RESEARCH ARTICLE

Deriving water quality criteria for China for the organophosphorus pesticides dichlorvos and malathion

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Abstract

Organophosphorus pesticides are effective, cheap, and used extensively but can harm aquatic organism and human health. Here, water quality criteria (WQCs) for dichlorvos (DDVP) and malathion (MAL) were derived. Nine aquatic organisms native to China were used in toxicity tests. Published toxicity data for aquatic organisms native and non-native to China were also analyzed. DDVP and MAL WQCs were derived using (log-normal model) species sensitivity distributions. Species sensitivity distribution curves indicated native and non-native species have different sensitivities to DDVP. The sensitivities of native and non-native species to MAL were not different because non-native species data for fewer than eight genera were available, so further research is required. The results indicated that native species need to be considered when deriving WQCs. The criteria maximum concentration (CMC) and criteria continuous concentration (CCC) were 1.33 and 0.132 μg/L, respectively, for DDVP, and 0.100 and 0.008 μg/L, respectively, for MAL. The CMCs for DDVP and MAL derived using ETX 2.0 software and species sensitivity ranks were different from the CMCs obtained using the SSD method because of parameter uncertainties. The DDVP and MAL WQCs were significantly lower than Chinese surface water quality standard thresholds. The results provide basic data for revising these thresholds.

Keywords Dichlorvos · Malathion · Water quality criteria · Species sensitivity distribution · Acute safety threshold · Chronic safety threshold

Introduction

Organophosphorus pesticides (OPs) are currently the most widely used pesticides in China. OPs are very effective against a wide range of organisms and are cheap (Chen et al. 2016;

Wang 2015). The widespread application of large amounts of OPs such as dichlorvos (DDVP) and malathion (MAL) has led to environmental media in large areas being contaminated with these chemicals (Xu et al. 2012). DDVP was detected at 80% of sites in the Pearl River Estuary at which pesticide

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contamination was assessed (Zhou et al. 2013). Gao et al. (2009) found MAL in 43.5% of surface water samples collected in 2003 and 2004 from > 600 sites in seven major river basins and three major internal river drainage areas in China. OPs in soil can migrate into aquatic systems in runoff (particularly from sloping fields). OPs can persist in the environment and can accumulate in and harm aquatic organisms (Ellison 2012; Guo et al. 2014). In addition, OPs can biomagnify through the food chain (Cholewa et al. 2015; Li et al. 2015). However, the standard thresholds for some OPs (including DDVP and MAL) in the current Chinese "Surface Water Environmental Quality Standard" (MEP 2002) are no longer appropriate for protecting aquatic organisms in either the short or long term (Zhu et al. 2017). Appropriate water quality criteria (WQCs) therefore need to be developed.

Appropriate WQCs are important for protecting and managing the aquatic environment. Good practical and theoretical understandings of the environmental behaviors and toxicities of OPs are required for an environmental protection agency to develop water quality standards (Wang et al. 2016). Unfortunately, managing OPs in aquatic systems in China is difficult because of a lack of data on the toxicities of OPs to aquatic organisms (Zhu et al. 2016). Research on WQCs started late in China and has mainly relied on data from developed countries, particularly the USA (Wu et al. 2010). However, data from outside China may not be appropriate for the Chinese environment because of the differences between native and non-native species (Zheng et al. 2017a; Zhang et al. 2017b). Data on the toxicities of DDVP and MAL to a small number of aquatic organisms found in China are available (Chen et al. 2016; Zhu et al. 2016), but toxicity tests have been performed using few taxa native to China. In United States Environmental Protection Agency (US EPA) guidelines (USEPA 1984) and other guidelines, it is recommended that WQCs for some toxicants should be derived using data for species native to the region of interest. Therefore, there is an urgent need for DDVP and MAL WQCs to be developed using data for aquatic organisms native to China.

This study was focused on DDVP and MAL, which are used in large quantities and are frequently detected in aquatic environmental media (Chen et al. 2016). OP WQCs for freshwater aquatic organisms were derived following US EPA guidelines using at least eight genera with a specified taxonomic diversity (Mu et al. 2013; Qin et al. 2014b). In this study, 17 acute and six chronic toxicity experiments were performed using nine organisms (from several taxa) native to China. The experiments were performed using three fish species (*Pseudorasbora parva*, *Misgurnus anguillicaudatus*, and *Gobiocypris rarus*), a planktonic crustacean (*Daphnia magna*), a benthic crustacean (*Neocaridina denticulata*), an insect (*Chironomus plumosus*), an annelid (*Limnodrilus claparedeianus*), an amphibian (*Bufo gargarizans*), and a

mollusk (*Cipangopaludina cahayensis*). The log-logistic species sensitivity distribution (SSD) method developed by the Netherlands National Institute for Public Health and the Environment was used to evaluate the toxicity test data and the other data (RIVM 2007; Jin et al. 2014; Yi et al. 2016). Differences in the sensitivities of species native and not native to China were assessed. The WQCs of DDVP and MAL for freshwater aquatic organisms were derived according to the SSD method based on the log-logistic model.

The aims of the work were as follows: (1) to extend the DDVP and MAL toxicity databases, (2) to compare the sensitivities of native and non-native species to DDVP and MAL, and (3) to derive WQCs for the toxicities of DDVP and MAL to aquatic organisms. The results will benefit environmental risk assessments for DDVP and MAL and will improve the management of DDVP and MAL in natural aquatic environments in China.

Materials and methods

Toxicity data collection and OP selection

Published ecotoxicity data for OPs were obtained by searching the ECOTOX database (http://cfpub.epa.gov/ecotox), the Elsevier publication database (http://www.sciencedirect.com), and the China Knowledge Resource Integrated Database (http://www.cnki.net). The keywords included dichlorvos, malathion, toxicity, risk assessment, water quality criteria, criterion maximum concentration (CMC), criterion continuous concentration (CCC), toxicity endpoints, environmental exposure, detection, and concentration.

