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Pesticide Contamination in Drinking and Surface Water in the Cienega, Jalisco, Mexico

Rosa J. Silva-Madera · Joel Salazar-Flores · A. Aarón Peregrina-Lucano · Judith Mendoza-Michel · Hazael R. Ceja-Gálvez · Daniel Rojas-Bravo · Mireya Z. Reyna-Villela · Erandis D. Torres-Sánchez

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Abstract Sixty percent of global agricultural production depends on the use of pesticides, despite their adverse effects on human health and the ecosystem. In Mexico, the application of these products has been exacerbated, including pesticides already banned in other countries. The objective of this study was to determine pesticide concentrations in samples of water purification plants and surface water from the Cienega area of Jalisco, Mexico. A survey of 119 farmers with occupational exposure to pesticides was carried out in order to obtain information on the most frequently used pesticides. Subsequently, 51 samples taken at 7 different sites were analyzed using liquid chromatography and mass-mass spectrometry. The most frequently used pesticides were organophosphates (28.87%), pyrethroids (12.89%), and the herbicide paraquat (31.95%). In surface water, the prevalent pesticides were glyphosate (56.96–510.46 ppb) and malathion (311.76–863.49 ppb). Glyphosate levels were higher than the limits acceptable in daily water intake in Cumuato. Malathion levels exceeded the limits permissible by EPA in water purification plants in urban public establishments (100 ppb for children, and 200 ppb for adults). In addition, a multidimensional scaling analysis showed that the sampled sites could be grouped into 2 different bodies of water, based on similarities in their glyphosate concentrations (stress = 0.005), while the concentrations of malathion were heterogeneous (stress = 0.001).

Keywords Pesticides · Water purification plants · Surface water · HPLC-MS/MS · Cienega-Jalisco

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Rosa J. Silva-Madera and Joel Salazar-Flores contributed equally to this work.

R. J. Silva-Madera · J. Salazar-Flores · H. R. Ceja-Gálvez · E. D. Torres-Sánchez (✉)
Department of Medical and Life Sciences. University Center of La Cienega, University of Guadalajara (CUCI-UdeG), Av. Universidad # 1115, C.P. 47810 Ocotlán, Jalisco, Mexico
e-mail: erandis.torres@academicos.udg.mx

A. A. Peregrina-Lucano · J. Mendoza-Michel
Department of Pharmacobiology, University Center of Exact Sciences and Engineering, University of Guadalajara, Blvd. Marcelino García Barragán 1421, esq Calzada Olímpica, Module E, 44430 Guadalajara, Jalisco, Mexico

D. Rojas-Bravo · M. Z. Reyna-Villela
Department of Technological Sciences. University Center of La Cienega, University of Guadalajara (CUCI-UdeG), Av. Universidad # 1115, C.P. 47810 Ocotlán, Jalisco, Mexico

1 Introduction

The Cienega area belongs to the states of Jalisco and Michoacán which cover an area of size 4892 km^2, with an average annual rainfall of 809 mm, and a semi-humid climate (CEA 2020). The Lerma-Santiago Basin and Chapala Lake are located in this region (CEA 2020). Previous evidence from the Lerma-Santiago River indicates high concentrations of fertilizers in the area (Ibarrarán et al. 2017; Pérez-Díaz et al. 2019). The water flowing from the Lerma and Zula rivers feeds the lake of Chapala (Camps and Arroyo 2018). The Zula River runs through 85.89% of the municipality of Ocotlán (a priority agricultural area) that feeds on the run-off, leading to a high level of fertilizers (CEA 2020; Sanchez et al. 2007). The tributary Palmar basin is nourished by the Chapala Lake, and it is a region popular for restaurant services (CEA 2020). On the other hand, Lerma river (section Cumuato) is located in the state of Michoacán, and it is a source of water supply to the agricultural areas of the community of Cumuato and its surroundings (INAFED 2020).

In 2015, it was reported that three-quarters of the water supply in the Cienega area was used for agricultural activities (CEA 2020). Thus, water supply in the Cienega area serves as its main economic engine (INAFED 2020). The application of pesticides has increased due to agricultural activities. A Jalisco-based study by Ortiz and collaborators (2013) reported increased frequencies in the use of organophosphates (19%), pyrethroids (20%), carbamates (14%), bipyridyls (6%), organochlorines (1%), amongst others, in 2012 (Ortíz et al. 2013). Semarnat (2020) has been reported the use of 140 bioactive components of pesticides banned in other countries and CONAGUA indicated the increased use of pesticides has accentuated pollution of water sources in the region, as indicated for 28 points for Chapala Lake, and 22 points for the Lerma River (Semarnat 2020). Therefore, the objective of this study was to determine the concentrations of pesticides in water purification plants and surface waters in the Cienega region: Chapala lake, Zula river, Palmar tributary, Lerma river section Cumuato, community of Cumuato and Ocotlán, Jalisco, Mexico.

