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Organochlorine and pyrethroid residue in fish and sediment of Lake Singkarak, a tropical deep lake

Aiman Ibrahim[1]*, Muhamad Suhaemi Syawal[1], Asep Nugraha Ardiwinata[3], Sugiarti[1], Moh. Awaludin Adam[2], Wathri Fitrada[4], Riky Kurniawan[1]

[1]Research Center for Limnology and Water Resources, Nasional Research and Innovation Agency (BRIN), Bogor, West Java, 16911, Indonesia
[2]Research Center for Marine and Land Bioindustry, Nasional Research and Innovation Agency (BRIN), Teluk Kodek, North Lombok, 83352, Indonesia
[3]Research Center for Horticultural and Estate Crops, Nasional Research and Innovation Agency (BRIN), Bogor, West Java 16911, Indonesia
[4]Department of Environmental Engineering, Sekolah Tinggi Teknologi Industri Padang, Padang City, 25171, Indonesia

*Corresponding author's e-mail: aima001@brin.go.id

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Abstract: Agricultural activities still involve the use of synthetic pesticides to support the increase of their products. On the other hand, the use of synthetic pesticides such as organochlorines and pyrethroids may contribute to the decline of aquatic ecosystem health due to the accumulation of their residues in sediments and organisms. The current study aimed to assess the levels of organochlorine and pyrethroids pesticide residue in endemic fish and sediment from Lake Singkarak. Bilih fish and sediment samples were taken in June 2021 at ten (10) sampling sites in Lake Singkarak. The samples were extracted and analyzed by gas chromatography. Seven organochlorine compounds were measured, including aldrin, endrin, dieldrin, DDT, heptachlor, lindan, and endosulfan. Meanwhile, three compounds chosen from the pyrethroid group, cypermethrin, permethrin, and α-cypermethrin, were also measured. Four organochlorine compounds, aldrin, dieldrin, DDT, and endrin, were detected in bilih fish in three different sites. Dieldrin had the highest concentration at nd-0.007 mg/kg, followed by DDT, endrin, and aldrin. Meanwhile, in the sediments, no organochlorine compounds were detected from all observed sites. Pyrethroid compounds were detected in bilih fish at six sites. The compound with the highest concentration was permethrin (nd-0.02 mg/kg), followed by cypermethrin and α-cypermethrin. The surface sediment from three sites contained two pyrethroid residues, permethrin and α-cypermethrin, at nd-0.002 and nd-0.001 mg/kg, respectively. Our findings show that the residual levels of organochlorine and pyrethroid in bilih fish still meet the standards set by the Codex Alimentarius Commission (CAC). Nevertheless, Bilih fish accumulate more pesticide than surface sediment, so it is essential to be aware of their potential accumulation in the human body as the final consumer. Restriction on synthetic pesticide application is necessary to reduce its residue input into the lake waters for ecological and human health.

Keywords: Bilih fish, Lake Singkarak, organochlorine, pyrethroid, sediment

1. Introduction

Farmers still use synthetic pesticides to eradicate pests and diseases in crops quickly and practically. However, its increasingly intensive use in agricultural or plantation areas can cause problems on land and aquatic systems. Generally, synthetic pesticides are toxic. Therefore, they become a potential source of pollution for the aquatic environment

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(Taufik, 2011). Pesticide residues can degrade water quality and accumulate in sediments and aquatic organisms (Lushchak, 2018; Shah & Parveen, 2023).

Organochlorine is a synthetic pesticide that began to be used worldwide in the 1950s to improve agricultural products. Although banned several decades ago, organochlorine residues are still detectable in soil, water, and agricultural commodities (Ardiwinata *et al.*, 2020). To date, some farmers still use organochlorine obtained through illegal routes (Egbe *et al.*, 2021). As a chemical with high environmental persistence, organochlorine has low solubility in water, high lipophilicity, and a low degradation rate (Jayaraj *et al.*, 2016 or 2017). Organochlorine properties cause it to have a short residence time in the water, as it can be quickly adsorbed to suspended materials, sediments, and organisms (Fernández-Bringas, 2008). Therefore, organochlorine also contributes to bioaccumulation in the food chain (Sharma *et al.*, 2009).