The median effect concentration (EC_{50}) and median lethal concentration (LC_{50}) were used as the acute toxicity measurement endpoints. The geometric mean was used if a number of toxicity data were available for one species.

Test chemicals and organisms

The DDVP and MAL used in the study were purchased from Spectrum Analysis Technology Co. (Beijing, China). The basic physicochemical properties of DDVP and MAL are shown in Table 1.

Tap water was dechlorinated by aerating it before being used as the dilution water in an experiment. The aerated tap water was at pH 8.08 ± 0.18 and had a dissolved oxygen concentration of 8.69 ± 0.16 mg/L, a total organic carbon concentration of 0.43 ± 0.13 mg/L, a conductivity of 353 ± 1 μS/cm, and a hardness (as $CaCO_3$) of 150 ± 0.10 mg/L. The algae growth medium (M11) used in the study contained 100 mg/L $NaNO_3$, 10 mg/L K_2HPO_4, 75 mg/L $MgSO_4 \cdot 7H_2O$, 40 mg/L $CaCl_2 \cdot 2H_2O$, 20 mg/L Na_2CO_3, 6 mg/L $FeC_6H_5O_7$, and 1 mg/L $Na_2EDTA \cdot 2H_2O$ and was at pH 8.0 (Lv and Ma 2013).

Table 1 Basic physicochemical properties of dichlorvos and malathion

Name	Abbr.	CAS no.	Chemical formula	Molecular weight (g/mol)	Purity (%)	Water solubility	Co-solvent
Dichlorvos	DDVP	62-73-7	$C_4H_7Cl_2O_4P$	220.98	99.6	1 g/100 mL	–
Malathion	MAL	121-75-5	$C_{10}H_{19}O_6PS_2$	330.358	99.8	0.0145 g/100 mL	Methanol

According to US EPA guidelines, WQCs such as CMCs must be derived from data for at least eight families of aquatic animals in three different phyla (Stephen et al. 1985). In this study, published DDVP and MAL ecotoxicity data were used, and new data were acquired by performing acute DDVP toxicity tests using eight aquatic species native to China and acute MAL toxicity tests using nine aquatic species native to China. The test organisms were obtained from Chaolai Market (Chaoyang, Beijing). *D. magna* (< 24 h old) from in-house cultures at the Chinese Research Academy of Environmental Sciences were used. Before a toxicity test was performed, the test organisms (except *D. magna*) were acclimatized in aerated tap water in the laboratory for at least 7 days (Zhang et al. 2017b). The toxicity tests were performed following Organisation for Economic Co-operation and development standard guidelines (OECD 2016).

General test conditions

The formal test concentrations used in the main toxicity tests were determined after performing preliminary experiments. The acute toxicity tests were performed using a static test method, and the chronic toxicity tests were performed using a semi-static method with the test solutions being completely replaced once every two days (OECD 1992, 2012; Zhang et al. 2017a; Wang et al. 2013a). Triplicate toxicity tests were performed at each formal test concentration, one blank control test was performed for DDVP, and one blank control test and one co-solvent control test were performed for MAL. Each test chamber contained seven organisms (Yin and Cai 2013; OECD 1992). The test conditions were described in detail by Yan et al. (2012). The tests were performed using a 14-h light, 10-h dark photoperiod. Each test chamber was immersed in a water bath kept at 22 ± 2 °C. The temperature, dissolved oxygen concentration, and pH in each test chamber were measured each day during each acute toxicity test and at least once each week during each chronic toxicity test. The mortality rates for the blank control and co-solvent control groups were < 10%.

Acute toxicity tests

Each acute toxicity test lasted 48 or 96 h. Test organisms with similar activities and body sizes were used in each test. The mean body length and wet weight were recorded. In the preliminary experiments, tests were performed at a series of concentrations, each a factor of ten different from the next, to allow the concentration ranges used in the full tests to be determined. Using the preliminary experiment results, at least

five concentrations between the minimum concentration at which all the organisms died and the maximum concentration at which all the organisms survived were selected for use in the full tests. In a test, *P. parva* or *M. anguillicaudatus* were placed in 4 L of a test solution in a 5-L beaker covered with a watch glass for 96 h. *D. magna* were placed in a 200-mL beaker containing 100 mL of test solution for 48 h. *C. plumosus* were placed in a 1-L beaker containing 300 mL of test solution for 48 h. *L. claparedeianus* were placed in 25 mL of test solution in a glass culture dish for 96 h. *B. gargarizans* were placed in a 2-L beaker containing 1 L of test solution for 96 h. *C. cahayensis* were placed in a 2 L beaker containing 1 L of test solution for 96 h. *G. rarus* were placed in 5 L of test solution for 96 h. As specified in Organisation for Economic Co-operation and Development standard 202 (OECD 2004), the test endpoint for *D. magna* was 48-h EC_{50} based on immobilization. *D. magna* unable to swim within 15 s of the test vessel being gently agitated were considered to be immobilized, even if they could still move their antennae. The test endpoint for *C. plumosus* was the 48-h LC_{50} based on the survival rate, and the test endpoint for the other aquatic species was the 96-h LC_{50} based on the survival rate.

The test organisms were not fed during the acute tests. The test organisms were observed daily, and any abnormal behavior was recorded. Dead organisms were removed promptly to avoid the water becoming contaminated. The dissolved oxygen concentration was > 60% of saturation throughout each test. The DDVP and MAL concentrations were within ± 20% of the mean measured concentrations throughout the toxicity tests. The test organism parameters and concentrations used in the main tests are shown in Table S1.

