MAGNITUDE OF AZOXYSTROBIN RESIDUES IN TOMATO FRUIT USING HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC) AND QUECHERS METHODOLOGY

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ABSTRACT: Field trail was led to determine the decline pattern of azoxystrobin fungicide in/on tomato fruits after application at the recommended rate under open Egyptian field states using QuEChERS technique coupled by high performance liquid chromatography (HPLC)

The technique was validated for azoxystrobin at different fortification levels (0.01, 0.1 and 1.0 mg/kg) in tomato fruit blank. The mean recoveries from 97.69% to 102.46%, and the relative standard deviations (RSDs) of repeatability of the azoxystrobin ranged from 1.42% to 1.69%. Good linearity was obtained over the concentration ranges (0.01 -5 µg mL^{-1}) with correlation coefficient R^2 = 0.999. The limit of quantification (LOQ) for azoxystrobin was estimated to be 0.01 mg/kg. Tomato plants were sprayed with the azoxystrobin commercial formulation at the recommended rate of 90 grams (active ingredient. /hectar.). Tomato fruit samples were taken randomly zero, 1, 3, 7, 10 and 15 days after trail and extracted , cleaned-up via QuEChERS technique. Azoxystrobin residues declined following first-order kinetics rate and half-life was 3.01 days. The azoxystrobin pre-harvest interval (PHI) on tomato fruit was one day constructed on the European Union maximum residue levels (MRL) after treatment at the mentioned dose and the residues were observed under detectable level of maximum residue levels (MRL 3 mg/kg). in the tomato fruits.

Keywords: Residues, QuEChERS, Azoxystrobin, Tomato.

1.INTRODUCTION

Strobilurins pesticides are considered the greatest significant groups of agriculture fungicides. Now the strobilurins comprise the massive selling fungicide in the world's namely azoxystrobin **Bartlett *et al.* (2002).** Azoxystrobin [methyl (*E*)-2-{2-[6-(2-cyanophenoxy) pyrimidin-4-yloxy] phenyl}-3-methoxyacrylate] **(Fig. 1**), is a broad-spectrum fungicide for systemic, curative, protectant, eradicant and translaminar properties and is used on a wide range of crops pertinence to the group of

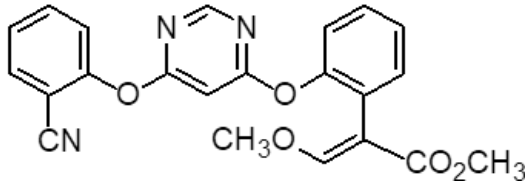

Fig 1. Chemical structure of Azoxystrobin

methoxyacrylates, that are extract from the naturally-happen strobilurins. It is effective to dominance the fungal diseases make by Basidiomycota, **Ascomycota, Oomycota and Deuteromycota Kondo *et al.* (2012**). Azoxystrobin dominance foliar and soil-borne diseases like downy and powdery mildew, early and late blight, and pathogens Sclerotinia, Alternaria, Ascochyta, Pythium, and Rhizoctonia on numerous crops **(PMRA, 2009).**

Tomato *Solanum lycopersicum* L. (Solanaceae) is one of the main food crops according to human diets **Yanbing *et al.* (2016)** and it is today the greatest common popular and widely grown vegetables everywhere at the world **Gupta *et al.* (2011).** As well as sixteen essential nutrients and several more chemical elements are beneficial in human health. Today tomato is cultivated almost everywhere at the world, Egypt, is the fifth producer largest tomato after China, India, United States and Turkey. Egypt produces about eight million tons of tomato every year **Dorais *et al.* (2008).** It is planted in around 221 thousand hectares which state around 34 % of the average zone of vegetables which representing 3% of Egypt's available planting land **(FAO 2018; Malhat *et al.* 2012; EL Sherif 1997).** Most of the tomatoes produced in Egypt are consumed locally and only small portion is exported to other countries. **Abdelhadi *et al.* (2015)**.The dissipation studies are essential to assay the suitability of pesticides application planning to understanding the hazardous influence of pesticide residues **Lu *et al.* (2014).**

