Sediment Pyrethroid Sampling in Irrigation Canal/Ditch System, Missoula, Montana

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Cover photo: Canal at intersection of 39th St. and Paxson St., Missoula, MT; photo credit C. Schmidt, MDA

Introduction

The Montana Agricultural Chemical Groundwater Protection Act was passed in 1989 (MCA Title 80, Chapter 15, Sections 80-15-101 through 80-15-414). Section 80-15-103 states that it is the policy of the state to: protect groundwater and the environment from impairment or degradation due to the use of agricultural chemicals, allow for the proper and correct use of agricultural chemicals, provide for the management of agricultural chemicals to prevent, minimize, and mitigate their presence in groundwater, and provide for education and training of agricultural chemical applicators and the general public on groundwater protection, agricultural chemical use, and the use of alternative agricultural chemicals. Under this Act, it is the directive of the Ground-Water Protection Program (GWPP) of the Technical Services Bureau of the Montana Department of Agriculture (MDA) to monitor the occurrence and concentration of agricultural chemicals in the waters of the State of Montana.

In 2009, the GWPP collected sediment samples from the extensive canal/ditch network in and around the City of Missoula in Missoula County, Montana. Sediment samples were analyzed for synthetic pyrethroids which are widely used in agricultural, residential, ornamental, turf and veterinary insecticide applications. They are structural analogues of natural pyrethrins of botanical origin. In general, they are more stable (to photochemical, chemical, and microbial degradation), less toxic to mammals, and more toxic to insects than natural pyrethrins. Pyrethroids induce repetitive activity in the nervous system by acting on the sodium channel in the nerve membrane.

Of increasing use, pyrethroids are a group of insecticides used nationwide in place of more heavily restricted organophosphates and are used in agriculture, commercial pest control and residential consumer use (Nowell et al., 1999, Amweg et al., 2005). Weston et al. reported that the majority of insecticides sold for consumer use contain pyrethroids and they are also used widely for structural pest control around homes (2005). Crops in Montana on which pyrethroids are commonly applied include alfalfa, barley, corn, potatoes, sugar beets and wheat.

Pyrethroids are largely insoluble, non-persistent chemicals, and are relatively immobile in the environment. They have high adsorption coefficients and bind tightly to the organic fractions in soils and sediment and have low risk of leaching to groundwater. Bound to soil particles, pyrethroids are prone to off-site transportation and deposition in surface waters following a precipitation or irrigation event. The proclivity of synthetic pyrethroids to bind to the organic fraction of sediment results in a strong correlation between sediment toxicity and organic carbon (OC) content (Michelsen, 1992). Therefore, soil and sediment samples are OC-normalized to provide a better estimate of toxicity and bioavailability providing a better risk assessment than dry weight alone. Studies have established that there is a linear decrease in bioavailability with increasing total organic carbon (TOC) (Maund et al., 2002; Budd et al., 2007). Gan et al. found that bifenthrin distribution in sediments was correlated with sediment OC (r^2=0.98) and clay content (r^2=0.96) (2005).

Once deposited in streams, pyrethroids are relatively persistent and can be toxic to aquatic macro-invertebrates at low concentrations (Gan et al., 2005). Studies in urban and agricultural parts of California have revealed extensive stream sediment pyrethroid contamination at levels acutely toxic to sensitive aquatic macro-invertebrate taxa (Amweg et al., 2005, 2006; Weston et al., 2004, 2005). Sediment contamination by pyrethroids is of concern due to their wide spectrum of toxicity (Gan et al., 2005, Amweg et al., 2006). Amweg et al. (2005) reported average 10-day LC_{50} for the amphipod *Hyalella azteca* for several commonly used pyrethroids in the range of 0.45 – 10.83 μg/g OC in several California streams. These insecticides have been observed to alter invertebrate physiology at concentrations much lower than established sediment toxicities. Phillips et al. (2005) examined the relative contributions to toxicity from chlorpyrifos, DDT, and pyrethroids including permethrin, esfenvalerate and fenvalerate detected in sediment. All the compounds were detected at below published toxicity data, but the authors determined that mortality of *H. azteca* was due to additive or synergistic effects of organic contaminants. Experimentation led the authors to conclude that the synthetic pyrethroids were the source of the sediment toxicity.