Pesticides in the form of pyrethroids have been widely used since the 1970s as an alternative to organochlorine (Yang *et al.*, 2020). In contrast to organochlorines, pyrethroids are characterized by their low persistence, high degradability, and low toxicity to mammals (Costa, 2015). However, it is highly toxic to fish and non-invertebrate targets (Li *et al.*, 2017). Like organochlorines, pyrethroids are also highly hydrophobic (Wang *et al.*, 2023). This characteristic allows them to bind to sediment particles easily, which can potentially be a source of secondary contaminants if released into the water column (Li *et al.*, 2014).

Lake Singkarak is the second largest lake on the island of Sumatera, with an area reaching 11,220 ha and a maximum depth of 270 m (Wils *et al.*, 2021; Syawal *et al.*, 2023). Locals use the lake water for various purposes. However, the agricultural activities around the lake cannot be separated from using insecticides to protect plants from pests. Pesticides from the pyrethroid group are also used in the lake to make it easier to harvest shrimp. Pesticide residue can potentially reduce the quality of lake water resources, including biotic components, such as the bilih fish, an endemic fish with high economic value (Triharyuni *et al.*, 2022). However, information regarding the occurrence and pesticide pollutant levels in tropical lakes is still limited, especially studies involving endemic biota as a high-value economic food source. This study aimed to determine the levels of organochlorine and pyrethroid pesticide residues in bilih fish and sediments in Lake Singkarak.

2. Materials and Methods

2.1 Study Area and Sample Collections

The study was conducted in June 2021 at 10 sampling sites, including Batu Taba, Sumpur, Guguk Malalo, Tikalak, Ombilin, Sumani, Saniang Baka, Muaro Pingai, Paninggahan, and Tanjung Mutuih (Figure 1). The sampling location was determined by considering several factors such as water resources, water use, land use, irrigation system, type of crops, various pesticide use, and the agricultural area. As much as 50-100 g of bilih fish samples were taken from each study site. They were wrapped in aluminium foil and immediately cleaned. 500 g of sediment samples were taken from each location using a shovel or grab sampler. In the laboratory, the fish and sediment samples were stored at -25 °C and 4°C, respectively, until the extraction process was conducted.

