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Abstract: Dieldrin is legacy organochlorine insecticide, which was listed in the Stockholm Convention because of its persistence, bioaccumulation and toxicity. But it is still present in the environment and in organisms two decades after its ban. The current criteria used to risk assessment in China are based on acute toxicity data in water column without considering the bioavailability and bioaccumulation, which accordingly lead to under-protection of aquatic organisms and wildlife. In this study, the water quality criteria (WQC) for dieldrin were derived in combination of tissue-based toxicity data and bioaccumulation factor (BAF) to better protect aquatic ecosystems. The dieldrin residue data in surface water in China were obtained by literature review and the ecological risk was assessed using quotient method. Combined with the BAF estimated by the model of 58884.37 L/kg, the WQC were calculated to be 3.07 and 1.5 ng/L to protect aquatic life and aquatic-dependent wildlife, respectively. The results of the risk assessment revealed the potential high risk posed by dieldrin bioaccumulation. This study provides a scientific guidance for the determination of water quality standard for dieldrin and ensure the risk management of aquatic environment in China.

Keywords: Dieldrin; Water quality criteria; Bioaccumulation; Ecological risk; Wildlife criteria

1. Introduction

Dieldrin(C12H8Cl6O) is both a synthetic organochlorine pesticides (OCPs) classified as a chlorinated cyclodiene compound that was primarily used on cotton, corn, and citrus crops, and the environmental and metabolic product of the related compound aldrin [1]. It is lipophilic and persistent in soil with half-life up to five years. Dieldrin possesses high potential for bioaccumulation and readily bioaccumulates in terrestrial and aquatic organisms [2–5], tending to bio-magnify in the food chain.

Endpoints of dieldrin have been clarified, including carcinogenicity, endocrine disruptions, reproductive/development effects and neurotoxicity [6–9]. With causing adverse effects on humans and the ecosystem, dieldrin's persistence in the environment became a major concern, prompting its inclusion as one of the 12 initial POPs under the Stockholm Convention, which imposed a ban on its manufacturing, usage, and commerce worldwide. While dieldrin has been widely detected in the environment in studies related to pesticides [10–14]. Its residues have also been monitored in human milk [15–17] and wildlife such as raptors globally [18], whales and polar bear in Arctic [19]. In China, dieldrin was never produced on an industrial scale and was not used as a pesticide in agriculture [20]. However, previous researches have unequivocally demonstrated its presence in Chinese environmental media such as soil, air, water as well as in wild animals [21–25], indicating long-range transport and latent illegal use of dieldrin. Meanwhile, research which conducted in two sediment cores from Lake Fuxain, the largest deep freshwater lake in China to track the historical pollution

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records, revealed that as one of the predominant OCPs dieldrin might pose a potential threat to exposed organisms [26].

For the protection of aquatic life, CCME developed Canadian interim sediment quality guidelines and probable effect levels for dieldrin, which can be used to assess the likelihood that exposure to dieldrin in sediments will have detrimental biological effects [27]. The procedures to derive dieldrin concentrations in sediment that are protective of benthic organisms were described in an equilibrium partitioning sediment benchmark document [28]. The dieldrin wildlife value for waters within the Great Lakes Basin was also determined, along with the calculations and avian values [29]. There are limited specific guidelines, standards or criteria for the risk that dieldrin might pose to aquatic ecosystem in China. Yang et al have deduced recommended values of the criteria maximum concentrations of dieldrin for domestic aquatic organisms using acute toxicity data [30]. Dieldrin in aquatic environments generally occurs at low concentrations, however, and might result in high accumulation within living organisms. Accordingly, the traditional water quality criteria (WQC) derived from water-based toxicity that does not consider bioaccumulation factors (BAFs) makes it challenging to provide comprehensive protection for aquatic life. Therefore, it is needful to adopt a tissue-based method for deriving aquatic life criteria because multiple routes of exposure (e.g., diet, sediment, water) may impact the bioavailability of substances [31]. Moreover, the basis for establishing water quality standards in China did not consider trophic magnification of chemical substances and effects on higher trophic levels. Wildlife criteria have been urgently needed to assessing risks posed by PBTs to birds or mammals in Chinese aquatic systems and to support national policy-making decisions.

