Tests of Water and Oil-Based Anionic Polyacrylamide Toxicity in Sediment Control Applications

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Abstract Anionic polyacrylamide (PAM) is effective at reducing sediment movement by flocculating particles together, which is important in maintaining water quality and erosion control. There are different formulations of PAM, including water and oil-based PAM. This study aimed to determine if the base composition of PAM affected water toxicity, sediment toxicity, and the amount of sediment in irrigation runoff. Sediment and runoff samples were collected from agricultural research plots treated with water and oil-based PAMs. Samples were analyzed using the survival rates of the test organisms *Hyalella azteca* and *Ceriodaphnia dubia*, along with the weights of suspended solids. Both water and oil-based PAMs increased the species mortality in water and sediment toxicity assays. All three PAMs were effective at decreasing the weight of sediment in runoff. Based on this study, there is not a correlation between PAM base composition and sediment or water toxicity because water and oil-bases decrease organism survival rates. Regardless of base, PAM greatly decreases the amount of sediment and "murkiness" in the irrigation runoff. PAM is effective at reducing sediment load and controlling soil movement, however there is uncertainty regarding water and sediment toxicity.

Introduction

Polyacrylamide, also called PAM, is a flocculating agent that binds suspended particles together. PAM causes loose sediment to remain in the soil rather than separate into the solution (Entry *et al.* 2002, Seybold 1994). PAM has been shown to reduce soil loss in creeks and agricultural settings (Seybold 1994, Weston *et al.* 2007), and improve water quality and infiltration (Wu 2001, Seybold 1994), and thus is a possible solution to soil erosion (Wu 2001). Other applications of PAM are the treatments of microbial borne disease and toxin control. When added to irrigation systems, PAM aggregates the treated and contaminated sediment, ideally leaving the runoff nontoxic and clear (Wu 2001). The runoff of PAM-treated water is “often cleaner than when it came in” and low doses can increase water infiltration by 60 percent (Wu 2001). The application of PAM in sediment control is recent and has been shown to be more effective at controlling sediment loss than common practices such as vegetating ditches and sediment traps (Weston *et al.* 2007). While its use for erosion and toxin control is promising, recent studies have found that PAM may be toxic depending on base and chemical compositions (Weston 2007).

The toxicity and use of PAM are being questioned because research investigating potential adverse effects has been limited (Wu 2001). When used at the directed concentrations, PAM produces dramatic results with no recorded toxicity (Sojka *et al.* 2007), and there is “no indication of any adverse impact on soil systems and plants when anionic PAMs are used in soil erosion applications” (Deskin 1996). Several properties of PAM allow it to not be regulated under the Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA) and lack permanent exposure limits, comprehensive toxicity, and risk assessments. PAM is a large molecule, hypothetically making it unable to enter the biological system to cause adverse effects, and anionic PAM does not belong to a class of chemicals known to cause health problems (Barvenik *et al.* 1996, Sojka *et al.* 2007, Young *et al.* 2007). Material Safety Data Sheets (MSDS) for anionic PAM only mention broad toxicity ranges for mammals, and lack toxicological information for aquatic organisms, species that are likely to intercept PAM (Accepta 2004). If we are to use PAM to reduce irrigation contamination, it needs to be evaluated through efficacy and toxicity endpoints to ensure that PAM is not contributing its own adverse effects.

The properties of PAM may change based on the carrier fluid used to apply the compound. The carrier fluid can also be referred to as the base. Although granular PAM can be used, PAM is typically prepared with a water or oil base for easier application. Previous studies have not examined how PAM solution bases, compare in erosion control or toxicity, but have examined the toxicity effects of water and oil bases used in drilling fluids, supplements, and dispersants (Addy *et al.* 1984, Cranford *et al.* 1999, Couillard 2002, Gulec *et al.* 1997, Myhre *et al.* 2003). The dispersant study showed that oil-based treatments increased toxicity relative to the control for amphipods and snails (Gulec *et al.* 1997), and oil-based drilling fluids also had higher toxicity than water bases (Cranford *et al.* 1999). Conversely, mud (Addy *et al.* 1984) and retinol supplements (Myhre *et al.* 2003) containing oil bases were less toxic than those with water bases. The types of oil-bases used in these substances are mainly mineral oils, which are used in PAM's oil emulsion. While the base composition is similar in the mentioned studies, the varying toxicity results do not conclude that either a water or an oil-base yields toxicity. This suggests that a base-compound interaction or other proprietary ingredients determine the toxicity of PAM and other substances, not the base composition alone. Couillard (2002) showed that mineral oil isolated as a base does not elevate toxicity, stressing the importance of the reaction between the base and compound. The interaction of PAM and an oil or water base may yield a specific reaction and different toxicity than these studies, so specific testing is necessary using biological models to safely and efficiently contain soils.

