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Pesticides Residue Analysis in Potato Produced in Iraq from Selected Markets Across Basrah Province

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Abstract

The purpose of this study was intended to reveal of the types of pesticide residues pre-sent and measure their levels in samples of nationally produced potatoes in Iraq by ex-tracting them according to the QuEChERS method for the first time in Basrah province and diagnose them by using Gas Chromatography Mass Spectrophotometry (GC-MS), then evaluate and discuss the human health implications as a result of the presence of such residues. Thirty-six potato samples were collected from the main local markets in Basrah throughout 2020 year, an average of three collections per month. The current study revealed that the contamination of pesticides residue of produced potatoes in Iraq during the year 2020 is not a cause for concern, as 91.6% of the studied samples after extraction and diagnosis did not contain any residues that could be mentioned or detected, while only three samples contained residues, these were largely owing to Methane, isocyanato- residues in potatoes (8.3 %, 3 samples). According to the study results, Methane, isocyanato- was the sole chemical compound found in potatoes and belonged to pesticides residue groups. For the time being, we can say that Iraqi pota-toes are almost free of pesticides residue and can be considered a healthy food product that poses no hazard to human health; this indicates that our farmers are highly com-mitted to use safe and appropriate pesticides and adhering to the recommended safety period; this trend would benefit both humanity and the environment.

Keywords: Pesticides Residue, Potato, Basrah, Methane, isocyanato-, QuEChERS.

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1.Introduction

In many nations, potato (*Solanum tuberosum* L.) is one of the most significant vegetable crops of the Solanaceae family and in world crop statistics, it is ranked third, just behind rice and wheat (yang,2010; Birch *et al*.,2012; Thomas-Sharma *et al*.,2016). Potatoes are considered as an essential source of energy, where, each 100 g of storage roots provides 76 calories, containing (17.1) g of carbohydrates, (2.1) g of proteins and (20) mg of vitamin C, the roots also rich in some other vitamins such as A, B complex, as well as β-carotene, dietary fiber, folate requirement, some minerals: potassium, magnesium, calcium, iron and high value of antioxidant depending on the flesh composition (Brown, 2005; Storey, 2007). Potatoes are cultivated on every continent except Antarctica, demonstrating the crop's adaptability and versatility in a variety of climates, Potatoes are nowadays farmed in 149 countries; approximately 50% of the crop will be consumed fresh, while the rest will be processed into culinary items, ingredients, starch production, animal feed, or seed tubers for the next seasons. (Birch *et al*.,2012).

Crops are susceptible to contaminate with different pesticides residue in the field due to either direct application of pesticides, soil borne pesticides, or contaminated water for irrigation (Ingham *et al*., 2005). It's worth noting that when pesticides are used according to the product label, any residues that stay are unlikely to exceed the Maximum Residue Levels (MRLs) recommended by FAO /WHO; additionally, a thorough understanding of pesticide concentration is required to properly assess human exposure; therefore, farmers should be educated in order to reduce the residual impact of

toxic pesticides, exact dose, and harvest time after spraying any crop. (Akoto *et al*.,2013). Pests and diseases pose a significant and obvious threat to crops production in many countries around the world, necessitating the use of large amounts of pesticides to eliminate these pests and control the spread of these diseases. Because of the potential health and environmental risks posed by these chemicals, pesticides residue on plant products are a major food safety and human health concern for governments and consumers. (Spence *et al*., 2020).

2.Materials and methods

2.1. Area of study

Basrah, is an industrial and agricultural province located in the far south of Iraq (situated at 31º 20'- 29º 05' north latitude, 48º 20'- 46º 40' east longitude; Iraq, Asia) with a total area of 19,070 km^2 and featured with two agricultural seasons as a result of the passage of Tigris, Euphrates, Shatt al-Arab river and its branches in addition to the availability of groundwater and produces very much crops either in greenhouses in winter or in open lands in summer. (Hadeel *et al*., 2010).