The aim of this study is to determine dieldrin WQC to protect aquatic organisms and aquatic-dependent wildlife using tissue-based toxicity data to better manage the environment. This study gives new insight into the ecological risk of dieldrin and provides a scientific foundation for enhancing WQC determination systems in China through serving as a reference for the derivation of bio-accumulative substances.

2. Methods

2.1 Toxicity Data Screening

The sources of tissue-based toxicity of aquatic organisms for dieldrin are the Environmental Residue Effects Database, ECO-TOX database and published literature. For wildlife, tissue-based toxicity data were obtained from literature which compiled by the USEPA. Internationally recognized, indigenous, and introduced species, including freshwater invertebrates, vertebrates, and wildlife were selected. The region of species was mainly defined according to the Global Biodiversity Information Facility (https://www.gbif.org/). Tissue-based values need to be developed based on ecologically relevant endpoints that protect populations [32]. Thus, the endpoints of the effect include morality, developmental growth, reproduction, behavior and accumulation. And the no-observed effect concentration (NOEC) was selected as the test endpoint to develop chronic criteria. All dieldrin residue data are obtained from whole-body measurements.

2.2 Derivation of Tissue-Based Criteria

The term "tissue-based criteria (TBC)" refers to criteria generated from toxicity data reported as concentrations in tissues of the target organisms or their diet (in wildlife) [33]. Tissue-based toxicity metrics are developed using the same methodology as that for external concentration-based toxicity tests [32]. On the premise of sufficient valid toxicity data, the species sensitivity distribution (SSD) method is selected to derive a baseline or toxicity reference value. This probability distribution function assumes that species sensitive can be characterized by statistical distributions. Designating the most sensitive species, a hazard concentration, the 5-th percentile of SSD which protects 95% of organisms is used to set the criteria.

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In the case of aquatic-dependent wildlife, toxicity data of avian or mammalian species expressed as tissue concentrations per day are used to generate the HC_5, represent the tolerable daily intake (TDI). While in the derivation of WQC, tissue concentrations need to manifest as concentrations in the aquatic diet. The tissue reference value (TRV) for aquatic-dependent wildlife is calculated using the TDI in conjunction with daily food ingestion rates (FI) and body weights (BW) of wildlife species [34], as shown in Eq. (1).

$$TRV = \frac{TDI}{FI:BW} \quad (1)$$

where TDI is derived using the SSD method using tissue–based toxicity data (mg/kg), and FI:BW is the ratio of food ingestion rates to body weights of representative wildlife species in China, which were selected from literature.

2.3 Derivation of WQC for Dieldrin

In order to translate the tissue criterion to corresponding water concentrations, it would be divided by the relevant bioaccumulation factors (BAFs) determined for each representative species [33]. Bioaccumulation can be viewed as the combination of bioconcentration and food uptake due to it occurs when chemicals accumulate in aquatic organisms through multiple exposure routes, including dietary, respiratory, and dermal absorption [35]. The BAF is the ratio of the concentration of the chemical in the organism to that in the environment. Fish have among the highest bioaccumulation rates for dieldrin and higher trophic levels in aquatic ecosystems. From there, empirical model based on field-derived BAFs is used to assess bioaccumulation potential and to translate tissue-based criteria to water concentrations [31]. Herein, a generic BAF model for fish development by Arnot and Gobas [36] was used to estimate the BAF of dieldrin in absence of site-specific measurements. The model is a quantitative structure-activity relationship (QSAR), with input parameters consisting only of the octanol-water partition coefficient (Kow) of the chemical and, if available, the metabolic transformation rate constant. The model provides predictions of BAF for fish species in lower, middle and upper trophic levels of aquatic food webs and can be used to predict dietary concentrations for higher trophic level predators (e.g., birds and mammals) from fish in their diet. Then with the estimates of BAF, TBC or TRV were converted to WQC expressed as the concentration in water using Eq. (2).

$$WQC_{aqua}(WQC_{wild}) = \frac{TBC(TRV)}{BAF} \quad (2)$$

where WQC_aqua and WQC_wild are for the protection of aquatic life and aquatic-dependent wildlife, respectively; TBC is derived from tissue–based toxicity data through the SSD method (mg/kg), TRV is calculated using Eq. (1), and BAF is the bioaccumulation factor (L/kg).

