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# Comparison of the Relative Efficacies of Granulated Activated Carbon and Biochar to Reduce Chlorpyrifos and Imidacloprid Loading and Toxicity Using Laboratory Bench Scale Experiments

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## Abstract

Pesticide loads and associated toxicity can be significantly reduced using integrated vegetated treatment systems, which remove moderately soluble and hydrophobic pesticides, but need a sorbent material to remove more soluble pesticides. Neonicotinoids such as imidacloprid are widely used insecticides, acutely toxic, and have been linked to a range of ecological effects. Laboratory experiments were conducted to test the sorptive capacity of granulated activated carbon and biochar for removing imidacloprid and the organophosphate insecticide chlorpyrifos in a scaled-down treatment system. Simulated irrigation water spiked with individual pesticides was treated with a bench-top system designed to mimic a 600 L carbon installation receiving 108,000 L of flow per day for sixteen days. Biochar reduced insecticides to less than detectable and non-toxic levels. Granulated activated carbon similarly reduced chlorpyrifos, but allowed increasing concentrations of imidacloprid to break through. Both media treated environmentally relevant concentrations, and would be effective if used under conditions with reduced particle loads.

**Keywords** Vegetated treatment system (VTS) · Neonicotinoid insecticide · Organophosphate insecticide · Granulated activated carbon (GAC) · Biochar

Integrated vegetated treatment systems have been shown to be effective at removing up to 100% of insecticide loads in agriculture and urban runoff (Anderson et al. 2011; Anderson et al. 2016; Phillips et al. 2017). These systems include basins for the settling of suspended sediment, and vegetation for the adsorption and uptake of contaminants. They are particularly effective for insecticides of low to moderate solubility, such as pyrethroids and organophosphates. More soluble insecticides, such as neonicotinoids, require additional sorption steps to reduce insecticide loading and concentrations to non-toxic levels.

Sorption of organic pollutants has been studied extensively (Sophia and Lima 2018), particularly the use of activated carbon (Dias et al. 2007), which has been described as the most widely used sorption medium. Activated carbon is usually a dense material, sometimes derived from coal or coconut charcoal. Most commercially available activated carbon products are manufactured by a few established chemical companies and optimized for specific adsorption characteristics. Granulated activated carbon (GAC) has been shown to be a useful component of integrated field treatment systems for agriculture, and is used as a polishing step for reducing the loading of water soluble insecticides not treated by vegetative treatment systems (Phillips et al. 2017).

Although activated carbon is used extensively, it can be cost prohibitive for some applications. A number of reviews have discussed low-cost alternatives for activated carbon for various applications, including agricultural wastes, and biochar (Bhatnagar and Sillanpaa 2010; Ahmed et al. 2014; Mohan et al. 2014; Cha et al. 2016). Biochar, a more recent collective term for carbon products produced by heating biomass in a closed system with little or no air (Lehmann and Joseph 2009), may provide a promising low-cost alternative to activated carbon. These substances are traditionally used as soil amendments, and can be prepared from agricultural

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waste materials, such as rice straw and corn stover (Taha et al. 2014). Biochar is produced at temperatures < 700°C, whereas activated carbons are biochar-type materials that have been activated with chemicals or temperatures > 700°C (Lehmann and Joseph 2009).

Biochar has been used successfully as remediation or treatment with contaminated soils (Yu et al. 2009; Jin et al. 2016), and drinking water (Kearns et al. 2014; Gwenzi et al. 2017), and some recent research has been conducted applying biochar to treat pesticides in simulated agricultural runoff (Taha et al. 2014; Cederlund et al. 2017). Recent research demonstrated the removal of the organophosphate insecticide chlorpyrifos by activated carbon in simulated agricultural runoff (Phillips et al. 2017), and a laboratory study showed similar success with removal of the neonicotinoid imidacloprid (Voorhees et al. 2017). No studies have demonstrated the long-term efficacy of activated carbon or biochar in a field setting. Potential limitations to practical applications of these substances include cost, carbon disposal, and longevity, which is affected by reductions of contaminant active binding sites under real-world flow conditions. Binding sites can be blocked by particulate and organic matter and natural organic compounds.

Both chlorpyrifos and imidacloprid are routinely detected in agricultural runoff at toxic concentrations to aquatic organisms (Anderson et al. 2018). These chemicals cause toxicity at low concentrations, particularly to organisms at the base of the food chain, such as aquatic stages of insects. Terrestrial flying insects often have larval aquatic stages, and world-wide declines in flying insects have been linked to current-use pesticides, including neonicotinoids, through impacts to aquatic insect larvae (Dirzo et al. 2014; Morrissey et al. 2015). Dirzo et al. (2014) reports a global decline of up to 35% of Lepidopteran species abundance over the last 40 years, although this decline has not been linked to individual insecticides.