2.2 Samples collection

A total of 36 Kg of nationally produced fresh Potatoes samples (*Solanum tuberosum* L.), were purchased from twenty-nine different markets center within Basrah city, an average of one time per 10 days, on (5^{th}, 15^{th} and 25^{th}) of each month, throughout the period from January to December 2020 as indicated in Fig.1, where many consumers purchased their daily food basket. The shop owners were quizzed about the provinces where the potatoes they sold came from, and it was established that they are all of Iraqi descent, with the majority hailing from the capital, Baghdad, central, and northern regions. The samples were collected from the upper, middle, and lower shelves to provide the most representative sample, packed in paper bags, sealed, tagged with a unique sample identity, and sent directly to Basrah University's laboratory for extraction process within 24 hours. Acetonitrile (MeCN) and formic acid (HCOOH) were synthesized (J. T. Baker, Germany). Anhydrous magnesium sulfate (anhy. MgSO4) and Sodium chloride (NaCl) were purchased from (Himedia, India). Triphenyl phosphate and primary secondary amine (PSA) sorbent were obtained from (Sigma-Aldrich). Alfa Aesar supplied the disodium hydrogencitrate sesquihydrate ($C_{12}H_{18}Na_4O_{17}$). Kimia Exir provided Trisodium citrate dihydrate ($C_6H_5Na_3O_7.2H_2O$). Dr. Ehrenstorfer provided Metalaxyl and Propamocarb pesticide reference standards. Individual standard solutions in Acetonitrile were produced at a concentration of 1000 µg ml^{-1}. Internal standard solution was made by combining the necessary amount of Triphenyl phosphate with Acetonitrile and diluting to a final concentration of (20 µg mL^{-1}). A 5% formic acid solution was prepared by mixing 5:95 (v: v) of formic acid and Acetonitrile respectively. All solutions were kept at 4 °C.

2.3. Sample processing

Potatoes samples were prepared and extracted by original version of QuEChERS method as described in (Anastassiades *et al*., 2003). Each 1Kg was washed with distilled water, dried, peeled and chopped into fine pieces, well-milled in an electrical blender, then a (10 g) of the comminuted and homogenous sample was weighted and dried with oven until the change in mass is constant to estimate, calculate and stabilize the water content in it.

2.4. Solid - Phase Extraction

Another (10 g) of same homogeneous mixture was weighted into a 50 ml Teflon centrifuge tube, 10 ml acetonitrile was added using a solvent-dispenser with 100 µL internal standard solution, a mixture of (4 g anhydrous magnesium sulphate coarsely grained,1 g sodium chloride, 1 g Trisodium citrate dihydrate and 0.5 g disodium hydrogencitrate sesquihydrate) were added to previous Teflon centrifuge tube then centrifuged for 5 min. 3000 U/min.

2.5. Purification

To achieve purification 2 ml of the supernatant was transferred to a clean 10 ml single-use desiccator with screw cap containing 50 mg primary-secondary amine and 300 mg anhydrous magnesium sulfate fine powder. The tubes were forcefully shaken before being centrifuged for 5 min. 3000 U/min. Following that, 1 ml of cleaned

extract was put into a 1.5 ml vial, and the pH was promptly corrected to 5 by adding a 10 µL of 5 % formic acid solution. The pH-adjusted extract which filled into special class vials, stored in a freezer at -20 °C and became ready for further analysis by Gas Chromatography- Mass Spectrometry (GC-MS).

2.6. Calibration curves

Stock solutions of Metalaxyl and Propamocarb pesticides (1000 µg mL^{-1}) and serial dilutions ranging from 0.01 to 10 µg mL^{-1} were prepared using acetonitrile. Areas under the peak versus concentrations were plotted and fit by simple linear regression to obtain the equation for the standard curves for the tested pesticides. The quantity of each diagnosed pesticide in each sample will be calculated based on the slope of the standard curve. (Fig. 2,3,4 and 5).

2.7. Gas Chromatography–Mass spectrometry) GC–MS

In comparison to other approaches, the most modern and powerful tool for assessing pesticides residue in vegetable samples is Gas Chromatography-Mass Spectrometry (GC-MS) because it is employed to perform a specific test, GC-MS has been widely hailed as the "Gold Standard" for forensic substance identification (Kwon *et al*.,2012). The residues and chemical components were analyzed by using Gas Chromatography–Mass Spectrometry (Agilent 7693) with Gas Chromatography (7890 B) and Mass Spectrometry (Agilent 5977 A) detector equipped with a Splitless injector in the range of 35 m z^{-1} to 650 m z^{-1}. HP-5MS capillary column (5% phenyl methyl siloxane: 30m × 0.25 mm × 0.25µm) was used in combination with the following oven temperature program: initial temperature 40 ^{O}C hold for 5 min, ramp to 280 ^{O}C at 10 ^{O}C min^{-1}, Final temp. 310 ^{O}C held to end-run.

