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# FATE, REMOVAL, RISK ASSESSMENT, AND BIOCHEMICAL EFFECTS OF INDOXACARB RESIDUES IN TOMATO FRUITS

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**ABSTRACT:** Field experiments were conducted on tomato fruits to investigate the residues, dissipation behavior, removal efficiency, health risk assessment, and biochemical impacts of indoxacarb (Tunchii 15% SC) during the winter season of 2023 in Mit Al-Qurashi village, Dakahlia Governorate, Egypt. Samples were collected at multiple intervals: 1 hour, 1, 3, 6, 9, 12, and 15 days post-application. The QuEChERS method was employed for residue extraction and clean-up, followed by analysis using HPLC. The initial indoxacarb residue level was 6.88 mg/kg, which declined gradually over time. The estimated half-life (t½) was 1.965 days, and the pre-harvest interval (PHI) was determined to be 12 days. Notably, no residues were detected in processed tomato paste made from contaminated fruits, indicating 100% removal. Furthermore, washing the tomatoes with tap water, acetic acid 5%, and sodium carbonate 5% for 5 minutes resulted in removal efficiencies of 28.84%, 34.58%, and 44.66%, respectively. While the health risk index indicated that tomatoes could be safely consumed after 15 days, converting the fruits into paste allowed for earlier safe consumption. Minor reductions were observed in certain quality parameters (e.g., total sugars and some minerals), whereas key nutrients such as protein, vitamin C, and beta-carotene showed no significant changes.

**Key words:** Indoxacarb, residues, Dissipation, Risk assessment, PHI, QuEChERS, tomato.

## INTRODUCTION

Tomatoes (*Solanum lycopersicum* L.) are among the most extensively cultivated and consumed vegetables worldwide, primarily due to their high nutritional value and numerous health-promoting properties. They are particularly rich in antioxidants, including ascorbic acid (vitamin C), vitamin E, carotenoids (such as lutein and lycopene), flavonoids, and phenolic acids. These compounds have been linked to a variety of health benefits, such as improved skin health, weight management, cardiovascular protection, regulation of blood pressure, prevention of cancer and diabetes, relief from constipation, and support for healthy vision **(Mahugija *et al*., 2021)**.

However, tomato cultivation is frequently challenged by various insect pests, including fruit borers, armyworms, *Tuta absoluta*, and leaf miners. To control these pests, synthetic insecticides are commonly used due to their fast-acting nature and high effectiveness. However, the widespread and prolonged use of such chemicals has raised serious concerns regarding their environmental impact, their role in fostering insecticide-resistant pest populations, and their potential harmful effects on human health **(Arowolo *et al*., 2022).**

Oxadiazine insecticides are derivatives of oxadiazine. Indoxacarb is the only member of this class. It is used for control of a wide range of lepidopterous insects in corn, vegetables, and fruit (**Simon, 2011**, added other new references). Indoxacarb is an insecticide that is readily metabolized by an esterase/ amidase to its corresponding N-decarbomethoxylated metabolite. The metabolite is a potent sodium channel

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blocker in insects, leading to flaccid paralysis and death (**Simon, 2011; Moustafa *et al.*, 2023**).

In recent years, growing public awareness regarding pesticide residues in food has sparked increased interest in developing simple and effective household methods for residue reduction. Numerous studies have examined the efficacy of domestic treatments such as rinsing with water, vinegar, baking soda, or commercial produce washes. While some of these approaches can significantly reduce surface-level residues, their effectiveness is influenced by several factors, including the physicochemical properties of the pesticide, the type and duration of the treatment, and the surface characteristics of the product. In certain cases, pesticides may penetrate the peel or systemically absorbed into the fruit tissue, thereby limiting the success of external washing methods **(Ahmad *et al*., 2024)**.

Therefore, this study aims to evaluate the residual behavior of indoxacarb on/in tomato fruits by determining its dissipation rate, half-life, and pre-harvest interval (PHI). It also examines the effectiveness of various household washing solutions and processing techniques (e.g., tomato paste preparation) in reducing residue levels. Additionally, the study explores the impact of indoxacarb on selected quality parameters and essential mineral content in tomatoes, analyzes matrix effects, and assesses the potential health risks associated with the consumption of contaminated fruits. The results of this research are intended to support food safety initiatives and guide informed pesticide management practices.

# MATERIALS AND METHODS

## Chemical and Reagents

Indoxacarb (>99% purity) reference analytical standard was obtained from Dr. Ehrenstorfer GmbH (Augsburg, Germany). HPLC-grade methanol, acetonitrile, and glacial acetic acid were purchased from Sigma (Sigma GmbH, Darmstadt, Germany). Primary secondary amine (PSA, 40 µm Bondesil) and graphitized carbon black (GCB) sorbents were obtained from Supelco (Bellefonte, Pennsylvania, USA). Analytical-grade anhydrous magnesium sulfate and sodium chloride were purchased from CARLO ERBA Reagents S.A.S. The commercial formulation of indoxacarb (Tunchii 15% SC) was procured from the local market and manufactured by Astra Nova Tarim Techart.

### Preparation of standard solutions

For HPLC analysis, a stock solution of indoxacarb was initially prepared at a concentration of 100 mg/L using acetonitrile as the solvent. This solution was then serially diluted to obtain the necessary working standards and spiking solutions. All prepared solutions, including standards and dilutions, were stored at 4 °C to ensure stability.

### Field experiment and sampling

During the fruiting stage, tomato plants grown in a private field in Mit Al-Qurashi Village, Dakahlia Governorate, Egypt, were treated with indoxacarb (Tunchii 15% SC) at the recommended application rate of 26.5 $cm^3$ per 100 liters of water. Control plots were sprayed with water only. After spraying, tomato samples were collected from each replicate at specific intervals: 1 hour, 1, 3, 6, 9, 12, and 15 days post-application. From each treatment, 2 kg of fruit was collected for residue analysis. Control samples were collected at the same time points. Immediately after collection, samples were placed in polyethylene bags and transported to the laboratory in an ice box. Upon arrival, the samples were roughly chopped and homogenized using a food processor (HOBART). The resulting homogeneous matrix was stored in sealable plastic bags at -20°C until analysis.

### Extraction and Clean-Up

Tomato samples were extracted and cleaned following the QuEChERS method described by **Lehotay (2007)**. Briefly, 10±0.1 g of homogenized tomato sample was weighed into a 50 mL centrifuge tube. Then, 10 mL of acetonitrile containing 1.0% acetic acid was added, and the mixture was shaken vigorously for 1 minute. Subsequently, 1 g of sodium chloride and 4 g of anhydrous magnesium sulfate were added, followed by another round of shaking and centrifugation at 4000 rpm for 5 minutes.