The details on residues of pesticide treated crops and appraisal of pre-harvest intervals (PHIs) are prerequisites for national registration of a new pesticide and for suitably setting the maximum residue limits (MRLs) to keep the general consumer versus the potential health hazard of exposure to pesticides. In Egypt, during the recording of a new compound to the regulatory list, the Central Agricultural Pesticide Laboratory (Giza, Egypt) has set the PHIs to prevent the distribution of agricultural products containing residues beyond the allowable limits **(Malhat *et al.* 2013; Malhat *et al.* 2014).** The establishment of the PHI and assessment of risks caused by pesticide residues in agricultural crops increased the demand for the evolution of analytical techniques.

Several analytical techniques had been utilized for detection and surveillance azoxystrobin residues in environmental matrices. These techniques were high performance liquid chromatography (HPLC) with diode array detector (DAD) **Polati *et al.* (2006)** mass spectrometry (MS) detector **Jørgensen *et al.* (2012)** and tandem mass spectrometry (MS/MS) **Utture *et al.* (2011)** and gas chromatography (GC) with electron capture detector (ECD) **Gajbhiye *et al.* (2011).**

In the existing study, a fast, simple and effective extraction technique with HPLC-DAD was used to determine azoxystrobin residues in tomato fruit succeeding application under Egyptian field states .

2.MATERIAL AND METHODS

2.1.Chemicals and Reagents:

Authenticated reference standard of azoxystrobin (>99 % purity) was bought of Dr. Ehrenstorfer GmbH (Augsburg, Germany). Every organic solvent was HPLC

grade, and bought of Sigma (Sigma GmbH, Darmstadt, Germany). Primary secondary amine (PSA, 40 µm Bondesil) and graphitized carbon black sorbent were bought of Supelco (Bellefonte, Pennsylvania, USA). Analytical class of anhydrous magnesium sulfate, sodium chloride were got of CARLO ERBA Reagents S.A.S.

2.2.Preparation of standard solutions:

Individual stock solution (100 ml/L) of azoxystrobin was prepared in acetonitrile. The consecutive working dilution and spiking standard solution for HPLC analysis were set by diluting the stock solution. All standard and working solutions were stored at 4 ºC.

2.3.Field trials:

Field experiment was conducted to determine the degradation of azoxystrobin fungicide in/on tomato fruits at El-Dakahlya Governorate, Egypt. Tomato plants were sprayed with one commercially available suspension concentrate formulation)Herose® 25% SC). One treatment was carried out on 2017 at the recommended dose 90 g (ai) / hec. according to pest control program, **(Ministry of Agriculture and Land Reclamation 2018)].** The treatments, including the untreated (control) and treated plots, were replicated three times in a complete randomized block design in open field. The pesticide treatment was conducted using knapsack hand sprayer fitted with one nozzle boom. Replicate samples, two kg tomato fruits were collected at random from sampling plots at intervals of one hour after application (zero time), 1, 3,7,10 and 15 days. As soon as the fruits picked up, and put in polyethylene bags were transferred in ice box to laboratory. Samples were roughly cut with a knife into small potions and homogenized in a food processor (HOBART). The homogeneous matrix was stored in sealable plastic bag at -20 ºC until the preparation day.

2.4.Sample extraction and clean-up:

The extraction and clean-up process was conducted on the authority of the unique virgin QuEChERS process **Anastassiades *et al*. (2003).** A 10 g (± 0.1) of homogenate tomato samples were weighted inside 50 ml centrifuge teflon tubes. Next, acetonitrile (10 ml) was addition and shake it strongly for 1 min up to down. Following, (4.0 g) of anhydrous magnesium sulfate was addition then sodium chloride (1.0 g) then shaken as mentioned above, after that was immediately centrifuged to 4000 rpm for 5 min in 5 ºC refrigerated centrifuge.