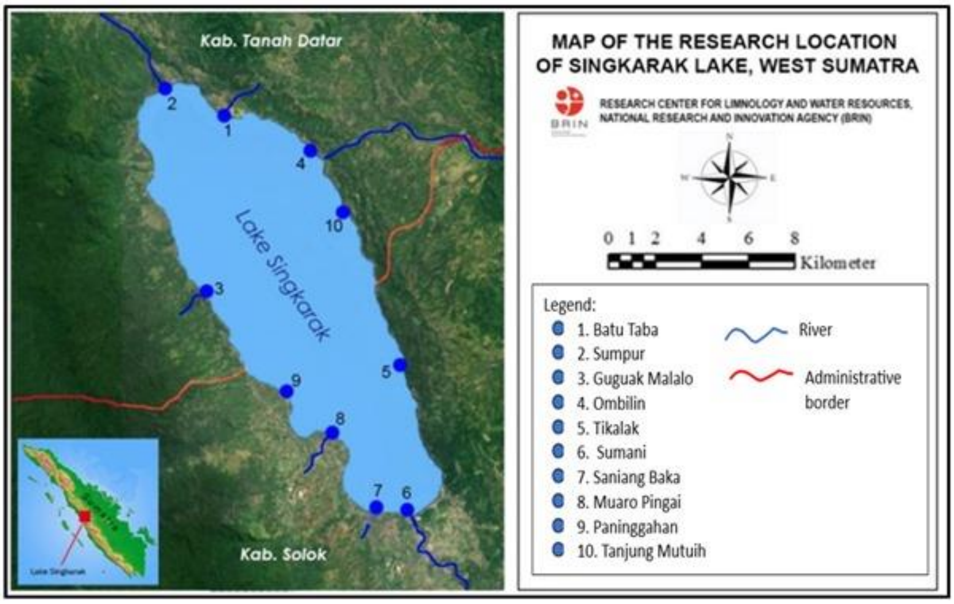

Figure 1. Map of sampling locations in Lake Singkarak

2.2 Sample Extraction-and Analysis

Sample extraction and analysis were carried out at the Laboratory of Agrochemical Material Residues in Bogor Regency. The procedure to extract sediment and bilih fish samples is shown in Figure 2 and Figure 3. The extraction was the step before the sample was measured in Gas Chromatography.

For chromatographic analysis, 1 µL of the solution sample was injected into the GC injector in the 450-Gas Chromatograph to determine the active ingredients of the pesticides classified as organochlorine. The temperature of the injector and detector were set at 300°C and 250°C, respectively. The initial temperature of the column was first set to 100°C for three minutes, then 250°C for 10 minutes (rate of 20°C/min), and then raised to 260°C for 20 minutes (rate of 2°C/min). The concentration of pesticide residues in the sample was calculated using the formula (Ardiwinata, 1999) as follows.

$$\text{Residue (mg/kg)} = (Ac \times Vis \times Cs \times Vfc) / (As \times Vic \times B \times R)$$

where:

Ac = Sample area (mm)
As = Standard area (mm)
Vic = Injection volume sample (µL)
Vis = Injection volume standard (µL)
Cs = Standard concentration (µg/mL)
B = Initial weight of sample (g)
Vfc = Sample final volume (mL)
R = Recovery (%)

Figure 2. Flowchart showing the extraction procedure of sediment samples for pesticide residue analysis (Rahmawati *et al.*, 2017; Oginawati *et al.*, 2021)