2 Material and Methods

This investigation was carried out as an exploratory and descriptive study. The determination of pesticides was done at the Laboratory of Applied Pharmacokinetics, University Center of Exact Sciences and Engineering (CUCEI) of the University of Guadalajara. Prior to sampling, a survey on the pesticides used and the frequencies of their applications in the region was conducted using 119 volunteer farmers. He wondered about the application season, which was at least twice a year, once when maize is grown (June to October) and the other when wheat is grown (January to May). On the other hand, questions were raised about the time of use of pesticides in the region, and the subjects indicate that they are used from about two decades to date. It should be mentioned that sampling from July to September was during the period of application of these pesticides (June–October).

2.1 Sampling

Fifty-one (51) water samples were taken from seven points: (*a*) Lake Chapala (5 samples), (*b*) River Zula (5 samples), (*c*) tributary Palmar (5 samples), (*d*) stretch of the Lerma Cumuato River (12 samples), (*e*) community Cumuato (5 samples), and the community of Ocotlán (19 samples) which is divided into: (*f*) 11 samples of water purification plants and (*g*) 8 samples from the University Center of La Ciénega, CUCI located in the border area where the crops start (Fig. 1). Samples taken from point (*a*) to (*e*) as well as dot (*g*) were surface water from tributaries, while those in item (*f*) correspond to water purification plant samples.

2.2 Processing

The sampling was carried out in the rainy season which occurs in the region from July to September 2019, the period of the year during which the use of pesticides increases. Such sampling was carried out in a single series and at the same time for each sampled point, during the indicated period. Water samples (50 mL) from the middle part of the flows were collected in sterile polyethylene vessels and kept at 4 °C prior to until transfer to the laboratory where they were filtered with 0.2 μm Whatman before injection to the chromatograph. On the other hand, 50 mL of water was taken from 11 purifying plants for human consumption following the same collection protocol as for surface water before injection to the chromatograph. The analysis of the samples was performed using multiple reactions at a flow rate of 0.5 mL per minute in an Agilent Technologies liquid chromatograph 1200 ® coupled to mass spectrometry 6430B with a column C18 Zorbax Eclipse XDB of dimensions 50 mm × 2.1 mm × 3.5 μm. The mobile phases comprised 0.1% formic acid in water and a gradient of 40–100% acetonitrile at a flow rate of 0.5 mL per minute. The curve range for each pesticide was 0.01–1000 μg/mL (Schaner et al. 2007; Arora et al. 2007). Twenty-two pesticides were analyzed. The standards used (99% purity) were as follows: parathion, picloram, ametrine, 2,4-dichlorophenoxyacetic acid (2,4-D), pyraclostrobin, malathion, diazinon, imazalil, dimethoate, carbofuran, atrazine, thiabendazole, molinate, acetachlor, carbendazin, Emamectin, cialotrin, meclizine, methomyl, methoxymers, oxandrolone, and glyphosate from AccuStandard ®. The electrospray interface conditions (EICs) were gas temperature of

