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Pesticides in surface freshwater: a critical review

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Abstract The objective of this study was to critically review studies published up to November 2021 that investigated the presence of pesticides in surface freshwater to answer three questions: (1) in which countries were the studies conducted? (2) which pesticides are most evaluated and detected? and (3) which pesticides have the highest concentrations? Using the Prisma protocol, 146 articles published from 1976 to November 2021 were included in this analysis: 127 studies used grab sampling, 10 used passive sampling, and 9 used both sampling techniques. In the 45-year historical series, the USA, China, and Spain were the countries that conducted the highest number of studies. Atrazine was the most evaluated pesticide (56% of the studies), detected in 43% of the studies using grab sampling, and the most detected in passive sampling studies (68%). The compounds with the highest maximum and mean concentrations in the grab sampling were molinate (211.38 µg/L) and bentazone (53 µg/L), respectively, and in passive sampling, they were oxyfluorfen (16.8 µg/L) and atrazine (4.8 µg/L), respectively. The levels found for atrazine, p,p′-DDD, and heptachlor in Brazil were higher than the regulatory levels for superficial water in the country. The concentrations exceeded the toxicological endpoint for at least 11 pesticides, including atrazine (*Daphnia* LC_{50} and fish NOAEC), cypermethrin (algae EC50, *Daphnia* and fish LC_{50}; fish NOAEC), and chlorpyrifos (*Daphnia* and fish LC_{50}; fish NOAEC). These results can be used for planning pesticide monitoring programs in surface freshwater, at regional and global levels, and for establishing or updating water quality regulations.

Keywords Surface freshwater · Pesticide contamination · Herbicides · Insecticides · Organochlorines · Organophosphates

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Introduction

Pesticides are widely used in the management of pests that affect agricultural quality and production (Mateo-Sagasta et al., 2017), and their use has increased over the years to meet the demand for food and other products

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for a growing population (FAO, 2020a). Currently, more than 1680 substances, active ingredients and metabolites, are included in the Pesticides Properties DataBase (PPDB, 2021). These are substances classified into different classes (e.g., herbicide, insecticide, and fungicide), chemical groups (e.g., organochlorines, organophosphates, and triazines), and modes of action (e.g., acetylcholinesterase enzyme and photosystem inhibition).

Despite the benefits of using pesticides in agriculture, these compounds are potential contaminants of surface freshwater (Caldas, 2019; Pirsaheb et al., 2017; Souza et al., 2020). Rockström et al. (2009) described chemical water pollution as one of the axes of the planetary boundary that is not yet quantified, and its damage to aquatic organisms and humans is still not totally understood. Environmentally sound management and a significant reduction in the release of chemical substances into water by 2020, such as pesticides, were also two of the goals of the 12th United Nations Sustainable Development Targets (UN, 2016). Therefore, efforts to assess the panorama of pesticides in surface freshwater at local and global scales are important.

Some review studies demonstrated the presence of pesticides in freshwater, from trace levels to high concentrations. Pirsaheb et al. (2017) focused on organochlorine and organophosphate chemical groups and restricted the study to the period 2000 to 2015, while Souza et al. (2020) analyzed studies published from 2012 to 2019. However, a review that covers all pesticides, without restriction of period, is important to understand the panorama of water contamination by these pollutants and the evolution of the problem in recent decades.

The objective of this study was to critically review studies on pesticides in surface freshwater. The study covered works published until November 2021, of all types and chemical groups of pesticides, to answer three questions: (1) in which countries were studies that analyze pesticides in surface freshwater conducted? (2) which pesticides are most evaluated and detected by the studies? and (3) which pesticides have the highest concentrations?

Methods

The Prisma protocol, used to prepare a systematic review and meta-analysis, was followed in this critical review in order to reduce the risk of bias and ensure study quality (Moher et al., 2015). Descriptors related to pesticides inserted into the search string were: {((pesticide* OR metabolite* OR agrochemical* OR agrichemical) AND detect*)} AND {(((surface AND freshwater* OR river* OR lake*) AND contaminant*) AND NOT soil*)}. Searches were performed in the ScienceDirect, Scopus, and Web of Science databases, focusing on titles, abstracts, and keywords, and include papers published up to November 08, 2021. Additionally, papers that escaped from the database search but were mentioned in some studies were also included.

Eligibility criteria

For inclusion criteria, were considered studies (1) published in peer-reviewed scientific journals, (2) in English, (3) about pesticides in surface freshwater, (4) cited the sampling environment and pesticide analysis technique, (5) generated analytical data, and (6) contained the names and the concentrations of the investigated pesticides. Exclusion criteria were studies that (1) were not original research (reviews, meta-analysis, letters, etc.), (2) had data from another study already included, (3) presented data for the sum of pesticide concentrations, and (4) showed data that were in non-comparable units and/or only displayed in graphs.

Study selection and data collection process

Two independent reviewers selected the publications based on information contained in titles and abstracts and considering the eligibility criteria. When there were disagreements regarding the inclusion or exclusion of a study, the reviewers evaluated the work and decided together. The studies included were fully read to verify if they met the eligibility criteria.

After assessing compliance with the eligibility criteria, the following information was extracted: (1) authors and year of publication; (2) country and/or region where the study was conducted; (3) landscape from the surrounding area to the sampling locations; (4) type of water body monitored; (5) sampling technique performed in the study (grab or passive); (6) pesticides evaluated and concentrations detected; (7) analysis technique and method limit of quantification

(LOQ), maximum detection limit (MDL) and/or limit of detection (LOD).

Data analysis

The name of each substance (pesticide and/or its metabolite) was first standardized according to the Pesticides Properties Data Base (PPDB, 2021), and, when the name was not found, the PubChem (2021) database was used. The included studies were separated into two groups, according to the sampling technique: grab and passive sampling. Grab sampling consists of carrying out the sample collection at an episodic moment in time, while passive sampling makes use of devices, called passive samplers, which are installed in the environment for a period, usually days, and makes up for the loss of occasional events of pollution and the variation of pollutants over time (Vrana et al., 2005).

The collected data were tabulated and analyzed using Excel software. Concentrations extracted from the studies and the LOD/MDL/LOQ of the methods were standardized in micrograms per liter. The number of studies conducted per country and by region was plotted on a map using Google Earth and QGis software.

The number of studies in which each pesticide was evaluated, and the number of studies in which it was detected at least once was counted. Concentration values equal to or above the LOD/MDL or LOQ were considered, as reported in the study. Pesticides with the highest concentrations were identified for both sampling techniques, considering the maximum and mean concentrations reported in the studies. The highest concentrations found were compared with regulations on pesticides in surface freshwater and ecotoxicological endpoints for aquatic biota, according to data availability.