Initial testing examining the potential toxicity of oil and water based PAM was conducted by the Weston Lab of UC Berkeley. The Weston Lab uses the U.S. Environmental Protection Agency's standardized organisms for freshwater sediment and aquatic toxicity testing to determine survival levels (Weston 2007, Smith *et al.* 1991). *Hyalella azteca* and *Ceriodaphnia dubia* were the most sensitive species to PAM (Weston 2007). Weston showed that the oil-based PAMs were toxic to the two organisms, but the water-based form was not. This implies that the toxicity of oil-based PAM is from the added oils in the solution, or the interaction between the oil and PAM, not anionic PAM itself. The Weston Lab's research was conducted in a controlled laboratory setting with spiked water and sediment samples, but PAM in a field situation has not been studied. Movement of the runoff in a field may, through interactions of its constituents with the soil, alter toxicity from what was observed in the laboratory PAM exposures (Weston 2008a).

My research consists of further testing on the toxicity of the oil and water-based forms of anionic PAM on *Hyalella azteca* and *Ceriodaphnia dubia* in a field situation. More specifically, what is the effect of oil- and water- based PAM on water toxicity (using *Ceriodaphnia dubia* survival)? Does the toxicity of oil- and water-based PAM change once it has interacted with the soil? What is the effect of oil- and water-based PAM on sediment toxicity (using *Hyalella azteca* survival)? Lastly, what is the effect of oil- and water-based PAM on the amount of total suspended solids (TSS)? Is the amount of sediment in runoff increased or decreased compared to the control?

I hypothesize that the interaction of PAM and an oil base is the reason for the toxicity in the initial testing conducted by the Weston lab (unpublished data), so in the oil based PAM samples: (i) the incoming irrigation will be toxic to *Ceriodaphnia dubia* (essentially the same results as the initial testing), (ii) the outgoing runoff will be toxic, (iii) there will be no sediment toxicity, and (iv) it will reduce the amount of sediment in the runoff (TSS). For the water based PAM, I hypothesize: (i) the incoming irrigation will be nontoxic to *Ceriodaphnia dubia* (same as initial testing), (ii) the outgoing runoff will be non-toxic, (iii) there will be no sediment toxicity, and (iv) it will reduce the amount of sediment in the runoff (TSS). The controls of plain irrigation water should all come out nontoxic with an increased TSS. If my hypotheses are correct, microbial movement and other contamination can be stopped via water-based PAM.

Methods

Study site Data were collected on 10/9, 10/12, and 10/17/2007 from a research field at the U.S. Department of Agriculture facility in Salinas, CA. The plot had just been planted with lettuce seed (for a future unrelated USDA study) when I began my testing, but there was no vegetative cover. The research area was divided equally into four plots, one for each type of treatment. Three sampling trips were taken, and the procedures for each date were as follows:

Experimental Design To determine PAM base toxicity and sediment binding effects, experimental field tests were conducted using oil and water-based PAM. Tests were conducted to measure water toxicity from incoming and outgoing irrigation, sediment toxicity, and the amount of solids in the runoff. The oil-based PAMs were Soilfix (Ciba-Geigy, Avonmouth, Bristol, UK) and Soilfloc 300E (Hydrosorb, Inc, Orange, CA, USA). PAM 25 (Terawet, San Diego, CA, USA) was used as the water-based PAM. The indicator species, *Ceriodaphnia dubia* and *Hyalella azteca*, were incubated in runoff or sediment with each PAM base and the percent mortality was measured to quantify toxic results. The weights of the runoff samples were measured to analyze the effectiveness of oil- and water-based PAM at reducing the amount of suspended particles.