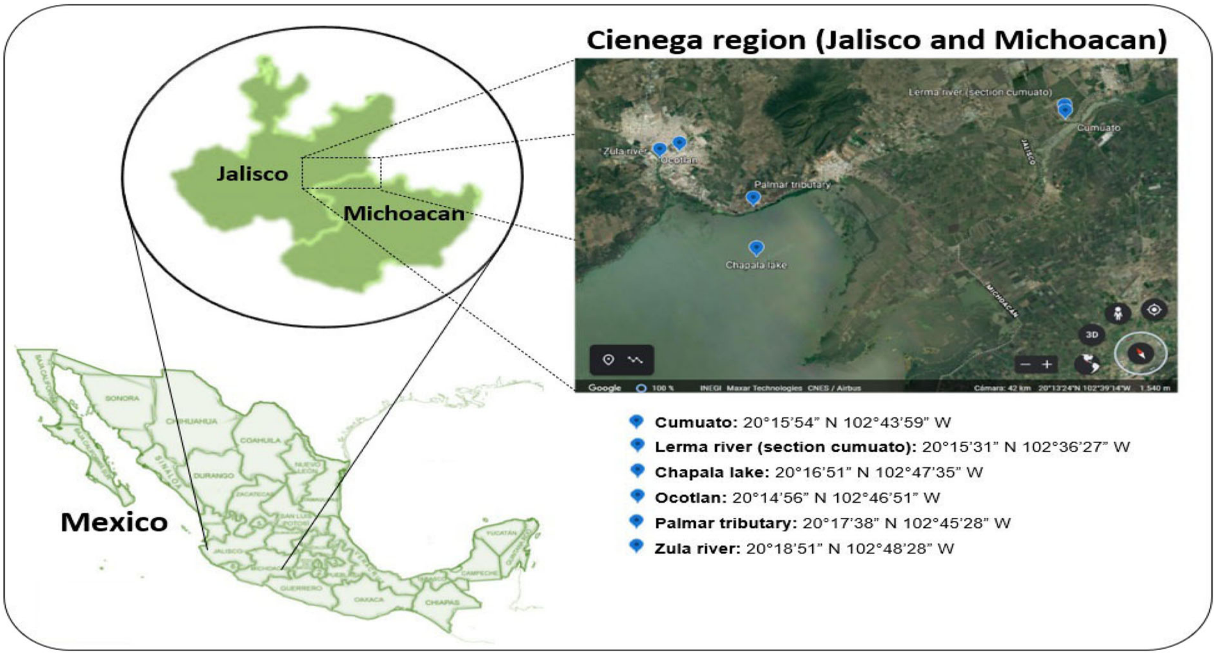

Fig. 1 Sampling sites in the Cienega region, located between the states of Jalisco and Michoacan, Mexico (indicated by blue dots). Image taken from Google Earth

350 °C, gas flow rate of 12 L/min, nebulizer pressure of 25 psi nebulizer, + 4000 capillary in precursor ion and − 4000 capillary in product ion. The time used to perform chromatographic analysis was 200 ms using multiple reaction monitoring (MRM) (Rodríguez-Aguilar et al. 2019). The mass spectrometry conditions for the determination of each pesticide are shown in Table 1. The validation of the method was performed according to Analytical Method Validation Guide Edited by The National College of Pharmaceutical Chemists Biologists Mexico, A.C. The analytical parameters evaluated were recovery (as a measure of accuracy), linearity, precision, limit of quantification, and limit of detection (Método Analíticos 2002).

2.3 Analysis

The results were analyzed with SPSS v19.0 software and are expressed as mean and standard deviation (SD). The data were compared using Student's *t* test for samples matched with normal distribution, while Mann-Whitney *U* test was used for samples that did not meet a normal distribution. Multidimensional scaling (MSD) was carried out to group the zones on the basis of similarity in the concentrations of pesticides. A value of $p \leq 0.05$ was taken as indicative of statistical significance.

3 Results

The survey carried out on farmers revealed that the most frequently used pesticides were bipyridyls (31.95%), followed by organophosphates (28.87%), while the least used were pyrethroids (12.89%). The frequencies of use of pesticides in the three groups are shown in Table 2. Within the organophosphates, glyphosate was the most frequently used pesticide (14.43%), while alpha-cypermethrin was the most frequently used pyrethroid pesticide. Paraquat (31.95&) was the most popular bipyridyl amongst the farmers.

Since glyphosate and malathion stood out amongst the 22 pesticides analyzed in water samples, subsequent studies were focused on these two pesticides. The remaining 20 pesticides were detected only in some samples at concentrations below 10 ppb. It is worth noting that picloram was the only pesticide that was present at concentrations higher than 10 ppb and up to 100 ppb in the samples of the Lerma river section Cumuato. In contrast, glyphosate and malathion were present at

Table 1 Mass spectrometry conditions and characteristics of pesticides analyzed

Compound	Precursor ion (m/z)	Product ion (m/z)	Fragment (v)	Polarity
Parathion	292	264	90	Positive
Parathion	292	236	90	Positive
Picloram	240.9	222.9	90	Positive
Picloram	240.9	194.9	90	Positive
Ametrine	228.1	186	120	Positive
Ametrine	228.1	96	120	Positive
2, 4-D	219	161.1	50	Negative
Pyraclostrobin	388	163	120	Positive
Malathion	331	99	80	Positive
Diazinon	305	153	160	Positive
Imazalil	297	159	160	Positive
Dimethoate	230	171	80	Positive
Carbofuran	222	123	120	Positive
Atrazine	216	132	120	Positive
Thiabendazole	202	131	120	Positive
Molinate	188.1	55.1	78	Positive
Acetochlor	270.1	224.2	60	Positive
Acetochlor	270.1	148.4	60	Positive
Carbendazin	192.1	160	110	Positive
Emamectin	887.1	158.1	60	Positive
Cialotrine	467.1	225.1	80	Positive
Meclizine	391.2	201.1	90	Positive
Methomyl	163.1	106	30	Positive
Methomyl	163.1	88.1	30	Positive
Metoxuros	229.1	72.1	93	Positive
Oxandrolone	325	289.2	100	Positive
Oxandrolone	307.2	271.2	100	Positive
Oxandrolone	307.2	229.1	100	Positive
Glyphosate	168	149.9	80	Negative
Glyphosate	168	124.2	80	Negative

concentrations greater than 100 ppb. These results are presented in Table 3 and Fig. 2.

Table 3 shows high concentrations of malathion, especially for purifiers (863.49 ppb) and the community of Cumuato (848.11 ppb), and in other sampling zones. Compared to malathion, the levels of glyphosate were much lower. The highest concentration of glyphosate (510.46 ppb) was obtained in the community of Cumuato Lake Chapala, while lower and comparable levels of this pesticide were recorded in Zula River, tributary Palmar, and Lerma river section Cumuato. In contrast, low level of glyphosate (56.96 ppb) was seen in the community of Ocotlán in purifiers and in the University Center.