Results and discussion

Selection process and study distribution around the world

The search returned 941 publications (Fig. 1). With the removal of duplicates and selection of articles according to eligibility criteria, 146 studies remained in this revision, the oldest published in 1976: 127 studies used grab sampling, 10 studies used

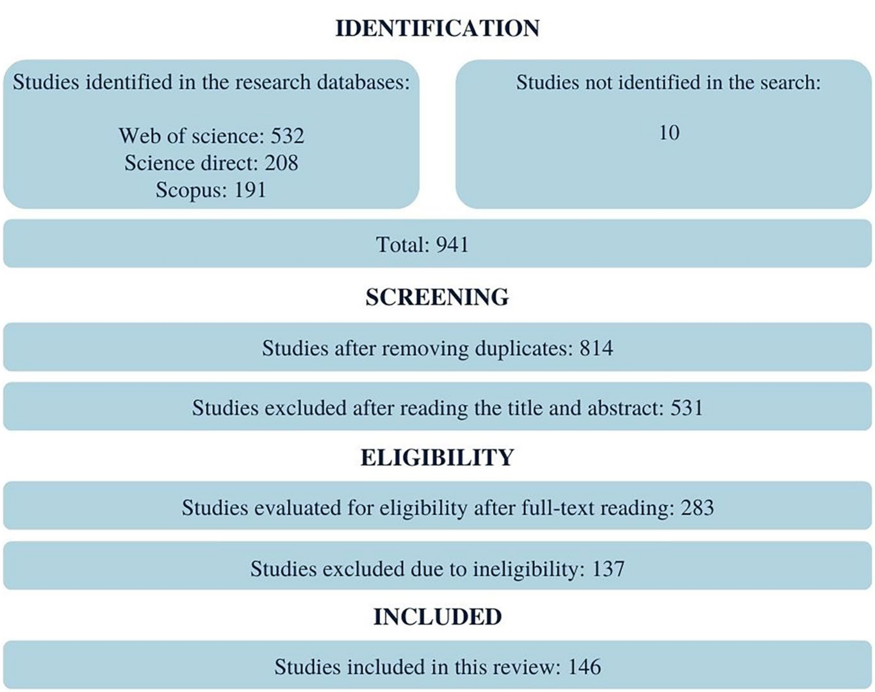

Fig. 1 Flowchart for selecting the studies that analyzed pesticides in surface water published up to November 08, 2021

passive sampling, and 9 studies used grab and passive samplings.

The 146 studies were conducted in 48 countries, mainly the USA (30), China (19), and Spain (16) (Fig. 2). Detailed information of each study is available in Table 1 and in the Supplementary Material (Tables S1 and S2). The studies conducted in the USA included several regions of the country and were carried out mainly by institutional agencies such as the US Geological Survey (USGS), Environmental Protection Agency (EPA), and California Department of Pesticide Regulation (Bai et al., 2018; Bradley et al., 2019; Bradley et al., 2017a, b; Elliott et al., 2017; Elliott & VanderMeulen, 2017; Ensminger et al., 2013),

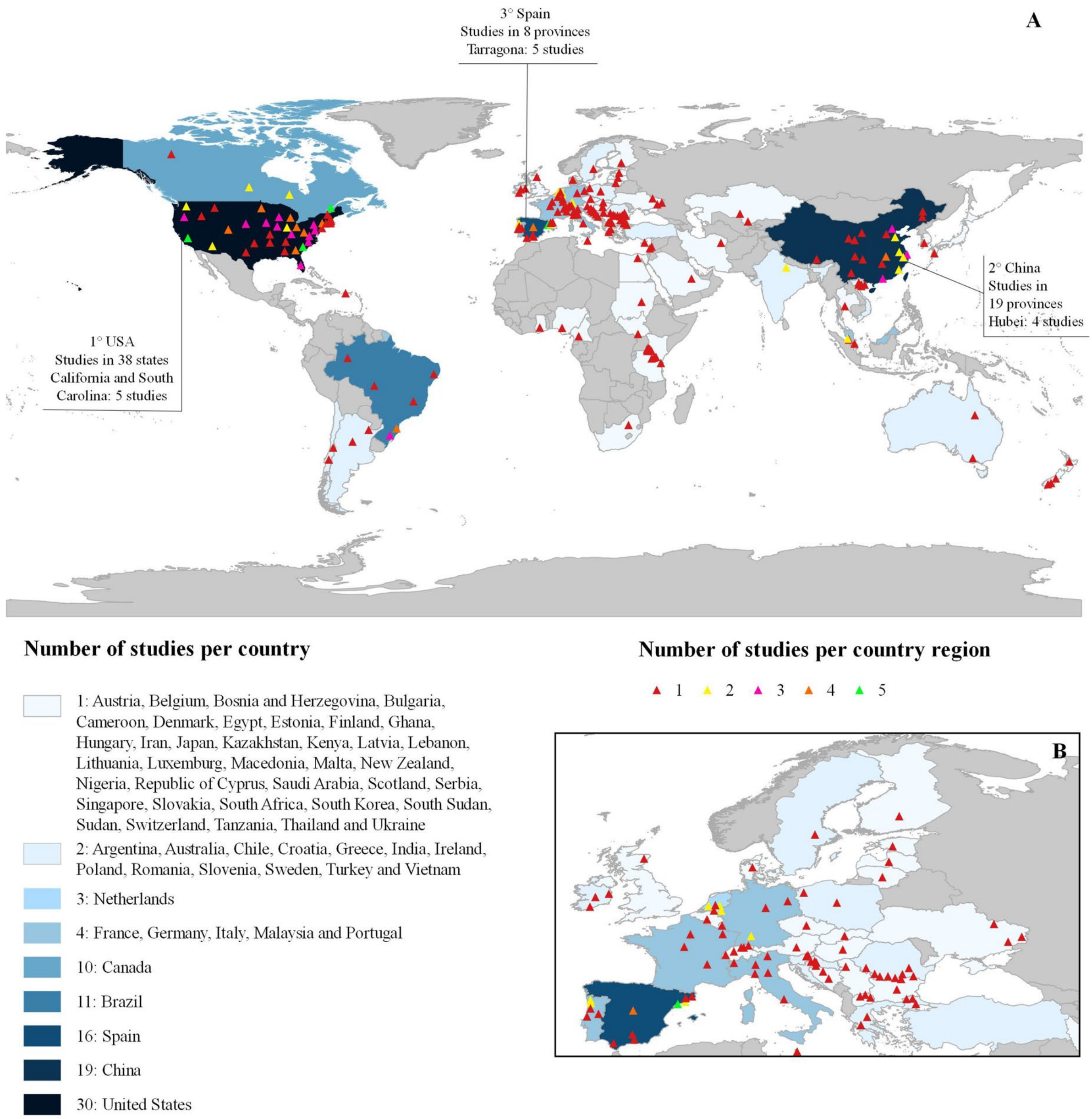

Fig. 2 (**A**) World distribution of the 146 studies published from 1976 to 2021 that investigated the occurrence of pesticides in surface freshwater. (**B**) Distribution of the 43 studies conducted in Europe

Table 1 General characteristics of the 146 studies included in this review that investigated the occurrence of pesticides in surface freshwater in the world, listed according to the publication year. G, grab sampling; P, passive sampling

