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Original article

Detection of Pesticide Residues (Imidacloprid, Aldicarb, Metalaxyl, Cypermethrin, Chlorpyrifos, DDA, and Endrin) in Peach Samples Collected from Jabal al Akhdar farms, Libya

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Abstract
This study was conducted to determine the presence of pesticide residues, specifically (Imidacloprid, Aldicarb, metalaxyl, cypermethrin, chlorpyrifos, DDA, and Endrin) in peach samples collected from various regions in eastern Libya, including (Qarnadah, Iqafnata, Abdul Wahid, Qasr Libya, and Al-Marj). The analytical methodology employed a (UV-Vis) spectrophotometer in conjunction with the Quick, Easy, Cheap, Effective, Rugged, and Safe (QuEChERS) extraction technique for the estimation of pesticide concentrations in the peach specimens. The quantitative detection limits of the (UV-Vis) spectrophotometer for standard pesticide solutions were determined as follows: cypermethrin (0.52, 1.58 μg/ml), chlorpyrifos (0.53, 1.61 μg/ml), Imidacloprid (0.64, 1.95 μg/ml), metalaxyl (0.27, 8.32 μg/ml), Aldicarb (0.90, 2.73 μg/ml), DDA (3.50, 10.62 μg/ml), and Endrin (1.89, 5.73 μg/ml). The findings revealed detectable residues of (cypermethrin and Imidacloprid) exclusively in samples from the Al-Marj region, according to the instrument's limit of detection (LOD). The detected concentrations were below the maximum residue limits established by the European Union (0.860 μg/ml).
Keywords: Residual Pesticide, Peach, Libya.

Introduction

Peach (Prunus persica), a member of the large plant family Rosaceae, is one of the most popular stone fruits worldwide. Peaches are nutritious and delicious summer fruits grown in temperate zones ranging from 30° to 45° latitude in both hemispheres [1]. In 2022, China was the biggest producer of peaches, with a production volume of 16.8 million metric tons, accounting for 63.7% of global production. Four Mediterranean countries (Italy, Turkey, Greece, and Spain) contribute about 15% to world production. Other top-producing countries were the United States (2.5%), Iran (2.2%), Chile (1.2%), Egypt (1.0%), and Morocco (0.9%). Turkey was the third-largest producer, with more than 1 million metric tons in 2022. Peaches are a rich source of health-promoting bioactive compounds such as flavonoids (flavan-3-ols, anthocyanins), phenolic compounds (hydroxycinnamic acids), and carotenoids (lutein, zeaxanthin, and beta-cryptoxanthin). These chemicals operate as free radical scavengers, supporting healthy aging and reducing the risk of numerous diseases [2-3]. Brown rot (Monilinia fructicola, Monilinia laxa), peach leaf curl (Taphrina deformans), powdery mildew (Sphaerotheca pannosa), peach scab (Cladosporium carpophilum), shot hole (Wilsonomyces carpophilus), and bacterial spot (Xanthomonas spp.) are among the most frequent diseases that affect peaches and nectarines [4]. There are also many insect pests (mites, aphids, thrips, and leafhoppers) that affect peach production in Turkey. Some of the most common of these are European red mites (Panonychus ulmi), brown mites (Bryobia rubrioculus), webspinning spider mites (Tetranychus urticae), black peach aphid (Brachycaudus persicae), Western flower thrips (Frankliniella occidentalis), peachtree borer (Synanthedon exitiosa), and Mediterranean fruit fly (Ceratitis capitata). Pest infestations pose a significant threat to agricultural productivity, leading to substantial losses in crop yields. According to the FAO, plant pests and diseases are responsible for a reduction of approximately 20–40% in global crop yields annually. These diseases and pests affecting stone fruits can be managed efficiently by employing a combination of sanitation, resistance, culture, and pesticide treatments. This comprehensive disease management method reduces dependence on a single type of control and typically leads to a high yield of top-quality fruit. Pesticides include insecticides for controlling a wide range of insects, fungicides for preventing mold and mildew growth, disinfectants for preventing bacterial transmission, herbicides for killing weeds and other undesired plants, and chemicals for controlling mice and rats. These plant protection products play a crucial role in plant health and food security. The primary goal of the current study was to determine the presence and concentration of multi-class residues in peaches. To achieve this, the quick, easy, cheap, effective, robust, and safe (QuEChERS) method is used. Several studies have been conducted in the Green Mountain region to estimate pesticide residues in locally produced vegetables and fruits. The results of these studies revealed the presence of various pesticide residues in several tested samples, indicating a potential risk to consumers' health due to the consumption of agricultural products containing pesticide traces [5-7]. In the last ten years the environmental studies take places in many studies for determination heavy metals in different samples [8-42], GC mass method is one of the most important methods used for determine pesticide and other