For the clean-up step, 1 mL of the resulting supernatant was transferred to a new tube containing 25 mg of primary secondary amine

(PSA), 150 mg of anhydrous magnesium sulfate, and 10 mg of graphitized carbon black (GCB). The mixture was shaken for 1 minute and centrifuged again at 4000 rpm for 5 minutes. The final supernatant was filtered through a 0.22 µm PTFE syringe filter (Millipore, Billerica, MA), and 0.5 mL of the filtrate was transferred into a vial for subsequent analysis by high-performance liquid chromatography (HPLC).

**Instrumentation**

Chromatographic analysis was performed using a high-performance liquid chromatography (HPLC) system (Agilent 1260 Infinity series) equipped with a quaternary pump, an autosampler, and a diode array detector (DAD). Separation was achieved using a Nucleosil C18 analytical column (30 × 4.6 mm i.d., 5 µm particle size). The mobile phase consisted of acetonitrile and water in a ratio of 90:10 (v/v), delivered at a flow rate of 1.0 mL/min. The injection volume was set to 20 µL, and detection was carried out at a wavelength of 205 nm.

**Method Validation**

According to the **SANTE guidelines (2021)**, the following validation parameters were examined: linearity, matrix effect, recovery, and LOQ (the lowest spiking level). Linearity was evaluated by constructing a six-point calibration curve using standard solutions at concentrations of 0.01, 0.1, 0.5, 1, 2.5, and 5 mg/L. The correlation coefficient ($R^2$) was calculated by plotting peak area responses against the corresponding concentrations in the solvent.

To account for potential matrix interferences, matrix-matched calibration (MMC) was employed. The matrix effect (ME) was defined as the alteration in analyte response due to co-extracted matrix components. It was determined by comparing the detector responses of the analyte in pure solvent with those in matrix-matched standards fortified at the same concentration levels (0.01, 0.1, 0.5, 1, 2.5, and 5 mg/ kg).

The matrix effect percentage (%ME) was calculated using the following equation:

$$ME (\%) = [(M_{matrix} - M_{solvent}) / M_{solvent}] \times 100$$

Where:

$\mathbf{M_{matrix}}$ = slope of the calibration curve prepared in the matrix

$\mathbf{M_{solvent}}$ = slope of the calibration curve prepared in pure solvent

## Recovery Assessment

To evaluate the efficiency of the extraction, cleanup, and quantification procedures, untreated tomato fruit samples were spiked with known concentrations of the insecticide standard solution at three levels: 0.1, 0.5, and 1.0 mg/kg. The spiked samples were processed and analyzed as previously described.

The recovery percentage was calculated using the following equation:

$$\% \text{ Recovery} = (\text{Amount detected} / \text{Amount added}) \times 100$$

The results of the recovery test are summarized in Table 1, showing that the mean recovery value was 99.32%, indicating high method reliability. All analytical results were adjusted based on the recovery rates obtained.

## Removal Trials of Indoxacarb Residues through Household Processing

To assess the effectiveness of common household processing methods in reducing indoxacarb residues in tomato fruits, approximately two kilograms of tomatoes were collected one day after insecticide application. The fruits were soaked for 5 minutes in jars containing different washing solutions: tap water, acetic acid ($CH_3COOH$5) %, and sodium carbonate ($Na_2CO_3$) 5%, following the method of **Pekel (2023)**.

After washing, the samples were air-dried on clean paper towels and stored appropriately. For paste preparation, the washed tomato juice was concentrated at 100 °C with the addition of 2.5% sodium chloride (NaCl) until a paste was formed, according to **Shalaby *et al.* (2022)**.

To quantify the impact of each processing step, Processing Factors (PFs) were calculated as the ratio of pesticide residue concentration (mg/kg) in the processed product to that in the corresponding raw (unprocessed) sample. A PF less than 1 indicates a reduction in residue levels due to processing, while a PF greater than 1 suggests an increase—typically resulting from dehydration or mass reduction during processing, rather than actual pesticide addition **(Bonnechère *et al*., 2012).**

**Table 1. Fortification levels and recovery percentage (±RSDr) of indoxacarb in tomato fruits for HPLC analyzed**

| Spiking level (mg/kg) (n=5) | Mean recovery (%RSD) | RSDr% |
|---|---|---|
| **0.1** | 98.42±1.36 | 1.38 |
| **0.5** | 98.86±4.83 | 2.41 |
| **1** | 100.68±2.47 | 1.006 |
| **Mean** | 99.32 | |

The formula used was:

PF = Residue concentration in processed product (mg/kg) / Residue concentration in raw product (mg/kg)

## Residual Effects of Indoxacarb on Biochemical Constituents of Tomato Fruits

To evaluate the residual effects of indoxacarb on the biochemical quality of tomato fruits, samples from both treated and untreated plots were collected at 3, 6, and 9 days post-application. The analysis focused on several key quality parameters, including total soluble sugars, glucose, titratable acidity, total soluble solids (TSS), ascorbic acid (vitamin C), β-carotene, crude protein, and dry matter content.

In addition, the concentrations of trace essential elements, namely nitrogen (N), phosphorus (P), potassium (K), iron (Fe), manganese (Mn), calcium (Ca), and zinc (Zn) were also determined according to the methods described by **Shalaby (2016).**

## Health Risk Assessment

To assess the potential health risks associated with insecticide residues in tomato fruits, the Estimated Average Daily Intake (EADI) and the Health Risk Index (HRI) also known as the Risk Quotient (RQ) were calculated using the following equations:

$$EADI=CRL\times FI\times 100/b.w$$

$$HRI \text{ or } RQ= EADI/ADI$$

Where:

CRL: Mean concentration of insecticide residues in tomato samples (mg/kg).

FI: Food intake, representing the average daily tomato consumption, estimated at 0.118 kg/day **(Ibrahim *et al.*, 2022)**.

100: A general safety factor commonly used in dietary exposure assessments **(Malhat *et al.*, 2014; Szpyrka *et al.*, 2015).**

b.w: Average adult body weight, assumed to be 80 kg **(Ahmed *et al.*, 2016; Taghizadeh *et al.*, 2019).**

ADI: Acceptable Daily Intake, as defined by **FAO/WHO (2010, 2015).**

An HRI (or RQ) value greater than 1 indicates a potential health risk, while values below 1 suggest the exposure is within acceptable safety limits.

Subsequently, the Health Risk Index (HRI), also known as the Risk Quotient (RQ), was calculated by comparing the Estimated Average Daily Intake (EADI) with the Acceptable Daily Intake (ADI) as defined by international regulatory guidelines.