Highest concentration [max.] and (mean), µg/L	Environment	Landscape	Country	Reference
G: tolylfluanid [201,56]	River	Agriculture, urban areas, and industry	Croatia	Malev et al. (2022)
G: diuron [1.37]	Rivers	Agriculture, urban area, and industry	Ukraine	Nikolopoulou et al. (2022)
G: δ-HCH [0.026] and o,p′-DDD (0.002)	River and spring water	Agriculture	China	Huang et al. (2021)
G: bentazone [180] and (53)	Channels	Agriculture and chemical industries	Spain	Barbieri et al. (2021)
G: metolachlor SA [0.09]	River and creeks	Agriculture, pasture, forests, and developed	USA	Thompson et al. (2021)
G: malathion [0.535]	Rivers and streams	Urban area	Brazil	Rico et al. (2021)
G molinate [211.38] and (15.56)	River and creeks	Agriculture, big urban regions, and industries	Turkey	Emadian et al. (2021)
G: metolachlor ESA [0.08]	River[a]	n.i	Canada	Picard et al. (2021)
G: imazethapyr [0.58]	Lake	Agriculture, urban areas, industries, and livestock	Brazil	Perin et al. (2021)
G: MCPA [23.7] and flufenacet ESA (1)	Streams	Agriculture and pasture	France	Le Cor et al. (2021)
G: metazachlor ESA [3]	River	Agriculture, urban area, industry, and forest	Italy	Carere et al. (2021)
G: 2,4-D [1.2]	Creeks	Urban area	USA	Cavallin et al. (2021)
G: aldrin (1.15)	River	Agriculture, urban area, and livestock	Portugal	Paíga et al. (2021)
G: carbendazim [0.02]	River	Agriculture, urban area, and forests	China	Liu et al. (2021)
G: bentazone [63.1]	Rivers and creeks	Agriculture and urban area	USA	Bradley et al. (2021)
G: thiabendazole [< MDL]	Reservoir	n.i	China	Zhang et al. (2021a, b)
G: atrazine [38]	River and creeks	Agriculture, urban area, pasture, and forest	USA	Smalling et al. (2021)
G: heptachlor [0.04] and β-HCH (0.02)	Lake	Agriculture and industry	China	Cao et al. (2021)
G: 2,6-dichlorobenzamide [0.06]	River	Urban area	Sweden	Golovko et al. (2021)
G: dichlorvos (0.04)	River	Urban area	China	Zhang et al. (2021)
G: atrazine [0.3]	Rivers and creeks	Park	USA	Bradley et al. (2020)
P: atrazine [4.1] and (0.35)	Rivers, lakes, channel, and creeks	Agriculture, urban area, and pasture	USA	Alvarez et al. (2021)
G: atrazine [0.3]	Streams	Agriculture, urban area, and pasture	USA	Guardian et al. (2021)
G: carbendazim [0.17]	Lakes and river	Agriculture and urban area	Vietnan	Wan et al. (2021)
P: p,p′-DDE [0.00007]	Lake and creeks	Urban area and industries	Canada	Zhang et al. (2020)
G: bentazone [0.18]	Lake and river	Agriculture, urban area, and industry	China	Meng et al. (2020)

Table 1 (continued)

Highest concentration [max.] and (mean), µg/L	Environment	Landscape	Country	Reference
G: metolachlor [3.8]	Rivers and chaneel	Agriculture, urban area, and industry	USA	Battaglin et al. (2020)
G: imidacloprid [0.41]	River	Agriculture	Japan	Hashimoto et al. (2020)
G: dieldrin [0.37] P: atrazine (0.001)	River and reservoir	Agriculture, wetlands, and pasture	Kazakhstan	Snow et al. (2020)
G: hexazinone [1.5]	Lake	Agriculture, urban area, industry, and grassland	Kenya	Kandie et al. (2020)
G: n.d	River	Urban area	Brazil	Huelsmann et al. (2020)
G: n.d	River	Agriculture and urban area	Brazil	Cancillier et al. (2020)
G: α-HCH [0.013] P: α-HCH (0.007)	River	Agriculture and livestock	China	Liu et al. (2020)
G: n.d	Lake and channek	n.i	Turkey	Turan et al. (2020)
G: diazinon [0.25]	River	Urban area, agriculture, industry, and mineral	Malaysia	Zainuddin et al. (2020)
G: acetamiprid [0.034] and (0.02)	River and creek	Urban area	China	Lu et al. (2020)
G: glyphosate [4.8] and AMPA (1.6)	Streams	Agriculture, urban area, industry, grassland, forest, and park	Australia	Okada et al. (2020)
G: propoxur (0.85)	River	Agriculture and urban area	Brazil	Gonçalves et al. (2020)
G: diazinon [1.01] and (0.14)	Lakes and channels	Agriculture, urban area, and industries	Saudi Arabia	Picó et al. (2020)
P: diazinon [0.05]	River	Agriculture, urban areas, and industries	Bosnia and Herzegovina	Toušová et al. (2019)
G: atrazine [76.8]	River and creeks	Agriculture, urban areas, forests, and grass	USA	Cipoletti et al. (2019)
G: tebuconazole [0.45] P: diuron [1]	Rivers and streams	Agriculture, urban areas, forested areas, and grasslands	Spain	Rico et al. (2019)
G: glyphosate [3] and (0.1)	Rivers	Agriculture, urban area, forest, and forestry	Canada	Montiel-Léon et al. (2019)
G: α-HCH (0.2)	River	Agriculture, urban area, and industry	Nigeria	Ogbeide et al. (2019)
G: 2,4-D (0.2) P: 2,4-D (0.07)	Streams	Agriculture, pasture, and native bush	New Zealand	Hageman et al. (2019)
G: terbutryn [0.43]	Rivers and stream	Agriculture, urban area, and industry	Spain	Rubirola et al. (2019)
G: carbendazim [0.15]	River and channel	Urban area	Thailand	Juksu et al. (2019)
G: imidacloprid [0.15] and (0.08)	River	Urban area	China	Yi et al. (2019)
G: n.d	River	n.i	Brazil	Silva et al. (2019)
G: prometryn [0.03] P: prometryn (0.05)	River	Urban area	China	Gao et al. (2019)
G: carbendazim [16.84] and (4.84)	Rivers	Agriculture, urban area, industry, and mining	Spain	Quintana et al. (2019)
G: thiamethoxam [0.27] and imidacloprid (0.006)	River	Agriculture and urban area	China	Mahai et al. (2019)

Table 1 (continued)