Chronic toxicity tests

Chronic toxicity tests using *P. parva*

The chronic toxicity tests using *P. parva* lasted 28 days, and the 28-day EC_{20}s were calculated. In each test, *P. parva* were placed in a 60-L beaker containing 50 L of test solution. The test solution was completely replaced every 2 days. When replacing the solution, the solution in the beaker was siphoned off and then fresh solution was poured in slowly. This avoided the test organisms being mechanically damaged. During each test, the fish were fed *Artemia* cysts (supplied by Tianjin Fengnian Aquaculture Co., Tianjin, China) once each day. The toxicity endpoint was the survival rate. Chronic toxicity tests were performed using seven test concentrations (0.000,

0.075, 0.113, 0.169, 0.253, 0.380, and 0.570 mg/L for DDVP and 0.000, 0.266, 0.400, 0.600, 0.900, 1.350, and 2.025 mg/L for MAL). A test was performed in triplicate at each concentration. A water control test was performed for DDVP, and a water control test and a co-solvent control test were performed for MAL. The DDVP and MAL concentrations were determined at the beginning and end of each test.

Chronic toxicity tests using *N. denticulata*

Each *N. denticulata* chronic toxicity test lasted 28 days, and the 28-day EC_{20} was calculated. The shrimps were fed with *Artemia* cysts (Tianjin Fengnian Aquaculture Co.) once each day, and the test solution was replaced every 2 days. The water was replaced in the same way as described above for the *P. parva* chronic toxicity tests. The toxicity endpoint was the same as used for the *P. parva* chronic toxicity tests. The test chemical concentrations used for the *N. denticulata* chronic toxicity tests were determined from acute toxicity data published by Zhu et al. (2017). Chronic toxicity tests were performed using seven test concentrations (0.000, 0.991, 1.388, 1.943, 2.720, 3.808, and 5.332 μg/L for DDVP and 0.000, 7.755, 15.200, 21.280, 29.792, 41.709, and 58.392 μg/L for MAL). Each test was performed in triplicate. A clean water control test was performed for DDVP, and a clean water control test and a co-solvent control test were performed for MAL. The DDVP and MAL concentrations were determined at the beginning and end of each test.

Chronic toxicity tests using *D. magna*

Each chronic toxicity test using *D. magna* (< 24 h old) lasted 21 days, and the 21-day EC_{20} was calculated. The test organisms were cultured in M4 medium under controlled conditions (OECD 211, 1998). For each test, *D. magna* were added to a 200-mL beaker containing 100 mL of test solution. The organisms were transferred to fresh medium every 2 days, and newborn juveniles in each beaker were counted and removed every 2 days. A second test vessel was prepared for each test, and the parent animals were transferred to it using a glass pipette when the medium was renewed. During a test, *D. magna* were fed each day with green algae (*Scenedesmus obliquus*) at a concentration of 1.0×10^5 cell/mL. The toxicity endpoint was the total number of offspring. The chronic toxicity test concentrations used in the DDVP tests were 0.000, 0.040, 0.080, 0.160, 0.320, 0.640, and 1.28 μg/L, and chronic toxicity test concentrations used in the MAL tests were 0.000, 0.060, 0.120, 0.240, 0.480, 0.960, 1.92, and 3.84 μg/L. Each test was performed in triplicate. A clean water control test was performed for DDVP, and a clean water control test and a co-solvent control test were performed for MAL. The DDVP and MAL concentrations were determined at the beginning and end of each test.

Chemical analyses

The DDVP and MAL concentrations in solution at the beginning and end of each test were determined by high-performance liquid chromatography using an Agilent 1200 instrument (Agilent Technologies, Santa Clara, CA, USA).

A sample was passed through a 0.45-μm membrane; then, the sample was analyzed by high-performance liquid chromatography using a ZORBAX Eclipse XDB-C18 column (150 mm long, 0.3 mm i.d., 5 μm particle diameter; Agilent Technologies). The mobile phase was a 70:30 *v/v* mixture of methanol and water, and the flow rate was 1.0 mL/min. The column temperature was 25 °C. An ultraviolet detector was used, and the wavelength was 230 nm for DDVP and 225 nm for MAL.

The DDVP and MAL method detection limits were 0.1 and 0.4 μg/L, respectively. A test solution containing DDVP or MAL at a concentration lower than the detection limit was passed through a solid-phase extraction system using a previously described method (Zheng et al. 2017b, 2017c). The extract was then evaporated to 1 mL and analyzed. The mean DDVP recoveries were 94.70–109.99%, and the measured concentrations in the acute and chronic toxicity test solutions were 92.59–106.81% of the expected concentrations. The mean MAL recoveries were 96.54–102.82%, and the measured concentrations in the acute and chronic toxicity test solutions were 109.68–115.82% of the expected concentrations. The DDVP and MAL concentrations before and after a water replacement were generally < 20% different, which met the toxicity test requirements (OECD 1994).

Statistical analyses and criteria derivation

Linear regression analyses were performed to calculate the 48-h EC_{50} and 96-h LC_{50} values and the 95% confidence intervals. As recommended by the US EPA, chronic toxicity was assessed using the EC_{20} and the geometric mean of the no observable effect concentration and lowest observable effect concentration, which was only used when the EC_{20} could not be calculated for the genus of interest (USEPA 2009, 2013, 2016). The chronic toxicity data were calculated using SPSS 18.0 software and were expressed as 21-day EC_{20} values for *D. magna* and 28 d-EC_{20} values for *P. parva* and *N. denticulata*. Independent-samples *t* tests were used to identify significant differences between sets of data. The *t* tests were performed using SPSS 18.0 software.

The HC_5 values (5% hazard concentrations, at which 5% of the aquatic organisms could have been negatively affected by exposure to the chemical of interest) were derived from the DDVP and MAL SSD curves using a log-logistic distribution model (Awkerman et al. 2008; Dyer et al. 2008). ETX 2.0 software (also used by the Netherlands National Institute for Public Health and the Environment (Van Vlaardingen et al. 2005)) and the species sensitivity rank (SSR) (Stephen et al. 1985) were used to verify these estimates. All toxicity data were determined using Origin Lab 9.0 software to construct SSD curves. The CMC was defined as the HC_5 value divided by an assessment factor of 2 (Yi et al. 2016). The CCC was extrapolated from the acute data to chronic data based on the final acute chronic ratio (FACR) because of a lack of chronic toxicity data (Dong et al. 2017).