2.4 Ecological Risk Assessment

The available literature data on concentrations of dieldrin in China were used for environmental risk screening via quotient method. Risk quotients (RQs) are the ratio of points estimate of exposure and toxicity. The exposure refers to actual monitored or model-estimated environment concentration. The toxicity refers to an effect level such as Predicted No Effect Concentration (PNEC), which is the ecological risk threshold predicted to have no adverse effects on organisms. RQs were calculated as shown in Eq. (3),

$$RQ = C/WQC \quad (3)$$

where C is the measured environmental concentration collected from literature; WQC for dieldrin is derived from Eq. (2), which express the ecological risk threshold. Then compared to EPA's levels of concern (LOCs). According to EPA's risk presumptions, the value of LOC is 1 for chronic risk. A resulting RQ below the value of 1 indicates no chronic risk concern.

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2.5 Data Analysis

The current study employs the R package "ssdtools" to generate the SSD [37] using R version 4.1.0 [38]. We utilized the "ssd_fit_dists" function using maximum likelihood to fit the distribution and the "ssd_gof" function to assess the goodness of fit. With bootstrapping to get confidence intervals, the "ssd_hc" function was used to estimate the model-averaged 5% concentration; that is, the HC_5 value. Together with the original data, the predictions were plotted using the "ssd_plot" function. The model BAF-QSAR v1.1 [36] coded in a Microsoft Excel workbook was used to obtain the BAF. The data screening and derivation of WQC were also using Microsoft Excel 2019 for Windows.

3. Results and Discussion

3.1 WQC for Protection of Aquatic Life

Table 1 presents toxicity data obtained from database for dieldrin of aquatic life. The selected species, including 10 fishes, 1 crustacean and 1 mollusk, met the minimum data requirement. As shown in Figure 1, the SSD curve with confidence interval was simulated from collated data. The TBC is 0.227 mg/kg (wet weight; WW) derived from estimated model-averaged predictions by parametric bootstrapping.

It was recommended to translate tissue criteria into water concentrations and develop site-specific criteria using paired water concentration and fish tissue data from a representative set of species. While data of dieldrin in environment and biota are insufficient, model approach was used to obtain the BAF. Both empirical models that leverage field-derived BAFs and mechanistic models can be used to assess bioaccumulation potential. Since the metabolic transformation rate constant was not available, the $\log_{10}$Kow value of 5.4 for dieldrin determined with the "slow-stirring" method [39] was used as input parameter for the BAF-QSAR model. The BAF predictions are considered generic in that they are not considered to be for a particular species of fish. The estimation of logarithm BAFs base 10 in lower, middle and upper trophic levels of fish species are 4.77, 5.09 and, 5.66 respectively. Arnot and Gobas [40] reviewed the status of bioaccumulation evaluations for organic chemicals in aquatic systems and summarized regression statistics for different organism classes before and after the confidence assessment on the reviewed data. In agreement with their empirical model, the $\log_{10}$BAF value of 4.77 was used to derive the WQC for dieldrin. The calculated BAF for fish is 58884.37 L/kg. Using Eq. (3), the calculated WQC for aquatic organisms is 3.86 ng/L.