Highest concentration [max.] and (mean), µg/L	**Environment**	**Landscape**	**Country**	**Reference**
G: imidacloprid [0.16]	Rivers[a]	Agriculture, urban area, industry, livestock, and poultry	China	Zhang et al. (2019)
G: imidacloprid (0.016)	Rivers	Agriculture, urban region, and industries	Spain	Borrull et al. (2019)
G: 2,4,5-trichloro-6-hydroxybenzene-1,3-dicarbonitrile [39.18]	Creeks	Agriculture, pasture, urban region, and forest	USA	Bradley et al. (2019)
G: n.d	River	n.i	Iran	Chahkandi et al. (2019)
P: oxyfluorfen [16.8]	Rivers, streams, and lakes	Agriculture	Brazil	Valenzuela et al. (2019)
G: quinoxyfen [0.006]	Rivers[a]	Agriculture, urban region, and industries	Ireland	Jones et al. (2019)
G: triallate [0.5]	Rivers[a]	Agriculture, pasture, urban region, and industries	Portugal	Sousa et al. (2019)
G: diazinon [< 0.00001]	River[a]	Urban region	Malasyan	Wee et al. (2019)
G: bentazone [0.85] and (0.23)	Rivers and channel	n.i	China	Xu et al. (2019)
G: chlorpyrifos and 2-phenylphenol [< 0.01]	Rivers*	Agriculture, urban area, and park	Australia	Scott et al. (2018)
P: atrazine (0.5)	River	Agriculture and urban area	Canada	Challis et al. (2018)
P: atrazine (0.14)	River	Agriculture and urban area	USA	Penland et al. (2018)
G: 2-Phenylphenol [0.04] and (0.04)	River, lake, and channel	Agriculture, urban area, and industry	Serbia	Škrbić et al. (2018)
G: acephate [4.47] and (1.67)	River[a]	Agriculture and urban area	China	Sun et al. (2018)
G: thiamethoxam [0.06] and imidacloprid (0.01)	Wetland	Agriculture	USA	Williams and Sweetman (2019)
G: triclopyr [5.2]	Rivers and creeks	Agriculture, urban region, industries, forests, recreation parks, and golf courses	USA	Bai et al. (2018)
P: atrazine (1.25)	Rivers, lake,[a] and creek	Agriculture and urban region	Canada	Challis et al. (2018, b)
G: bromacil [0.02]	Dam catchment and rivers[a]	Agriculture, urban region, and industries	South Africa	Rimayi et al. (2018)
G: thiacloprid (0.76)	Rivers[a]	Agriculture, urban region, and industries	Portugal	Barbosa et al. (2018)
G: malathion [0.94]	River	Agriculture and vegetation native	Brazil	Berton et al. (2018)
G: diuron [13.9] and (1.63) P: diuron [0.37] and (0.21)	Rivers	Agriculture, urban regions, industries, and forests	Cameroon	Branchet et al. (2018)
G: hydroxyatrazine [9.7]	River	Agriculture, urban region, and industry	China	Brauns et al. (2018)

Table 1 (continued)

Highest concentration [max.] and (mean), µg/L	Environment	Landscape	Country	Reference
G: carbendazim [0.12]	Streams and river[a]	Industry	Germany	Merel et al. (2018)
G: terbuthylazine [0.002]	Stream and lake	Glaciers	Italy	Ferrario et al. (2017)
G: β-Endosulfan [0.004]	Tributaries and channels	Urban area and industry	Singapore	Wang and Kelly (2017)
G: phorate sulfoxide (38.9)	River	n.i	India	Asati et al. (2017)
G: 2,4-D [0.8] and (0.2)	Rivers	Agriculture, urban region, and industries	Brazil	Bianchi et al. (2017)
G: simazine [3.14]	Rivers, creeks, lakes, and streams	Agriculture, urban region, industry, pasture, forest, wetland, and grassland	USA	Elliot and VanderMeulen (2017)
G: pentachlorophenol [0.2]	Rivers and creeks	Agriculture, urban region, forest, and wetland	USA	Bradley et al. (2017a)
G: 3,4-dichloroaniline [80.02]	Rivers, creeks, canals, swamp, dam, lakes, and sloughs	Agriculture, urban regions, pasture, forests, shrubs, and wetlands	USA	Bradley et al. (2017b)
G: metolachlor [1.53]	Rivers, lakes, creeks, and channel	Agriculture, urban region, animal feeding operations, forest, and wetland	USA	Elliot et al. (2017)
G: permethrin (0.94) P: deltamethrin (0.02)	Creeks	Urban region	USA	Liao et al. (2017)
G: desphenyl-chloridazon [0.42] and (0.37)	Rivers[a]	Agriculture, urban region, and industries	Germany	Seitz and Winzenbacher (2017)
G: molinate [0.55]	Rivers	Agriculture, urban region, factories, industries, and mining	Macedonia	Stipaničev et al. (2017)
P: chlorpyrifos [0.12] and diazinon (0.05)	Lake and river	Agriculture and urban region	Lebanon	Aisha et al. (2017)
G: chlorotoluron (0.02) P: chlorotoluron (0.01)	River[a]	Agriculture and urban area	Scotland	Zhang et al. (2016)
G: simazine [0.46] P: 2,4-D (0.25)	Creeks and river	Agriculture, forests, and bare rock	USA	Hapke et al. (2016)
G: aclonifen [0.01]	Rivers	n.i	Romania	Iancu et al. (2016)
G: chlorpyrifos (0.07)	Rivers[a]	Agriculture, urban region, industries, mining, forest, and poultry farm	Malaysia	Wee et al. (2016)
G: metolachlor [0.44]	Creeks	Agriculture, pasture, and urban region	USA	Fairbairn et al. (2016)
G: atrazine [0.004]	River	Agriculture, urban region, and forest	Slovenia	Koroša et al. (2016)
G: heptachlor epoxide [1.57] and (0.67)	River	Agriculture and urban region	Sudan and South Sudan	Nesser et al. (2016)
G: atrazine (0.16)	Rivers	Agriculture, urban region, and forest	France	Camilleri et al. (2015)
G: n.d	Rivers and channel	Agriculture and urban region	Spain	Luque-Espinar et al. (2015)

Table 1 (continued)

Highest concentration [max.] and (mean), µg/L	Environment	Landscape	Country	Reference
G: chloridazon-desphenyl [2.2]	Rivers	Agriculture and urban regions	Switzerland	Moschet et al. (2014)
G: chlorpyrifos (0.02)	River[a]	Agriculture, urban region, industries, and park	Spain	Pintado-Herrera et al. (2014)
G: p,p′-DDE [0.02]	Lake	Agriculture and urban region	China	Zhang et al. (2014)
G: dimethoate [5.17] and prometon (4.1)	Rivers and reservoirs[a]	Agriculture, urban region, and industries	Spain	Robles-Molina et al. (2014)
G: chlorpyrifos [0.04]	Rivers and tributaries	Agriculture and industry	South Korea	Lee et al. (2014)
G: α-HCH [0.004] and (0.003)	Rivers	Agriculture, urban area, industry, and livestock	Tanzania	Hellar-Kihampa et al. (2013)
G: endosulfan sulfate [0.03]	Rivers	Urban region	Spain	Nallanthigal et al. (2013)
G: simazine [2]	Rivers[a]	n.i	USA	Anumol et al. (2013)
G: diuron [17.6]	Creeks[a]	Urban region	USA	Ensminger et al. (2013)
P: atrazine (4.8)	River	Agriculture	USA	Knight et al. (2013)
G: carbaryl [0.09]	Rivers	Agriculture and forests	Chile	Retamal et al. (2013)
G: AMPA [2.28]	Rivers, channel, intake, and sluice	Agriculture, urban region, industry, stock farming, and nature	Netherlands	Houtman et al. (2013)
G: γ-HCH [0.025]	Rivers	Agriculture, urban area, and forest	Malaysia	Santhi and Mustafa, (2013)
G: endosulfan sulfate [0.004]	River	Agriculture	Argentina	Schreiber et al. (2013)
G: clopyralid [3.5]	Reservoir and rivers	Agriculture, urban region, and industries	China	Wolf et al. (2013)
G: diazinon [0.27] and (0.09)	River	Agriculture, urban area, and industry	Spain	Gómez et al. (2012)
P: endosulfan sulfate [0.0006]	Lakes	Agriculture, forest, and park	USA	Mast et al. (2012)
G: 21 pesticides [< LOD]	Ponds, ditches, and canals	Agriculture	Vietnan	Hoai et al. (2011)
G: diazinon [0.15] and MCPA (0.1)	River	Agriculture and urban area	Spain	Calderón-Preciado et al. (2011)
G: atrazine [0.2]	Creeks	Agriculture and pasture	USA	Sellin et al. (2011)
G: aldrin [0.16]	Rivers	Agriculture	Brazil	Bedendo and Carasek (2010)
G: diuron [0.06]	Rivers	Agriculture, urban region, and industries	Spain	Bueno et al. (2010)
G: MCPA [0.38] and (0.1)	Rivers	n.i	Spain	Matamoros et al. (2010)
G: isoproturon [0.3] and (0.16)	River*	Agriculture and urban region	Greece	Stamatis et al. (2010)
G: endrin [0.28] and (0.04)	River	Agriculture, urban region, industry, and wetland	Poland	Tomza-Marciniak and Witczak (2010)
G: atrazine [7.3]	Rivers	Agriculture, urban region, pasture, wetland, and forests	USA	Kolpin et al. (2010)