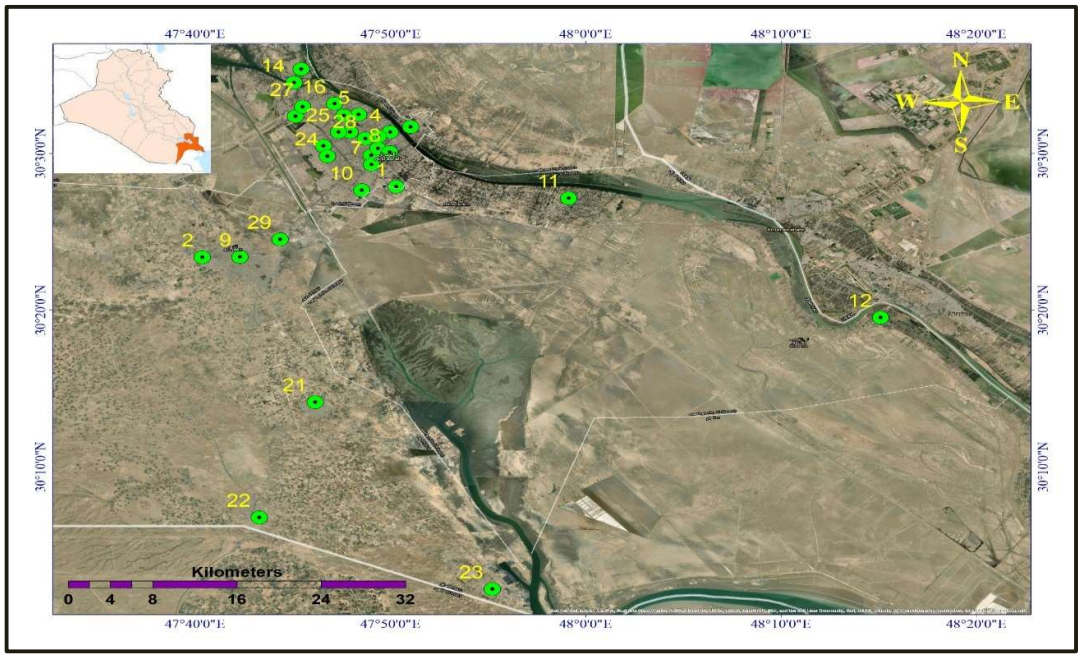

Fig.1. Map showing samples collection sites.

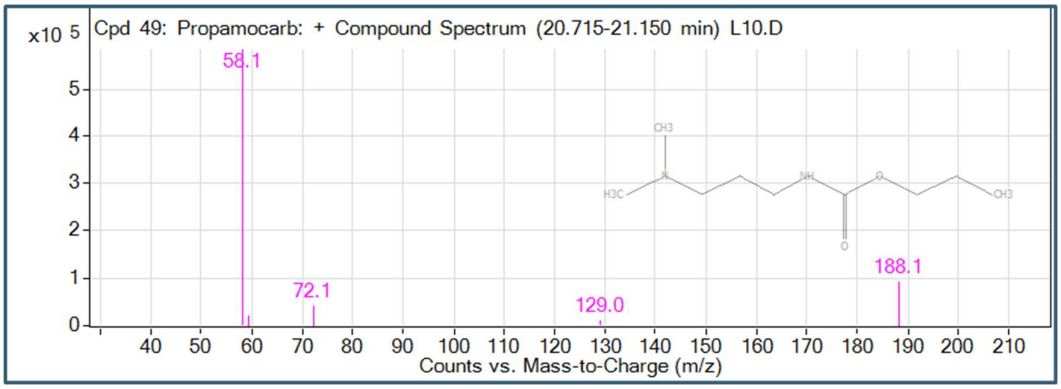

Fig. 2. Spectrum of Propamocarb ion 58 selected as quantification ions.