As shown in Fig. 2, for the seven sampling points studied, malathion concentrations were significantly and consistently higher than those of glyphosate levels.

3.1 Glyphosate and Malathion Concentrations Between Sampling Areas

Based on Mann-Whitney *U* test, comparison of glyphosate concentrations amongst sampling areas was

Table 2 Pesticides used by farmers in the Cienega region

Pesticides type	Used percentage (%)	Classification IUPAC	PubChem CID	Classification EPA
Organophosphates				
Glyphosate	14.43	2-(Phosphonomethylamino)acetic acid	3496	D
Ethyl chlorpyrifos	5.67	Diethoxy-sulfanylidene-(3,5,6-trichloropyridin-2-yl)oxy-λ^5-phosphane	2730	E
Glufosinate ammonium	5.67	2-Amino-4-[hydroxy(methyl)phosphoryl]butanoic acid;azane	11,564,649	E
Terbufos	1.03	*tert*-Butylsulfanylmethylsulfanyl-diethoxy-sulfanylidene-λ^5-phosphane	25,670	E
Diazinon	1.03	Diethoxy-(6-methyl-2-propan-2-ylpyrimidin-4-yl)oxy-sulfanylidene-λ^5-phosphane	3017	E
Parathion	0.51	Diethoxy-(4-nitrophenoxy)-sulfanylidene-λ^5-phosphane	991	C
Malathion	0.51	Diethyl 2-dimethoxyphosphinothioylsulfanylbutanedioate	4004	No evaluation
Pyrethroids				
Alphacypermethrin	6.18	[Cyano-(3-phenoxyphenyl)methyl] 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropane-1-carboxylate	2912	C
Tefluthrin	3.09	(2,3,5,6-Tetrafluoro-4-methylphenyl)methyl (1*S*,3*S*)-3-[(*Z*)-2-chloro-3,3,3-trifluoroprop-1-enyl]-2,2-dimethylcyclopropane-1-carboxylate	11,534,837	No evaluation
Lambda cyhalothrin	2.06	[(*S*)-Cyano-(3-phenoxyphenyl)methyl] (1*R*,3*R*)-3-[(*Z*)-2-chloro-3,3,3-trifluoroprop-1-enyl]-2,2-dimethylcyclopropane-1-carboxylate	6,440,554	No evaluation
Deltamethrin	1.03	[(*S*)-Cyano-(3-phenoxyphenyl)methyl] (1*R*,3*R*)-3-(2,2-dibromoethenyl)-2,2-dimethylcyclopropane-1-carboxylate	40,585	E
Cypermethrin	0.51	[cyano-(3-phenoxyphenyl)methyl] 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropane-1-carboxylate	2912	C
Bipyridyls				
Paraquat	31.95	1-methyl-4-(1-methylpyridin-1-ium-4-yl)pyridin-1-ium	15,939	C

EPA Classification: *C*, possible human carcinogen; *D*, not classified as human carcinogen; *E*, evidence of non-carcinogenicity. The IUPAC, PubChem CID, and EPA classifications were taken from PubChem's website, 2020

Table 3 Average value of glyphosate and malathion concentrations by sampling area

Sampling area	Mean concentration of malathion (ppb)	SD	Mean concentration of glyphosate (ppb)	SD
Chapala lake (*n* = *5*)	671.97	83.88	258.28	41.91
Zula river (*n* = *5*)	713.28	108.01	250.66	66.86
Palmar tributary (*n* =5)	717.34	66.03	228.53	37.29
Lerma river, section Cumuato (*n* = *12*)	311.76	84.67	252.17	78.70
Community of Cumuato (*n* = *5*)	848.11	53.78	510.46	442.51
Community of Ocotlan, Purifier (*n* = *11*)	863.49	111.41	LOD*	LOD*
Community of Ocotlan, Cuci (*n* = *8*)	771.498	91.08	56.96	59.73

*Compound with concentration below the limit of detection. *SD*, standard deviation

performed. The results showed statistically significant differences between the community of Cumuato and the area of Cuci ($p \leq 0.05$). A similar result was also seen with analysis using MDS (Fig. 3a), where a homogeneous distribution was distinguished in two dimensions. The first zone of similarity comprised the lake of Chapala, the river Zula, and the tributary of Palmar, while the second similar covered Lerma river section Cumuato, the community of Cumuato, and Cuci (*stress* = *0.005*).

Malathion concentrations were also compared amongst sampling zones using the Mann-Whitney *U* test. There were statistically significant differences in most of the paired comparisons ($p \leq 0.05$). Moreover, MDS analysis showed a heterogeneous distribution amongst these areas (*stress* = *0.001*; Fig. 3b).