Field Study The research field consisted of four plots (Table 1). Samples were collected for irrigation runoff, sediment, and TSS using both oil- (Soilfix and SoilFloc) and water-based (TeraWet) PAM solutions for treatments and sediment containment and toxicity effects were compared relative to pre-treatment controls.

Table 1. Experimental design for sampling trips. The 10/9 trip had temporal pre-treatment controls, and a spatial control was taken in plot 1 on 10/12 and 10/17. There were two plots of each oil- and water-based PAM. The Soilfloc plots were treated five days earlier with Soilfix which could have influenced results. (O) = oil-based PAM, (W) = water-based PAM.

Sample date	Plot 1	Plot 2	Plot 3	Plot 4
10/9/07	Control	Control	Control	Control
10/12/07	Control	Soilfix (O)	Soilfix (O)	PAM25 (W)
10/17/07	Control	Soilfloc (O)	Soilfloc (O)	PAM25 (W)

Sampling 10/9/07 All plots were untreated to ensure the existing plots were not already toxic, and to get a baseline reading for comparison to 10/12 and 10/17 samples. Outgoing irrigation runoff was gathered in ditches at the end of plots 1-4 that were independent from each plot.

Sampling 10/12 and 10/17/07 Plot 1 was the control in all sampling trips (Table 1). In the 10/12 sample, Soilfix was used in plots 2 and 3. In the 10/17, was used as the oil-based PAM. Due to a miscommunication error, Soilfloc plots 2 and 3 were previously treated with Soilfix (Table 1). This could have altered the water and sediment toxicity of Soilfix, but it would not have affected the amount of TSS because PAM's flocculating properties would be removed after the Soilfix treatment five days earlier. In any case, plots 2 and 3 were still treated with oil-based PAMs in the 10/12 and 10/17 samples. PAM25 was used in plot 4 for both samplings (Table 1). The PAMs were added to irrigation pump supplies (plots 2 and 3 shared same pump) at concentrations of 5 ppm. Instead of gathering runoff from ditches at the end of each plot, the plots were organized so each plot had a main furrow where runoff and sediment samples were collected independently of the other plots.

Sampling procedures Plots were irrigated for three hours for each sampling. The samples taken were:

Incoming irrigation Water directly from sprinkler head which has not interacted with sediment yet. Periodic sampling was taken approximately every hour. Each hour, one third of a four liter jug was filled with incoming irrigation water using a tube attached to the sprinkler head, so that by the end of the irrigation the jug was full. This helps to ensure a more representative water sample. This sample is used for water toxicity testing with *Ceriodaphnia dubia*, and for water quality testing in lab.

Outgoing irrigation Separate sample to examine water toxicity after it interacts with soil. Water collected from the runoff ditch (in 10/9 sampling) and furrows (10/12 and 10/17 samplings). Once furrows had runoff, a four liter jug was filled with outgoing irrigation water (in the same one third increments as incoming irrigation). Using a 400mL beaker, water was taken from the runoff ditch/furrows and put into the jug. This sample is also used for water toxicity testing with *Ceriodaphnia dubia*, and water quality tests in lab.

Sediment Collected from runoff ditch or furrows, as described above. Once irrigation had stopped and furrows/ditches had preferably drained, sediment samples were obtained from treated furrows/ditches. The top layer of fine silt or clay sediment was gathered using a stainless steel scooper until about half of a four liter jar was filled. Sediment samples were taken along the length of each plot’s ditch/furrows to get a representative sample. This sample is used for sediment toxicity testing with *Hyalella azteca*.

Total Suspended Solids (TSS) Collected from runoff ditch or furrows, as described above. TSS samples were only gathered for 10/12 and 10/17 samples because the 10/9 sampling did not have any PAM treatments to measure. After runoff had started to flow, small jars were filled with runoff from furrows by submerging the jar under the stream until full. Samples were gathered four to six times throughout the irrigation, depending on time constraints, so the samples were representative of the entire irrigation. These TSS samples are used to calculate the amount of sediment in the runoff, and if PAMs reduced the sediment load.