An HRI (or RQ) value greater than 1 indicates a potential health risk resulting from insecticide exposure through tomato consumption. Conversely, a value equal to or less than 1 suggests that dietary exposure is within acceptable safety limits and is considered safe for human health **(Hamilton and Crossley, 2004; Darko and Akoto, 2008).**

### Statistical analysis and kinetic studies

A one-way ANOVA and Fisher's LSD test in Microsoft Excel 2021 were used to assess field findings and identify significant differences between groups; a P value of less than 0.05 was deemed significant. The degradation rate constant (K) and the associated half-life value (t½) for the insecticide indoxacarb was calculated based on the following formulas: K=2.303× slope, and t½ = 0.693/K. **(Gomaa and Belal, 1975)**.

# RESULTS AND DISCUSSION

## Method Validation

In this study, the analytical method was validated according to the SANTE/11312/2021 guidelines, using several standard validation parameters:

### Linearity

To assess the linearity of the method for indoxacarb, calibration curves were constructed using six concentration levels ranging from 0.01 to 5 mg/L. The relationship between analyte concentration and detector response was evaluated using the least squares regression method.

The calibration curve exhibited excellent linearity, with a correlation coefficient ($R^2$) greater than 0.9986, indicating a strong linear relationship.

The regression equation was found to be: y = 61.689 + 12.526x, where y represents the peak area and x the concentration of indoxacarb in mg/kg. Fig. 1 illustrates the calibration curve for indoxacarb.

## Matrix Effect (ME%) Evaluation

To evaluate the impact of co-extracted matrix components on the detection sensitivity of HPLC analysis, the matrix effect (ME%) was assessed. The matrix effect reflects any alteration in analyte response, either enhancement or suppression—due to interfering substances present in the sample matrix.

In this study, the matrix effect for indoxacarb was evaluated by comparing the slopes of calibration curves prepared in pure solvent (acetonitrile) and in tomato matrix extract. The ME% was calculated using the following formula:

ME% = [(Slope of matrix-matched calibration – Slope of solvent calibration) / Slope of solvent calibration] × 100

### Interpretation of ME% values

0% indicates no matrix effect,

Positive ME% suggests signal enhancement,

Negative ME% indicates signal suppression.

For indoxacarb, a positive matrix effect of +240% was observed, indicating a significant enhancement in detector response due to the presence of matrix components in the tomato extract.

This result emphasizes the critical need for matrix-matched calibration to ensure accurate quantification of pesticide residues in complex food matrices such as tomato.

### Limit of quantification (LOQ)

The limit of quantification (LOQ) is a significant measure for evaluating the accuracy and precision of analytical methods. It designates the minimum concentration of target substances that can be reliably detected within a specified matrix, corresponding to a signal-to-noise ratio of 10 **(Su *et al.*, 2020)**. The LOQ has been established at 0.1 mg/kg. According to the guidelines provided by SANTE/11312/2021, LOQ value is considered acceptable if they do not exceed the maximum residue limit (MRL) set for these substances. The established MRLs for indoxacarb in tomato is 0.5 mg/kg according to Codex Alimentarius.

### Residues of indoxacarb insecticide

The data presented in Table 2 and Fig. 2 illustrate the initial deposits and dissipation behavior of indoxacarb residues on and within tomato fruits following foliar application. One-hour post-application, the initial residue level was 6.88 mg/kg. Residue concentrations subsequently declined over time, reaching 5.05, 3.21, 2.04, 0.73, 0.41, and 0.013 mg/kg after 1, 3, 6, 9, 12, and 15 days, respectively. These values correspond to dissipation percentages of 26.59%, 53.34%, 70.34%, 89.38%, 97.96%, and 99.81%, respectively.

Based on dissipation dynamics, the Pre-Harvest Interval (PHI) was established at 12 days, ensuring that residue levels remain within the Maximum Residue Limit (MRL) for consumer safety.

The half-life ($RL_{50}$) of indoxacarb was calculated to be 1.965 days, indicating a relatively fast degradation rate. The corresponding dissipation

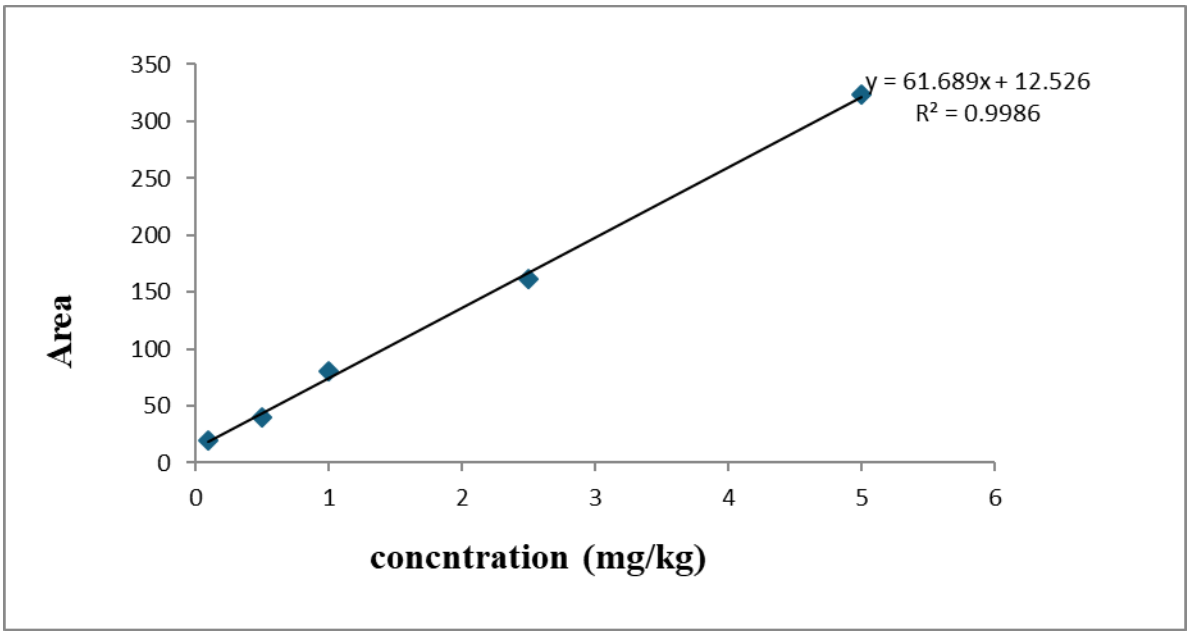


**Fig. 1. Calibration curve of indoxacarb with HPLC-DAD analysis**

**Table 2. Residue levels and dissipation behavior of indoxacarb in tomato fruits under field conditions**

| Intervals (days) | Residues (mg/kg) | % Loss | % persistence |
|---|---|---|---|
| **0** | 6.88±0.40 | 0 | 100 |
| **1** | 5.05±0.25 | 26.59 | 73.41 |
| **3** | 3.21±0.48 | 53.34 | 46.66 |
| **6** | 2.04±0.10 | 70.34 | 29.66 |
| **9** | 0.73±0.08 | 89.38 | 10.62 |
| **12** | 0.41±0.04 | 97.96 | 2.04 |
| **15** | 0.013±0.023 | 99.81 | 0.19 |
| **MRL** | 0.5 mg/kg(Codex) | | |
| **PHI (days)** | 12 days | | |
| **RL50 (days)** | 1.965 days | | |
| **K (days)** | 0.3527 days | | |