This project was designed to determine the relative capacities of GAC and biochar to remove current-use insecticides from simulated runoff over extended simulated irrigation regimes under controlled laboratory conditions. The study also evaluated the carbon media in sequence to determine whether a greater mass of insecticide could be removed when the treatments were used together. The removal efficiencies were tested using two representative insecticides, the organophosphate chlorpyrifos and the neonicotinoid imidacloprid. Both insecticides are commonly used in California and have been linked to surface water toxicity (Anderson et al. 2018). (However, state-wide sales of chlorpyrifos in California will end in February 2020.) The treatment systems were simulated in the laboratory using glass columns filled with the carbon media (after Voorhees et al. 2017). Column breakthrough was determined with a combination of insecticide analysis and toxicity testing. The relative efficacies were compared in terms of loading capacity, toxicity reduction, relative cost, and potential for recycling the two carbon media. These results are intended to provide growers and resource managers a comparative analysis for consideration of on-farm implementation of carbon treatment as part of integrated runoff treatment systems.

Chlorpyrifos toxicity thresholds are typically in the low parts-per-trillion range for aquatic invertebrates such as the daphnid *Ceriodaphnia dubia* and the amphipod *Hyalella azteca*, whereas toxicity thresholds for imidacloprid are in the low parts-per-billion range. These insecticides are not the most toxic, but were chosen to represent a range of different insecticide uses, toxicity, and solubilities. Chlorpyrifos also has proven to elicit human nervous system and neurodevelopmental effects, which led to a ban on its use by homeowners in 2001 (Lovasi et al. 2011), and the more recent agricultural ban. Imidacloprid has had significant effects on pollinators with potential secondary effects on the human food supply (van der Sluijs et al. 2013; Simon-Delso et al. 2015).

## Materials and Methods

Glass columns were constructed by cutting the tops off of 50 mL disposable Fisherbrand® (Fisher Scientific, Pittsburgh, PA, USA) serological pipettes. Each column was packed with 50 mL of either GAC, biochar, or a combination of 50% (by volume) each GAC and biochar. The activated carbon, Aquacarb® NS, is a reactivated coal/coconut shell charcoal combination GAC, is an economic alternative to virgin activated carbon products, and was sourced from Evoqua Water Technologies (Benicia, CA). The biochar, Evergreen Biocarbon®, was derived from organic sustainably-grown yellow pine wood pyrolized at 900°C, and was sourced from the Leland Agriculture Group (Fairfield, CA). The columns were packed with the desired carbon product(s), wetted with distilled water, and stirred to release trapped air. The average mass of GAC in each column was approximately 28 g and the average mass of biochar was approximately 15 g.

Chemical stock solutions were prepared using certified reagent-grade insecticides (AccuStandard, New Haven, CT, USA) in 30 L of laboratory well water. Target stock solution concentrations were 1 µg/L for chlorpyrifos and 5 µg/L for imidacloprid, and were based on environmentally relevant concentrations detected in local receiving systems. Stock solutions were pumped through 3 columns (GAC, biochar, GAC/biochar combination) simultaneously using a positive pressure peristaltic pump (Masterflex, Cole Parmer, Vernon Hills, Il, USA.). Each column treatment had a paired treatment blank to quantify possible toxic effects from the carbon-packed columns. Un-spiked water was passed through the blank columns. Working with one chemical at a time, each experiment proceeded for four weeks, and simulated sixteen irrigation events of 3 h each. Experiments evaluated treatment at laboratory-scaled flow rates (50 mL/min) comparable to field flow rates of 10 L/s and a total irrigation flow of approximately 108,000 L per event. The chosen flow rate was based on measured flow rates in agricultural practices and was the same as those tested by Voorhees et al. (2017), but greater than flow rates tested by Phillips et al. (2017). Each column packed with 50 mL of carbon was equivalent to a field installation of 600 L GAC and/or biochar.

Breakthrough events were assessed using a combination of analytical chemistry and toxicity testing, and were defined as the first detectable insecticide measured or toxicity observed in the column effluent after the maximum mass of insecticide has been loaded on the column. Chemistry and toxicity were measured every fourth event for a total of four measurements for each experiment.