Techniques of analysis Water toxicity, sediment toxicity, TSS, and water quality tests are performed to determine the toxicity and efficacy of oil and water based PAM. The analyses will be broken down as follows:

Water toxicity Samples from incoming and outgoing irrigation were sent to Pacific EcoRisk (Fairfield, CA, USA) to determine the water toxicity using *Ceriodaphnia dubia.* This species acts a biological model for aquatic toxicity. An acute 96-hour test (the 48-hour results were the values recorded) and a chronic seven-day test were performed for each sample, and the percent survival was used to determine if oil- or water- based PAM affect survival.

Sediment toxicity Sediment samples obtained from the field were kept moist and preserved via refrigeration. Each plot's sample was homogenized and 75mL was put into a 400mL beaker topped off with D.I. water. Five replicates were made for each plot. Ten *Hyalella azteca* were added to each beaker and the survival was calculated after ten days. The *Hyalella azteca* were fed once a day and water was changed daily so sediment composition is the only variable. The survival rates obtained from these tests were analyzed to determine if oil- or water-based PAM affect survival. Two sets of replicates were tested for each treatment because of high variability in the first set. The data with a lower standard deviation from the replicates was used to display the results.

TSS A recorded volume of TSS sample was filtered, dried, and weighed for each bottle. The grams/liter values were calculated and correlation between treatment and sediment load was checked to see if PAM base yielded different results.

Water quality Dissolved oxygen, pH, temperature, ammonia, hardness, alkalinity, and conductivity levels are recorded on the first and last day of *Hyalella azteca* sediment testing, and also a couple days in between. These values are monitored to make sure that samples are uniform and to check if water quality is affecting the survival rates.

Analysis There were two controls present in my studies, the temporal pre-treatment control from 10/9 and the spatial control from plot 1 on the same sampling date (Table 1). The water and sediment toxicity survival rates were compared to the spatial control. They were not measured relative to the pre-treatment control because the survival rates from each plot were very similar, and there was greater variation within the spatial controls on each sampling date. Therefore, the spatial control of plot 1 was more representative of the actual sampling conditions, including changes in weather and other uncontrolled variables. Only the acute results were analyzed and reported for water toxicity tests because the chronic values were the same.