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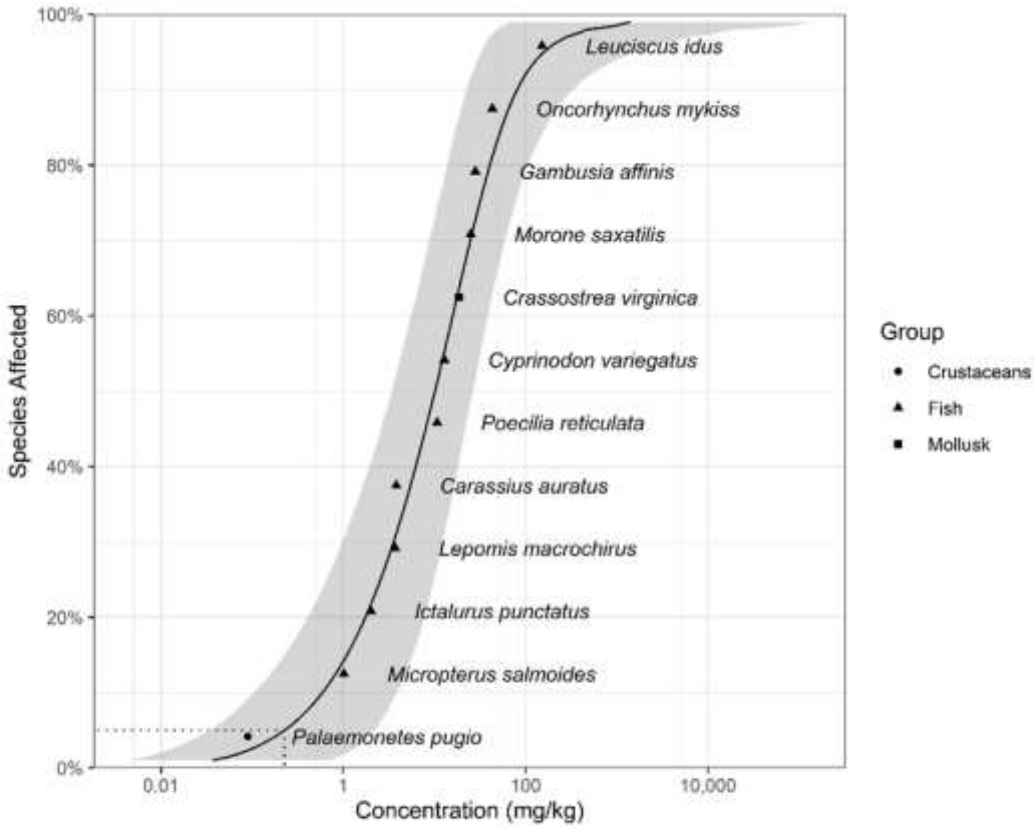

Figure 1. Species sensitivity distribution for dieldrin for aquatic organism tissue-based toxicity data.

Table 1. Tissue-based toxicity data of aquatic life for dieldrin.

Species	Common name	NOEC (mg/kg)	Taxa	Reference
Palaemonetes pugio	Grass shrimp	0.09	Crustaceans	[41]
Micropterus salmoides	Largemouth bass	1.01	Fishes	[42]
Ictalurus punctatus	Channel catfish	2	Fishes	[43]
Lepomis macrochirus	Bluegill	3.7	Fishes	[44]
Carassius auratus	Godfish	3.8	Fishes	[44]
Poecilia reticulata	Guppy	10.7	Fishes	[45]
Cyprinodon variegatus	Sheepshead minnow	12.8	Fishes	[41]
Crassostrea virginica	Eastern oyster	18.6	Mollusks	[46]
Morone saxatilis	Striped bass	25	Fishes	[47]
Gambusia affinis	Mosquito fish	28	Fishes	[48]
Oncorhynchus mykiss	Rainbow trout	43	Fishes	[49]
Leuciscus idus	Golden ide	151	Fishes	[50]

3.2 WQC for Protection Of Aquatic-Dependent Wildlife

Table 2 presents the toxicity data for dieldrin of wildlife, including 11 avians and 10 mammalians which met the minimum data requirement. As shown in Figure 2 and Figure 3, the SSD curves with confidence intervals were simulated for each taxa group from collated data. Calculated from the SSD

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curves, TDIs for avian and mammalian are 0.0379 and 0.0914 mg/kg-day, respectively. The highest FI:BW value among representative avian species in China is 0.43 [51] and the higher ratio of FI to BW among representative mammalian species in China is 0.05 [52–54]. TRVs are 0.088 and 0.1828 mg/kg WW, respectively. Using Eq. (3) and the previous calculated BAF for fish, the calculated WQC are 1.50 and 3.10 ng/L, respectively. To better protect aquatic-dependent wildlife, the smallest WQC (1.50 ng/L) was selected as the dieldrin WQC for protecting aquatic-dependent wildlife in China.

Table 2. Tissue-based toxicity data of aquatic-dependent wildlife for dieldrin.