Results and discussion

Toxicities of DDVP and MAL to aquatic organisms native to China

The acute toxicity values for DDVP for eight aquatic species and for MAL for nine aquatic species are shown in Table 2. No mortality was observed in the control groups. The acute toxicity tests showed that *D. magna* was the most sensitive species to DDVP (48-h EC_{50} = 2.87 μg/L), and the next most sensitive species (in decreasing sensitivity order) were *N. denticulata*, *L. claparedeianus*, *P. parva*, *M. anguillicaudatus*, *B. gargarizans*, *C. plumosus*, and *C. cahayensis*. *C. cahayensis* was the least sensitive species (96-h LC_{50} = 3.64×10^3 μg/L). In a previous study, the 48-h LC_{50} of DDVP for *D. magna* was found to be 1.59×10^2 μg/L, about 55 times higher than the 48-h EC_{50} found here (Saler and Saglam 2005a). The different results would have been caused by the different toxicity endpoints used in the studies. In the study by Saler and Saglam, the endpoint was lethality. The acute toxicity tests showed that *D. magna* was the most sensitive species to MAL (48-h EC_{50} 12 μg/L), and the next most sensitive species (in decreasing sensitivity order) were *N. denticulata*, *G. rarus*, *B. gargarizans*, *P. parva*, *C. cahayensis*, *M. anguillicaudatus*, *C. plumosus*, and. *L. claparedeianus*. *L. claparedeianus* was the least sensitive species (96-h LC_{50} = 4.13×10^4 μg/L). In a previous study, the 48-h LC_{50} of MAL for *D. magna* was found to be 1.20×10^2 μg/L, 10 times higher than the 48-h EC_{50} found here (Saler and Saglam 2005b). Like for DDVP, the different results would have been caused by the different toxicity endpoints

used in the studies. Our data indicated that DDVP is more toxic than MAL to *D. magna*. *P. parva* and *C. cahayensis* had similar sensitivities to MAL. *L. claparedeianus* was the species least sensitive to MAL (96-h LC_{50} = 4.13×10^4 μg/L).

The chronic toxicity test results are shown in Table 3. The 28-day EC_{20} for *P. parva*, 28-day EC_{20} for *N. denticulata*, and 21-day EC_{20} for *D. magna* were 1.26×10^2, 1.41, and 0.87 μg/L, respectively, for DDVP and 4.73×10^2, 14.30, and 1.13 μg/L, respectively, for MAL. *D. magna* was the most sensitive species to chronic exposure to both DDVP and MAL.

Published data on the toxicities of DDVP and MAL to freshwater organisms

Published data on the acute toxicities of DDVP and MAL to freshwater organisms were collected and are shown in Tables S2–S5. Acute DDVP toxicity values for 26 species native to China and acute MAL toxicity values for 30 species native to China were found. Chronic toxicity data for DDVP and MAL are not shown because few values were available. Acute DDVP and MAL toxicity data for species not native to China are shown in Tables S6.

All calculated species mean acute values (SMAVs) for the toxicity of DDVP to 26 species (six fish, eight crustaceans, five amphibians, three insects, and four mollusks) and for the toxicity of MAL to 30 species (15 fish, nine crustaceans, three mollusks, one amphibian, and two insects) are shown in Table 4. The most sensitive organism to DDVP was found to be *Daphnia pulex*, which had a SMAV of 0.068 μg/L, and the least sensitive species was *Bufo melanostictus*, which had a SMAV of 5.16×10^4 μg/L. The most and least sensitive species to MAL were *Gammarus pulex* and *L. claparedeianus*, which had SMAVs of 1.01 and

Table 2 Acute toxicities of dichlorvos (DDVP) and malathion (MAL) to aquatic organisms native to China

OPs	Species	Exposure time(h)	Functions	R^2	p	LC_{50}/EC_{50} (μg/L)
DDVP	*P. parva*	96	$y = 1.6602x - 0.9291$	0.8856	< 0.05	3.73×10^3 ($2.56–5.42 \times 10^3$)
	M. anguillicaudatus	96	$y = 2.7983x + 3.0679$	0.9866	< 0.01	4.90×10^3 ($4.22–5.70 \times 10^3$)
	D. magna	48	$y = 1.7507x + 4.1977$	0.8892	< 0.01	2.87 (2.08–3.98)
	*N. denticulate**	96	$y = 3.2668x - 0.0072$	0.9732	< 0.01	34.1 (27.6–42.2)
	C. plumosus	48	$y = 2.4286x + 1.3034$	0.9858	< 0.01	3.33×10^4 ($2.50–4.43 \times 10^4$)
	L. claparedeianus	96	$y = 3.062x - 3.2488$	0.9599	< 0.01	4.94×10^2 ($4.03–6.06 \times 10^2$)
	B. gargarizans	96	$y = 2.5821x + 1.9586$	0.8952	< 0.05	1.51×10^4 ($1.18–1.92 \times 10^4$)
	C. cahayensis	96	$y = 1.7999x + 2.1894$	0.9348	< 0.01	3.64×10^4 ($2.66–5.00 \times 10^4$)
MAL	*P. parva*	96	$y = 5.6215x - 1.1452$	0.8220	< 0.05	1.24×10^4 ($1.10–1.40 \times 10^4$)
	M. anguillicaudatus	96	$y = 4.5364x - 0.7650$	0.8284	< 0.05	1.87×10^4 ($1.63–2.14 \times 10^4$)
	D. magna	48	$y = 4.1351x + 0.4850$	0.9836	< 0.01	12.0 (11.0–14.0)
	*N. denticulate**	96	$y = 1.9884x - 0.8212$	0.9712	< 0.01	8.46×10^2 ($5.96 \times 10^2–1.201 \times 10^3$)
	C. plumosus	48	$y = 2.4498x + 1.6995$	0.9755	< 0.01	2.23×10^4 ($1.80–2.76 \times 10^4$)
	L. claparedeianus	96	$y = 6.4572x - 5.4332$	0.9685	< 0.01	4.13×10^4 ($3.26–5.23 \times 10^4$)
	B. gargarizans	96	$y = 0.84569x - 1.0220$	0.9400	< 0.01	7.38×10^3 ($4.95–11.01 \times 10^3$)
	C. cahayensis	96	$y = 2.1895x + 2.5034$	0.8194	< 0.05	1.38×10^4 ($1.18–1.62 \times 10^4$)
	G. rarus	96	$y = 14.581x - 5.8627$	0.9803	< 0.01	5.56×10^3 ($5.12–6.04 \times 10^3$)