Results

Water toxicity as a result of PAM base

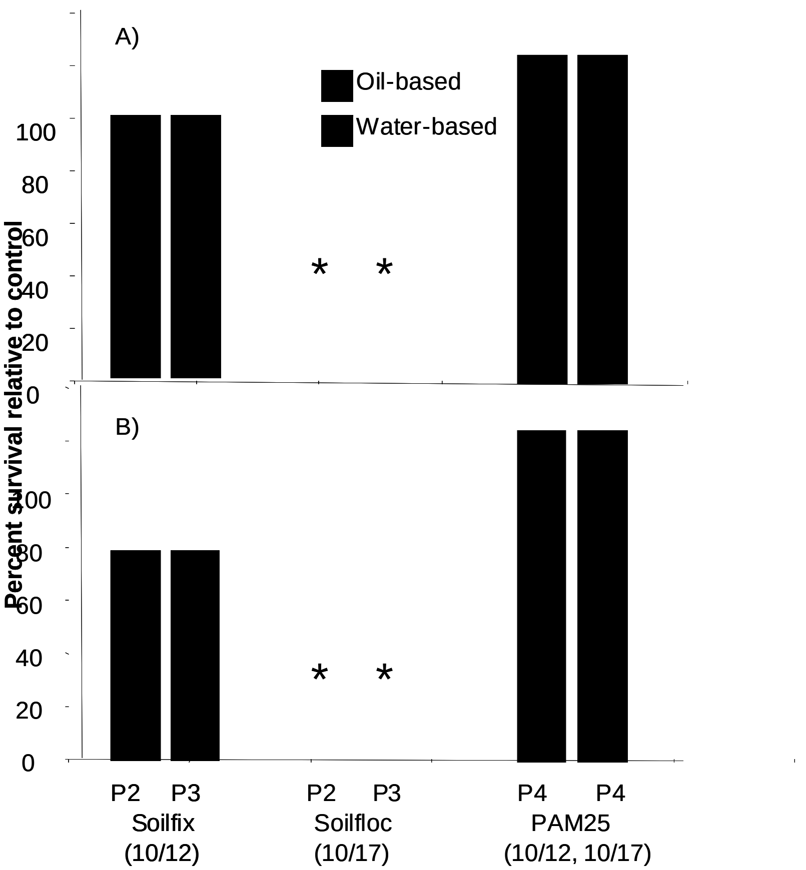

Figure 1. Effects of Soilfix, Soilfloc, and PAM25 on *Ceriodaphnia dubia* survival relative to the spatial control (plot1). A) Incoming irrigation water toxicity for plots before and during PAM treatment. B) Outgoing irrigation water toxicity for plots before and during PAM treatment. Results based on a 48 hour acute test. Soilfix treatment n=2, Soilfloc n=2,PAM25 n=1 (two separate treatments) Standard deviations for all tests were zero. * Results significantly less than the lab control ($p<0.05$)

*

In the incoming irrigation, Soilfloc decreased survival by 100%, while Soilfix had no effect on survival compared to the control, and PAM25 increased survival by 25% compared to the control. The control from plot 1 on 10/12 and 10/17 had 80% survival. The outgoing irrigation sample showed that Soilfix decreased survival by 20%, and Soilfloc decreased survival by 100%. Again, PAM25 had 25% greater survival than the spatial control (Fig.1).

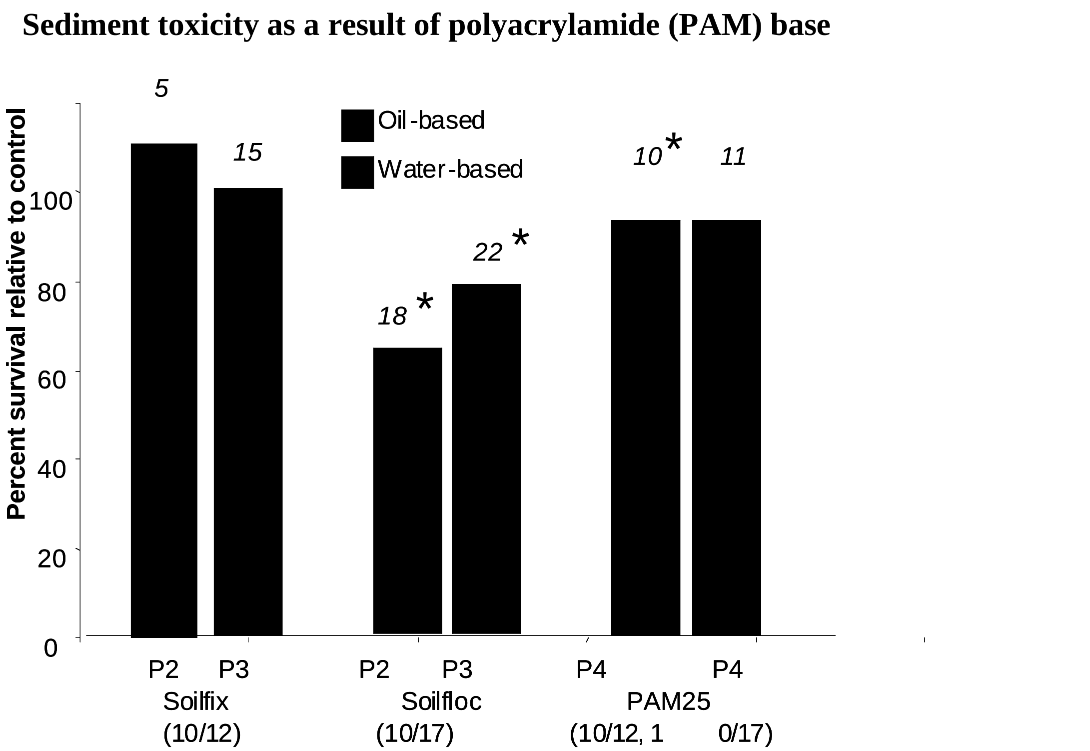

Figure 2. Effects of Soilfix, Soilfloc, and PAM25 on *Hyalella azteca* survival. Standard deviations are listed above each treatment. * Significantly toxic by t-test ($p < 0.05$)

The plot 1 controls for 10/12 and 10/17 had survivals of 86% and 90%, respectively. Soilfix did not reduce survival in plot 2 or 3, and increased survival in plot 2 by 11% (Fig. 2). Soilfloc reduced *Hyalella azteca* survival by an average of 29%. PAM25 decreased percent survival by 7% on average between the two dates. Based on this data, Soilfloc was toxic in all treatments, PAM25 slightly reduced survival, and Soilfix did not decrease percent survival relative to the control.