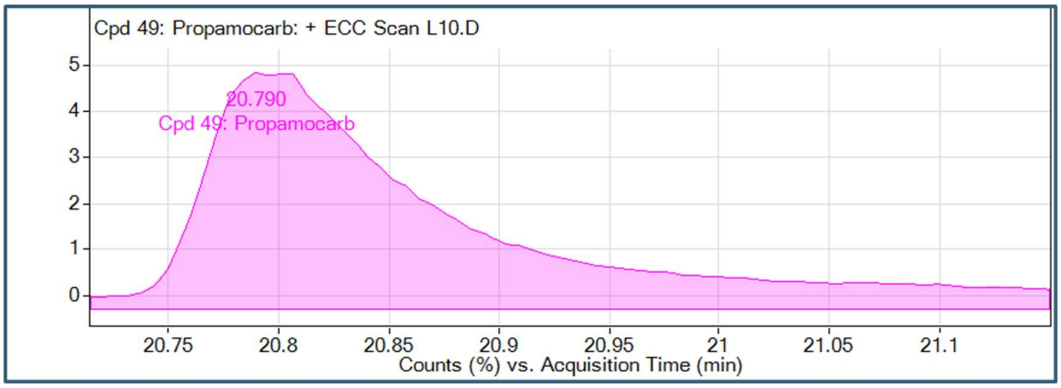

Fig. 3. Chromatography curve of GC-MS for Propamocarb.

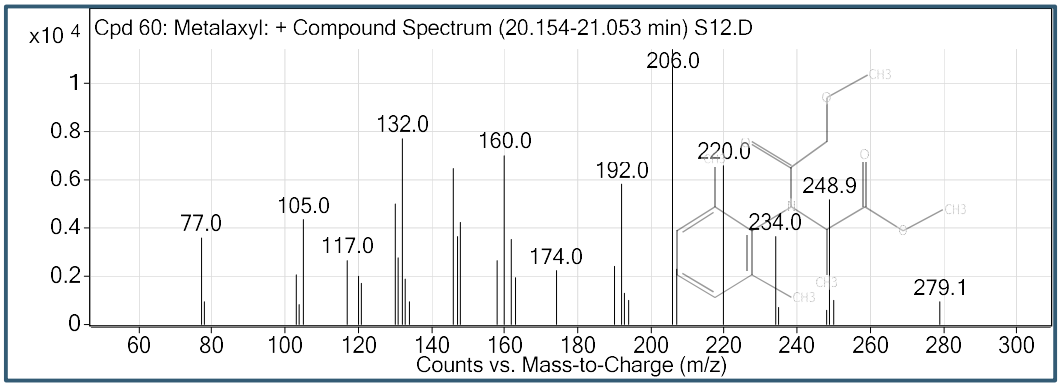

Fig. 4. Spectrum of Metalaxyl ion 206 selected as quantification ions.

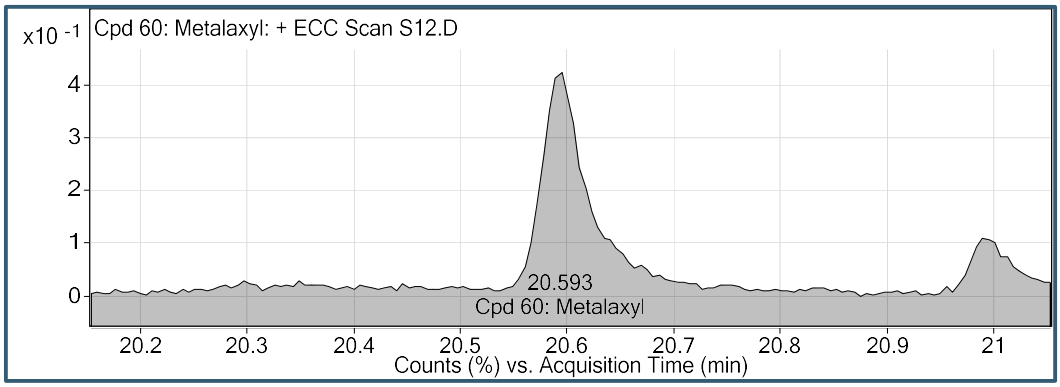

Fig. 5. Chromatography curve of GC-MS for Metalaxyl.

3. Results and discussion

The detection of pesticides residue in the nationally produced potatoes throughout 2020 year, which were sold from several markets in Basrah province was the basic goal of this study.