*Acute toxicity data from a study performed by Zhu et al. (2017)

Table 3 Chronic toxicities of the organophosphorus pesticides (OPs) dichlorvos (DDVP) and malathion (MAL) to three aquatic organisms native to China

OPs	Species	Endpoints	EC_{20} (µg/L)
DDVP	*P. parva*	Mortality	1.26×10^2
	N. denticulata	Mortality	1.41
	D. magna	The total number of offspring (n)	0.87
MAL	*P. parva*	Mortality	4.73×10^2
	N. denticulata	Mortality	14.30
	D. magna	The total number of offspring (n)	1.13

4.13×10^4 µg/L, respectively. Planktonic crustaceans were generally relatively sensitive to both DDVP and MAL, so it could be used as biological indicators of OP contamination of aquatic environments. It can be seen from Table 4 that invertebrates are

more sensitive than vertebrates to DDVP and MAL. Like other insecticides, DDVP and MAL more strongly affect invertebrates, particularly insects and plankton, than vertebrates. It is therefore very important to assess the harm OPs can cause to invertebrates.

It can be seen from the toxicity data shown in Table 4 that the SMAVs for fish species vary widely, indicating that different fish species have different relative sensitivities to DDVP and MAL. In a previous study, *P. parva* was found to be more sensitive than other fish species to the brominated flame retardant tetrabromobisphenol A, and the LC_{50} was 0.860 mg/L (Yang et al. 2012). Wang et al. (2013b) also found that *P. parva* is particularly sensitive to some organic contaminants, particularly pesticides such as triazophos ($LC_{50} < 0.01$ mg/L). In contrast, we found that *P. parva* is not particularly sensitive to DDVP ($LC_{50} = 3.73$ mg/L) or MAL ($LC_{50} = 12.4$ mg/L). The contrasting toxicity values may be explained by

Table 4 Ranked species mean acute values (SMAVs) for dichlorvos (DDVP) and malathion (MAL) for species native to China

DDVP			MAL		
Rank	Species	SMAV (µg/L)	Rank	Species	SMAV (µg/L)
1	*D. pulex*	0.068	1	*G. pulex*	1.01
2	*C. dubia*	0.130	2	*C. dubia*	1.41
3	*S. serrulatus*	0.267	3	*D. pulex*	1.90
4	*L. stagnalis*	0.400	4	*S. vetulus*	2.90
5	*D. magna*	1.23	5	*S. serrulatus*	4.02
6	*N. denticulata*	34.1	6	*D. magna*	4.39
7	*Moina* sp.	1.06×10^2	7	*D. carinata*	13.0
8	*M. cephalus*	2.24×10^2	8	*L. macrochirus*	71.6
9	*Cyclopoida*	2.26×10^2	9	*N. undulata*	80.0
10	*Peltodytes* sp.	3.00×10^2	10	*G. aculeatus*	85.0
11	*L. claparedeianus*	4.94×10^2	11	*S. fontinalis*	1.25×10^2
12	*C. plumosus*	7.65×10^2	12	*O. mykiss*	1.38×10^2
13	*M. ornata*	7.80×10^2	13	*O. kisutch*	1.68×10^2
14	*L. macrochirus*	8.69×10^2	14	*C. batrachus*	2.50×10^2
15	*S. serrata*	1.22×10^3	15	*G. affinis*	3.48×10^2
16	*A. granosa*	1.79×10^3	16	*N. denticulata*	8.46×10^2
17	*L. variegatus*	2.66×10^3	17	*P. reticulata*	1.46×10^3
18	*P. parva*	3.73×10^3	18	*P. clarkii*	1.53×10^3
19	*M. anguillicaudatus*	4.90×10^3	19	*G. rarus*	1.60×10^3
20	*C. batrachus*	8.88×10^3	20	*C. orientalis*	7.23×10^3
21	*C. carpio*	9.41×10^4	21	*C. auratus*	7.25×10^3
22	*F. multistriata*	1.05×10^4	22	*B. gargarizans*	7.38×10^3
23	*P. megacephalus*	1.29×10^4	23	*C. striata*	8.00×10^3
24	*B. gargarizans*	1.51×10^4	24	*C. carpio*	1.15×10^4
25	*C.cathayensis*	3.64×10^4	25	*C. plumosus*	1.15×10^4
26	*B. melanostictus*	5.16×10^4	26	*C. cathayensis*	1.38×10^4
			27	*P. parva*	1.39×10^4
			28	*M. anguillicaudatus*	1.87×10^4
			29	*L. variegatus*	2.05×10^4
			30	*L. claparedeianus*	4.13×10^4

tetrabromobisphenol A and triazophos being very toxic but DDVP and MAL not being very toxic. OP pesticides inhibit acetylcholinesterase (Pope 1999), and there is concern about the potential for OPs in surface water to pose risks to non-target organisms. The chemical-specific sensitivities of particular organisms might be attributed to different contaminants having different modes of action (Raimondo et al. 2010; Wu et al. 2015; Yan et al. 2013).