Table 1 (continued)

Highest concentration [max.] and (mean), µg/L	Environment	Landscape	Country	Reference
G: α-HCH [26.8]	River	Agriculture, urban region, and industry	India	Najam et al. (2010)
G: isoproturon [1.96] and (0.05)	Rivers and creeks	n.i	Union European	Loos et al. (2009)
G: atrazine [2.1]	River and creek[a]	Agriculture, urban region, and forests	USA	Alvarez et al. (2009)
G: desethylatrazine [0.48]	Rivers	Agriculture and urban region	Canada	Garcia-Ac et al. (2009)
G: γ-HCH [1.61] and (0.07)	Lake	Agriculture	Ghana	Darko et al. (2008)
G: atrazine [0.17]	River	Agriculture, urban area, and industry	Spain	Gómez-Gutiérrez et al. (2006)
G: n.d	Lake	n.i	China	Xiao et al. (2006)
G: parathion-methyl [0.13]	Creeks[a]	Agriculture, urban region, pasture, and forests	USA	Barber et al. (2006)
G: o,p′-DDT [0.16] and p,p′-DDE (0.05)	Reservoir	Agriculture, urban region, and industries	China	Xue and Xu (2006)
G: n.d	River	Agriculture, forestry, and rocks	Chile	Barra et al. (2005)
G: aldrin [0.11]	River[a]	Agriculture, urban region, and industry	Spain	Brossa et al. (2005)
G: atrazine (0.09)	River	Agriculture and urban region	Canada	Sabik et al. (2003)
G: atrazine [0.08]	Rivers[a]	Agriculture and industry	Netherlands	van Stee et al. (2002)
G: molinate [0.36]	River	Agriculture	Italy	Agradi et al. (2000)
G: β-HCH [0.0001]	River[a]	Agriculture, urban region, and industry	Egypt	Yamashita et al. (2000)
G: heptachlor [57.8]	River	Agriculture and urban regions	Brazil	Araújo et al. (1998)
G: atrazine [0.05]	River	Agriculture and urban region	Canada	Sabik and Jeannot (1998)
G: mecoprop and dichlorprop [0.1]	Canals, river, and lake	n.i	Germany	Heberer et al. (1998)
G: 2,4,6 trichlorophenol [0.04]	River	Agriculture and industry	Canada	McCarthy et al. (1997)
G: p,p′-DDT [0.02] and (0.005)	River	Agriculture and industry	Argentina	Janniot et al. (1994)
G: atrazine [0.8]	Rivers[a]	Agriculture	France	Legrand et al. (1991)
G: aldicarb sulfoxide [10.9]	River[a]	Agriculture and urban region	USA	Foran et al. (1986)
G: hexachlorobenzene [0.03]	River	Industry	Canada and USA	Kauss and Hamdy (1985)
G: atrazine [42]	Rivers, reservoirs, creek, lake, and pond[a]	Agriculture and urban region	USA	Junk et al. (1976)

n.i. not informed

[a]Environment: exclusion of one or more collection points due to the study classifying it in an environment other than freshwater or the data being graphically displayed

which demonstrated a strong government effort to monitor pesticides in water. As one of the five countries with the largest export, import, and use of pesticides in the world (FAO, 2020a, b), the USA is extremely susceptible to the environmental impacts arising from the use of these substances.

China is the largest exporter and user of pesticides in the world (FAO, 2020a, b) and has the largest population on the planet (The World Bank, 2019). Since the 2000s, China has been implementing agricultural policies to guarantee food security and stabilize prices (Gale, 2013). Indeed, the first study conducted in the country that investigated the levels of pesticides in freshwater retrieved in this review was published in 2006, and in total, 19 studies were conducted up to November 2021, indicating also a growing concern over the impact of pesticide use on the environment.

Member countries of the European Union are required to monitor water quality for priority substances and other pollutants, including pesticides, but no concentration limits are established (European Commission, 2008). A total of 43 studies were conducted in the European region in surface water eligible for this critical review (Fig. 2B). Spain was the country that most conducted these studies (16), followed by Portugal, France, Italy, and Germany, with four studies each (Table 1; Tables S1 and S2).

In South America, only Brazil, with 11 studies, Argentine and Chile (2 studies each) had studies included in this review, and 9 studies were conducted on the African continent (Fig. 2). Analytical techniques for pesticide detection involve complex and expensive instruments that require specific training for use and ongoing maintenance (Kot et al., 2000; Ong et al., 2020), which may be limiting factors for some developing countries, including in Central and South American and Asian countries. It is interesting to note that no studies conducted in Russia were retrieved in this review, a developed country where, in principle, technical limitations do not apply (Fig. 2A).

General aspects of selected studies

The review covered a period of 45 years, and the oldest study was conducted in the USA using grab monitoring technique (Junk et al., 1976). This study evaluated the levels of atrazine, DDE, the degradation product of DDT (1,1′-(2,2,2-trichloroethane-1,1-diyl) bis(4-chlorobenzene)), and dieldrin in water bodies in Iowa.

The USGS pioneered the development of passive sampling techniques (USGS, 1999), and some studies describe these devices for use in surface water (Alvarez, 2010; Brumbaugh et al., 2002). van Stee et al. (2002) was the first study found during the article search process that used a passive sampler (Semipermeable Membrane Device, SPMD); however, the study was not included in this review because the concentrations were expressed in non-comparable units (ng/g fat). In addition to SPMD, the studies used other passive sampling devices such as POCIS (Polar Organic Chemical Integrative Sampler), o-DGT (diffusive gradients in thin films for organics), and PU (polyurethane film), with exposure from 4 to 460 days in water (Table S2). Alvarez et al. (2009) used POCIS and SPMD to evaluate various pesticides in the Potomac River watershed, USA, and four other studies using this technique were carried out in the country.