Whereas; MRL = Maximum Residue Limit, PHI = Pre-Harvest Interval, RL50 = Time required for 50% residue dissipation, K=rate of degradation.

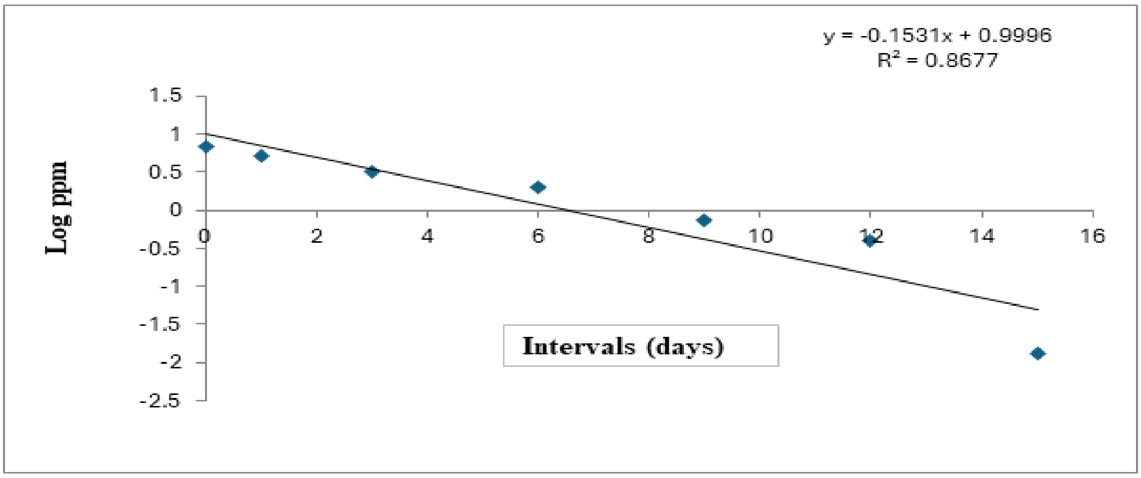


**Fig. 2. Log residue-day regression line of indoxacarb residue in tomato fruits under field conditions**

rate constant (K) was 0.3527 day$^{-1}$, confirming the rapid breakdown of the insecticide under the experimental conditions.

These findings indicate that indoxacarb dissipates significantly over time and reaches safe residue levels before harvest, supporting its suitability for tomato application when Good Agricultural Practices (GAPs) are followed.

Moreover, the observed residue behavior aligns with previous studies that reported similar dissipation trends of indoxacarb and other insecticides in various vegetable crops (**Gaaboub, 2015; Anita *et al*., 2018; Kaur *et al*., 2023; Shalaby *et al*., 2022; Sardar *et al*., 2023**), thereby reinforcing the validity and reliability of the current findings.

## Removal Trials of Indoxacarb Residues from Treated Tomato Fruits

This study investigated the effects of common household and processing techniques on the removal of indoxacarb residues from contaminated tomato fruits. The evaluated processes included washing with tap water, acetic acid ($CH_3COOH$) 5%, sodium carbonate ($Na_2CO_3$) 5%, and tomato paste preparation (thermal processing).

The results, presented in Table 3, clearly demonstrate that all tested washing treatments reduced indoxacarb residues compared to untreated (unwashed) tomato fruits collected one day after spraying, which initially contained 5.05 mg/kg of the insecticide. Washing with tap water reduced the residue to 3.59 mg/kg, representing a 28.84% reduction. Washing with 5% acetic acid further decreased the residue to 3.30 mg/kg, equating to a 34.58% reduction. Washing with sodium carbonate solution proved most effective among washing methods, reducing the residue to 2.79 mg/kg, or 44.66%.

Notably, thermal processing by converting the tomatoes into paste resulted in a complete removal of indoxacarb residues, reducing them to undetectable levels, equivalent to a 100% removal rate. These findings suggest that residue reduction efficiencies of the tested methods can be ranked in descending order as follows: Tomato paste preparation (thermal processing) > Sodium carbonate washing > Acetic acid washing > Tap water washing. In terms of Processing Factor (PF), lower PF values indicated more effective residue removal, as seen clearly in Table 3.

Furthermore, although safe consumption based on MRL was achieved after 12 days post-application under field conditions, this interval can be significantly shortened if the tomatoes are processed into paste just one day after treatment. These results agree with findings from previous studies investigating the removal of indoxacarb and other pesticide residues from tomatoes and various vegetables (**Andrade *et al*., 2015; Kwon *et al*., 2015; Rodrigues *et al*., 2017; Sakthiselvi *et al*., 2020; Shalaby *et al*., 2022; Qi *et al*., 2023**).

**Table 3. Removal Residue levels of indoxacarb in tomato fruits after one day of application.**

| PROCESS | Residues (ppm) | % Loss | Processing Factor |
|---|---|---|---|
| **Unwashed** | 5.05±0.25 | 26.59 | - |
| **Water** | 3.59±0.51 | 28.84 | 0.71 |
| **Acetic acid** | 3.30±1.11 | 34.58 | 0.65 |
| **$Na_2CO_3$** | 2.79±1.71 | 44.66 | 0.55 |
| **Paste** | UND | 100 | 0 |

## Risk Assessment of Indoxacarb Residues in Tomato Fruits

The concentrations of indoxacarb residues in both treated and untreated tomato samples were assessed in comparison with the Maximum Residue Limits (MRLs) established by international regulatory authorities, including Codex Alimentarius, the European Union, and the U.S. Environmental Protection Agency (EPA). This evaluation aimed to determine an appropriate Pre-Harvest Interval (PHI) that ensures consumer safety.