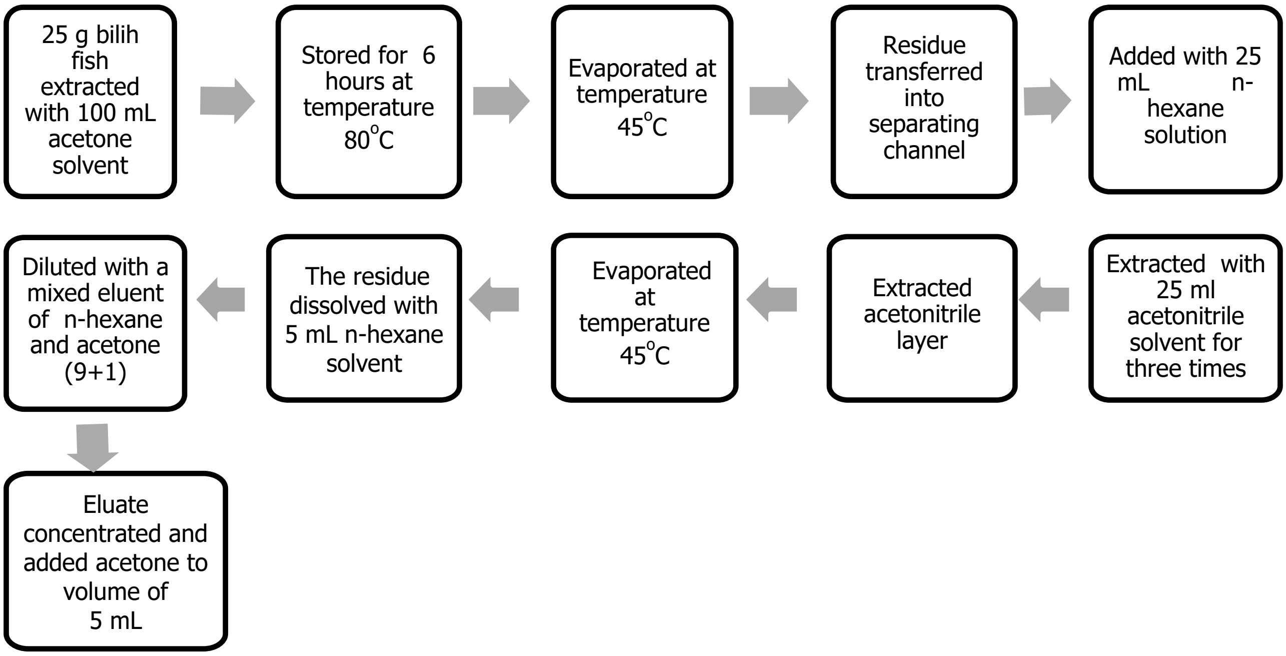

Figure 3. Flowchart showing the extraction procedure of fish samples for pesticide residue analysis (modified from Kanazawa, 1981)

3. Results and discussion

3.1. Organochlorine and Pyrethroid Residue of Bilih Fish

The types of organochlorine residues detected in bilih fish in Lake Singkarak during this study are shown in Table 1. Only four types of organochlorines were detected in fish, including aldrin, dieldrin, DDT, and endrin. Aldrin was only found in Batu Taba at 0.002 mg/kg, while endrin was only found in Sumpur at 0.005 mg/kg. Dieldrin and DDT were detected in bilih fish at the Tanjung Mutuih site at 0.007 mg/kg and 0.005 mg/kg, respectively. The three types of organochlorines (heptachlor, lindan, and endosulfan) were not detected in all observed sites. Meanwhile, at seven other sites, no organochlorine was detected.

The discovery of four types of organochlorines at three sites is thought to be caused by their use in the past and their high persistence in the environment. Their uncontrolled use also affects the presence of organochlorine, especially in Tanjung Mutuih sites where rice fields are found with channels (canals) that flow directly into lake waters. The presence of dieldrin in bilih fish in Tanjung Mutuih is thought to be due to the breakdown of aldrin compounds by bilih fish tissues. In addition, detecting aldrin in fish indicates the recent use of pesticides that contain this active compound (Mensah *et al.*, 2021).

This study found higher organochlorine levels in bilih fish than in the surface water of Lake Singkarak (Ibrahim *et al.*, 2022). In bilih fish, organochlorine levels were also higher than in the sediment. These high organochlorine levels are probably related to their low solubility in water and high lipophilic properties (Kafilzadeh, 2015). Taufik (2011) also reported that pesticide levels in pond fish are higher than in water and sediments. The lipophilic nature of organochlorines allows it to bind to the fatty tissue in fish easily.

The organochlorine levels in bilih fish in this study are generally smaller when compared to levels in tilapia and catfish in Lake Geriyo, Nigeria (Shinggu *et al.*, 2015). This rate is also still below the Maximum Residue Limit (MRL) based on the Codex Alimentarius Commission (CAC) 39th Session (2016). However, the dieldrin recorded in this study exceeds the levels detected in Lake Edko of 0.0004 mg/kg (Abbassy *et al.*, 2021).

Table 1. The concentration of organochlorine residue (mg/kg) in bilih fish from Lake Singkarak

Station	**Aldrin**	**Dieldrin**	**DDT**	**Endrin**	**Heptachlor**	**Lindan**	**Endosulfan**
Batu Taba	0.002	nd	nd	nd	nd	nd	nd
Sumpur	nd	nd	nd	0.005	nd	nd	nd
Guguak Malalo	nd	nd	nd	nd	nd	nd	nd
Ombilin	nd	nd	nd	nd	nd	nd	nd
Tikalak	nd	nd	nd	nd	nd	nd	nd
Sumani	nd	nd	nd	nd	nd	nd	nd
Saniang Baka	nd	nd	nd	nd	nd	nd	nd
Muaro Pingai	nd	nd	nd	nd	nd	nd	nd
Paninggahan	nd	nd	nd	nd	nd	nd	nd
Tanjung Mutuih	nd	0.007	0.005	nd	nd	nd	nd
LoD	0.001	0.001	0.001	0.001	0.019	0.015	0.017
MRL	0.2	0.2	5	-	0.2	0.01	0.2