Differences in the sensitivities of species native and not native to China to DDVP and MAL

Data on the toxicities of DDVP and MAL to species native and not native to China

Toxicity data for species native and not native to China acquired in this study and from previous publications are shown as a box plot in Fig. 1, and the data are shown in detail in Tables 3 and S2–S5. Fish native to China are more sensitive than fish not native to China to DDVP (the 96-h LC_{50}s for native and non-native fish were 2.23×10^2–9.41×10^2 and 2.70×10^2–2.88×10^4 µg/L, respectively). However, crustaceans not native to China are more sensitive than crustaceans native to China to DDVP (the 96-h LC_{50}s for native and non-native crustaceans were 0.068–1.22×10^2 and 0.400–4.00 µg/L, respectively). This could be because data for eight species native to China (including planktonic crustaceans and benthic crustaceans) but only three species not native to China (all benthic crustaceans) were used. The sensitivities of mollusks and insects to DDVP were also different for species native and not native to China. The acute toxicity of DDVP to amphibians native and not native to China could not be compared because insufficient data for species not native to China were available.

Fish native and not native to China had similar sensitivities to MAL except for *Alonella* spp. (the 96-h LC_{50}s for native and non-native species were 71.6–1.87×10^4 and 34.3–2.31×10^4 µg/L, respectively). The data for crustaceans not native to China were only for benthic crustaceans, and these were found

to be more sensitive than native benthic crustaceans (except *G. pulex*) to MAL. However, native planktonic crustaceans were found to be more sensitive than benthic crustaceans to MAL. Amphibians not native to China were found to be more sensitive than native amphibians to MAL. The toxicities of MAL to other species native and not native to China could not be compared because insufficient data on MAL toxicities to species not native to China were available.

Independent-samples *t* tests are usually used to identify significant differences between sets of data (Borja et al. 2000). Differences between the toxicities of DDVP and MAL to species native and not native to China were identified by performing independent-samples *t* tests. A significant difference was found between the DDVP toxicities to species native and not native to China (n1 = 26, n2 = 19, $P = 0.002$ (i.e., < 0.05)) but no significant difference was found between the MAL toxicities to species native and not native to China (n1 = 30, n2 = 18, $P = 0.358$ (i.e., > 0.05)).

Overall, species native to China were found to be less sensitive than species not native to China to both DDVP and MAL but the relative sensitivities of native and non-native species to DDVP and MAL varied.

DDVP and MAL SSDs for species native and not native to China

The DDVP and MAL SSDs for species native and not native to China are shown in Fig. 2. Few chronic toxicity data for DDVP and MAL were available, so SSD curves were constructed using acute toxicity data (shown in detail in Tables 4 and S2–S5).

The DDVP SSD for species native to China was to the right of the DDVP SSD for non-native species when the cumulative probability was < 0.5. HC_5 values were determined from the fitted curves to allow the sensitivity distributions of the native and non-native species to be compared (Fig. 2). The HC_5 for native aquatic organisms (2.66 µg/L) was about eight times higher than the HC_5 for non-native aquatic organisms (0.324 µg/L), indicating that aquatic organisms native to China are less sensitive than non-native aquatic organisms to DDVP. The SSD curves for native species were left of the curves for non-native species when the cumulative probability was > 0.50, suggesting that aquatic organisms native to China are more sensitive than non-native aquatic organisms to DDVP. These results were consistent with the results of a study performed by Zheng et al. (2017a) in which native and non-native species were found to have different sensitivities to As(III) in terms of the SSD. We concluded that there were differences between the SSD curves for native and non-native species, particularly for the HC_5 of DDVP.

Unlike for DDVP, the MAL SSD curves for species native and not native to China were not markedly different when the cumulative probability was < 0.1. This agreed with the results of independent-samples *t* tests. The SSD curve for species native to China was right of the curve for non-native species
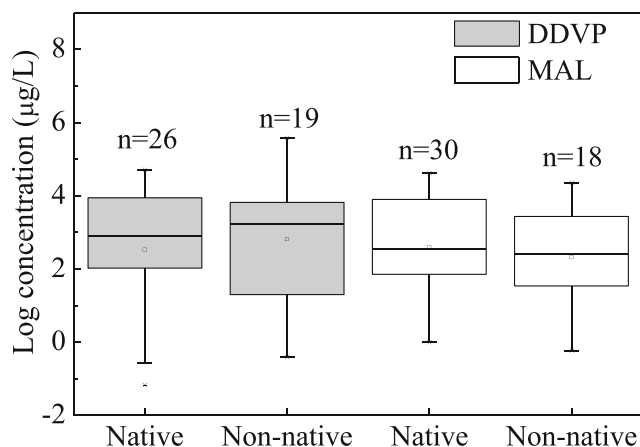

Fig. 1 Toxicities of dichlorvos (DDVP) and malathion (MAL) to species native and not native to China. *n* is the number of toxicity data, × indicates the maximum and minimum values, and the horizontal line inside each box is the median
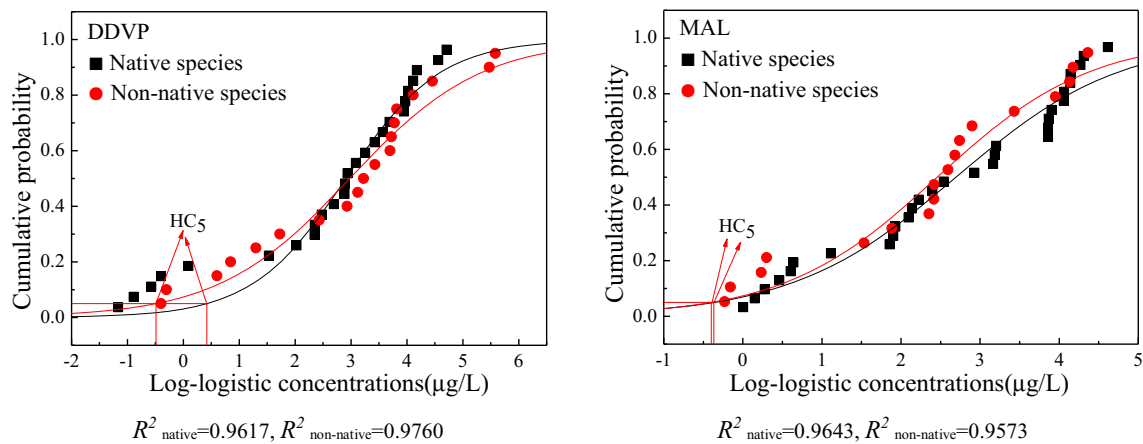
Fig. 2 Species sensitivity distribution curves for the acute toxicities of dichlorvos (DDVP) and malathion (MAL) to species native and not native to China