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aromatic compounds [43-51], many of studies on the treatment of pollutants were suggested [52-58], The contents of radioactive elements and their contamination effects were studied [59-62], also the studies on estimation the constituents in plant samples were carried out [63-99].In this study, the residual of one of the most dangerous compounds (pesticides) was estimated in some peach samples collected from farms around Al-Gabal Akhder region (Libya).

Methods

Sample Collection Areas

Samples were collected from different peach farms at various regions of (Qarnadah, Iqafnata, Abdul Wahid, Qasr Libya, and Al-Marj - Jabal al Akhdar – Libya), (Figure 1).

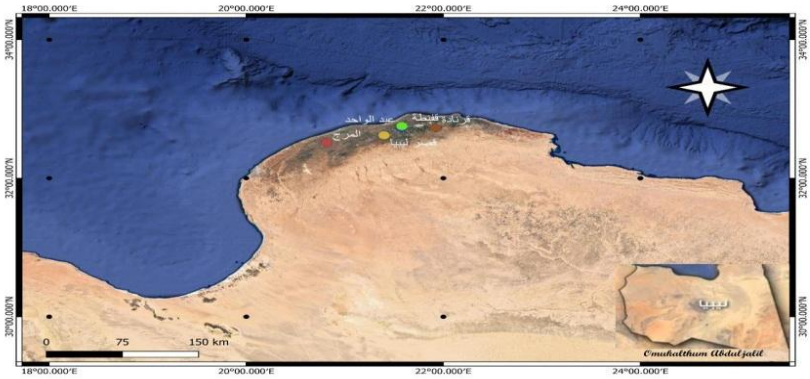


Figure 1. Study Locations

Peach Sample Collection

Six different species of peach fruits were collected during August and early September [2024]. Five hundred grams were collected from each sample. The region name and date of sample collection were recorded. The samples were transported in a cooled container directly to the laboratory. The collected samples were stored in a refrigerator at a temperature of 4°C until analysis (Hunt and Wilson, 1986; APHA, 1995. Seven standard pesticides were used: Imidacloprid, Aldicarb, Metalaxll, cypermethrin, chlorpyrifos, DDA, and Endrin). The purity of the standard pesticides was 99% (Augsburg, Germany).

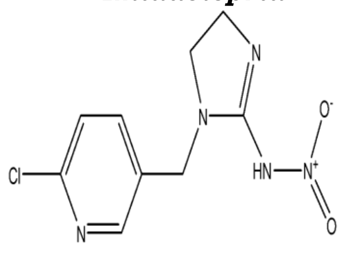
Chemicals Used

Acetonitrile 99.9%, primary secondary amine, anhydrous sodium sulfate 90%, and sodium chloride 90%.

Pesticide Selection

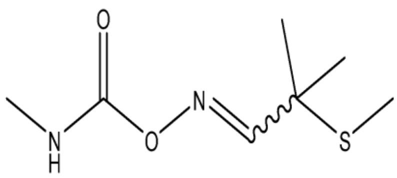
Most of the commonly used pesticides, as well as those banned globally, were used in this study. This selection was based on their presence in the samples and on instances where the maximum residue limits for the pesticides under study were exceeded.