Species	Common name	NOEC (mg/kg)	Taxa	Reference
Tyto alba	Barn owl	0.0445	Avian	[55]
Numida meleagris	Crowned guinea fowl	0.0537	Avian	[56]
Phasianus colchicus	Ring-necked pheasant	0.26	Avian	[57]
Colinus virginianus	Bobwhite quail	0.27	Avian	[58]
Streptopelia risoria	Ring dove	0.331	Avian	[59]
Coturnix japonica	Japanese quail	0.852	Avian	[60]
Phasianus colchicus	Pheasant	0.905	Avian	[61]
Coturnix coturnix	Quail	1.36	Avian	[62]
Columba livia	Homing pigeon	2	Avian	[63]
Anas platyrhynchos	Mallard	2.21	Avian	[64]
Gallus domesticus	Chicken	10	Avian	[65]
Canis familiaris	Dog	0.05	Mammalian	[66]
Mus musculus	Mouse	0.133	Mammalian	[67]
Damaliscus pygargus	Blesbuk	0.449	Mammalian	[68]
Odocoileus virginianus	White tailed deer	0.72	Mammalian	[69]
Rattus novegicus	Rat	0.81	Mammalian	[66]
Ovis aries	Sheep	0.87	Mammalian	[70]
Peromyscus leucopus	White footed mouse	1.14	Mammalian	[71]
Sus scrofa	Pig	1.20	Mammalian	[72]
Oryctolagus cuniculus	Rabbit	1.25	Mammalian	[73]
Cavia porcellus	Guinea pig	1.76	Mammalian	[74]

Considering the bioaccumulation of dieldrin via piscivorous food webs, the threshold contaminant body burden in wildlife was calculated, which is then back-calculated to an equivalent concentration in fish or water using the combination of food and chemical assimilation efficiencies and bioconcentration/ bioaccumulation factors. For wildlife criteria derived from dietary toxicity data, BAFs would be applied and appropriately weighted for each component of the aquatic diet of the representative wildlife species [33]. Using this method, the dieldrin wildlife value for waters within the Great Lakes Basin was determined to be 7.1×10^{-5} μg/L [29].

According to Protocol for the Derivation of Canadian Tissue Residue Guidelines for the Protection of Wildlife that Consume Aquatic Biota [34], reference concentrations are calculated using information on BW and FI for wildlife species as well as the TDI derived from toxicity studies, then the result can be compared to the generic tissue residue guideline developed to protect all wildlife. For substances with a high potential to bio-magnify within food chains, it is important that the guideline derived from lowest reference value be applied to the highest aquatic trophic level (e.g., level 4 fish) in order to protect predators feeding at that level