$R^2_{\text{native}}=0.9617$, $R^2_{\text{non-native}}=0.9760$

$R^2_{\text{native}}=0.9643$, $R^2_{\text{non-native}}=0.9573$

when the cumulative probability was > 0.1, indicating that non-native aquatic organisms are more sensitive than native aquatic organisms to MAL. The HC_5 values for native aquatic organisms (0.430 µg/L) and non-native aquatic organisms (0.398 µg/L) were of the same order of magnitude. The data for non-native species were more variable than the data for native species and did not meet the requirement (in guidelines published in 1985) that data should be available for at least eight genera with a specified taxonomic diversity. This introduced some uncertainty into our conclusions, and further research will be required to resolve this.

The different sensitivities of organisms native and not native to China to DDVP and MAL may be attributed to differences in the species composition and the numbers of toxicity data used in the SSD analyses. The DDVP toxicity data for fish accounted for 23% and 53% of the toxicity data used in the native and non-native SSD analyses, respectively. The DDVP toxicity data were for five taxonomic groups of native species and only four taxonomic groups of non-native species. The MAL toxicity data for fish accounted for 77% and 47% of the data used in the non-native and native SSD analyses, respectively. The MAL toxicity data were for five taxonomic groups of native species and only three taxonomic groups of non-native species. Other factors, such as the organism age, could have affected the test results and therefore affected the comparisons of the data for native and non-native species, and further research will be required to address this.

Differences in sensitivity distributions for species native and not native to China have been assessed in some studies. Wu et al. (2015) found that SSDs did not indicate statistically significant differences between native and non-native species. Zheng et al. (2017a) found that SSDs for As(III) indicated a statistically significant difference between the sensitivities of native and non-native species to As(III) ($P = 0.029$ (i.e., < 0.05)). However, no difference was found between the sensitivities of native and non-native species to As(V) ($P = 0.194$ (i.e., > 0.05)). No statistically significant difference between

the sensitivities of native and non-native species to 2,4-dichlorophenol was found by Jin et al. (2011). Differences in the sensitivities of native and non-native species to a potential toxicant may depend on the nature of the toxicant. Further research will be required to address this. There are therefore uncertainties in and risks associated with using toxicity data for species not native to China when developing WQCs for aquatic organisms in China. Toxicity data for native species should be used to derive WQCs for China. Indeed, in US EPA guidelines, it is recommended that only toxicity data for species native to an area of interest should be used to develop WQCs for that area to minimize uncertainties associated with differences in natural history, aquatic system characteristics, taxonomic groups, habitats, and geographical distributions of species (Stephen et al. 1985). It has been recommended that ecotoxicological data for only aquatic organisms indigenous to Australia or regions adjoining Australia should be used when using the SSD method to derive aquatic ecological benchmarks for Australia (ANZECC and ARMCANZ 2000; Hose and Brink 2004). Wu et al. (2015) found marked differences between the SSD curves for phenanthrene constructed using data for species native and not native to China.

Only aquatic organisms that are native to and widespread in China should be taken into consideration when deriving WQCs for China. Data for some alien species (e.g., *Oncorhynchus mykiss*) (Lower 2008), however, should be included because cultures of these species have been used widely in toxicological tests performed in China.

Derivation of DDVP and MAL WQCs

Acute toxicity tests were performed using nine aquatic species native to China, and acute DDVP toxicity data for 39 other aquatic organisms (20 native and 19 not native to China) and acute MAL toxicity data for 43 other aquatic organisms (25 native and 18 not native to China) were used. The acute DDVP and MAL toxicity data were used to generate cumulative sensitivity probability distributions using a log-logistic distribution model (Fig. 2) (Yi et al. 2016).

The acute SSD curves for native and non-native species are shown in Fig. 2. Insufficient chronic DDVP and MAL toxicity data were available for chronic SSD curves to be drawn. The HC$_5$ values were calculated from the log-logistic SSD curves to allow DDVP and MAL WQCs to be derived (Zheng et al. 2017b; MEP 2017). The calculated DDVP and MAL HC$_5$ values for aquatic organisms native to China were 2.66 and 0.430 μg/L, respectively. The final CMCs were calculated by dividing the HC$_5$ values by a factor of 2 to allow for uncertainties involved in calculating the HC$_5$ values (Stephen et al. 1985; Chen et al. 2016; Yan et al. 2012). The estimated DDVP and MAL CMCs were 1.33 and 0.215 μg/L, respectively.

Insufficient chronic DDVP and MAL toxicity data were available for the process described above to be used to calculate chronic toxicity WQCs. It is stated in US EPA guidelines that a CCC can be calculated by dividing the CMC by the FACR if inadequate chronic data are available. We used both acute and chronic toxicity data for *P. parva*, *N. denticulata*, and *D. magna* to calculate DDVP and MAL FACRs. The FACR was defined as the geometric mean of the species acute–chronic ratio. The species acute–chronic ratios and FACRs are shown in Table 5. The chronic safety threshold was calculated by dividing the HC$_5$ by the FACR. The estimated DDVP and MAL CCCs were 0.100 and 0.008 μg/L, respectively.