The objectives of the Missoula pyrethroid study were to investigate the existence and potential aquatic invertebrate toxicity of synthetic pyrethroids in the sediments of the canal/ditch system in Missoula County. Aquatic invertebrate LC_{50}[1] sediment toxicities were not measured in this study. All sediment toxicity data were obtained from peer-reviewed literature. Utilizing this data, a toxic unit (TU) analysis was performed in conjunction with organic carbon-normalized detections to examine possible toxic effects of pyrethroids on aquatic invertebrates due to exposure to multiple sediment-bound pyrethroids in the canal/ditch water delivery network.

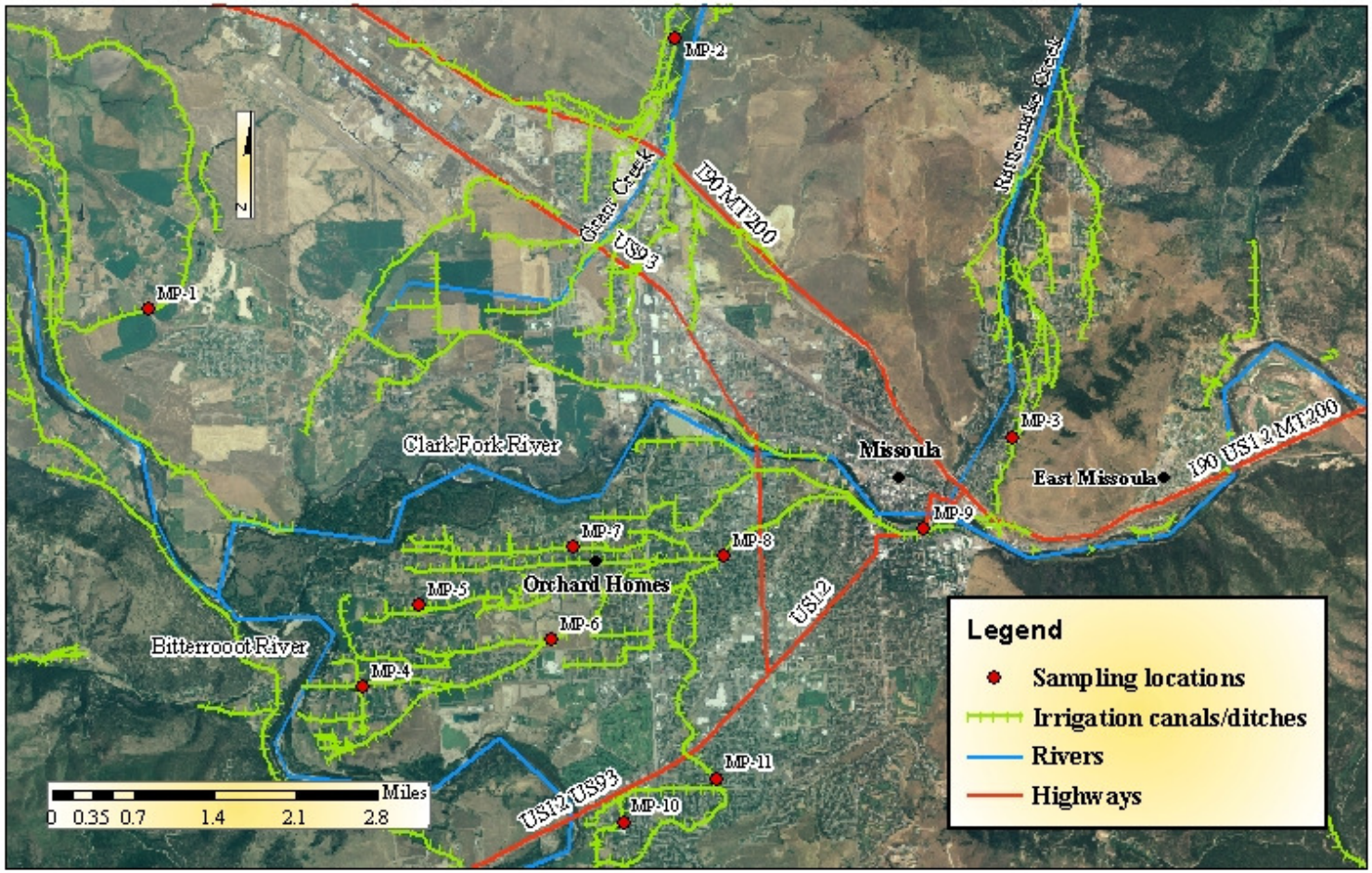
Sediment Sampling Methodology

Missoula County is located in west-central Montana and contains the City of Missoula, which is the 2nd largest city in Montana with an estimated population of 64,000 (July, 2006 (est.) and 100,000+ in the metropolitan area. The city has experienced rapid growth since 2000 (+12.3%). The Upper Clark Fork flows westward and joins the north-flowing Bitterroot River northwest of the City of Missoula. The impacts of a growing population on important fisheries utilized by the tourism and outdoor recreation industries may be significant and is cause for investigation. It has also been suggested that urban use of pyrethroids is more frequent than use in agriculture which often utilizes a short application window (Weston et al., 2004).

Sampling areas were initially located using aerial photos and ArcGIS mapping software for a coarse screening. On-site reconnaissance was then used to verify and select locations. Sediment samples were collected from the extensive canal/ditch network that delivers water to residential and agricultural users in Missoula and the surrounding area (Figure 1). According to the Montana Department of Natural Resources and Conservation (DNRC), there are 36.6 miles of canals/ditches in the Missoula and Orchard

[1] An LC_{50} is the concentration in a medium (water, sediment, etc) that is lethal to 50% of a population.

Figure 1.
Montana Department of Agriculture
2009 Pyrethroid Sampling
Missoula County, MT