3.2 Glyphosate and Malathion Concentrations in Water Purification Plant Samples from Ocotlan

Samples for human consumption from 11 water purification plants show high concentrations of malathion (above 600 ppb, Table 4) and glyphosate values in a range above 10 ppb and below 100 ppb for 10 samples, except in the water purification plant 9 with a value of 120.1 ppb (Table 4). The levels of malation of our results exceed the limits indicated by EPA in drinking water in urban public establishments (100 ppb for children and 200 ppb for adults). With regard to glyphosate concentrations, they are within the permissible limits according to the World Health Organization (WHO). On the other hand, local and municipal government regulations monitor water

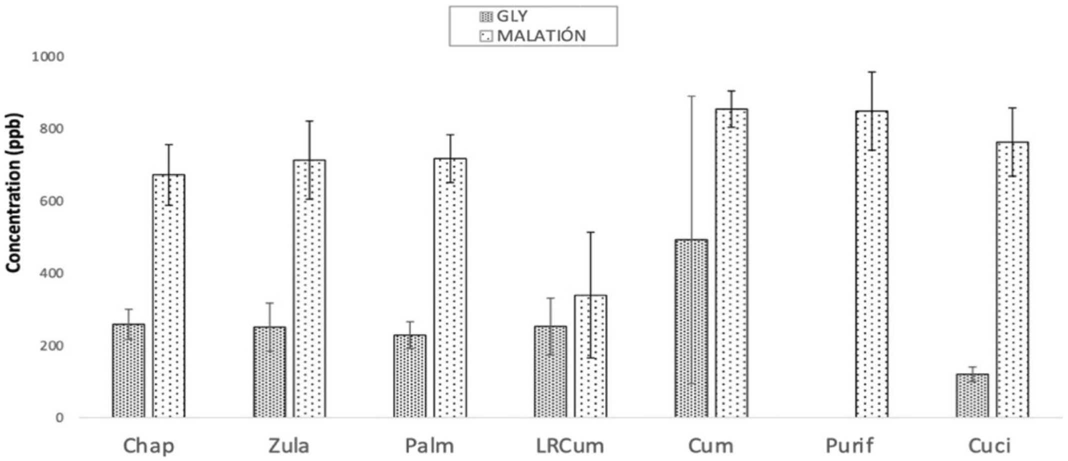

Fig. 2 Comparison of glyphosate and malathion concentrations (ppb) in 51 water samples from 7 different regions of the Cienega zone, Jalisco, Mexico