The use of grab sampling for water is widespread and consolidated (CETESB, 2011; European Commission, 2009); however, the chemical profile and concentrations of contaminants are restricted to the time of sampling, and the conditions between collections are unknown (European Commission, 2009). On the other hand, passive sampling provides weighted mean concentrations over the exposure time, which covers the conditions of the entire sampled period and eliminates extreme variations, such as fluctuations in contaminants (Valenzuela et al., 2020). However, these devices still have some limitations, including the effects of environmental conditions on analyte absorption; low sampling rate, which requires longer sampling time for lower concentrations (Namieśnik et al., 2005); and device theft. Additionally, some require complex mathematical models to calculate the sampling rate (Valenzuela et al., 2020). Thus, the two techniques are not mutually exclusive, but complementary. In Europe, for example, passive sampling is used as a method complementary to grab sampling (European Commission, 2009), and 9 studies included in this review also used both methods (Table 1).

Most studies were conducted close to agricultural regions (Table 1), an activity identified as the main source of pesticide contamination in water (WHO, 2016). However, some studies also evaluated pesticides in urban regions (Table 1), such as Liao et al.

(2017), who investigated insecticides for urban use and Wee et al. (2019), who focused on endocrine disruptors in urban rivers.

Rivers were the most monitored water bodies, although it must be noted that terms that describe lower order water bodies, such as streams, were not included in the search string. The European water quality directive, for example, suggests monitoring points in large rivers, because they are strategic environments for checking the state of a hydrographic basin (European Commission, 2000). In any case, the monitoring of water bodies of various orders in the hydrographic basin is important.

Pesticides evaluated and detected by studies on surface freshwater

The improvement of chromatography, with the development of new equipment and techniques from the 1960s onwards, allowed advances in the analysis of pesticides, with more sensitive and specific detectors, such as the mass spectrometer (MS), and made it possible to measure concentrations in the order of ng and pg (Solomon & Stephenson, 2010). Tables S1 and S2 show the analytical methods used in the studies and the reported LOD/MDL and/or LOQ. All studies included in this review used chromatographic methods for analyte separation. While non-polar and thermostable compounds, such as organochlorines and pyrethroids, are more easily evaluated by gas chromatograph (GC) techniques, (Ibáñez et al., 2008; Wille et al., 2012), more polar and thermolabile molecules are preferentially analyzed by liquid chromatography (LC) (Ibáñez et al., 2008). GC was the equipment used in most studies, and tandem mass spectrometry (MS/MS) was the most frequent detector, coupled with GC and/or with LC; many studies used different equipment, including high resolution (HR) GC or LC for screening before quantitation. The lowest LOD reported in the studies was 0.8 pg/L, obtained in UPLC-ESI-QqQ-MS/MS equipment (ultra-performance liquid chromatography equipped with an electrospray ion source coupled to triple quadrupole tandem mass spectrometry).

A total of 1064 pesticides were evaluated in the 127 studies that used grab sampling, with the reported number of samples collected varying from 2 to 370, information that was not included in most studies (Table S1). Almost half of the investigated pesticides (636) were detected. The pesticides most evaluated were atrazine (56%), simazine (46%), and chlorpyrifos (36%), and the most detected were atrazine (43%), simazine (29%), metolachlor (28%), and imidacloprid (28%) (Fig. 3A). Atrazine, simazine, chlorpyrifos, and metolachlor were more evaluated and detected in the USA (Table S1).

Nineteen studies were conducted using passive samplers (Fig. 3B), mainly in regions with agricultural proximity, with samples collected in South and North America, Europe, Africa, Asia, and Oceania, of which nine studies also used grab sampling (Table S2). A total of 164 pesticides were investigated in the 19 studies, with 5 to 460 passive samplers used and 132 pesticides detected at least once. Chlorpyrifos (74%), atrazine (68%), and diazinon (63%) were the most investigated compounds, while atrazine (68%), simazine (42%), chlorpyrifos (42%), p,p′-DDD (42%), and p,p′-DDE (42%) both DDT degradation products, were the most detected (Fig. 3B), with percentages similar to those of grab sampling (Fig. 3A).

Atrazine is a selective and systemic herbicide of the triazine group, used in the pre- and post-emergent stages of many crops, mainly corn, soybean, wheat, cotton, sorghum, and sugarcane (ANVISA, 2021; PPDB, 2021; USGS, 2017). Atrazine degradation products (desethyl and deisopropylatrazine) were also detected in surface water samples (Fig. 3). In 2017, the use of atrazine in the corn crop in the USA was 10,508 ton/km^2 (USGS, 2017). In Brazil, this herbicide was the fifth highest-selling active ingredient, with more than 23,000 tons sold in 2019 (IBAMA, 2020). In the European Union, the use of this herbicide has been banned (European Commission, 2004), but it is still monitored in food (European Commission, 2016) and in surface water (European Commission, 2008).

Simazine, another triazine herbicide, is also used in the pre- and post-emergent stages in various crops, including fruits, canola, chickpeas, beans, corn, sorghum, and sugarcane (ANVISA, 2021; PPDB, 2021; USGS, 2017). Metolachlor is a selective herbicide of the chloroacetamide group, which inhibits the synthesis of very long chain fatty acids in plant tissue and can be used in various crops, including corn, soybeans, sorghum, potatoes, cotton, and ornamental plants (PPDB, 2021; USGS, 2017). Imidacloprid is a systemic neonicotinoid insecticide used in various crops, including rice, maize, cotton, sugar cane, and various vegetables (PPDB, 2021; ANVISA, 2021).