Then, (1 ml) supernatant was set to clean-up using 25 mg primary secondary amine and 150 mg anhydrous magnesium sulfate and 10 mg GCB, the tube was shaken vigorously to 1 min, afterwards centrifuged to 4000 rpm until 5 min. Finally, (0.5 ml) supernatant was move to a vial after filtered through a 0.22 µm PTFE filter (Millipore, Billerica. MA), followed by injection to High performed liquid chromatography system

2.5.Method validation:

The technique was performed to provide evidence that the method is appropriate for the extraction and quantitative determination of azoxystrobin in tomato fruit. The technique was validated following a conservative validation that included the succeeding parameters: Matrix effect assessment, accuracy, limit of quantification LOQ, precision, linearity and trueness (bias) according to Guidance document on analytical and validation methods (**SANTE/11813/2017)**.

The linearity of the technique was intended from the results directly relative to the concentration of azoxystrobin which diluted in solvent. Linearity was evaluated by the correlation coefficient (R^2) resulted from the 5-point calibration curve at series (0.01, 0.1, 0.5, 1.25, 2.5 and 5) µg/ml for HPLC analysis.

The matrix match effect was verified by the response from the azoxystrobin in a pure solvent with spiked tomato samples with azoxystrobin in the identical solvent after extracted at the same concentration points (0.01, 0.1, 0.5, 1.25, 2.5 and 5) mg/kg for HPLC analysis.

The limit of quantitation (LOQ) explained as the lowest concentration of the azoxystrobin in tomato that has been validated with tolerable trueness (70-120%) and precision (RSDr ≤ 20%) by passing the complete analytical technique defined as a signal-to-noise ratio (S/N) of 10:1. On the authority of the document (**SANTE/11813/2017**) the Limit of quantification should be ≤ MRL. The maximum residue limit (MRL) for azoxystrobin is (3 mg/kg to tomato**, (European Union 2016).**

A recovery experiment (trueness) was estimated by extraction and analysis of 5 replicates of blank sample at 3 spiked levels (0.01, 0.1, and 1) mg/kg of azoxystrobin in tomato fruit. According to the document SANTE/11813/2017)., tolerable mean recoveries are those within the range of 70-120%.

The precision was stated by (Repeatability (r)) as standard deviation RSD. The similar method on the similar samples in a laboratory through a compact period was used. The limit value of reproducibility relative standard deviation (RSDr) was ≤ 20%. Five replicates for individually recovery levels (0.01, 0.1, and 1) mg/kg over day through three several days were studied to realize the precision.

2.6. HPLC determination:

Chromatographic analyses were achieved utilizing the HPLC system (Agilent 1260 infinity series) equipped with a quaternary pump, variable wavelength diode array detector (DAD), for an analytical column: Nucleosil C18 (30 ×4.6 mm (i.d) × 5 um film thickness), auto sample valve. Chromatographic separation was done by a mobile phase acetonitrile: water (90:10 v/v) at a flow rate of 1 ml min^{-1}. Injection volume was 20 µl and the wavelength was 230 nm. The retention time was 5.52 min.

2.7.Decomposition rate:

The dissipation kinetics of azoxystrobin residues in tomato fruit was determined by plotting residue concentration versus pass time after application and equations of best curve fit with maximum coefficients (R^2) were determined. For dissipation of azoxystrobin in tomato, exponential relationships were found to be applicable corresponding to the general first-order kinetics equation:

$$Ct=C0^{e-kt}$$

Where Ct symbolized the concentration of the pesticide residue at the time of t, C0 symbolized the initial deposits after application and k is the constant rate of pesticide dissipation per day **(Wang and Hoffman 1991).**

The half-life periods (RL50) of azoxystrobin were calculated as follows: (ln 2/k) **Moye *et al.* (1987)**. The degradation percentage was calculated by the following equation:

$$\%degradation = C0 - Ct/C0 \times 100$$

where: C0 is the concentration of the pesticide (ppm) at 0 time and Ct is the concentration

of the pesticide (mg/kg) during time t

3.RESULTS

3.1.Method validation:

Good linearity was achieved over the concentration ranges (0.01 -5 µg mL^{-1}) with correlation coefficient R^2 = 0.999. (Fig. 2). The standard curve equation was:

y = 83.292 x + 2.5097. where y = peak area and x = concentration (mg/kg).