In addition to MRL comparisons, Health Risk Indices (HRIs) were calculated to estimate the potential dietary risk associated with indoxacarb residues. The Estimated Average Daily Intake (EADI) was computed based on average tomato consumption rates and residue levels detected in samples collected at different intervals post-application. The EADI values were then compared to the Acceptable Daily Intake (ADI) to evaluate potential acute and chronic health risks.

According to the established risk assessment guidelines:

An HRI value > 1 indicates a potential health risk for consumers.

An HRI value ≤ 1 suggests that dietary exposure remains within acceptable safety margins **(Hamilton and Crossley, 2004; Darko and Akoto, 2008; Ahmed *et al.*, 2016; Liu *et al.*, 2019; Ibrahim *et al.*, 2022).**

As shown in Table 4, the calculated EADI and corresponding HRI values for indoxacarb residues over time indicate that the insecticide residues decrease to safe levels 15 days after application. Therefore, a minimum waiting period of 15 days is recommended before the tomatoes treated are considered safe for consumption.

These findings are in line with those of prior studies evaluating indoxacarb and other pesticide residues in tomato fruits and other vegetable crops **(Shalaby *et al.*, 2020; Ibrahim *et al.*, 2020; Hunter and Helmy, 2021; Odewale *et al.*, 2021; Ibrahim *et al.*, 2022; El-Sheikh *et al.*, 2022; El-Sheikh *et al.*, 2023)**.

## Effect of Indoxacarb Residues on Internal Quality Parameters and Trace Elements in Tomato Fruits

Regarding the impact of indoxacarb application on internal quality parameters and both macro- and micronutrient elements in tomato fruits, the following findings were observed, as presented in Tables (5–10).

The application of indoxacarb resulted in slight reductions in the concentrations of macronutrients (N, P, K, and Ca), with reduction percentages ranging between 0.502% and 4.619%. Similarly, micronutrients (Fe, Mn, and Zn) showed decreases ranging from 0.277% to 1.77%, as illustrated in Tables 5 and 6. The concentrations of N, P, and Ca in both treated and control samples increased significantly over time, suggesting a possible natural accumulation during fruit development. In contrast, the levels of K, Fe, Mn, and Zn in treated and control samples decreased significantly over time.

These findings suggest that indoxacarb has a minor impact on certain internal quality parameters. Slight reductions were observed in soluble solids and total sugar content, but there were no statistically significant changes in major

**Table 4. Health risk assessment of indoxacarb on treated tomato fruits**

| Time after spraying (days) | Residues (mg/kg) | EADI | HRI | Health risk |
|---|---|---|---|---|
| **Initial** | 6.88±0.40 | 1.0148 | 101.48 | Yes |
| **1** | 5.05±0.25 | 0.744875 | 75.4875 | Yes |
| **3** | 3.21±0.48 | 0.473475 | 47.3475 | Yes |
| **6** | 2.04±0.10 | 0.357 | 35.4 | Yes |
| **9** | 0.73±0.08 | 0.107675 | 10.7675 | Yes |
| **12** | 0.41±0.04 | 0.060475 | 6.0475 | Yes |
| **15** | 0.013±0.023 | 0.0019175 | 0.19175 | No |

**Initial**: One hour after spraying, **EADI:** Estimated Average daily intake, **HRI:** Health risk Indices and **ADI:** Acceptable daily intake for indoxacarb was 0.01 mg/kg.

**Table 5. Effect of indoxacarb on some internal macronutrient elements (N, P, K, and Ca) on tomato fruits**

| Treatments | Days after treatment | | | | | | General means | |
|---|---|---|---|---|---|---|---|---|
| | 3 | | 6 | | 9 | | | |
| | Levels | %Reduction | Levels | %Reduction | Levels | %Reduction | Levels | %Change |
| | | | | **Nitrogen %** | | | | |
| **Control** | 1.55803 a | _ | 1.8550 a | _ | 1.9923 a | _ | 1.809 a | 100.00 |
| **Indoxacarb** | 1.5173 b | 4.619 | 1.8300 b | 1.348 | 1.9823 b | 0.502 | 1.777 a | -1.679 |
| | | | | **Phosphorus %** | | | | |
| **Control** | 0.4830 a | _ | 0.6527 a | _ | 0.7910 a | _ | 0.6422 a | 100.00 |
| **Indoxacarb** | 0.4777 a | 1.097 | 0.6413 b | 1.747 | 0.7810 b | 1.264 | 0.6333 ab | -1.38 |
| | | | | **Potassium%** | | | | |
| **Control** | 2.1010 a | - | 2.4803 a | - | 2.5843 a | - | 2.3886 a | 100.00 |
| **Indoxacarb** | 2.0520 b | 2.332 | 2.4600 b | 0.818 | 2.5437 b | 1.571 | 2.3519 b | -1.536 |
| | | | | **Calcium%** | | | | |
| **Control** | 0.4467 a | - | 0.5900 a | - | 0.6967 a | - | 0.5783 a | 100.00 |
| **Indoxacarb** | 0.4433 a | 0.761 | 0.5800 b | 1.695 | 0.6900 a | 0.968 | 0.5710 a | -1.262 |

Levels with the same letter in each column are not significantly different.

**Table 6. Effect of indoxacarb on some internal micronutrient elements (iron, manganese and zinc) on tomato fruits**

| Treatments | Days after treatment | | | | | | General means | |
|---|---|---|---|---|---|---|---|---|
| | 3 | | 6 | | 9 | | | |
| | Levels | %Reduction | Levels | %Reduction | Levels | %Reduction | Levels | Change |
| | | | | Iron % | | | | |
| Control | 46.320 a | - | 36.187 a | - | 33.987 a | - | 38.831 a | 100.00 |
| Indoxacarb | 46.097 b | 0.481 | 36.123 a | 1.77 | 33.890 a | 0.285 | 38.703 a | -0.329 |
| | | | | Manganese % | | | | |
| Control | 34.467 a | - | 24.847 a | - | 22.707 a | - | 27.340 a | 100.00 |
| Indoxacarb | 34.173 b | 0.853 | 24.740 b | 0.431 | 22.603 a | 0.458 | 27.172 b | -0.614 |
| | | | | Zinc % | | | | |
| Control | 17.877 a | - | 17.497 a | - | 16.997 a | - | 17.457 a | 100.00 |
| Indoxacarb | 17.737 a | 0.783 | 17.433 a | 0.366 | 16.950 a | 0.277 | 17.373 a | -0.481 |

Levels with the same letter in each column are not significantly different.