Note: nd : not detected or below detection limit; LoD: Limit of Detection; MRL: Maximum Residue Limit

Three compounds from the pyrethroid group have been analyzed in bilih fish at ten sites in Lake Singkarak. Table 2 shows the results of the analysis of the pyrethroids accumulated in bilih fish. Permethrin is the dominant pyrethroid accumulated in bilih fish, followed by α-cypermethrin and cypermethrin. The permethrin level found in this study was nd-0.02 mg/kg. Meanwhile, the concentrations of cypermethrin and α-cypermethrin detected were nd-0.008 mg/kg and nd-0.007 mg/kg, respectively. Permethrin was found in three sites, with its highest concentration at Muaro Pingai. The highest levels of cypermethrin and α-cypermethrin were found at Tanjung Mutuih and Sumpur sites, respectively. The three compounds of pyrethroids analyzed in this study were not detected in four other sites.

The levels of pyrethroids measured in bilih fish was relatively higher than in sediments. The higher lipid content in fish leads to an increased potential absorption of hydrophobic compounds (Spacie & Hamelink, 1982). As mobile organisms, fish can be exposed to these compounds in other parts of the hydrological system. Similarly, the accumulation rate of pesticide residues increases with temperature (Charles *et al.*, 2000). Riaz *et al.* (2018) also reported that higher pyrethroids levels were detected in fish than in the Chenab River sediment. Similar results were seen in Mahboob *et al.* (2015) study on the Ravi River. Riaz *et al.* (2018) explained that fish are highly susceptible organisms to pesticide accumulation due to their lipophilic properties. Pesticide contamination in aquatic organisms is caused by direct uptake from water through gills or skin, via uptake of suspended particles, and even contaminated food (van der Oost *et al.*, 2003).

The pyrethroid (cypermethrin) levels in bilih fish in this study were lower than the levels detected in fish in the Red River Basin, Vietnam, at 0.109-0.802 mg/kg (Pham *et al.,* 2011). Mahboob *et al.* (2015) findings also showed more significant levels of cypermethrin in the Ravi River. Conversely, wild fish in the Iberian River Basin in Spain had a lower accumulation of cypermethrin due to the use of local insecticides (Corcellas *et al.,* 2015). Xie *et al.* (2022) reported slightly higher levels of permethrin and slightly lower levels of cypermethrin in fish compared to this study's findings. This difference may be correlated with the increase in population and pesticide use.

Regarding the CAC 39th Session (2016), the three types of pyrethroids contained in bilih fish in Lake Singkarak still meet the required standard values. Nevertheless, we must be mindful of the increased use of pesticides in Lake Singkarak as their potential accumulation in bilih fish can have lethal and sublethal effects. Lethal effects can be in the form of fish death, while the sublethal effect can be physiological and biochemical changes (Taufik, 2011).

Table 2. The concentration of pyrethroid residue (mg/kg) in bilih fish from Lake Singkarak

Station	Permethrin	Cypermethrin	α-Cypermethrin
Batu Taba	nd	0.003	0.004
Sumpur	nd	nd	0.007
Guguak Malalo	nd	nd	nd
Ombilin	nd	nd	nd
Tikalak	0.0040	0.0030	nd
Sumani	0.0020	0.0020	nd
Saniang Baka	nd	nd	nd
Muaro Pingai	0.02	nd	nd
Paninggahan	nd	nd	nd
Tanjung Mutuih	nd	0.008	0.0060
LoD	0.0053	0.0015	0.0010
MRL	1	2	2

Note: nd: not detected or below detection limit; LoD: Limit of Detection;
MRL: Maximum Residue Limit