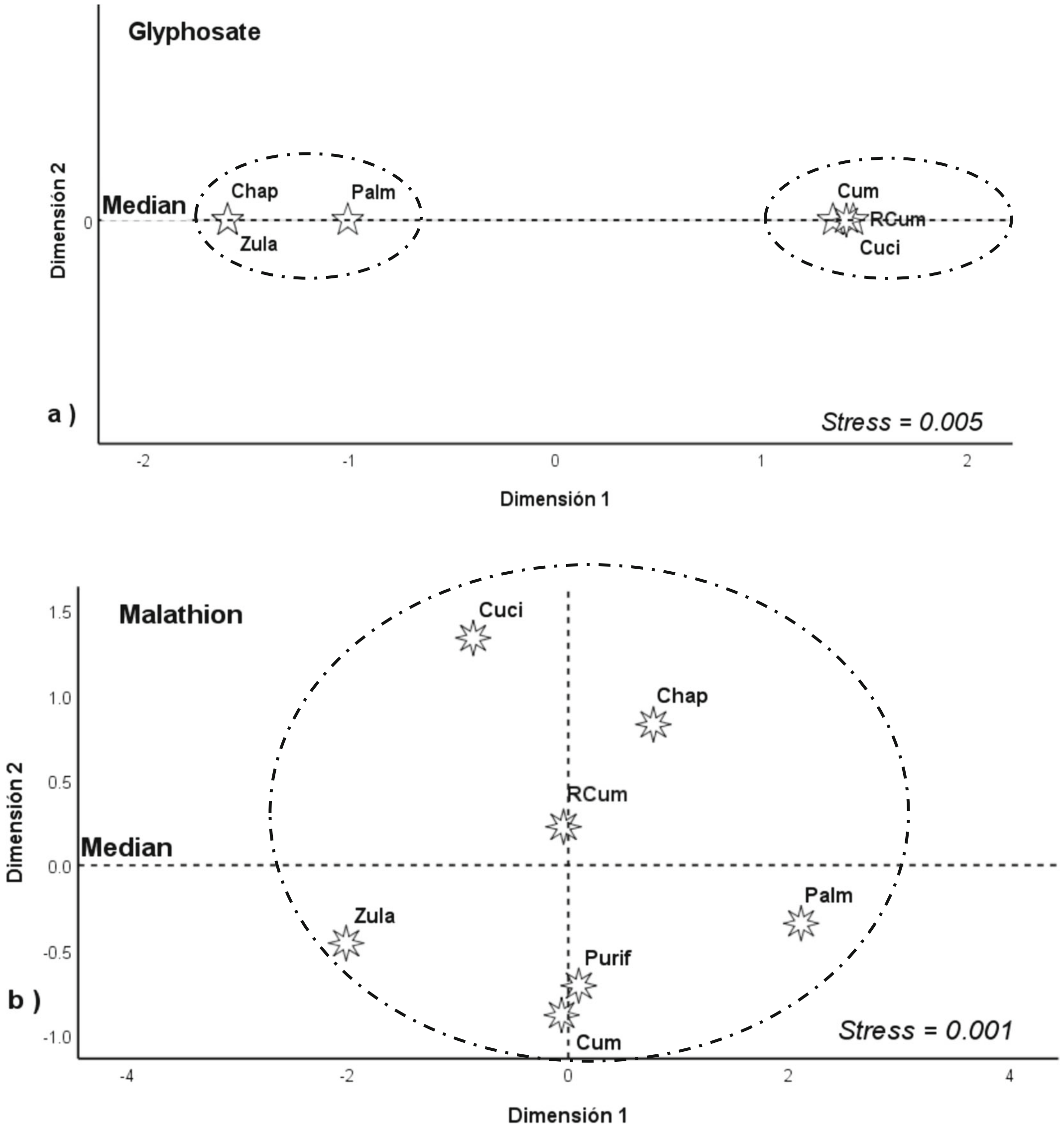

Fig. 3 **a** A multi-dimensional scaling (MDS) for the concentrations of glyphosate in different areas of the Cienega region; **b** MDS for the concentrations of malathion in the same areas in region

quality with respect to the concentration of organochlorine pesticides (NOMX-AA-071-1981, water analysis-determination of organochlorine pesticides), but not for organophosphates, it is therefore necessary to update an Official Mexican Standard that regulates the permissible limits of these pesticides.

4 Discussion

The Cienega region Jalisco is important at national level for its contributions to the production of wheat (68.05%), sorghum (48.05%), corn (27.75%), agave (4.8%), and red tomato (4.0%) (Transparencia 2004). It has been reported that the most frequently applied pesticides in agricultural fields in Jalisco, Mexico (Ayuquila-Armería river basin in Autlán de Navarro) are glyphosate, glufosinate, diazinon, parathion, malation, cypermethrin, lambda cialotrina, and paraquat (Pérez-Herrera et al. 2018; Rodríguez-Aguilar et al. 2019; Guzmán-Plazola et al. 2016). In this study, results from the Cienega region are consistent with the high frequencies of application of glyphosate, glufosinate, cypermethrin, and paraquat, with a 60.49% prevalence in the use of

Table 4 Pesticide concentrations in 11 water purification plants in Ocotlan, Jalisco, Mexico

Pesticide (ppb)	Water purification plants										
	1	2	3	4	5	6	7	8	9	10	11
Acetochlor	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD
Ametrin	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD
Atrazine	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD
Carbendazim	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD
Carbofuran	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD
Diazinon	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD
Dimethoate	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD
Emamectin	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD
Imazalil	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD
Cyhalothrin	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD
Malathion	811.6	732.2	880.2	1051.2	784.7	901.2	903.8	747	857.4	1056.9	665.1
Meclizine	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD
Methomyl	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD
Metoxuros	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD
Molinate	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD
Oxandrolone	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD
Parathion	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD
Picloram	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD
Pyraclostrobin	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD
Thiabendazole	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD
Glyphosate	< 100	< 100	< 100	< 100	< 100	< 100	< 100	<100	120.1	<100	<100
2,4-D	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD	LOD

herbicides, followed by fungicides with 39.05%, and insecticides with a 20.92% (Sierra-Diaz et al. 2019).

4.1 Pesticides with Concentrations Below 10 ppb

Acetochlor, ametrine, atrazine, carbendazin, carbofuran, diazinon, dimethoate, emamectin, imazalil, cialotrin, meclizine, methomyl, methxurs, molinate, oxandrolone, parathion, pyraclostrobin, thiabendazole, and 2, 4 D had concentrations less than 10 ppb. This may be associated with multiple factors and is complex to elucidate, so we propose to carry out individual studies for each pesticide in the region.

4.2 Concentrations of Glyphosate

Glyphosate is not susceptible to photochemical degradation, and it has low mobility in aquatic sediments, with half-life in soil varying from months to years depending on environmental conditions (Vereecken 2005). The interaction of glyphosate with clay, iron oxide, phosphates, and organic matter (characteristics of the soil in the Cienega region) facilitates the formation of colloids, thereby reducing its adsorption in the soil and decreasing its concentration in groundwater (IIEG 2018; Vereecken 2005; Saunders and Pezeshki 2015; OMS 2004). Increased colloid formation decreases the microbial degradation of glyphosate (Vereecken 2005). A study has reported that 88.1% of applied glyphosate is retained in the surface layer of the soil and is dragged by runoff (Lupi et al. 2019). These factors, as well as high frequency of application of the herbicide in the region (14.43%), are responsible for the high concentrations of glyphosate in the lake of Chapala, river Zula, tributary Palmar, Lerma river section Cumuato, and its community (228.53–510.46 ppb). The community of Cumuato had the highest concentration of glyphosate (442.51–510.46 ppb). Cumuato town is known for increases in the annual application of glyphosate. For example, glyphosate use increased from

408.1 g/ha in the year 2012 to 2 kg/ha in 2014 (Bautista-Ávalos et al. 2014).

