وتشير نتائج نسبة الخطورة الي وجود بقايا من البروباجريت، الكاربيندازيم، الثياكلوبرايد، OPP، ثيوفانات-ميثيل ودايمثيمورف. من بين 84 مبيدًا التي تم تقديرها وصنفت علي انها خطرة.