3.2. Organochlorine and Pyrethroid Residue of Surface Sediment

Organochlorine residues were not detected in the sediments of all sampling sites of Lake Singkarak (Table 3), DDT metabolites could be detected due to the use of DDT in the past. Only DDT metabolites were found in sediments, whereas concentrations of organochlorine pesticides (heptachlor, dieldrin, DDT) were detected below detection limits in several different lakes (Dvoršćak *et al.,* 2019; Mergia *et al.,* 2022; Hu & Tao, 2023). Meanwhile, the sedimentary concentrations of organochlorine pesticides ranged from 0.00 to 14.83 ng/g dry weight in the Eldrin-dominated Iznik Lake (Aydın & Albay, 2022). Organochlorine pesticide residues in the sediment were also found in the Tono Reservoir with average levels of 0.09, 0.04, and 0.047 µg/g for aldrin, DDE, and DDD (Akoto *et al.*, 2016). The study also showed higher levels of aldrin in the sediment compared to fish and lower DDE and DDD levels in the sediment. Other studies showed organochlorines were found to be higher (the highest pesticide aldrin was 2-1438 µg/L) in sediments compared to water, molluscs, and fish (Barlas *et al.*, 2006; GA, 2016; Mensah *et al.*, 2021; Oginawati *et al.,* 2022).

Table 3. Concentration of organochlorine residue (mg/kg) in sediment from Lake Singkarak

Station	Aldrin	Dieldrin	DDT	Endrin	Heptachlor	Lindan	Endosulfan
Batu Taba	nd	nd	nd	nd	nd	nd	nd
Sumpur	nd	nd	nd	nd	nd	nd	nd
Guguak Malalo	nd	nd	nd	nd	nd	nd	nd
Ombilin	nd	nd	nd	nd	nd	nd	nd
Tikalak	nd	nd	nd	nd	nd	nd	nd
Sumani	nd	nd	nd	nd	nd	nd	nd
Saniang Baka	nd	nd	nd	nd	nd	nd	nd
Muaro Pingai	nd	nd	nd	nd	nd	nd	nd
Paninggahan	nd	nd	nd	nd	nd	nd	nd
Tanjung Mutuih	nd	nd	nd	nd	nd	nd	nd
LoD	0.001	0.001	0.001	0.001	0.019	0.015	0.017
TEL (ug/kg)	-	2.850	-	2.670	0.600	0.940	-

Note: nd: not detected or below detection limit; LoD: Limit of Detection;
TEL: Threshold-Effects Level

Table 4. The concentration of pyrethroid residue (mg/kg) in sediment from Lake Singkarak

Station	**Permethrin**	**Cypermethrin**	**α-Cypermethrin**
Batu Taba	nd	nd	nd
Sumpur	nd	nd	nd
Guguak Malalo	0.0020	nd	nd
Ombilin	nd	nd	nd
Tikalak	nd	nd	nd
Sumani	nd	nd	nd
Saniang Baka	nd	nd	nd
Muaro Pingai	0.0010	nd	nd
Paninggahan	nd	nd	0.0010
Tanjung Mutuih	nd	nd	nd
LoD	0.0053	0.0015	0.0010
TEL	-	-	-

Note: nd: not detected or below detection limit; LoD: Limit of Detection;
TEL: Threshold-Effects Level

We detected organochlorine compounds in sediments that can be assessed based on the freshwater sediment quality assessment guide. This is done to evaluate the possible ecological risks posed by pesticides. The organochlorine levels obtained can also be evaluated based on the Threshold-Effects Level (TEL), which indicates the level below which adverse biological effects are expected to occur rarely (Smith *et al.,* 1996).

Pesticide residues in surface water and sediments are critical because of their negative impacts on aquatic ecosystems and their implications for drinking water sources. Pesticides can accumulate in sediments through the indiscriminate use of pesticides that leads to their entry to the bottom sediments, where they are absorbed in the sediment's particles and, thus, may become the consistent source of aquatic pollution (Shah & Parveen, 2023).