The MDS analysis showed similarity in glyphosate contamination for two statistically significant groups (stress = 0.005): Group 1 covered the areas of Lerma river section Cumuato, the community of Cumuato, and Cuci. The homogeneity of the river and the Cumuato community were similar since they are dependent areas. However, the distance from Cumuato to Cuci is 35.1 km. Thus, the homogeneity in MDS may be related to the high concentration of glyphosate in the water samples analyzed from Cuci (99.3–119.43 ppb). The surface tributaries of the University Center come from direct runoff from agricultural fields subjected to continuous application of pesticides. Group 2 covered the sampling points of Chapala Lake, Zula River, and tributary Palmar. The glyphosate concentrations for these 3 regions were similar, ranging from 228.53 to 258.28 ppb. This homogeneity of concentration is as a result of their interdependence on water exchange from Chapala Lake to the tributary of Palmar and the Zula River to Chapala Lake.

Regarding the levels of glyphosate in runoffs, surface water, and streams, in previous studies, variable concentrations of this herbicide ranging from 0.01 to 700 ppb were reported. The highest concentrations of glyphosate were obtained from runoff basins near agricultural areas (Rodríguez-Aguilar et al. 2019; Coupe et al. 2012; Shipitalo and Owens 2011; Battaglin et al. 2005; Kjær et al. 2005 and Edwards et al. 1980). This is agreement with the results obtained in Cumuato, Cuci, and Lake Chapala.

4.3 Concentrations of Malathion

Malathion is a broad-spectrum organophosphate used in agriculture and also in public health for the control of vector transmission of insects (Singh et al. 2014). It has a moderate water solubility of 145 mg/L at 25 °C (OMS 2004). However, it is chemically degraded at temperatures of 27 to 32 °C from malathion to malathion monocarboxylic acid, malathion dicarboxylic acid and malaoxon. It is broken down at $pH > 7$ by bacterial enzymes such as organophosphate hydrolases, esterases, carboxylesterases, phosphatases and oxidoreductases, amongst others (Kumar et al. 2019). However, it has been confirmed that high concentrations of malathion saturate the enzyme active sites, thereby decreasing its decomposition (Kumar et al. 2019; Singh 2002). The photolytic half-life of malathion is 156 days under environmental conditions, but it decreases to 107 days under aqueous conditions (Kumar et al. 2019; Gao et al. 2018). The adsorption and mobility of malathion in the soil are almost zero, but they vary in response to degree of solid-liquid phase partition, soil pH, organic matter content, and soil type (Al-Wabel et al. 2010). Sandy soil leads to the high adsorption of malathion (Kulluru et al. 2010). Once applied on crops, most of the malathion remains in the area of application, and due to its low absorption, rain, fog, or wind can mobilize the insecticide (OMS 2004). In the zone of Cienega, Jalisco, one of the most used insecticides for control of the population of mosquitoes is malathion in 44% emulsion with water (CENAVECE 2014). This explains why high concentrations of this insecticide were found in all sampling areas, despite its low use by farmers (0.51%). In general terms, the concentration of malathion tripled that of glyphosate in the same samples, with the lowest concentration observed in Lerma river section Cumuato (311.76 ± 84.67).

It was observed that the results for malathion were high and variable. This variation may be due to periodic fumigations of malathion by the Ministry of Health in the Cienega area, which is also reflected in the dispersion of the MDS data. Over time, the application of malathion (manual and fumigation) allows the accumulation of this insecticide in tributaries, runoff, and streams. This is compounded by its poor adsorption in the clay type of soil in the zone Cienega, thereby contributing to its high levels in aqueous surfaces (Kulluru et al. 2010; IIEG 2018). Analysis of various reports in the literature on the concentrations of malathion in surface water and runoff shows that the highest levels in water were reported in the Ayuquila-Armería River in the states of Jalisco-Colima, Mexico (average: 500120 ppb), a value that is very close to that reported in drainage waters in Damietta, Egypt, with a range of 71.9 to 466 ppb (Burgos-Hernández et al. 2006; Morales et al. 2019; Rodríguez-Aguilar et al. 2019; Abdel-Halim et al. 2006; Anderson et al. 2018; Sankararamakrishnan et al. 2005; Mekonen et al. 2016; Derbalah et al. 2019). In this study, the results obtained in the Cienega region are disturbing.