Homes area south of the Clark Fork River and east of the Bitterroot River. Bounded by Interstate 90, Rattlesnake Creek and Grant Creek drainages contain 14.6 and 15.3 miles of canals/ditches respectively. For comparison, Missoula County contains a total of 546.9 miles of canal/ditch reaches.

The following criteria were used to select sampling locations: 1) proximity to pyrethroid applications, 2) proximity of vulnerable canal/ditch reaches[2], and 3) access to the canal/ditch network.

Pyrethroids primarily sorb to organic matter and colloidal particles. Therefore, samples were preferentially collected from recently deposited fine sediments and organic matter. Three sampling environments were encountered during this project: slow stream flow with a soft bottom (type 1); relatively fast stream flow with coarse bottom material and pockets of fine material (type 2); slow stream flow with primarily coarse bottom material covered with filamentous algae and a thin layer of fine material (type 3). Different sample collection techniques were used for each environment. For type 1 environments, a trowel was used to remove the upper sediment layer (0.5 inches or less). For type 2 environments, latex gloved hands were used to grab sediment from the streambed. For type 3 environments, latex gloved hands were used to collect sediment from the algae and cobble surfaces. All subsamples were placed into a clean stainless steel bucket and homogenized before being transported in sample bottles.

A United States Geological Survey (USGS) publication was utilized for development of a pyrethroid collection SOP (Hladik et al., 2009). All sampling protocols are outlined in the MDA SOP GWPP-14. Two sediment samples were collected at each site for separate analyses of pyrethroids and TOC. All pyrethroid analyses were performed by the Fish and Wildlife Water Pollution Laboratory administered by the California Department of Fish and Game in Rancho Cordova, CA. TOC analyses were performed by Energy Labs in Helena, MT. All samples were put on ice immediately after collection and stored at 4°C. Samples were allowed to settle overnight and then excess liquids were decanted prior to shipment.