Pyrethroids were analyzed in sediments to determine their possible contamination. The pyrethroid residues that were studied for their content in the Singkarak Lake sediments were permethrin, cypermethrin, and α-cypermethrin (Table 4). Permethrin was found at two sites, namely Guguak Malalo and Muara Pingai, with a concentration range of 0.001-0.002 mg/kg. Another type of pyrethroid found was α-cypermethrin at Paninggahan with a concentration of 0.001 mg/kg. Around the Guguak Malalo, Muara Pingai, and Paninggahan areas are agricultural areas. In that area, there are many rice fields with rice as the main crop, besides other agricultural products such as chilies, beans, corn, and shallots. It is possible that the sedimentary pyrethroid pesticides originated from pesticides in agricultural areas, were carried into the lake water column and accumulated in the sediments. Pesticides enter water bodies through runoff from agricultural areas that use many pesticides (Effendi, 2003).

Sediments at Sumpur, Batu Taba, Ombilin, Tikalak, Tanjung Mutuih, Sumani, and Saniang Baka sites were not detected with pyrethroid residue. Ombilin, as an outlet area of the lake quite distant from agricultural areas, resulted in the absence of residues in the sediment. While the other six locations, despite being agricultural areas, did not detect the presence of residues in the sediment. This absence of residues in the sediment is thought to be related to the pattern of pyrethroid use and the half-life of pyrethroid types in the sediment (Li *et al*., 2017). Additionally, the surface area and organic content of sediment can influence the level of residue adsorption (Mensah *et al.*, 2021).

Compared to our findings, the pyrethroids levels in sediments in Lake Weija tend to be higher, where the levels of permethrin and cypermethrin were nd-0.0066 mg/kg and nd-0.0032 mg/kg, respectively (Afful *et al*., 2013). Lake Weija is located in Ghana, where its condition is similar to Lake Singkarak, where agriculture is one of the primary land uses surrounding the lake. Meanwhile, this study's α-cypermethrin levels tend to be the same as the findings reported by Merga (2021) of <0.00071-0.00197 mg/kg in sediments in Lake Ziway, Ethiopia. Moreover, Lake Ziway got pressure from agricultural activities, besides its urbanization activities. Pesticides released from large- and small-scale agricultural activities are posing ecological risks to biotic components in Lake Ziway. Pyrethroids in sediment in this study align with Li et al. (2017) who state that permethrin concentrations in the sediment will always be high. Due to pyrethroids' high hydrophobic properties, in aquatic environments, they can be absorbed into particles, deposited into the sediment, and accumulate in the body of organisms.

The presence of residual pyrethroid pesticide in bilih fish and sediment in this study confirms the use of synthetic pesticides from that class in the area around Lake Singkarak. Similarly, the detection of organochlorines such as aldrin in bilih fish may indicate their current use through illegal routes despite being banned since 1970. Residual pesticide compounds in the lake waters, with increasing types and concentrations, will affect the health of aquatic biota and can also have long-term implications for human health. Proper use and restriction of synthetic pesticide use can contribute to minimizing pollutant inputs into the lake for the sake of a healthy lake ecosystem.

4. Conclusion

This study reveals the presence of organochlorine and pyrethroid pesticide residues in Lake Singkarak, with their levels higher in bilih fish compared to sediment. Our findings enrich information on the presence of pollutant compounds in tropical lake ecosystems, including in endemic biota that serve as a high-value economic food source. Future study is needed to measure the levels of other pesticide classes in various biotic and abiotic components in Lake Singkarak during the dry and rainy seasons. Moreover, the human health and ecological risks of synthetic pesticide application are also essential to investigate. This investigation is crucial to support efforts in the protection and management of tropical lake ecosystems amidst increasing anthropogenic activities.

Data availability statement

Data used in this study are available from the corresponding author upon request.

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Conflict of interests

The authors declare no conflict of interest.

Contributor statement

AI: Conceptualization, Investigation, Writing - original draft. **MSS**: Investigation, Methodology, Project Administration. **ANA**: Resources, Methodology, Formal Analysis. **S**: Writing - original draft, Writing - review & editing. **MAA**: Writing - original draft. **WF**: Writing - original draft. **RK**: Resources

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