**Table 7. Effect of indoxacarb on total sugars and glucose (as quality parameters) on tomato fruits**

| Treatments | Days after treatment | | | | | | General means | |
|---|---|---|---|---|---|---|---|---|
| | 3 | | 6 | | 9 | | | |
| | Levels | %Reduction | Levels | %Reduction | Levels | %Reduction | Levels | Change |
| | | | | Total sugars % | | | | |
| Control | 4.0800 a | - | 3.3400 a | - | 3.3700 a | - | 3.5967 a | 100.00 |
| Indoxacarb | 4.0233 a | 1.389 | 3.2700 b | 2.096 | 3.1867 b | 5.439 | 3.4933 a | -2.875 |
| | | | | Glucose% | | | | |
| Control | 21.917 a | - | 18.947 a | - | 17.863 a | - | 19.576 a | 100.00 |
| Indoxacarb | 21.823 a | 0.429 | 18.800 a | 0.776 | 17.700 b | 0.913 | 19.441 a | -0.689 |

Levels with the same letter in each column are not significantly different.

**Table 8. Effect of indoxacarb on acidity, total soluble solids and dry weight on tomato fruits**

| Treatments | Days after treatment | | | | | | General means | |
|---|---|---|---|---|---|---|---|---|
| | 3 | | 6 | | 9 | | | |
| | Levels | %Reduction | Levels | %Reduction | Levels | %Reduction | Levels | Change |
| | | | | Acidity% | | | | |
| Control | 2.2767 a | - | 2.6800 a | - | 2.3200 a | - | 2.4256 a | 100.00 |
| Indoxacarb | 2.2267 b | 2.196 | 2.6200 ab | 2.231 | 2.2467a | 3.159 | 2.3645 b | -2.520 |
| | | | | Total soluble solids % | | | | |
| Control | 8.8233 a | - | 8.4667 a | - | 8.5267 a | - | 8.605 ab | 100.00 |
| Indoxacarb | 7.7367 ab | 12.375 | 7.3800 b | 12.835 | 8.0467 b | 5.629 | 7.7211 b | -10.278 |
| | | | | Dry weight % | | | | |
| Control | 20.730 a | - | 20.247 a | - | 18.857 a | - | 19.944 a | 100.00 |
| Indoxacarb | 20.617 a | 0.026 | 20.187 a | 0.296 | 18.643 a | 1.135 | 19.816 a | -0.642 |

Levels with the same letter in each column are not significantly different.

**Table 9. Effect of indoxacarb on ascorbic acid and beta carotene (as quality parameters) on tomato fruits**

| Treatments | Days after treatment | | | | | | General means | |
|---|---|---|---|---|---|---|---|---|
| | 3 | | 6 | | 9 | | | |
| | Levels | %Reduction | Levels | %Reduction | Levels | %Reduction | Levels | Change |
| | | | | Ascorbic acid % | | | | |
| **Control** | 14.820 a | - | 15.437 a | - | 17.000 a | - | 15.752 a | 100.00 |
| **Indoxacarb** | 14.760 a | 0.405 | 15.337 a | 0.648 | 16.880 a | 0.706 | 15.659 a | -0.590 |
| | | | | Beta carotene | | | | |
| **Control** | 4.9100 a | - | 5.1500 a | - | 5.6900 a | - | 5.2500 a | 100.00 |
| **Indoxacarb** | 4.8333 a | 1.562 | 5.1133 a | 0.713 | 5.6033 b | 1.524 | 5.1833 a | -1.270 |

Levels with the same letter in each column are not significantly different.

**Table10. Effect of indoxacarb on protein (as quality parameters) on tomato fruits.**

| Treatments | Days after treatment | | | | | | General means | |
|---|---|---|---|---|---|---|---|---|
| | 3 | | 6 | | 9 | | | |
| | Levels | %Reduction | Levels | %Reduction | Levels | %Reduction | Levels | Change |
| | | | | Protein | | | | |
| **Control** | 9.8771 a | - | 11.594 a | - | 12.452 a | - | 11.3680 a | 100.00 |
| **Indoxacarb** | 9.4833 b | 4.094 | 11.438 b | 1.346 | 12.390 b | 0.498 | 11.1030 a | -1.813 |

Levels with the same letter in each column are not significantly different.

nutritional indicators such as protein, ascorbic acid, or beta-carotene. This limited impact may be attributed to the rapid degradation of indoxacarb residues and the relatively low application rate used. These results are consistent with previous studies **(Malhat *et al.*, 2014; Darko and Akoto, 2008),** which also reported minimal changes in fruit quality following insecticide application, with all parameters remaining within acceptable limits for human consumption.

The current findings are in agreement with those reported by **Shalaby and Gad (2016), Shalaby (2017), Rodrigues *et al*. (2017), Abrokwah *et al.* (2019), Salem (2020), Amin *et al*. (2022), Mhya *et al.* (2024) and Abdelfatah *et al.* (2024)**, who confirmed that insecticides such as indoxacarb have limited effects on fruit nutritional quality when applied according to recommended guidelines.

## Conclusion

This study provides essential data on indoxacarb residues in tomatoes under Egyptian field conditions. The results underscore the importance of respecting appropriate pre-harvest intervals to ensure food safety. Moreover, proper washing of tomato fruits prior to consumption is recommended as an effective step to reduce insecticide residues and associated health risks. The findings also indicate that indoxacarb residues can affect certain fruit quality attributes and alter levels of trace and essential nutrients, particularly at the mature stage. Therefore, careful management of insecticide application is crucial. Overall, the study highlights the need for strict regulatory control and routine monitoring of insecticide residues in food commodities to protect consumer health and support safe agricultural practices.

## REFERENCES

Abdelfatah, R.M., Soliman, H.M. and Helmy, R. (2024). Depreciation, risk assessment study of pymetrozine, pyriproxyfen, and acetamiprid residues using QuEChERS and HPLC (DAD) and their biochemical effects on eggplant under greenhouse conditions. Journal of Plant Protection and Pathology, 15 (7), 191–197.

Abrokwah, F.K., Matthew, M., Danso, P.C. and Affoh, A. A. (2019). Study on the effects of acetamiprid and lambda-cyhalothrin application on some biochemical parameters of onion and tomato. International Research Journal of Food and Nutrition, 12, 7237–7252.

Ahmad, M.O.H., Abdel-Tawab, S.A., Ahmed, L. A.R. and Roby, M.H. (2024). Quality Control of Tomato Products and it's Influenced by Some Technological Treatments on Reducing Pesticide Residues. Labyrinth: Fayoum Journal of Science and Interdisciplinary Studies, 2 (1), 21-34.