Imidacloprid



N-(1-((6-chloropyridin-3-yl)methyl)-4,5-dihydro-1*H*-imidazol-2-yl)nitramide

Aldicarb



2-methyl-2-(methylthio)propanal *O*-methylcarbamoyl oxime

Metalaxll

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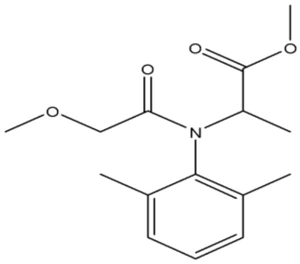
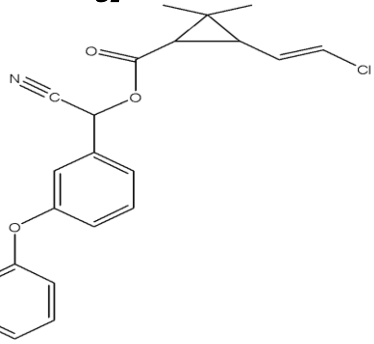


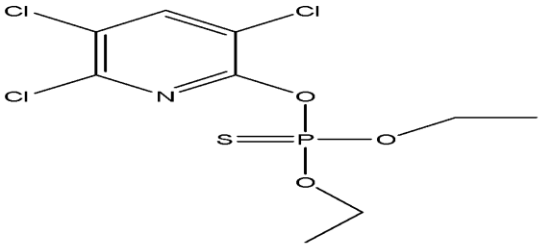
methyl 2-(N-(2-methoxyacetyl)-2,6-dimethylanilino)propanoate

Cypermethrin



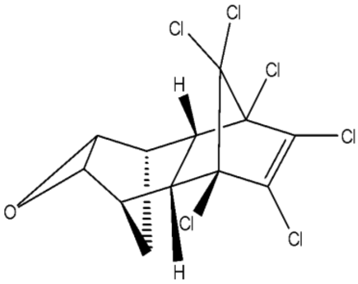
cyano(3-phenoxyphenyl)methyl (*E*)-3-(2-chlorovinyl)-2,2-dimethylcyclopropane-1-carboxylate

Chlorpyrifos



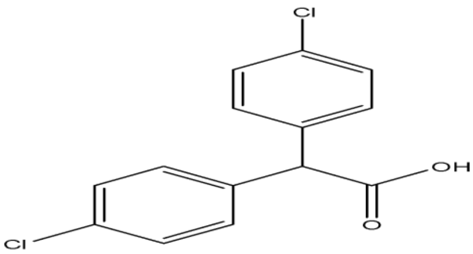
O,*O*-diethyl *O*-(3,5,6-trichloropyridin-2-yl) phosphorothioate

Endrin



(1a*R*,2*R*,2a*R*,3*R*,6*S*,6a*S*,7*S*,7a*S*)-3,4,5,6,9,9-hexachloro-1a,2,2a,3,6,6a,7,7a-octahydro-2,7:3,6-dimethanonaphtho[2,3-*b*]oxirene

DDA



2,2-bis(4-Chlorophenyl)acetic acid

Instrumentation for Pesticide Estimation

All spectrophotometric measurements were performed using a dual-beam UV/Vis spectrophotometer (Thermo Scientific, UK; Biotech Engineering Ltd., UK), with a wavelength range of 190-1100 nm, a spectral bandwidth of 2.0 nm, and a scanning speed of 400 nm/min. A quartz cell with a 1 cm path length was utilized for all UV/Vis spectrophotometric analyses.

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Preparation of Standard Pesticides

Accurately, 5 mg of each pesticide was weighed and dissolved in 50 mL of acetonitrile in a volumetric flask to obtain a stock concentration of 100 µg/ml. A series of concentrations was then prepared by diluting the stock solution to generate a calibration curve with different concentrations for each pesticide.

Extraction

The peach samples were extracted and analyzed following the QuEChERS method [6]. The peach samples were chopped using an electric blender. Approximately 10 g of the sample was transferred to a 50 ml centrifuge tube, followed by the addition of 10 ml of acetonitrile and (4 g of anhydrous magnesium sulfate + 1 g of sodium chloride). The mixture was then placed on a shaker for 30 seconds, followed by centrifugation at 5000 rpm for 5 minutes. Subsequently, 3 ml of the supernatant was transferred to a 15 ml centrifuge tube containing 600 mg of anhydrous magnesium sulfate and 120 mg of PSA. This was placed on a shaker for 30 seconds, followed by centrifugation at 5000 rpm for 5 minutes. Finally, the extract was transferred to a clean quartz cell for UV/Vis measurement.