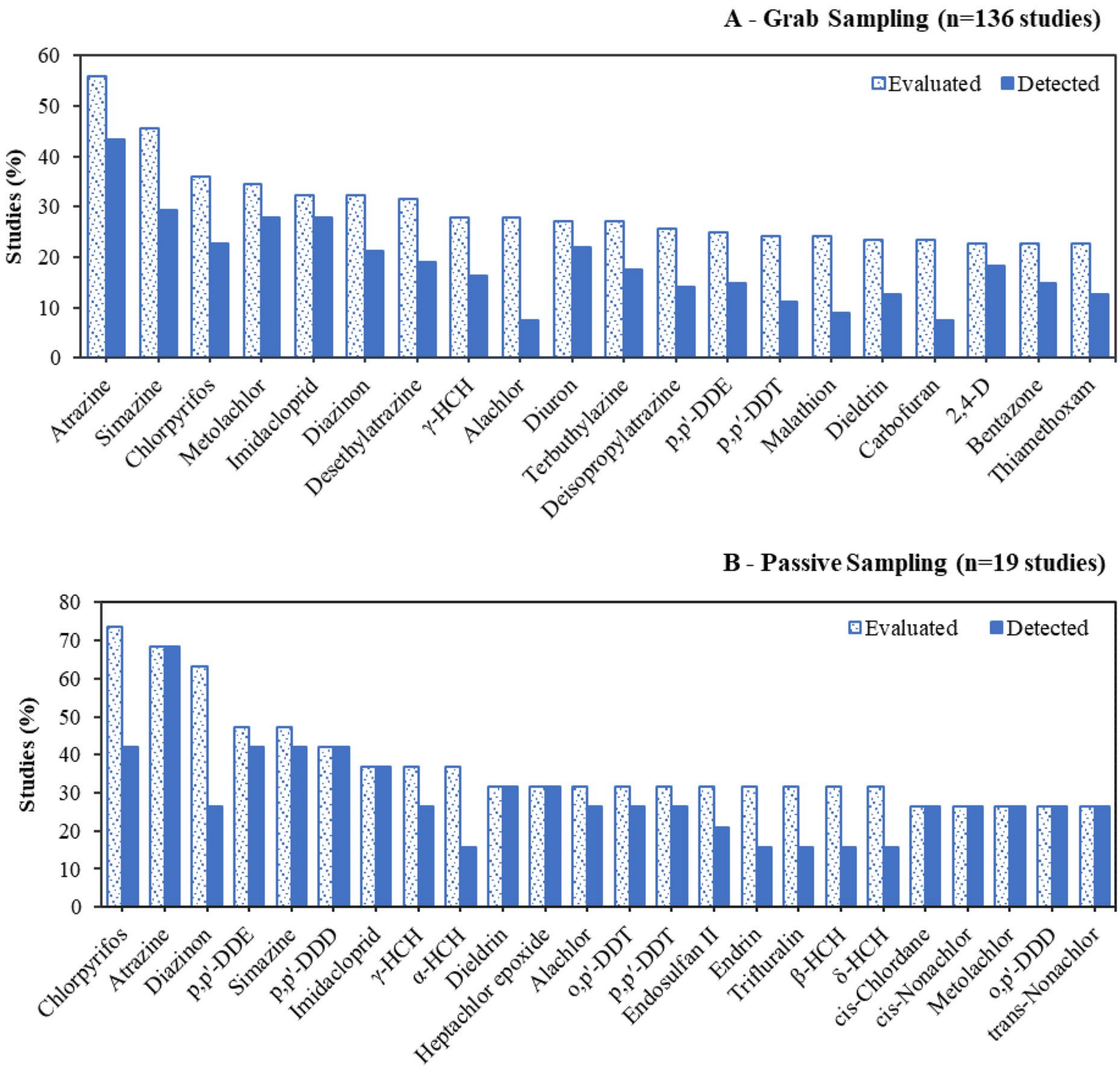

Fig. 3 Pesticides most evaluated and detected in the 146 studies (≥LOD/MDL/LOQ) using (**A**) grab sampling and (**B**) passive sampling as monitoring techniques

Several organophosphate insecticides were evaluated and detected in the studies, including chlorpyrifos, diazinon, and malathion (Fig. 3). These compounds are neurotoxic, acting as inhibitors of the enzyme acetylcholinesterase (AChE) in mammals, insects, and other organisms (Colovic et al., 2013). Several persistent organochlorine pollutants (POPs) were also detected in surface water samples, including the insecticides lindane (γ-HCH), DDT and its metabolite DDE, aldrin and dieldrin, and heptachlor and its epoxide (Fig. 3). These compounds are no longer used in agriculture in most countries or have restricted use, but their chemical characteristics make them persistent in the environment and susceptible to bioaccumulation in ecosystems (Chopra et al., 2011). Aiming to reduce and eliminate the release of these organochlorine pollutants, and to safeguard human health and the environment, the Stockholm Convention determined that the signatory parties carry out national and international research on these compounds (UN, 2001).

Pesticide concentrations in the surface freshwater

It is important to note that, for both sampling techniques, the most detected pesticides are not necessarily those with highest concentrations (Tables S1 and S2; Fig. 4). In general, the maximum and mean concentrations of compounds detected in samples collected by the grab monitoring technique were higher than those found in passive sampling (Fig. 4), similar to what was reported for some pesticides by Hapke et al. (2016). This is expected due to the dilution factor of concentration peaks that occurs during the passive sampling period.

The highest maximum and mean concentrations detected using grab sampling were for molinate (211.38 μg/L; Fig. 4A) and bentazone (53 μg/L; Fig. 4B), found, respectively, in the Ergene River hydrographic basin (Turkey; Emadian et al., 2021) and in Fangay Bay, Ebro River (Spain; Barbieri et al., 2021) (Table 1). For the passive sampling, the highest concentrations were for oxyfluorfen (16.8 μg/L; Fig. 4C) and atrazine (4.8 μg/L; Fig. 4D), detected in the San Francisco river basin (Brazil) and in the Elkhorn river (USA) (Table 1; Knight et al., 2013; Valenzuela et al., 2019). In all cases, the herbicides were detected