Results

Total organic carbon and pyrethroid analyses revealed a wide range of results and detections. TOC had a range of 0.7 – 7.8%; mean of 3.2% for the July sample collection and 1.1 – 11%; mean of 3.9% in September. Site locations and TOC (%) values for the study are found in Table 1.

[2] Factors considered include the distance between the canal/ditch and probable application areas, the length of canal/ditch near probable application areas, the extent of irrigation return flow from probable source areas into the canal/ditch system, stream discharge, and the degree to which fine sediment is present in the canal/ditch.

Table 1. Site location information and TOC values for 2009 sampling events			
Site ID	**Location**	**Total Organic Carbon (%)**	
		7/7/09	**9/10/09**
MP-1	Canal at Mullan Road bridge SE of Kona Ranch Rd	2.7	2.6
MP-2	Canal east of Prospect Dr and S of Comstock Ct.	7.8	11.0
MP-3	Ditch at intersection of Van Buren St. and Holly St.	5.8	6.0
MP-4	Canal at intersection of South Ave. and Humble Rd.	3.8	3.3
MP-5	Canal south of intersection of Clements Rd. and Spurgin Rd.	3.4	3.8
MP-6	Canal along walking trail east of Tower St. on DNRC nursery[1]	0.7	1.5
MP-7	Canal at Hiberta St. bridge at SW corner of Hawthorne School	1.9	4.6
MP-8	Canal at terminus of 6th St west off of Garfield St.	3.5	3.6
MP-9	Canal on west side of Arthur Ave. overpass south of the Clark Fork River	0.8	1.3
MP-10	Canal at Briggs St. between Cold Spring Ct. and Orchard Ave.	1.4	1.1
MP-11	Canal at intersection of 39th St. and Paxson St.	3.0	3.6
[1] Montana Department of Natural Resources, Montana Conservation Seedling Nursery			

Due to unanticipated difficulties encountered by the laboratory, all samples from both collection events violated the 40-day holding period. However, there is no scientific basis for the holding period length. At the laboratory, pyrethroid analytical standards are prepared and used for 6 months before being discarded. Laboratory comparisons of old standards versus fresh standards have not yielded any degradation of the target analytes (D. Crane[3], person. comm., 2010). Therefore, MDA sample extracts most likely remained stable though they exceeded the 40 day hold time by 34 days. The laboratory analyzed sediment samples for 15 different synthetic pyrethroids and piperonyl butoxide (PBO). Estimated detection and reporting limits are found in Table 2.

[3] David B. Crane, Laboratory Director, California Department of Fish and Game, Fish and Wildlife Water Pollution Control Laboratory

Table 2. Pyrethroid analytes detection and reporting limits		
Pyrethroid Pesticides by GC/MS/MS	**Estimated Method Detection Limit[1]**	**Estimated Reporting Limit[1]**
	(ng/g) Dry wt.	**(ng/g) Dry wt.**
Bifenthrin	0.010	0.020
Cyfluthrin	0.100	0.200
Cyhalothrin, Lambda	0.040	0.100
Cypermethrin	0.100	0.200
Deltamethrin / Tralomethrin	0.100	0.200
Esfenvalerate	0.100	0.200
Fenpropathrin	0.100	0.200
Permethrin, Cis	0.040	0.100
Permethrin, Trans	0.040	0.100
Allethrin	0.100	0.200
Prallethrin	0.100	0.200
Resmethrin	0.100	0.200
Tetramethrin	0.100	0.200
Phenothrin	0.100	0.200
Piperonyl butoxide	0.100	0.500
[1]Estimated MDL and RL values based on 50% moisture		

Pyrethroid detections were OC-normalized using the results of the TOC analysis (Table 3). To assess *H. azteca* toxicities for individual pyrethroid detections, concentrations were divided by the decimal value of TOC per respective sampling location. This calculation is expressed in the following formula.

$$\text{ng/g OC} = \frac{\text{ng/g dry weight}}{\text{ng TOC/g dry weight}}$$

A total of 20 sediment samples were collected from the canal/ditch system from 11 different sites. Five different pyrethroids were detected including bifenthrin, cypermethrin, lambda-cyhalothrin, permethrin (*cis*- and *trans*-), and allethrin. With the exception of MP-3[4], all sites had at least one pyrethroid detection in 2009. Frequent detections in the July sampling event

[4] July 9, 2009 samples from MP-3 and MP-6 were broken in transport to the laboratory and were not analyzed for pyrethroids.