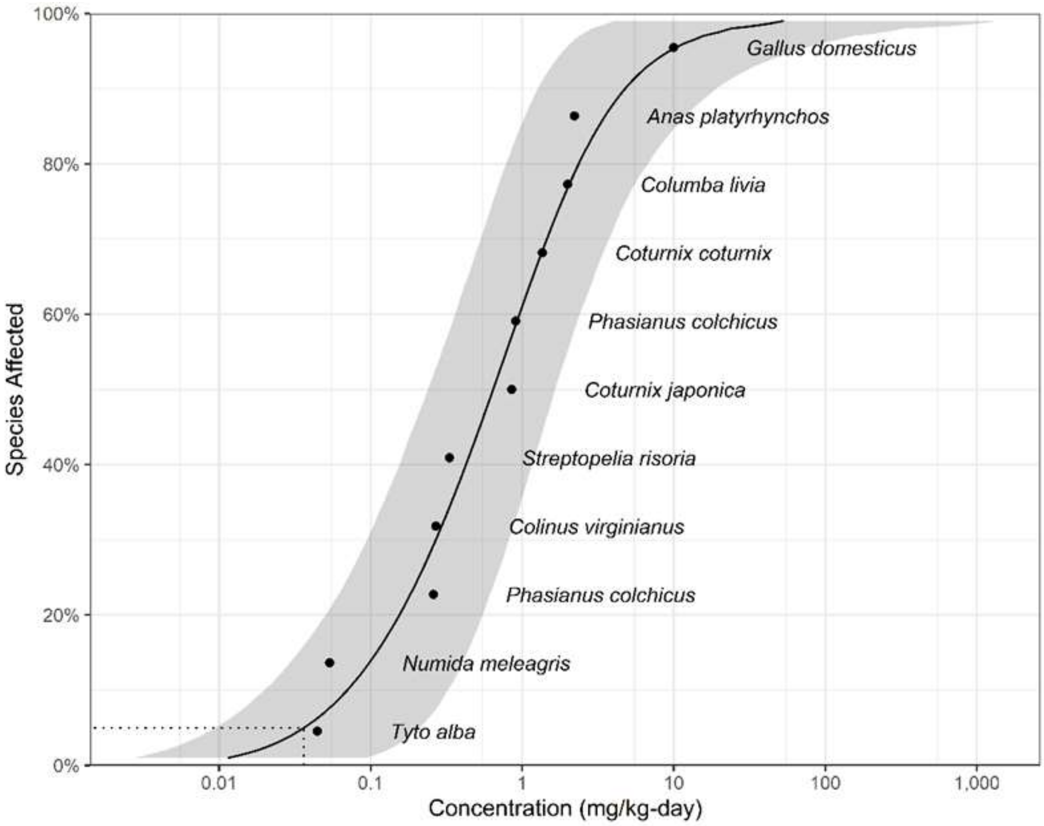

Figure 2. Species sensitivity distribution for dieldrin for avians tissue-based toxicity data.

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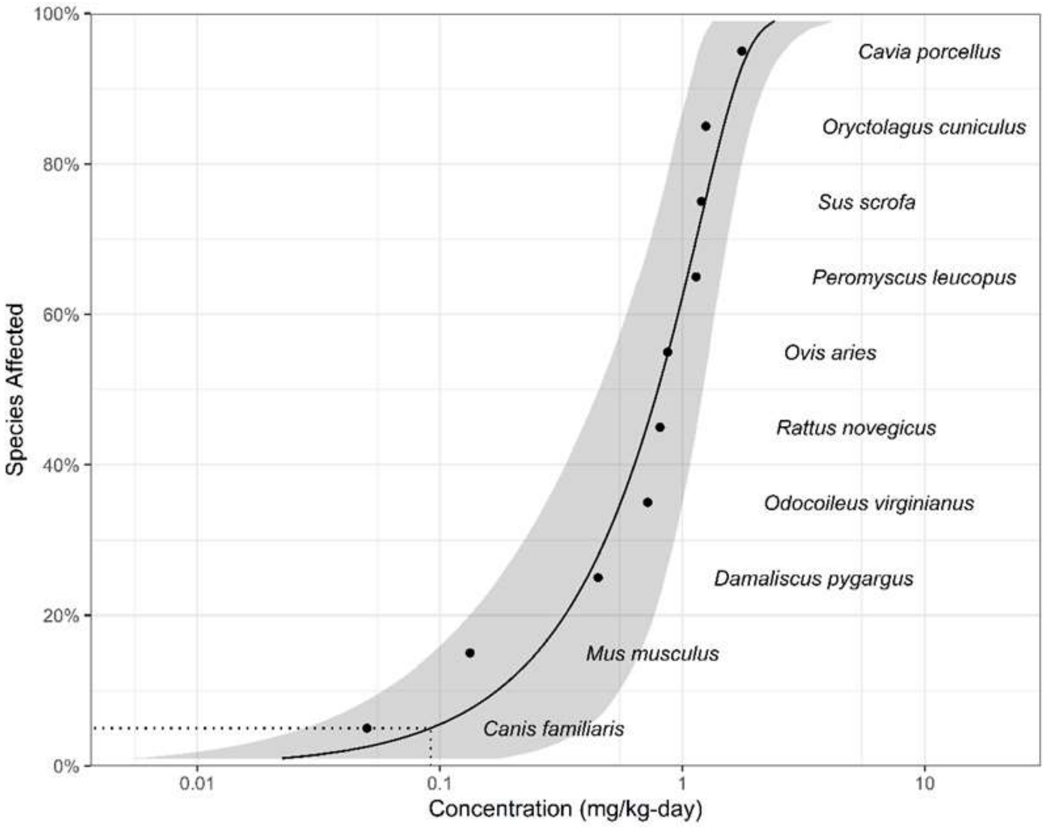

Figure 3. Species sensitivity distribution for dieldrin for mammalians tissue-based toxicity data.