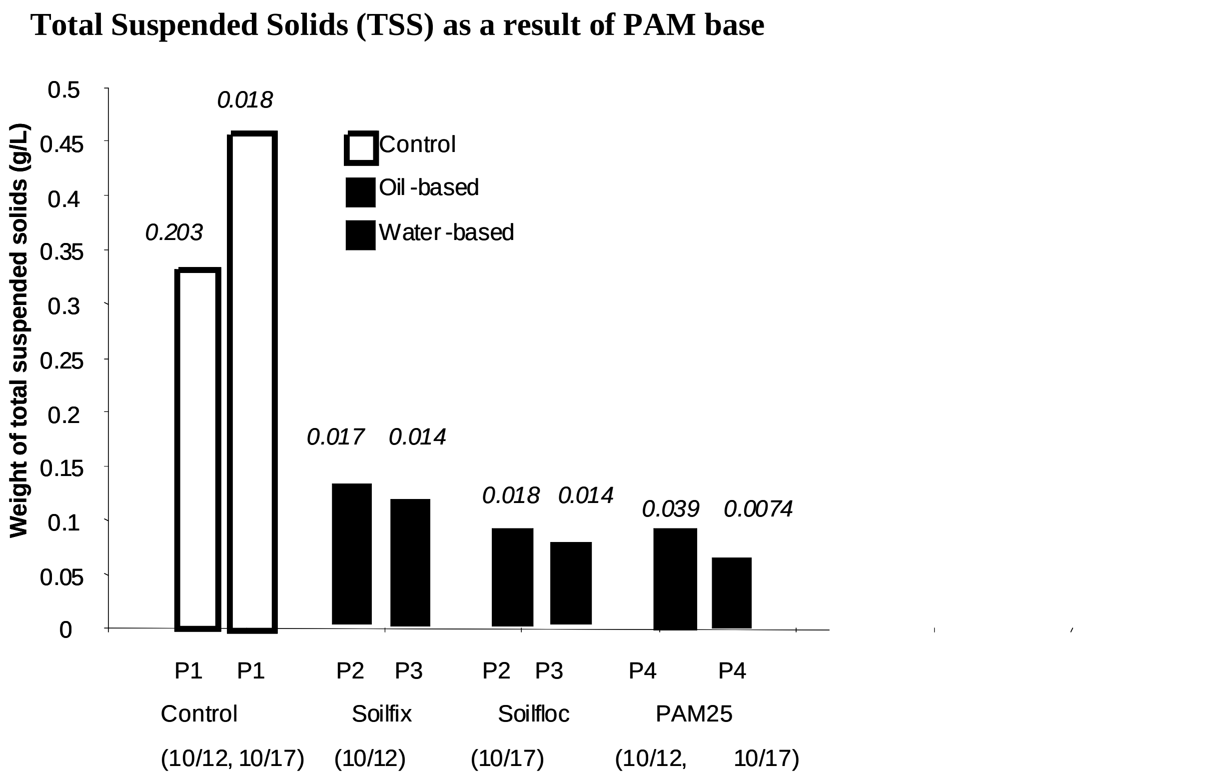

Figure 3. Effects of Soilfix, Soilfloc, and PAM25 on TSS. The standard deviation for each set of replicates is listed above the treatment.

On average, Soilfloc and PAM25 reduce solids by 79%, and Soilfix by 69% compared to the plot 1 control (Fig. 3). PAM base type does not seem to make a difference in efficacy of reducing TSS because PAM25 and Soilfloc are more effective than the Soilfix (Fig. 3).

Discussion

Based on results of this study, differences in PAM base composition potentially yield different toxicities. I did not observe consistent trends in toxicity related to PAM base and instead found decreased survival in both bases. Soilfix and Soilfloc decreased percent survival in the water toxicity tests, while PAM25 actually increased survival compared to the control (Fig. 1a&b). Soilfloc and PAM25 reduced survival in the sediment assays, and Soilfix increased the survival on average (Fig. 2). All PAMs were effective at reducing suspended solids by an average of 76%, but Soilfloc and PAM25 were most effective.