UV Detection and Quantification Limits of the Instrument

The detection and quantification limits for the following pesticides were calculated. For cypermethrin, the values were between 0.52 and 1.58 µg/ml; for chlorpyrifos, 0.53 and 1.61 µg/ml; for Imidacloprid, 0.64 and 1.95 µg/ml; for metalaxyl, between 0.27 and 8.32 µg/ml; for Aldicarb, 0.90 and 2.73 µg/ml; for DDA, 3.50 and 10.62 µg/ml; and for Endrin, 1.89 and 5.73 µg/ml, respectively.

Results

The results indicated the detection of Cypermethrin and Imidacloprid residues in the Al-Marj region. No pesticides (Imidacloprid, Aldicarb, chlorpyifos, metalaxyl, cypermethrin, DDA, and Endrin) were detected in Qarnadah, Iqafnata, Abdul Wahid, or Qasr Libya. The residue of Imidacloprid recorded in Al-Marj was 0.04 µg/ml, and the residue of Imidacloprid was 0.02 µg/ml at Qarnadah area (Table1). These values were below the internationally permissible limits set by the European Union of 0.860 µg/ml [100].

Table 1. The peach samples in the study areas.

Al-Marj	Abdul Wahid	Qasr Libya	Iqafnata	Qarnadah	Pesticides
ND	ND	ND	ND	ND	Cypermethrin
ND	ND	ND	ND	ND	Metalaxll
ND	ND	ND	ND	ND	Endrin
ND	ND	ND	ND	ND	DDA
ND	ND	ND	ND	ND	Chlorpyrifos
ND	ND	ND	ND	ND	Aldicarb
0.04	ND	ND	ND	0.02	Imidacloprid

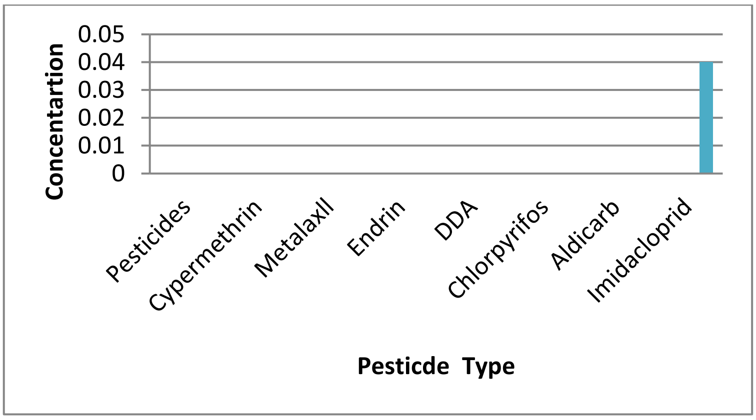

Figure 2. The residue of the Imidacloprid pesticide

Discussion

As illustrated in (Figure 2), there are statistically significant differences between the Al-Marj region and the other study areas. From these results, we can conclude that the presence of Imidacloprid residues may be attributed to the pesticide degradation period, and the values were within the globally permissible limits. In a similar study, the results obtained by previous studies [101], showed that pesticide concentrations were below the Maximum Residue Limits (MRLs). From these findings, it can be concluded that organophosphate

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and other insecticide residues were more significant in vegetables and fruits during the spring and summer seasons, when insect populations are more active. This leads to increased application of various insecticides, consequently resulting in the presence of residual pesticide concentrations in the study samples. However, excessive use of pesticides as well as other inappropriate applications of these chemicals can have a detrimental effect on the biodiversity of agricultural soil, water, and plants. This can have adverse effects on the well-being of humans, animals, and plants. Currently, global pesticide use surpasses 3.5 million tons per year, with over 1000 commercial pesticide formulations available, including chemical, microbiological, and botanical agents. Low concentrations of pesticide residues enter the human diet because of the extensive application of agricultural chemicals in food production [102-103].

Conclusion

No pesticides from the studied list (Imidacloprid, Aldicarb, metalaxyl, cypermethrin, DDA, Chlorpyriphos, Endrin) were detected in the peach samples from the study areas, apart from the Al-Marj region. In Al-Marj, residues of cypermethrin and Imidacloprid were found, with values below the maximum permissible limit set by the European Union. The presence of these insecticide residues is attributed to their widespread and extensive use on vegetable and fruit crops globally, owing to their high efficacy against insects. However, their residues pose a potential risk to public health. Therefore, from a health perspective, it is essential to control and regulate the application of pesticides on various crops in Libya.

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Conflict

No conflict of the results recorded in this study with other studies.

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