4.4 Glyphosate and Malathion Levels in Water Purification Plants

In 2017, Mexico was ranked the third country in the world in bottled water consumption (Fortune 2019; IBWA 2017). About 7000 micro- and small companies participate in the market of water purification at national level (Pacheco-Vega 2015). The purification process of water treatment plants in Mexico is based on Official Mexican Standards NOM-230-SSA1-2002, NOM-014-SSA1-1993, NOM-041-SSA1-1993, NOM-092-SSA1-1994, Nom-112-SSA1-1994, NOM-117-SSA1-1994, NOM-127-SSA1-1994, NOM-160-SSA1-19941995, and NOM-201-SSA1-2002. The water purification plants in Mexico use filtration techniques with sand beds and activated carbon filters to exclude particles of up to 30 μm, thereby eliminating residues of chlorine, dyes, and organic pollutants such as pesticides, herbicides, and hydrocarbons. Subsequently, the filtered water passes through a softener in order to remove hard minerals via ion exchange. The softened water is then directed to a multi-retention polishing filter that removes particles of sizes 25–5 μm. Once the liquid is free of residues, it passes into a reverse osmosis chamber of one or more membranes to remove germs and reduce traces of salts and minerals, followed by exposure to ultraviolet light and an ozone generator, broad-spectrum germ neutralizer where up to 99.9% of microorganisms are eliminated (Vera 2008). There are few studies regarding the water quality of purifier plants. A study evaluated the risk points during the water purification process in establishments in Veracruz, Mexico, and identified weaknesses in the purification plants. These weaknesses were as follows: poor quality of the municipal water from which they are supplied, low frequency of physico-chemical analysis of the water, and poor maintenance of calibration filters and purification systems (Nexticapa et al. 2018). Moreover, there are reports of purifiers in Mexico with dubious quality of water processing which lead to public health risk (Mesa 2010).

High concentrations of malathion in 100% of the samples analyzed (665.1–1056.9 ppb) were obtained in water purification plants samples for human consumption. In contrast, much lower malathion concentrations were obtained in water for human consumption in Venezuela, ranging from 0.012 to 2.033 ppb (Flores et al. 2011). The high levels of malathion found in bottler water consumption in the Cienega region may be related to the quality of water from which the treatment plants of these establishments get their supply. The water supply comes the tributaries of the Cienega area, i.e., lake of Chapala, river Zula, and tributary the Palmar; all of which had elevated levels of malathion. The concentrations of malathion reported in water purification plants were above the EPA-suggested limit (100 ppb for children and 200 ppb for adults). Exposure to malathion, in particular its malaoxon metabolite and isomalathion, is associated with genetic damage in lymphocytes, increased risk of incidence to different carcinomas, damage to the hematopoietic system, diffuse parenchymal degeneration of hepatocytes (Tchounwou et al. 2015), and effects associated with neurotoxicity (Salama et al. 2015). Therefore, there is urgent need for close monitoring of water purification plant supply facilities in the Cienega region of Jalisco.

The levels of the herbicide of glyphosate were close to 150 ppb. This may be due to the high frequency of use of the herbicide within the Cienega region (14.43%). Hepatic and renal glyphosate exposure has increased enzymatic activity of glutathione and glutathione peroxidase. In the central nervous system, glyphosate has a mechanism similar to glutamate agonist inducing neuronal death (Silva-Madera et al. 2019). On the other hand, glyphosate is associated with non-Hodgkin's lymphomas, renal tubule carcinoma, hemangiosarcoma, pancreatic islet adenomas, and breast cancer (Silva-Madera et al. 2019; Berry 2020).

It is important to emphasize that there is insufficient information in the literature about the quality of water purifiers, their processing, and the concentration of pesticides. Thus, this study is a pioneering effort in this type of analysis in the region and the country.

4.5 Weaknesses of the Study

Further studies should include the following: (a) assessment of the concentration of metabolites such as breakdown products of malathion and glyphosate; (b) determination of the concentrations of malathion and glyphosate in dry weight of filters in purifying plants to corroborate saturation with these pesticides; (c) increasing the number of samples and using consecutive sampling at different times of the year in both surface water and water for human consumption.

5 Conclusion

Efficient techniques are now required for the detection and resolution of surface and drinking water quality problems in many countries. This study shows the potential use of HPLC MS/MS for pesticide detection in surface and drinking water samples in the Cienega region of Jalisco, Mexico. The results indicate that despite analyzing a total of 22 pesticides, only the concentrations of glyphosate and malathion were high. Glyphosate was present in most of the sampled sites, but only the community of Cumuato showed values higher than those consistent with the Daily Intake Acceptable in Water according to the WHO. However, this did not apply to urban public establishments sampled in the city of Ocotlán Jalisco. Malathion was also present in all the sampled sites, even in urban public establishments, at levels above the limits recommended by EPA. This may pose a public health risk. Multidimensional scaling analysis (MDS) showed that the sampled sites could be grouped into 2 different bodies of water, according to their homogeneity in glyphosate concentrations, while malathion had more heterogeneous concentrations. This is the first study to assess water quality in relation to pesticide contamination in the Cienega region. It is therefore hoped that these results will help federal and local authorities adopt new guidelines for assessing water quality so as to prevent drinking water shortages and water-borne diseases that endanger human health.

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Compliance with Ethical Standards

Competing Interests The authors declare that they have no conflicts of interest.

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