The matrix effects may influence the repeatability and accuracy of the method. Hence, to reduce the matrix effect and obtain much preferable results, a calibration was done for azoxystrobin through adding the external azoxystrobin standards into tomato fruit extract. The effect of matrix on the response of HPLC was checked by comparing the slope of the calibration curve for azoxystrobin prepared in pure solvent (acetonitrile) with that prepared in tomato fruit matrix. A zero value symbolized that there is no matrix match effect, a positive value was compound response is fortified by the different matrixes, and a negative value displays that the compound restraint is inhibited by the various matrixes **Yanbing *et al.* (2016).** In current research, the matrix effect for HPLC analysis for tomato was -10.5 %. For more accurate quantitation matrix matched calibration was used for determination of azoxystrobin in field incurred tomato fruit samples.

As explained in Table 1, recovery experiment (trueness) of azoxystrobin at different fortification levels (0.01, 0.1 and 1.0 mg/kg) in tomato samples were validated. The precision of the technique was set by analyzing the relative standard deviation (RSD < 20 %) in the observed area. It is indicated that azoxystrobin can be noticed with valid precision on condition that the extraction technique adopted good recoveries. The mean recoveries ranged from 97.69% to 102.46%, and the relative standard deviations (RSDs) of repeatability of the azoxystrobin ranged from 1.42% to 1.69%. It is verifying that the method is suitably consistent for the azoxystrobin residue analysis in tomato according to. Guidance document on analytical quality and method validation for pesticide residues (**SANTE/11813/2017**).

In this study LOQ for azoxystrobin was predestined to be 0.01 mg/kg, assuring LOQ values significantly minimum than the MRLs (3 mg/kg) confirmed by the European union **(European Union 2016).**

Table 1. Fortification level and recovery percentage (±RSD) of azoxystrobin in tomato.

Spiking level (mg/kg) (n*=5)	Mean recovery (%±RSD)	RSDr%
0.01	97.69±1.39	1.42
0.1	102.46±1.57	1.53
1	101.48±1.71	1.69

*: Number of replicates

3.2.Decline of azoxystrobin in tomato fruits:

The residues of azoxystrobin in tomato fruits through field conditions are revealed in Table 2. The initial deposits of azoxystrobin at the recommended dose were 1.11 mg/kg. Residue levels decreased to reach 0.11 mg/kg after 10 days from application indicating that 90.09 % of azoxystrobin residue was dissipated and undetectable after 15 day of application. The half-lives and coefficient (R) of azoxystrobin residue dissipation was studied from the experimental data and abstracted in Table 2. The dissipation curve of azoxystrobin in tomatoes is depicted in Fig 3. The dissipation regressive equation could be described by the following equations: $y = 1.2303e^{-0.23x}$ The results of azoxystrobin residue analysis showed that the dissipation of follow the first order kinetics. The correlation coefficient was (R^2 = 0.98). The half-life value was 3.01.

4.DISCUSSION.

The results of method validation demonstrated that the QuEChERS sample technique, coupled by HPLC-DAD analysis was a valid method and fitted to detect and quantified azoxystrobin in tomato fruits. The results of validation are within agreeable criteria for pesticide residue analysis according to **(SANTE/11813/2017).**

There are various factors that effect the fate of pesticide in plants, as well as different species, weather conditions, the characteristics of the pesticide (including its total stability either as parent compound or metabolites, its volatility, solubility, formulation), the effect of physical and chemical factors such as light, heat, pH and moisture, the method and site of application. Also, the growth dilution factor might have played an important role **(Malhat *et al.* 2016 and Malhat *et al.* 2017)**

The best fit detected in tomato fruit was first order kinetics for the most of the azoxystrobin usages trials.