My hypotheses were partially supported by these results. Only the oil-based Soilfloc reduced survival in the water and sediment tests, and Soilfix reduced survival in just the water tests (Fig 1a&b). While the water-based PAM25 did not negatively affect survival in the water toxicity tests, it slightly reduced survival in the sediment tests (Fig. 1a&b, Fig. 2). Both oil- and water-based PAMs were effective at reducing sediment load, so that hypothesis was the only one completely supported.

The product formulation of the oil-and water-based PAMs should be considered when assessing their potential toxicity and effectiveness. The ingredients in the PAM mixtures are proprietary so the actual oil-base used in Soilfloc and Soilfix, along with the other ingredients, are unknown and may not be the same. Similarly, the ingredients aside from PAM and water in PAM25 are also unidentified. Changes in percent survival and flocculation could be attributed to those instead of just PAM and an oil- or water-base.

This supports the idea that there is a different reaction or ingredient going on in Soilfloc that Soilfix lacks that causes excessive toxicity. Formulation information on Soilfix mentions that the majority of the compound is formed by organic material, with only the remaining 0.25% to 5.00% consisting of a polymer (Harrison 2004). The Material Safety Data Sheet for Soilfloc lists it as a combination of calcium sulfate, cobalt chloride, and magnesium perchlorate (Crossings, Inc.). Soilfix is mainly composed of organic material, while the bulk of Soilfloc is composed of metals and non-organic material. These components could be the reason why Soilfloc demonstrates higher toxicity in both sediment and water tests than the other oil-based PAM, Soilfix. It is also important to take notice of the high standard deviations in the Soilfloc replicates for sediment testing (Fig. 2), so the data may not be completely reliable.

Study limitations There were numerous factors that could have affected the results of my research. The Soilfloc plots were pre-treated with Soilfix due to miscommunication. Even though they are both oil-based, it could influence the water and sediment toxicity of Soilfloc. It would not however, influence the ability to aggregate sediment because the leftover Soilfix residue is unable to continue flocculating sediment after being disrupted by the sprinkler irrigation (Weston 2008a). The TSS samples must be filtered immediately after being mixed and evenly distributed, and the lack of this could result in TeraWet's fluctuation between replicates. While the average of these values was taken to be more representative of conditions like this, there could still be some error. This could not be controlled, but the weather conditions varied greatly during the sampling process. On 10/9 it was dry, it was raining on 10/12, and 10/17 was somewhere in between. Other contaminants could have been present as a result of rain-induced transport and may have affected the toxicity results.

With these restraints in mind, the data that has been collected and analyzed still stresses the necessity for further testing and investigation to be done on base composition and other ingredients in PAM compounds. The sediment control applications are effective, as shown by the TSS testing, and can help control microbial borne diseases in agricultural and urban settings. While the sediment load is drastically reduced, uncertainty of water and sediment toxicity still remain. The chemical composition needs to be analyzed for Soilfix, Soilfloc, and PAM25 because the composition of the base does not strictly determine the toxic effect. The results of this study agree with the varying conclusions of Gulec *et al.* (1997), Cranford *et al.* (1999), Addy *et al.* (1984), and Myhre *et al.* (2003). Chemicals with oil-bases had higher toxicity than water-based compounds in the first two projects, and water-base demonstrated adverse effects in the last studies. To clear this overlapping variability, compounds in the PAM mixtures could be isolated and tested in sediment and water toxicity assays to pinpoint the cause of toxicity. Further work can also be done on PAM regulation. Threshold levels for toxicity on *Hyallela azteca* and *Ceriodaphnia dubia* have not been established either, so maybe a 10% reduction in survival compared to the control is acceptable. Once this has been researched and hopefully solved, the use of PAM in sediment control applications can be widespread without known ecotoxicological risks.

My research has shown that both oil- and water-based PAM have the ability to prevent contaminated from polluting water sources. However, it is still uncertain if these forms of PAM contribute to their own water or sediment toxicity. Although definite answers on PAM base toxicity were not achieved in this study, it can be said that the water-based PAM25 is the most effective option with the lowest reduction in survival overall. Expanded research will be able to determine the actual risk of different PAM bases in the management of water and land resources.

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