Ahmed, M.A.I., Abd El Rahman, T.A. and Khalid, N.S. (2016). Dietary intake of potential pesticide residues in tomato samples marketed in Egypt. Research Journal of Environmental Toxicology, 10 (4), 213-219.

Amin, S., Salim, M., Nawaz, T. and Anjum, F. (2022). Pesticide residue analysis of three different pesticides used against *Helicoverpa armigera* (Hubner) in tomato crop. Sarhad Journal of Agriculture, 38 (2), 448–455. https://doi.org/10.17582/journal.sja/2022/38.2.448.455

Andrade, G.C., Monteiro, S.H., Francisco, J.G., Figueiredo, L.A., Rocha, A.A. and Tornisielo, V. L. (2015). Effects of types of washing and peeling in relation to pesticide residues in tomatoes. Journal of the Brazilian Chemical Society, 26, 1994-2002.

Anita, V. K., Ahlawat, S. and Chauhan, R. (2018). Dissipation pattern and effect of household processing on reduction of Indoxacarb residues in tomato fruits (*Lycopersicon esculentum* Mill.). Journal of Entomology and Zoology Studies, 6(6), 814-819.

Arowolo, O. K., Gbaye, O. A. and Aborisade, A. T. (2022). Effect of household processing methods on insecticide residues in two tomato cultivars from the market in Ondo State, Nigeria. Toxicology and Environmental Health Sciences, 14(4), 371-378.

Bonnechère, A., Hanot, V., Bragard, C., Bedoret, T. and van Loco, J. (2012). Effect of household and industrial processing on the levels of pesticide residues and degradation products in melons. Food Additives & Contaminants: Part A, 29(7), 1058-1066.

Darko, G. and O. Akoto (2008). Dietary intake of organophosphorus pesticide residues through vegetables from Kumasi, Ghana. Food and Chemical Toxicology, 46: 3703–3706

El-Sheikh, E. S. A., Li, D., Hamed, I., Ashour, M. B. and Hammock, B. D. (2023). Residue analysis and risk exposure assessment of multiple pesticides in tomato and strawberry and their products from markets. Foods, 12 (10), 1936.

El-Sheikh, E. S. A., Ramadan, M. M., El-Sobki, A. E., Shalaby, A.A., McCoy, M.R., Hamed, I. A., Ashour, M-B. and Hammock B.D. (2022). Pesticide residues in vegetables and fruits from farmer markets and associated dietary risks. Molecules, 27 (22), 8072.

FAO/WHO (2010) Pesticides residues in food and feed. Acceptable Daily intake; Codex Alimentrarius Commission, FAO/WHO Food standards.

FAO/WHO (2015). Pesticide residues in food and feed. Acceptable Daily Intake; Codex Alimentarius Commission, FAO/WHO Food standards.

Gaaboub, I. A. (2015). Potential Impacts of Climatic Changes on Indoxacarb Persistence and its Pre-harvest Interval in Tomato Fruits.

Gomaa, E.A.A. and Belal, M.H. (1975). Determination of dimethoate residues in some vegetables [tomatoes, cucumber, common bean] and cotton plants [in Egypt]. Zagazig Journal of Agricultural Research, *2*.

Hamilton, D. and Crossley, S. (Eds.). (2004). Pesticide residues in food and drinking water: human exposure and risks (Vol. 2). John Wiley and Sons.

Hunter, H. and Helmy, R.M.A. (2021). Validation using QuEChERS method, risk assessment and preharvest intermission using GC-MS for determination of azoxystrobin in tomato and cucumber. Egyptian Journal of Chemistry, 64 (12), 7421–7429.

Ibrahim, A., Khorshed, M. A., Eshmawy, M. R., and Kandil, M. A. (2020). Pesticide residues in horticultural crops from domestic markets of Giza, Egypt: Occurrence and risk assessment. Plant Archives, 20 (1), 3862–3869.

Ibrahim, M.A., Belal, M.H., Abdallah, I.S. and El-Sawi, S.A.E. (2022). Monitoring and Risk Assessment of Pesticide residues in some locally produced vegetables and fruits. Egyptian Journal of Chemistry, 65 (7), 429-439.

Kaur, H., Sharma, S. and Kang, B. K. (2023). Estimation of indoxacarb and thiamethoxam residues in chilli. International Journal of Environmental Analytical Chemistry, 103 (17), 4924-4941.

Kwon, H., Kim, T. K., Hong, S. M., Se, E. K., Cho, N.J. and Kyung, K. S. (2015). Effect of household processing on pesticide residues in field-sprayed tomatoes. Food Science and Biotechnology, 24, 1-6.

Lehotay, S. (2007). AOAC official method 2007. 01 pesticide residues in foods by acetonitrile extraction and partitioning with Magnesium Sulfate. Journal of AOAC International, 90 (2), 485-520.

Liu, S., Kou, H., Mu, B., Wang, J. and Zhang, Z. (2019). Dietary risk evaluation of tetraconazole and bifenazate residues in fresh strawberry from protected field in North China. Regulatory Toxicology and Pharmacology, 106, 1-6.

Madan, A. V., Ahlawat, S. and Chauhan, R. (2018). Dissipation pattern and effect of household processing on reduction of Indoxacarb residues in tomato fruits (*Lycopersicon esculentum* Mill.). J. Entomol. Zoo Stud, 6, 814-819.

Mahugija, J., Ngabala, F. and Ngassapa, F. (2021). Effectiveness of common household washing of tomatoes on the removal of pesticide residues. Tanzania Journal of Science, 47 (1), 390-404.

Malhat, F., Badawy, H. M., Barakat, D.A. and Saber, A.N. (2014). Residues, dissipation and safety evaluation of chromafenozide in strawberry under open field conditions. Food Chemistry, 152, 18-22

Mhya, D.H., Muhammad, J.S., Urmar, N.S. and Mohammed, A. (2024). Impact of chemical pesticides on antioxidant constituents and free radical scavenging capacity of pesticide-treated tomato (*Solanum lycopersicum* L.) fruits. Journal of Agriculture and Environment, 20 (1), 187–199.

Moustafa, M.A., Fouad, E.A., Ibrahim, E., Erdei, A.L., Kárpáti, Z. and Fónagy, A. (2023). The comparative toxicity, biochemical and physiological impacts of chlorantraniliprole and indoxacarb on Mamestra brassicae (Lepidoptera: Noctuidae). *Toxics*, *11*(3), 212.