Since species specific and site-specific data for dieldrin were not available in China, we selected the highest ratio of FI to BW of representative wildlife species to calculate TRV from TDI and used BAF for fish to convert TRV to water concentration. Dieldrin was found in one fish liver sample (0.07 μg/g WW) collected from aquaculture cages in coastal waters of Xiamen [75], and in various organs and tissues of Indo-Pacific humpback dolphins from the Pearl River Estuary, ranging from 0.74 to 6.8 ng/g WW [24]. Our result of WQC is much higher than the value within the Great Lakes Basin, but compared to residue records in biota, it can be effective to protect wildlife.

3.3 Risk Assessment

Studies have shown that chronic exposure to dieldrin can cause harm to aquatic organisms and wildlife [9,76]. Therefore, it is important to assess the ecological risk of dieldrin in aquatic environments. The results of presence of dieldrin in surface water in the last dozen years from the literature review and ecological risk assessment results are shown in Table 3. Samples were collected from the Yangtze River, the Qinhuai River and the Xuanwu Lake in Nanjing [23] and Shaying River Basin [77], and the mean concentrations of dieldrin were 1.31, 2.32, 4.38 and 4.6 ng/L, respectively. RQ values were calculated separately using WQC for the protection of aquatic life and wildlife. The results between 0.34 and 3.07 were observed, suggesting that dieldrin post ecological risk to both aquatic organism and wildlife in Xuanwu Lake and Shaying River Basin, potential ecological risk to avians in Qinhuai River and no chronic risk concern in Yangtze River.

Table 3. RQs estimation.

Location	Conc/ (ng/L)	RQ_aqua	RQ_wild	Sampling Time	Reference

Yangtze River, Nanjing	1.31	0.34	0.87	May to July 2016	[23]
Qinhuai River, Nanjing	2.32	0.60	1.55		
Xuanwu Lake, Nanjing	4.38	1.13	2.92		
Shaying River Basin	4.60	1.19	3.07	November 2013	[77]

The sampling approach, which the timing and site selection are unable to adequately capture the periodic occurrence of pesticides and investigate surface waters particularly susceptible to pesticide risks may contribute to inappropriate estimation of risk [78]. Since dieldrin has been banned and not used in China, its main source is environment migration. With hydrophobicity and low water solubility, dieldrin is prone to binding to organic materials [1]. Research on distribution of organochlorine pesticide pollution in Indonesia revealed that sediments showed higher organochlorine concentrations than water, mollusk, and fish [14]. Sediment is still a potential source and is rarely detected. It was demonstrated that the effect concentration in sediments can be accurately predicted by multiplying the effect concentration in water by the chemical's organic carbon partition coefficient [28]. Thus, it significative to detect dieldrin concentrations in water to better understand its ecological risk to integral aquatic ecosystem.

4. Conclusions

This study set out to determine the dieldrin WQC using the tissue-based toxicity data which have not been used previously. In this study, the estimated BAF of 58884.37 L/kg was used to derive WQC in the absence of field-based data. The tissue-based criteria obtained with screened dieldrin toxicity data and SSD method are 0.227 mg/kg (WW) of aquatic life, 0.088 and 0.1828 mg/kg-day food (WW) of avian and mammalian, respectively. Using the statistic presented above, the dieldrin WQC for the protection of aquatic organisms and wildlife in China is 3.86 and 1.5 ng/L, respectively. The risk assessment results indicate that dieldrin levels in surface water may pose a high ecological risk to aquatic organisms and wildlife, especially avians. This work contributes to provide more scientific protection for aquatic organisms and wildlife from the harm of bioaccumulation of dieldrin, supporting the environmental management and risk assessment in China. The results also enhance China's water environment criterion system, enabling the development of the "water ecological civilization". The major limitation of this study is the lack of data of dieldrin presence in surface water, further study on monitoring should be taken.

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