3.1. Water content in potatoes

Water content (moisture) in all potatoes samples has exceeded 83%. These results allow us to apply the QuEChERS method directly without needing to raise the moisture content by adding a distilled water.

3.2. The diagnosed pesticides residue.

Based on a preliminary review of pesticides available on the Iraqi market and in the updated pesticides database registration published by National Committee for the Registration and Approval of Pesticides in the Iraqi Ministry of Agriculture, which a hard copy of it was obtained by Basrah Agriculture Directorate. It was found that Cymoxanil, Copper oxychloride, Spirodiclofen, Metalaxyl, Chlorothlonil, Pendimethalin, Fosetyl, Metribuzin, Propamocarb and Fenamidone are only the licensed pesticides to be used in the Republic of Iraq to control various diseases and pests that threaten the potatoes crop. So the 36 potato samples were examined for full pesticide scans to detect Metalaxyl and
Propamocarb or more of listed above pesticides in the range of 35 m z^{-1} to 650 m z^{-1}.
The results of analysis revealed that none of the examined samples of nationally produced potatoes showed measurable amounts of pesticide residues. It was not surprising that overall residue levels were rather low because Iraqi farmers use modern farming technologies, are completely committed to use safe and appropriate pesticides, and strictly stick to the recommended safety period day after day. The examination showed that the most frequent toxic chemical with only three replicates on 15th June, 25th October and 15th December samples was Methane, isocyanato- (Fig. 6 and 7).

Methane, isocyanato- (CH_3NCO) is not registered as an active ingredient in Codex Alimentarius Commission, European Commission or any other pesticide database, also, no trace of it was found in any other study of pesticide residues. But it is mainly utilized as a chemical intermediary in the synthesis of very hazardous pesticides derived from the carbamate insecticide and herbicide families (Bingham *et al*., 2001). Methane, isocyanato- (CH_3NCO) is highly toxic, insoluble in water, flammable, evaporates quickly when exposed to air, has a sharp and strong odor, enters the body by inhalation and is absorbed through the skin may cause severe injury or permanent burns on contact with eyes or skin more lethal than gas tear several times and may lead to death if inhaled in sufficient quantity, it causes severe bronchospasm and asthma-like breathing and was the main toxic substance implicated in the Bhopal disaster (Eckerman, 2001).

Based on what the reference described above, the reason for the presence of such toxic compounds in potato crops despite their relatively low molecular weight (57.05 g $mole^{-1}$) lies in its ability to easily penetrate organic plant tissues while insoluble in water. This was proved by the research through communicating with potato farmers in the capital, who confirmed that

they wash and clean the crop with abundant water (using strong water sprays) immediately after harvesting to remove the mud and pesticides residue. Furthermore, the research samples were rinsed many times by using tap and distilled water, and their scales were removed before starting the extraction and analysis process. Despite all these procedures, detectable residues of this toxic compounds were found where Soliman (2001) proved that washing and peeling operations reduce the amount of residues by a large percentage.

Another possible reason for its presence is its accumulated residues in the soil, which may be as a result for spraying different types of pesticides in earlier times in predisposition for preparing the soil in order to plant the crops or potato contamination by sources other than pesticides such as fertilization practices in potato production. Unfortunately, we were unable to determine which of these factors is mostly to blame caused by the lack of pesticides residue surveillance in soils, as well as the fact that farmers are not obligated to keep track of pesticides applied, as Lozowicka *et al* (2015) highlighted.

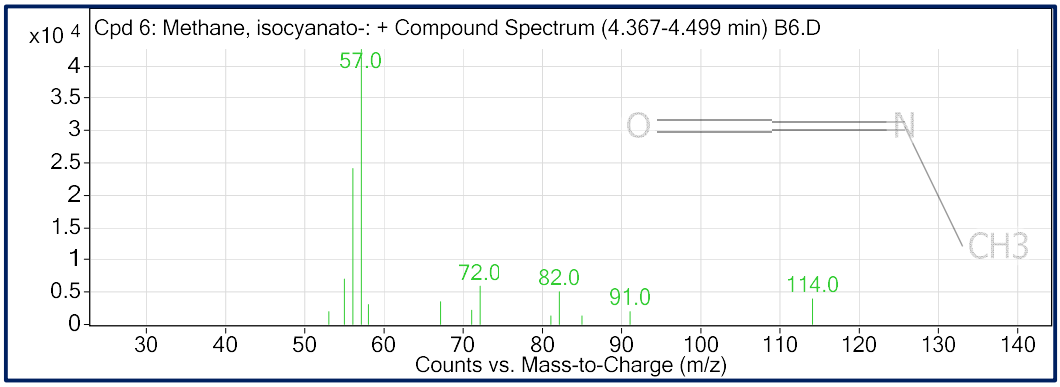

Fig. 6. Spectrum of Methane, isocyanato-; ion 57 selected as quantification ions.