Odewale, G.O., Sosan, M.B., Oyekunle, J.A. O. and Adeleye, A. O. (2021). Human health risk assessment of dichlorodiphenyltrichloroethane (DDT) and hexachlorocyclohexane (HCH) pesticide residues in fruits and vegetables in Nigeria. Environmental Science and Pollution Research, 28, 33133–33145.

Pekel, F. (2023). Effectiveness of household processes in the reduction of pesticide residue concentrations on fruit and vegetables. Environmental Science and Pollution Research, 30 (10), 27375–27388.

Qi, Y., Cao, J., Li, C., Ren, P., Qin, S. and Li, J. (2023). Dissipation, processing factors and dietary exposure assessment of myclobutanil in tomato. Molecules, 28 (16), 5978.

Rodrigues, A. A., De Queiroz, M. E. L., De Oliveira, A. F., Neves, A. A., Heleno, F. F., Zambolim, L. and Morais, E. H. C. (2017). Pesticide residue removal in classic domestic processing of tomato and its effects on product quality. Journal of Environmental Science and Health, Part B, 52(12), 850–857. https://doi.org/10.1080/03601234.2017.1357785.

Sakthiselvi, T., Paramasivam, M., Vasanthi, D., and Bhuvaneswari, K. (2020). Development and validation of gc-ms method for indoxacarb residues in vegetables and soil. Pesticide Research Journal, 32 (2): 253-262.

Salem, R.E. (2020). Efficacy of certain insecticides for controlling the two spotted spider mite, *Tetranychus urticae* (Koch) and determination residues of abamectin on some quality parameters of cucumber fruits. Egyptian Academic Journal of Biological Sciences, F. Toxicology & Pest Control, 12 (1), 217–226.

Simon, J.Y. (2011). The toxicology and biochemistry of insecticides. CRC press.

Sardar, S.W., Choi, J.Y., Jo, Y.J., Ishag, A.E. S.A., Kim, M.W. and Ham, H. J. (2023). Residues and safety assessment of cyantraniliprole and indoxacarb in wild garlic (*Allium vineale*). Toxics, 11(3), 219.

Shalaby, A. (2017). Residues of lambda-cyhalothrin insecticide and its biochemical effects on sweet pepper fruits. Journal of Product Development, 22 (1), 65–81.

Shalaby, A. A. (2016). Residues of profenofos with special reference to its removal trials and biochemical effects on tomato. Journal of Plant Protection and Pathology, 7(12), 845-849.

Shalaby, A. A., Seloma, A.S.O. and Shalaby, M. A. (2022). Study of Bifenazate, Indoxacarb and Emamectin Benzoate Residues on Tomato. Journal of Plant Protection and Pathology, 13 (7), 169-174.

Shalaby, A., Hendawy, M.A., Aioub, A.A.A., and Saleh, K.M. (2020). Health risk assessment of abamectin and buprofezin residues in eggplant and pepper plants. Journal of Plant Protection and Pathology, 11 (12), 693–699.

Shalaby, S. and Gad, N. (2016). Effects of insecticide residues on some quality attributes in tomato fruits and determination of their residues. International Journal of Pharm. Tech. Research, 9 (12), 360–371.

Szpyrka, E., Kurdziel, A., Matyaszek, A., Podbielska, M., Rupar, J., and Słowik-Borowiec, M. (2015). Evaluation of pesticide residues in fruits and vegetables from the region of south-eastern Poland. Food Control, 48, 137-142.

Su, Yue, Wang, Weijun, Hu, Jiye and Liu, Xiaolu, (2020). Dissipation behavior, residues distribution and dietary risk assessment of tembotrione and its metabolite in maize via QuEChERS using HPLC-MS/MS technique. Ecotoxicol. Environ. Saf. 191, 110187.

Taghizadeh, S. F., Goumenou, M., Rezaee, R., Alegakis, T., Kokaraki, V., Anesti, O. and Karimi, G. (2019). Cumulative risk assessment of pesticide residues in different Iranian pistachio cultivars: applying the source specific HQS and adversity specific HIA approaches in Real Life Risk Simulations (RLRS). Toxicology letters, 313, 91-100.

# مصير متبقيات مبيد الإندوكساكارب في ثمار الطماطم، وطرق ازالتها، وتقييم المخاطر الصحية المرتبطة بها وتأثيراتها البيوكيميائية

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تم إجراء تجارب حقلية على ثمار الطماطم لدراسة متبقيات مبيد الإندوكساكارب (Tunchii 15% SC)، وسلوك تحلله، وكفاءة إزالته، وتقييم المخاطر الصحية، والتأثيرات البيوكيميائية، وذلك خلال موسم الشتاء لعام 2023 في قرية ميت القرشي، بمحافظة الدقهلية، مصر. تم جمع العينات في فترات زمنية متعددة: بعد ساعة واحدة من الرش، ثم بعد 1، 3، 6، 9، 12، و15 يومًا من المعاملة. استُخدمت طريقة كاتشرز (QuEChERS) لاستخلاص المتبقيات وتنقيتها، ثم تحليلها باستخدام جهاز الكروماتوجرافيا السائلة عالية الأداء (HPLC). أظهرت النتائج ان كمية متبقي الإندوكساكارب الإبتدائية 6.88 ملجم/كجم، وانخفضت تدريجيًا مع مرور الوقت. وقد تم تقدير نصف العمر (t½) للمادة بـ 1.965 يوم، بينما تم تحديد فترة ما قبل الحصاد (PHI) بـ 12 يومًا. ومن المثير للإهتمام، أنه لم تُكتشف أي متبقيات في صلصة الطماطم المصنعة من الثمار الملوثة، مما يدل على إزالة كاملة بنسبة 100%. كما أن غسل الطماطم بماء الصنبور، وحمض الأسيتيك 5%، وكربونات الصوديوم 5% لمدة 5 دقائق أدى إلى نسب إزالة بلغت 28.84%، 34.58%، و44.66% على التوالي. أظهر مؤشر الخطر الصحي إلى أن الطماطم يمكن استهلاكها بأمان بعد 15 يومًا من المعاملة، إلا أن تحويل الثمار إلى صلصة يمكن أن يقلل من هذه الفترة ويجعلها آمنة للإستهلاك في وقت اقصر. وقد لوحظت انخفاضات طفيفة في بعض مؤشرات الجودة مثل السكريات الكلية وبعض المعادن، في حين لم تتأثر العناصر الغذائية الأساسية مثل البروتين، وفيتامين C، والبيتا-كاروتين بشكل ملحوظ.

**الكلمات الإسترشادية:** الإندوكساكارب، متبقيات مبيدات، التحطم، تقييم المخاطر، فترة ما قبل الحصاد (PHI) ، كاتشرز، الطماطم.

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