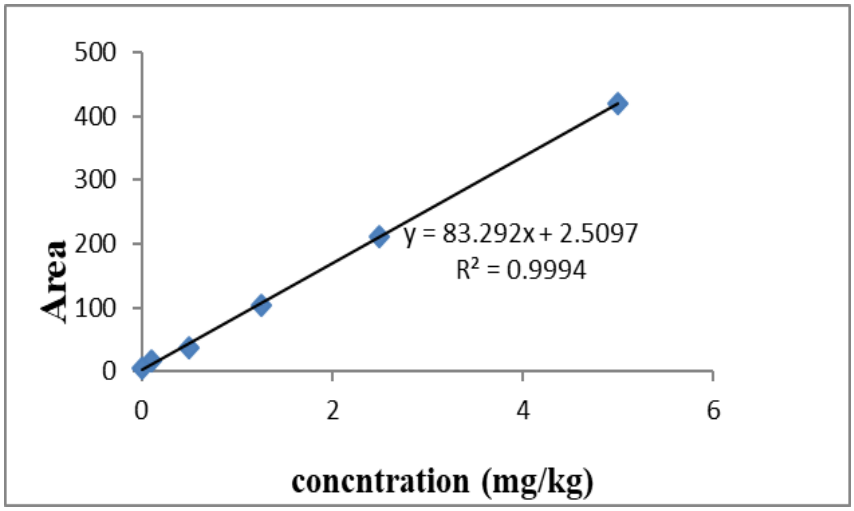

Fig.2. Calibration curve of azoxystrobin with HPLC-DAD analysis.

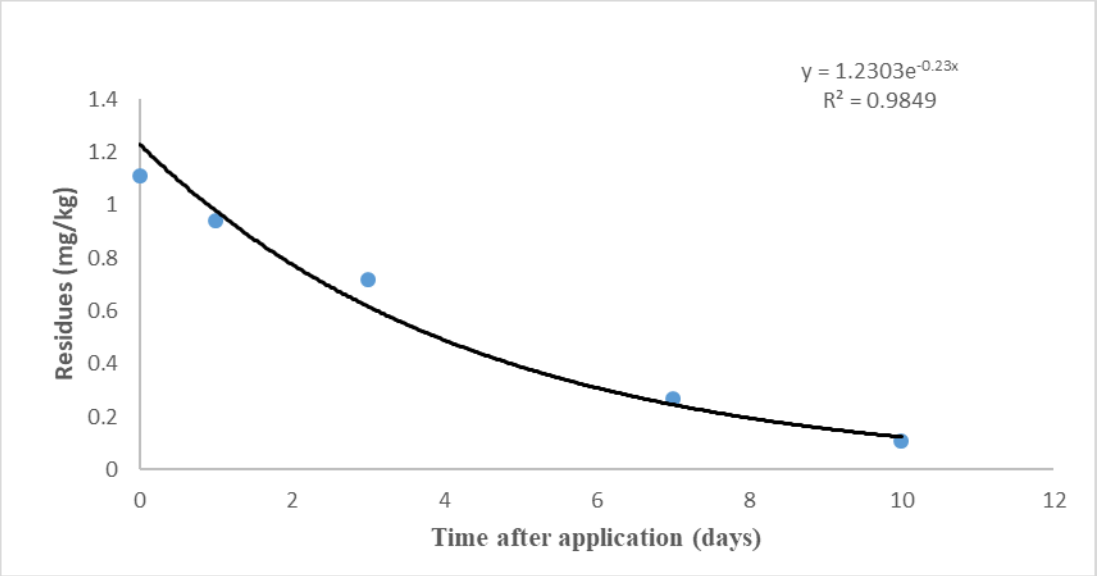

Fig.3. Dissipation behavior of azoxystrobin in tomato.

The half-life value was 3.01. Considering the maximum permissible residue limit of 3.0 mg/kg for tomato fruit, the proposed waiting period after spraying of azoxystrobin was one day for more safety. The residues of azoxystrobin were found at below detectable level of maximum residue levels in the tomato fruits. (Table 2).

Table (2) Residue levels and dissipation behavior of azoxystrobin in tomato.

Time after treatment (days)	Residues (mg/kg)	% Loss
initial*	1.11±0.09	0.00
1	0.94±0.05	15.31
3	0.72±0.11	35.13
7	0.27±0.08	75.67
10	0.11±0.02	90.09
15	ND	100
MRL (mg/kg)	3	
PHI (days)	1	
RL50 (days)	3.01	

*: one hour after application.