Chlorpyrifos stock solution and post-column effluent concentrations were measured using enzyme-linked immunosorbent assays (ELISA, Modern Water, New Castle DE) with a level of detection of 50 ng/L. Imidacloprid was measured using liquid chromatography/mass spectrometry (LC/MS) analysis. Extraction and analysis methods for LC/MS were as follows. Approximately one liter of each water sample was passed through an Oasis HLB cartridge (Waters, Massachusetts, USA) in a vacuum setting. After the water was poured out, sodium sulfate was added to the empty water bottles to absorb excess water. Bottles were rinsed three times with 4 mL methanol to recover residues remaining in the container. This solvent was transferred to a glass evaporation tube, and cartridges were dried for at least 1 h prior to elution. Ten milliliters of methanol were used to elute the cartridges into evaporation tubes. The methanol eluent was combined with the bottle rinse and evaporated to a final volume of 0.2 mL using Turbovap (Biotage) followed by the addition of 0.8 mL milliQ water. Internal standard was added and the extract was injected into an Agilent 1260 Infinity HPLC coupled to an Agilent 6530 QTOF-MS. The gradient used was a mobile phase of 98% Optima water + 1 mM ammonium fluoride and 2% acetonitrile for 16.5 minutes and a mobile phase of 100% acetonitrile for 4 mins. The flow rate was 0.35 mL/min. The total acquisition time was 23.5 mins including 3 mins post-run. Imidacloprid level of detection was 1 ng/L.

Toxicity to chlorpyrifos was determined using the cladoceran *Ceriodaphnia dubia*, and toxicity to imidacloprid was determined using the midge *Chironomus dilutus* (U.S. EPA 2002). Briefly, tests with *C. dubia* consisted of five replicate chambers containing 15 mL of test solution and five organisms. Organisms were counted, and test solutions were renewed daily for 96 h with survival measured as the test endpoint. Tests with *C. dilutus* consisted of four replicate chambers containing 5 mL of sand, 200 mL of test solution and twelve organisms. Tests solutions were renewed every other day for 10 days. Survival and midge larval growth endpoints were determined at the end of the exposure. In addition to the column treatment blanks, negative controls with clean laboratory culture water were tested with each batch of column samples. Significant toxicity was determined using separate-variance t-tests and comparisons to a toxicity threshold of 80% of the control.

## Results and Discussion

Survival in all control and treatment blanks exceeded test acceptability criteria of 90% survival for *C. dubia* and 80% survival for *C. dilutus*, and *C. dilutus* larvae showed adequate growth in control and blank solutions (data not shown). Chlorpyrifos standard reference material recovery was 122% for ELISA analysis, and imidacloprid recovery was 80% for LC/MS analysis. The first imidacloprid toxicity test with *C. dilutus* did not include a combination treatment post-column effluent sample due to loss of sample.

Stock solution concentrations for chlorpyrifos ranged from 1100 to 1360 ng/L, and were high enough to cause significant mortality to *C. dubia* (Table 1). Biochar, GAC and the combination treatment all reduced concentrations of chlorpyrifos below the method detection limit of 50 ng/L. The ELISA MDL was close to the median lethal

**Table 1** Mean percent survival results for *C. dubia* in chemical stock solution and post-column effluents, listed with chlorpyrifos stock solution concentrations and recovery concentrations in post-column effluents for the three column types

| | 1/22/2018 | | | 1/29/2018 | | | 2/5/2018 | | | 2/12/2018 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Mean % survival | SD | CHL (ng/L) | Mean % survival | SD | CHL (ng/L) | Mean % survival | SD | CHL (ng/L) | Mean % survival | SD | CHL (ng/L) |
| Stock | 0 | 0 | 1100 | 0 | 0 | 1360 | 0 | 0 | 1157 | 0 | 0 | 1131 |
| Biochar | 100 | 0 | ND | 96 | 9 | ND | 88 | 18 | ND | 93 | 12 | ND |
| GAC | 100 | 0 | ND | 100 | 0 | ND | 100 | 0 | ND | 100 | 0 | ND |
| Combo | 100 | 0 | ND | 100 | 0 | ND | 100 | 0 | ND | 100 | 0 | ND |

*CHL* chlorpyrifos, *GAC* granulated activated carbon; *ND* non-detect; *SD* standard deviation concentration (LC50) for *C. dubia* [53 ng/L (Bailey et al. 1997)], but no toxicity was observed in any of the post-column effluent samples

Stock solution concentrations for imidacloprid ranged from 4130 to 4145 ng/L, which were high enough to cause significant mortality to *C. dilutus* and significantly reduce larval growth (Table 2). All three treatments reduced imidacloprid concentrations below the level of detection of 1 ng/L, but GAC allowed increasing concentrations of imidacloprid through the column as the 4-week experiment progressed. Although the GAC column had increasing breakthrough throughout the experiment, concentrations were well below the 96-h LC50 for *C. dilutus* [11,800 ng/L (Raby et al. 2018)]. No significant toxicity was observed in the post-column imidacloprid effluent samples (Table 2)

These experiments were designed to saturate all active binding sites on the sorption media to the point of column breakthrough as a means to compare the relative binding capacities of GAC, biochar and the combination GAC/biochar treatment. The goal was to determine the practical life span of each filter media type. Results were intended to be used to inform growers on how long filters can be left in the field before they are saturated and become ineffective. The experiment was conducted for four weeks and included sixteen simulated irrigation events. When scaled up to field conditions, the design was equivalent to treatment of 108,000 L of insecticide-laden water per simulated irrigation event through a carbon installation containing 600 L of media. The total simulated flow in these experiments scales up to approximately 1.7 million liters of water. Conservative estimates indicate that approximately 60 acres of irrigated central California lettuce with 5% runoff could produce this amount of water during a crop cycle, but in most cases growers typically have 1 to 2% runoff. Low breakthrough concentrations and absence of toxicity demonstrate that these media, when used correctly, have the potential to last in the field for an entire growing season, but results would vary depending on individual farming practices and biochar quality.