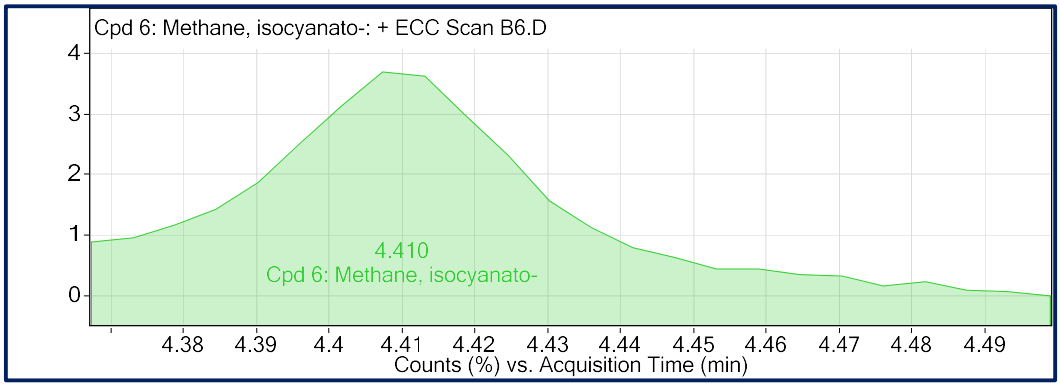

Fig. 7. Chromatography curve of GC-MS for Methane, isocyanato-.

Overall, the findings of this research were comparable to others of other studies (Česnik *et al*., 2006) who discovered that no residues were detected in potatoes samples except for a few Dithiocarbamates residues and (Wumbei *et al*., 2018) who decided that the levels of the pesticides residue under study identified in potatoes are extremely low and unlikely to pose a negative impact or harm on human health.

3.3. The nutrient content.

The nutrient content of potato has been characterized for some cultivars, and it has been demonstrated that Vitamin C, B6, and B1 are all abundant in potatoes, this content depends

largely on the genetic features of the variety, environmental conditions in which it is grown and the crop's subsequent storage., (Storey, 2007). Vitamin E can be also added to the previous list of vitamins, as it was diagnosed by chance in all tested samples in addition to other many hydrocarbons and acids during the process of detecting pesticides presence (Fig. 8 and 9).

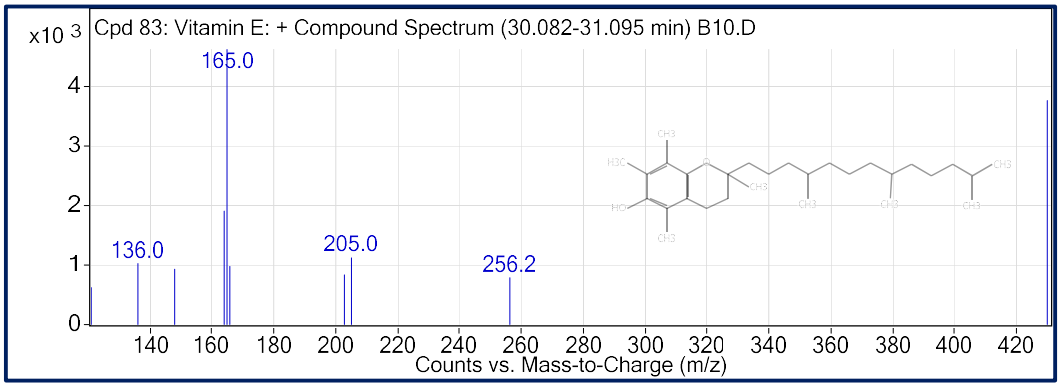

Fig. 8. Spectrum of Vitamin E; ion 165 selected as quantification ions.