(*n*=9) included bifenthrin (9), *cis*-permethrin (6), and *trans*-permethrin (6). From the September sampling event (*n*=11), common pyrethroid detections again included bifenthrin (8) and *trans*-permethrin (4), but also the previously undetected allethrin (7).

Table 3. Organic carbon-normalized sediment pyrethroid concentrations, Missoula County, 2009

Site ID	Date	Allethrin	Bifenthrin	Cyfluthrin	Cyhalothrin, Lambda	Cypermethrin	Deltamethrin / Tralomethrin	Esfenvalerate	Fenpropathrin	Permethrin, Cis	Permethrin, Trans	Phenothrin	Piperonyl butoxide	Prallethrin	Resmethrin	Tetramethrin
MP-1	9-Jul	ND	**17.68**	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	10-Sep	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
MP-2	9-Jul	ND	**12.97**	ND	ND	ND	ND	ND	ND	**36.42**	**12.25**	ND	ND	ND	ND	ND
	10-Sep	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
MP-3	9-Jul	*Sample broken in transport*														
	10-Sep	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
MP-4	9-Jul	ND	**13.94**	ND	ND	ND	ND	ND	ND	**21.73**	**14.08**	ND	ND	ND	ND	ND
	10-Sep	**9.15**	**18.63**	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
MP-5	9-Jul	ND	**5.98**	ND	ND	ND	ND	ND	ND	**11.49**	**17.24**	ND	ND	ND	ND	ND
	10-Sep	ND	**0.51**	ND	**1.87**	ND	ND	ND	ND	**4.15**	ND	ND	ND	ND	ND	ND
MP-6	9-Jul	*Sample broken in transport*														
	10-Sep	**13.96**	**2.27**	ND	ND	ND	ND	ND	ND	**7.08**	**10.32**	ND	ND	ND	ND	ND
MP-7	9-Jul	ND	**163.53**	ND	ND	ND	ND	ND	ND	**24.32**	**36.07**	ND	ND	ND	ND	ND
	10-Sep	**6.09**	**70.23**	ND	ND	ND	ND	ND	ND	**2.57**	ND	ND	ND	ND	ND	ND
MP-8	9-Jul	ND	**46.77**	ND	**23.16**	ND	ND	ND	ND	**13.76**	**19.07**	ND	ND	ND	ND	ND
	10-Sep	**13.64**	**46.87**	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
MP-9	9-Jul	ND	**34.37**	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	10-Sep	**36.21**	**58.28**	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
MP-10	9-Jul	ND	**9.50**	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	10-Sep	**22.66**	**7.61**	ND	**6.67**	ND	ND	ND	ND	**18.51**	**33.74**	ND	ND	ND	ND	ND
MP-11	9-Jul	ND	**0.63**	ND	ND	**14.12**	ND	ND	ND	**19.97**	**37.89**	ND	ND	ND	ND	ND
	10-Sep	**16.49**	**14.11**	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND

All concentrations are in ng/g (ppb); all samples violated pyrethroid laboratory 40-day holding time; piperonyl butoxide is a synergist frequently found in pyrethroid products to increase potency

OC-normalized pyrethroid concentrations were then divided by published *H. azteca* sediment toxicities for selected pyrethroids in order to calculate toxic units (TUs) per sediment sample (Table 4). This is expressed in the following formula.