Biochar provides promising potential as an alternative to GAC because it may serve as a more sustainable method for treating insecticides. There are numerous studies assessing the effectiveness of biochar as a soil amendment for water retention and infiltration, and the adsorption of pesticides (Yu et al. 2009; Jin et al. 2016). Few studies have assessed biochar filtration as a means for reducing pesticide concentrations in agricultural or urban runoff. Taha et al. (2014) demonstrated the ability of biochar to remove fifteen pesticides from spiked water, and Cederlund et al. (2017) looked at the reduction of chlorpyrifos, diuron, glyphosate and MCPA in sand columns amended with biochar. Ulrich et al. (2017) used biochar to treat a number of spiked organic contaminants in simulated stormwater. Mohanty et al. (2018) discusses the use of biochar as a filtration medium in bioswales as a component of low impact development and reviewed other applications. These authors suggest biochar can be used in filter strips, vegetated ditches and wetlands, tree boxes and green roofs. These studies demonstrated the effectiveness of biochar as a treatment medium, but did not discuss the practical application of biochar as a polishing step in field integrated treatment systems. Biochar is considered a collective term for various pyrolized carbon products, and can be prepared with a variety of source materials at a variety of temperatures (Lehmann and Joseph 2009). It should be noted that not all biochar products will perform similarly.

Biochar used in this study was equally effective to GAC at removing chlorpyrifos, and more effective at removing

**Table 2** Mean percent survival results and mean ash-free dry weight (AFDW) results for *C. dilutus* in chemical stock solution and post-column effluents, with imidacloprid stock solution concentrations and recovery concentrations in post-column effluents for the three column types

| | Mean % survival | SD | Mean AFDW(g) | SD | IMI (ng/L) | Mean % survival | SD | Mean AFDW(g) | SD | IMI (ng/L) |
|---|---|---|---|---|---|---|---|---|---|---|
| 5/14/2018 | | | | | | 5/21/2018 | | | | |
| Stock | 2 | 4 | NA | NA | 4130 | 0 | 0 | NA | NA | 4145 |
| Biochar | 96 | 8 | 5.44 | 1.08 | ND | 92 | 17 | 1.62 | 0.79 | ND |
| GAC | 98 | 4 | 3.60 | 1.61 | 22 | 94 | 4 | 0.83 | 0.19 | 31 |
| Combo | NA | NA | NA | NA | ND | 88 | 8 | 1.18 | 0.14 | ND |
| 5/29/2018 | | | | | | 6/4/2018 | | | | |
| Stock | 6 | 4 | 0.17 | 0.06 | 4135 | 73 | 14 | 0.19 | 0.02 | 4130 |
| Biochar | 98 | 4 | 3.00 | 1.80 | ND | 98 | 4 | 2.86 | 1.37 | ND |
| GAC | 98 | 4 | 3.64 | 1.67 | 46 | 98 | 4 | 2.59 | 1.24 | 73 |
| Combo | 100 | 0 | 1.93 | 0.86 | ND | 98 | 4 | 2.42 | 1.45 | ND |

*IMI* imidacloprid, *GAC* granulated activated carbon, *ND* non-detect; *NA* not analyzed, *SD* standard deviation, *AFDW* ash-free dry weight imidacloprid, but both materials completely eliminated insecticide-related toxicity. Biochar has two attributes that may allow it to be a more cost-effective and sustainable sorption media. The biochar used in these experiment retails for <$1/pound versus $0.70-$2/pound for GAC. Biochar may also be less expensive to dispose of because it is currently used as a soil amendment in agriculture, and it may be possible to re-till it into farm soils or roads after it has been used to treat irrigation runoff. Insecticides bound to the biochar will have the opportunity to degrade in place. Use of biochar as a soil amendment post-treatment would have a major cost advantage over bituminous GAC which requires disposal as hazardous waste after use.

Future studies include a field comparison between GAC and biochar using simulated irrigation runoff spiked with imidacloprid and the pyrethroid insecticide permethrin. Carbon will be installed as a polishing step at the terminal end of a vegetated ditch. The first year of the study compares the efficacy of each carbon, whereas the second year will test the ability of the biochar to reduce insecticide concentrations in runoff from a cultivated test field.

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