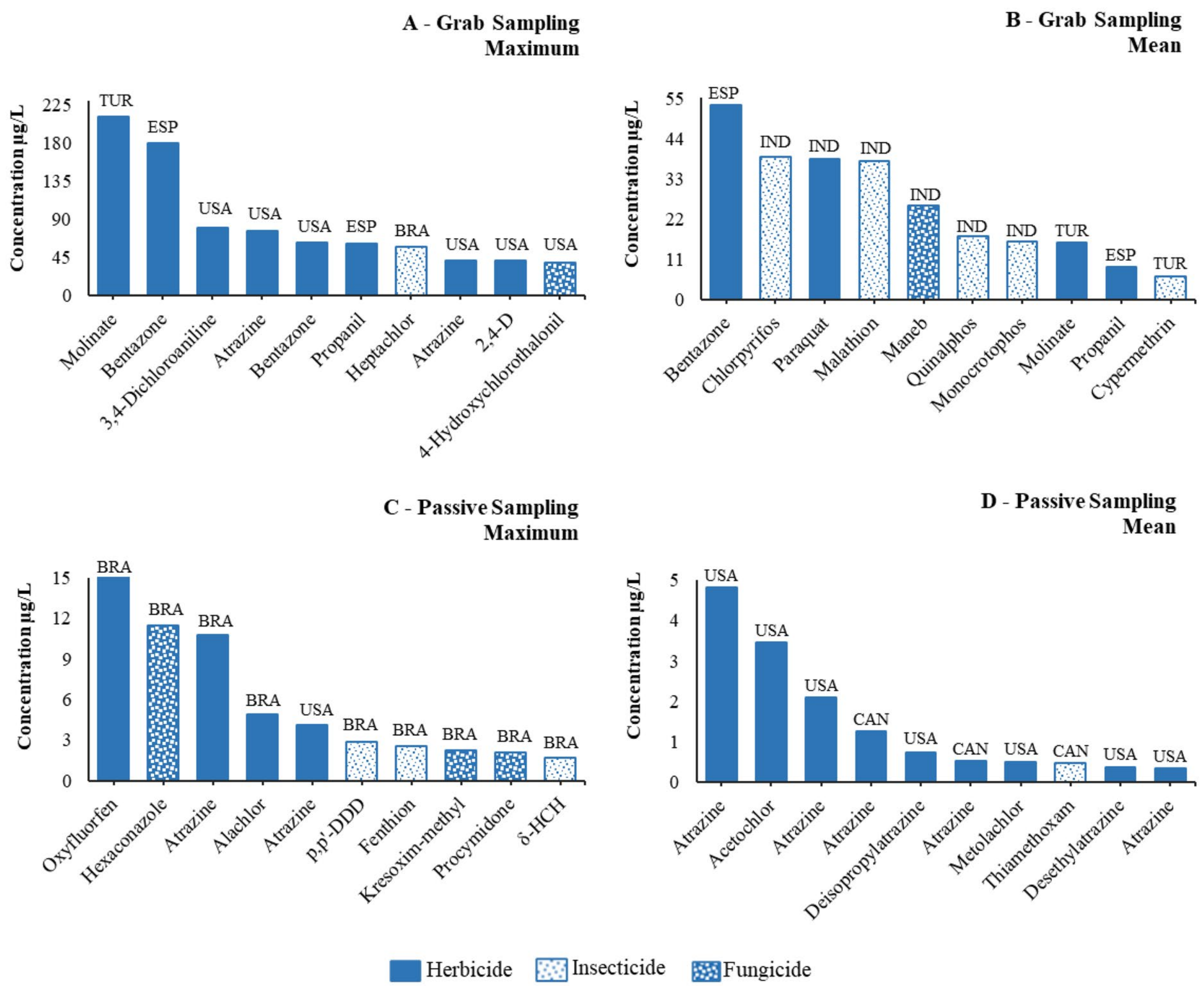

Fig. 4 The ten highest concentrations of pesticides or metabolites found in the 146 studies according to the sampling technique: (**A**) grab sampling — maximum concentrations; (**B**) grab sampling — mean concentrations; (**C**) passive sampling — maximum concentrations; (**D**) passive sampling — mean concentrations. Source: BRA: Brazil: Araújo et al. (1998); Valenzuela et al. (2019); CAN: Canada: Challis et al. (2018a, b); ESP: Spain: Barbieri et al. (2021); IND: India: Asati et al. (2017); TUR: Turkey: Emadian et al. (2021) and USA: United States: Alvarez et al. (2009, 2021); Bradley et al. (2021, 2017b); Cipoletti et al. (2019); Junk et al. (1976) and Knight et al. (2013)

Table 2 Pesticides for which the highest maximum or mean concentration (Fig. 4) is higher than the ecotoxicological parameter (μg/L)

Pesticide	Algae EC_{50} (72 h)[a]	*Daphnia* LC_{50} (48 h)[a]	Fish (LC_{50})[b]	Fish (NOAEC)[b]
Atrazine	68.02	30,031.17	2650	5
Chlorpyrifos	203.03	0.81	0.9	0.57
Cypermethrin	0.08	0.0006	0.195	0.051
Hexaconazole	0.5	10.96	2.44[a]	
Malathion	6371.78	20.32	2.05	8.6
Maneb	0.229	2.41	21	6.1
Molinate	18,455.77	17,783.15	105	390
Oxyfluorfen	507.17	231.22	100	1.3
Propanil	4589.33	1678.35	1150	9.1
Quinalphos	275.96	2.46	-	-
δ-HCH	2.03	1.39	0.0857[a]	-

LC_{50} lethal concentration, *EC_{50}* effective concentration, *NOAEC* non-observed adverse effect concentration

[a]Norman (2022)

[b]USEPA (2021)

near agricultural areas, which are indeed the major source of pesticide water contamination.

The European Commission (2020) established a parametric value of 0.1 μg/L for water for human consumption for any pesticide, except for organochlorine compounds (0.03 μg/L), but no value is established for surface water. Indian, Canadian, and USA regulations also establish values for water for human consumption (India, 2012; Canada, 2020, USEPA, 2018). Brazilian regulation for maximum pesticide levels in surface water depends on the water use (Brazil, 2005) and includes heptachlor (0.01 or 0.03 μg/L), atrazine (2 μg/L), and ΣDDTs (0.002 or 1 μg/L). These levels are much lower than the maximum concentrations found in the many studies conducted in the country (Fig. 4A, C). No regulation for pesticide in water was found in Turkey, in which a study showed the highest molinate concentration (Emadian et al., 2021).

The presence of pesticides and other chemicals in water bodies can have an important impact on aquatic organisms, reducing biodiversity and compromising the functioning of ecosystems (Carvalho, 2017). Table 2 shows the pesticides from Fig. 4 for which the highest maximum or mean concentrations extrapolated at least one ecotoxicological endpoint (EC_{50} and LC_{50} for acute exposure and NOAEC for fish chronic exposure), obtained from NORMAN (2022) and/or USEPA (2021).

Algae EC_{50} was extrapolated for atrazine (Fig. 4A; 76.8 μg/L) and cypermethrin (Fig. 4B; 6.24 μg/L) concentrations and *Daphnia* LC_{50} for chlorpyrifos, cypermethrin, and quinalphos (Fig. 4B; 6.24 to 38.9 μg/L) and δ-HCH (Fig. 4C; 1.65 μg/L). Fish LC_{50} was extrapolated for molinate (Fig. 4A; 211.4 μg/L); chlorpyrifos, cypermethrin, malathion, and maneb (Fig. 4B; 6.24 to 38.9 μg/L); and hexaconazole (Fig. 4C; 11.4 μg/L). Fish NOAEC was extrapolated for atrazine (Fig. 4A, C; 76.8, 10.7 μg/L); chlorpyrifos, cypermethrin, malathion, maneb, and propanil (Fig. 4B; 6.24 to 38.9 μg/L); and oxyfluorfen (Fig. 4C;16.8 μg/L).

This systematic review study has some limitations that should be pointed out. One limitation is that some publications may have been missed during the literature search, which was restricted to the previously defined descriptors and did not include other types of water bodies, such as streams and ponds. Another limitation is that most studies that used grab sampling did not report the number of samples collected, which hampered the estimation of the incidence of positive samples for each pesticide.

Conclusions

The USA, China, and Spain were the countries with the largest number of studies on pesticides in surface freshwater, and few economically less developed countries have also conducted studies, including those with high agricultural activity. Atrazine was the most evaluated and detected pesticide until 2021, and it is also among the compounds detected at higher concentrations, in addition to molinate, bentazone, and oxyfluorfen, detected in samples collected in the USA, Turkey, Spain, and Brazil. The levels of atrazine, p,p′-DDD and heptachlor were higher than the legal maximum levels for surface water in Brazil. The concentrations exceeded the ecotoxicological endpoint for at least 11 pesticides, including atrazine, cypermethrin, and chlorpyrifos.

Regulations that establish maximum concentration limits for pesticides in surface freshwater are limited in the world, and they were only identified in Brazil. Therefore, the results of this review can be used in planning monitoring of surface freshwater quality, at regional and global levels, and for implementing or updating regulations on the subject, which are essential for the protection of aquatic ecosystems.

Future studies in this area should include the use of landscape ecology tools to understand the dynamics that occur in the watershed and the flow of polluting sources to water bodies, thus identifying priority areas for water monitoring, including those for water intake for human consumption.

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Author contribution EPA and ECOF designed the study and reviewed the articles included in the review. EPA wrote the first draft of the manuscript, which was reviewed by ECOF and EDC. All authors approved the final version of the article.

Declarations

Conflict of interest The authors declare no competing interests.

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