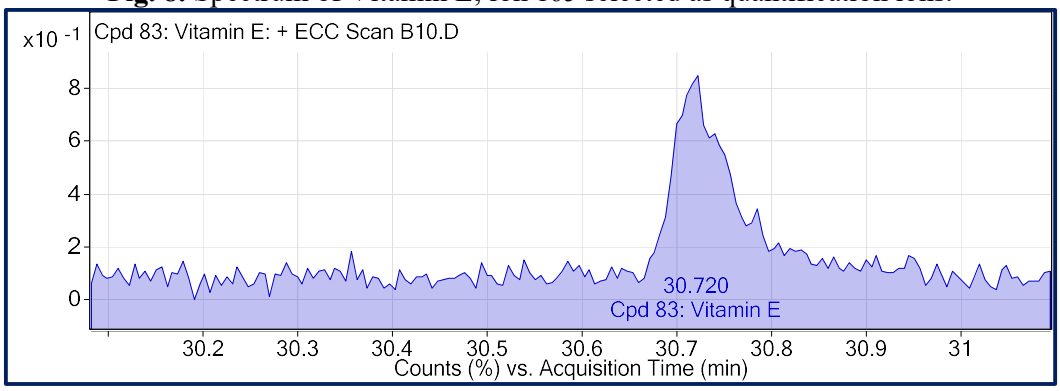

Fig. 9. chromatography curve of GC-MS for Vitamin E.

4.Conclusions

Potatoes grown in Iraq are considered a crop approximately free of pesticide residues with high nutritional value which provides real opportunities and gives a major impetus for breeding and development programs to increase the cultivated areas and become a supporter of the national economy by exporting the surplus after meeting the requirements of local markets, but these comfortable and reassuring results do not negate the necessity and inevitability of availability a comprehensive database and periodic examination centers to diagnose pesticides residue levels any foodstuffs and detect the dietary intake of pesticides by the Iraqi population.

5.Acknowledgment

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تحليل بقايا المبيدات في البطاطا المنتجة في العراق من أسواق مختارة في محافظة البصرة

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المستخلص
هدفت هذه الدراسة إلى الكشف عن أنواع بقايا المبيدات الموجودة وقياس مستوياتها في عينات البطاطا المنتجة محليًا في العراق، وذلك من خلال استخلاص بقايا المبيدات باستخدام طريقة QuEChERS، ولأول مرة في محافظة البصرة، وتشخيصها باستخدام جهاز الكروماتوغرافيا الغازية المقترنة بمطياف الكتلة (GC-MS)، ومن ثم تقييم ومناقشة الآثار المحتملة لهذه البقايا في صحة الإنسان نتيجة وجودها. تم جمع 36 عينة من البطاطا من الأسواق المحلية الرئيسية في محافظة البصرة على مدار عام 2020، وبمعدل ثلاث عمليات جمع للعينات شهريًا.أظهرت نتائج الدراسة الحالية أن تلوث البطاطا المنتجة في العراق ببقايا المبيدات خلال عام 2020 لا يمثل مصدرًا للقلق؛ إذ إن 91.6% من العينات المدروسة لم تُظهر، بعد الاستخلاص والتشخيص، وجود أي بقايا يمكن الإبلاغ عنها أو الكشف عنها، في حين احتوت ثلاث عينات فقط (8.3%) على بقايا، وكانت في معظمها ناتجة عن وجود مركب Methane, isocyanato- في البطاطا.ووفقًا لنتائج الدراسة، كان Methane, isocyanato- هو المركب الكيميائي الوحيد الذي تم العثور عليه في عينات البطاطا، وقد صُنّف ضمن مجموعات بقايا المبيدات.وبناءً على ذلك، يمكن القول إن البطاطا العراقية، في الوقت الراهن، تكاد تكون خالية من بقايا المبيدات، ويمكن اعتبارها منتجًا غذائيًا صحيًا لا يشكل خطرًا على صحة الإنسان. وتشير هذه النتيجة إلى أن المزارعين العراقيين ملتزمون بدرجة عالية باستخدام المبيدات الآمنة والمناسبة والالتزام بفترة الأمان الموصي بها قبل حصاد المحاصيل؛ ومن شأن هذا الاتجاه أن يعود بالنفع على كل من صحة الإنسان والبيئة.

الكلمات المفتاحية: بقايا المبيدات، البطاطا، البصرة، QuEChERS.