$$\text{Toxic Unit (TU)} = \frac{\text{Actual concentration (organic carbon-normalized)}}{\text{Reported } H.\ azteca\ LC_{50} \text{ concentration (organic carbon-normalized)}}$$

Table 4. Synthetic pyrethroid sediment toxicities from published literature

Pyrethroid	10 d LC_{50} for *H. azteca* in sediment[1] (ng/g OC[2])	Reference
bifenthrin	520	Amweg et al., 2005
cyfluthrin	1,080	Amweg et al., 2005
cyhalothrin, lambda	450	Amweg et al., 2005
cypermethrin	380	Maund et al., 2002
deltamethrin	790	Amweg et al., 2005
esfenvalerate	1,540	Amweg et al., 2005
fenpropathrin	8,900	Ding et al., 2009
permethrin	10,830	Amweg et al., 2005
tefluthrin	2,900	Ding et al., 2009

[1] No sediment toxicity data could be found for *allethrin*; *permethrin* is sum of *cis*- and *trans*- isomers
[2]OC = organic carbon normalized

This indirect assessment of toxicity was necessary to evaluate toxicity due to exposure to multiple sediment-bound pyrethroids. TUs had a range of 0.01 – 0.32 TUs with a mean of 0.07 TUs (n=9) for the July 9th sampling event and a range of 0.00 – 0.14 TUs (n=11) and a mean of 0.04 TUs for September 10th samples.

Table 5. Toxic Unit analysis per sampling site and event		
Site ID	**Toxic Units (TUs)[1,2]**	
	7/9/2009	**9/10/2009**
MP-1	0.03	0.00
MP-2	0.03	0.00
MP-2	0.03	0.00
MP-3	-	0.00
MP-4	0.03	0.04
MP-5	0.01	0.01
MP-6	-	0.01
MP-7	0.32	0.14
MP-8	0.14	0.09
MP-9	0.07	0.11
MP-10	0.02	0.03
MP-11	0.04	0.03

[1] Toxic unit analysis does not include allethrin for which no published sediment toxicity data could be found
[2] Permethrin TUs based on sum of *cis*- and *trans*-isomers

Discussion

In total, 5 different synthetic pyrethroids, including the *cis*- and *trans*- isomers of permethrin, were detected in the sediments of the canal/ditch system within and around the City of Missoula. The most common detections in the sediments were of bifenthrin, permethrin and allethrin. Bifenthrin and permethrin are two of the most commonly used synthetic pyrethroids. The half-life of bifenthrin is 12-16 months and the half-life of permethrin is 3-4.7 months for *cis*-permethrin and 2-10 months for *trans*-permethrin (Gan et al., 2005). Allethrin has a half-life in soil of 2 months (PPDB, 2009, 2010). The more recently synthesized pyrethroids such as bifenthrin have far greater aquatic toxicity than first generation pyrethroids such as allethrin. Permethrin and allethrin have numerous household uses while bifenthrin is used for structural pest control and lawn and garden applications. It is likely that retail sales and structural pest control and residential maintenance and control by professional applicators are the source of detected pesticides in the project area. A notable exception may be overland runoff from commercial nursery operations of the pyrethroid bifenthrin which is commonly used for insect control in potting soil.

Results of studies on the cumulative toxicity of sediment-bound pyrethroids to aquatic organisms have varied. Toxic Unit analyses by Weston et al. (2005) and Amweg et al. (2006) observed that

sediments at less than 1 TU of pyrethroids were non-toxic while there was little or no survival of *H. azteca* above 4 TUs. However in Amweg et al. (2005), authors observed that growth was typically inhibited at concentrations approximately 1/3 to 1/2 of the LC_{50}. The average, animal biomass was 38% below the level of the controls when exposed to pyrethroid sediment concentrations at these levels, and a majority of samples displayed >40% mortality of *H. azteca* when total pyrethroids reached about 0.4 TUs. However, *H. azteca* sensitivity to sediment-bound pyrethroids is neither a linear nor logarithmic relationship. Therefore, there is a significant difference in mortality between 0.3 TUs and the 0.4 TU threshold identified by Amweg et al. (2005). Mortality curves have not been formulated due to a paucity of data and the site-specific nature of pyrethroid toxicity on aquatic invertebrates. Authors theorized that sample mortality between >0.1 to 0.4 TUs was likely due to factors other than pyrethroids. Near total mortality of *H. azteca* was observed when total pyrethroids exceeded 3 TUs. This analysis also used data previously collected by Weston et al. (2004).