We also calculated DDVP and MAL CMCs using ETX 2.0 (as used by the Netherlands National Institute for Public Health and the Environment) (Van Vlaardingen et al. 2005)) (see Fig. S1) and using the SSR method (Stephen et al. 1985) (see Table S6). The DDVP CMCs estimated using ETX 2.0 and the SSR method were 0.214 and 0.080 μg/L, respectively. The MAL CMCs estimated using ETX 2.0 and the SSR method were 0.890 and 0.535 μg/L, respectively. The DDVP CMCs derived using ETX 2.0 and the SSR method were higher than the DDVP CMCs calculated using the SSD method, whereas the MAL CMCs derived using ETX 2.0 and the SSR method were lower than the MAL CMCs calculated using the SSD method. The method and fitting equation used in the ETX 2.0 software are not specified. HC$_5$ values can only be obtained by inputting toxicity data through the operating interface, and correlation coefficients, root-mean-square errors, and other parameters characterizing the fitting curve are not provided. The SSR method is typically controlled by the four most sensitive genera in the sensitivity distribution and the number of genera in the dataset because the fifth percentile of the distribution is calculated, meaning toxicity values for other species are ignored (Zheng et al. 2017a). These uncertainties lead to variability when estimating HC$_5$ values. Nevertheless, the results given using the SSR method and ETX 2.0 software were in the same order of magnitude as the results of the SSD curve method using a log-logistic model.

A previous study gave MAL CMC and CCC values of 170 and 28 ng/L, respectively (Palumbo et al. 2012). These results were similar to our results. The US EPA calculated that the MAL CCC is 0.1 μg/L, which is 12.5 times higher than the CCC we calculated (USEPA 1986). DDVP CMC and CCC values have not been calculated by the relevant organization in Canada, the European Union, or the Netherlands, possibly because DDVP and MAL are used less in these regions than in others. Our results are therefore a welcome addition to the toxicity data available for DDVP and MAL around the world.

The DDVP and MAL CMCs of 1.33 and 0.215 μg/L, respectively, were calculated taking into account the acute toxic effects of DDVP and MAL on aquatic organisms. These values can be used to evaluate the risks posed by short-term exposure to DDVP or MAL. The DDVP and MAL CCCs (0.100 and 0.008 μg/Lm, respectively) were calculated taking into account the chronic toxic effects of DDVP and MAL on aquatic organisms. The CCCs can be used to evaluate the risks posed by long-term exposure to DDVP or MAL. The CMCs and CCCs (except the MAL CCC) were higher than were found in a study performed by Qin et al. (2014a), in which the DDVP and MAL CMCs were 0.447 and 0.085 μg/L, respectively, and the DDVP and MAL CCCs were 0.030 and 0.056 μg/L, respectively. The current Chinese surface water quality standard thresholds (MEP 2002) for DDVP and MAL are both 50 μg/L. This is markedly higher than the WQCs suggested by our results. Rapid agricultural

Table 5 Species acute–chronic ratios (SACRs) and final acute–chronic ratios (FACRs) for dichlorvos (DDVP) and malathion (MAL)

	Species	Acute toxicity LC$_{50}$/EC$_{50}$ (μg/L)	Chronic toxicity EC$_{20}$ (μg/L)	SACRs	FACRs
DDVP	*P. parva*	3.73×10^3	1.26×10^2	29.60	13.32
	N. denticulata	34.1	1.41	24.18	
	D. magna	2.87	0.87	3.30	
MAL	*P. parva*	1.24×10^4	4.73×10^2	26.22	25.72
	N. denticulata	8.46×10^2	14.30	59.16	
	D. magna	12.4	1.13	10.97	

development in China has led to great increases in the amounts of pesticides produced and used. However, DDVP and MAL standard thresholds have not been updated to meet the need to manage pesticide use to protect the environment and are not appropriate for protecting aquatic organisms in either the short or long term. The current Chinese freshwater threshold for DDVP (50 μg/L) is not appropriate for protecting six sensitive species (*D. pulex*, *Ceriodaphnia dubia*, *Simocephalus serrulatus*, *Lymnaea stagnalis*, *D. magna*, and *N. denticulata*) according to the SMAVs (Table 4). The entire populations of the four most sensitive species (*D. pulex*, *C. dubia*, *S. serrulatus*, and *L. stagnalis*), which have SMAVs < 1 μg/L, could be destroyed if the Chinese freshwater threshold for DDVP is not reassessed. The situation is the same for MAL. The SMAVs for the seven most sensitive species (*G. pulex*, *C. dubia*, *D. pulex*, *Simocephalus vetulus*, *S. serrulatus*, *D. magna*, and *Daphnia carinata*) were lower than the MAL threshold (50 μg/L). These species will be harmed by MAL if the Chinese freshwater threshold for MAL is not reassessed.

There is an urgent need to reevaluate the Chinese surface water quality standards for DDVP and MAL. Useful information on which to base revised DDVP and MAL standards is provided here.

Conclusions

Crustaceans were found to be more sensitive than the other test organisms to DDVP and MAL. The SSD curves indicated that there are significant differences between the sensitivities of species native and not native to China to DDVP but not to MAL. However, the MAL data for species not native to China did not meet the requirement that eight genera with a specified taxonomic diversity should be included, so the MAL results were rather uncertain. Further research will be required to address this. Our results support the conclusion that native species should be taken into account when deriving WQCs for an area of interest. The DDVP and MAL CMCs were 1.33 and 0.215 μg/L, respectively, and the DDVP and MAL CCCs were 0.1 and 0.008 μg/L, respectively. These DDVP and MAL WQCs were markedly lower than the current Chinese surface water quality standard thresholds; therefore, the existing Chinese water quality standard thresholds for DDVP and MAL urgently need reevaluation. The data presented here will be useful for revising the DDVP and MAL standards.

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