The results are in contract with the results of **Gareur *et al.* (2002**) who calculated dissipation of azoxystrobin on tomato fruit samples in green house, at the preharvest interval, the residues were under maximum residue level.

The use of fungicides belonging to strobilurin groups are successful in controlling numerous plant diseases just their excessive, irrational and indiscriminate use can produce to unwanted residues and may causes pose problems concerning to the safety of the consumer when the levels exceed the MRLs or when they are applied at the maturing stage and a minimum waiting period is not followed. As numbers of the fruits and vegetables are consumed as raw products, fungicide residues on them may cause to health related problems

. If the dose used is high and it is applied at the improper time and a total number of sprays exceed than the proposed ones, there is every chance that the residues left in the crops at yield time are higher than the acceptance limits prescribed. Standardization of fungicidal residue is an important activity as the quality parameters are interlinked with inherent toxicity, residual effects and phytotoxicity, etc. **Anand *et al.* (2010)**

CONCLUSION

Residues of azoxystrobin were successfully extracted from tomato samples using the QuEChERS method. The method demonstrated acceptable accuracy and precision and was successfully applied to the dissipation kinetics of azoxystrobin in tomato from the field. The dissipation rates of azoxystrobin in tomato fruits were evaluated under Egyptian field conditions. The obtained results indicated that tomato fruit could be safely consumed after application where the azoxystrobin residues were found at below detectable level in the harvested fruits of tomato according to the recommended maximum residue limit (MRL) (3 mg / kg).

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تقدير متبقيات مبيد الازوكسى استروبين على ثمار الطماطم باستخدام جهاز الكروماتوجرافى السائل عالي الأداء HPLC وطريقة QuEChERS

رانيا محمد عبد الحميد

قسم بحوث متبقيات المبيدات وتلوث البيئة-المعمل المركزي للمبيدات- مركز البحوث الزراعية.الدقى – الجيزة- مصر.

الملخص العربى

تم إجراء تجربة تقدير انهيارمبيد الازوكسى استروبين الفطرى في / على ثمار الطماطم بعد التطبيق بالمعدل الموصى به تحت الظروف الحقلية المصرية باستخدام تقنية QuEChERS و تقديرها بواسطة جهاز الكروماتوجرافى السائل عالي الأداء(HPLC)

تم التحقق من دقة الطريقة لمبيد الازوكسى استروبين عند مستويات تلوث مختلفة (0.01 ، 0.1 و 1.0 مجم / كجم) في ثمارالطماطم الغير معاملة. وكان متوسط معدل الاسترجاع من 97.69 ٪ إلى 102.46 ٪ ، وتراوحت الانحرافات القياسية النسبية (RSDs) لمبيد لازوكسى استروبين بين 1.42 ٪ إلى 1.69 ٪. وتم الحصول على الخطية الجيدة على مدى تركيز (0.01 -5 ميكروجرام /مل) مع معامل الارتباط 0.999 .وتم تقدير الحد الكمى للطريقة (LOQ)وكان 0.01 مجم / كجم. تم رش محصول الطماطم بمبيد الازوكسى استروبين بمعدل 90 جم (مادة فعالة / هكتار). وتم أخذ عينات الطماطم المعاملة عشوائيا علي فترات زمنية بعد ساعة من الرش (صفر)، 1، 3، 7، 10 و 15 يوما بعد التطبيق.

تم استخلاص العينات وتنقيتها باستخدام طريقة QuEChERS .وتشير النتائج ان منحنى اختفاء مبيد الازوكسى استروبين يتبع معادلة الخط المستقيم من الدرجة الأولى وكانت فترة نصف العمر 3.01 يوما.

وكانت فترة ما قبل الحصاد (PHI) على ثمار الطماطم ا يوم من المعاملة بمبيد الازوكسى استروبين وفقا للحدود القصوي المسموح بها للاتحاد الأوروبي بعد التطبيق بالجرعة الموصى بها حيث تم رصد المتبقيات تحت الحدود القصوي المسموح بها للمبيد (3مجم/كجم) على ثمار الطماطم.

الكلمات الدالة: متبقيات – QuEChERS – مبيد الازوكسى استروبين – الطماطم.