Differences in sediment toxicities may be due to undetermined factors affecting bioavailability or toxic elements that remained undetected in the samples. Pyrethroid distributions have been found to be dependent upon adsorption coefficients (K_d) which increase with increasing organic carbon and clay contents of sediments (Gan et al., 2005). Preferential accumulation and deposition occurs where stream sediments contain a large fraction of these fractions. As total concentration increases with increasing organic carbon and clay, bioavailability may simultaneously decrease. Selective transport via erosion and subsequent enrichment of fine particles is the main mechanism for transportation of pyrethroids off-site (Gan et al., 2005). However, net export of pyrethroid contaminated sediments to receiving water bodies may be limited to extreme precipitation events capable of flushing sediments downstream.

The TU analysis for MDA samples did not yield a sample in excess of 0.4 TUs. The highest observation was the July 9, 2009 sample collected at MP-7 which contained 0.32 TUs. As observed in previous studies, bifenthrin was responsible for 80% of sediment toxicity on average including 98% of total toxicity in the MP-7 sample discussed above. Amweg et al. (2005) observed growth effects at 1/3 to 1/2 the LC_{50}. However, no MDA pyrethroid detections fell within these more stringent criteria. The TU analysis was limited because no LC_{50} was available for allethrin which was detected in 7 of 11 samples collected in September.

It is worth noting that the pesticide synergist piperonyl butoxide (PBO) was not detected in sediments in this study. PBO does not have pesticidal properties itself but when added to insecticide mixtures including pyrethroids, PBO considerably increases chemical potency. The detection of PBO would have provided a potential marker for pyrethroid use and deposition. In sediment, PBO half-life is up to 24 days (Arnold, 1998). This is significantly less than the half-lives of those pyrethroids detected in the study and may explain why PBO was not detected. For example, in March 2010 there were 147 products containing permethrin registered for use in the State of Montana and each product also contained PBO. However, although permethrin was detected at 8 of 11 sampling sites over the course of the study, PBO was not detected at a single site.

Conclusion

No sediment toxicity testing was done as part of this investigation. Therefore, peer-reviewed literature from pyrethroid studies performed outside the state was used to gather LC_{50} data for selected pyrethroids. In referencing the literature, it was determined that sediment-bound pyrethroids in the canal/ditch system in and around the City of Missoula are not at concentrations that would be considered toxic to *H. azteca*. It is worth considering that there may be areas where pyrethroid sediment toxicities exceed known LC_{50} data in the study area. Sediment toxicity studies using sediment samples specific to the study sites are needed to conclusively state that observed pyrethroid detections are not hindering aquatic organism life cycles. However, given the weight-of-evidence from previous studies this is not an urgent necessity to validate MDA results. It must be noted that pyrethroid samples did exceed laboratory holding times, but it is not known how this may have affected analytical results if at all.

Future Work

Sediment sampling for the purpose of pyrethroid detection will be conducted as part of a larger surface water/groundwater investigation in the City of Billings in 2010. Sediment toxicity testing as a means of quantifying toxicity to aquatic invertebrate populations is outside the scope of the GWPP of the MDA. However, given the site-specific nature of sediment toxicity, such experiments in Montana surface waters where pyrethroids are detected are necessary to conclusively quantify risk. In-state sediment toxicity experiments would be more preferable and more accurate than relying upon toxicity assessments conducted in climes unlike those found in Montana. Synthetic pyrethroids will continue to be of interest and the MDA will continue to conduct sediment sampling projects in Montana. It is likely that such a project will return to Missoula in the future.

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