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6 **A novel method to rapidly assess the suitability of water treatment residual and crushed**  
7 **concrete for the mitigation of point and nonpoint source nutrient pollution**

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21     **Abstract**

22     Freshwater ecosystems worldwide are at risk of becoming degraded as a result of excessive  
23     inputs of phosphorus (P) associated with terrestrial activities. This study describes a novel  
24     methodology to rapidly assess the potential of low-cost adsorbents which might be used to  
25     combat this issue. The ability of aluminum drinking water treatment residual (Al-WTR) and  
26     crushed concrete (CC) to remove P from dairy wastewater (DW) and forestry runoff  
27     (wastewaters representative of point and nonpoint P pollution sources, respectively) was  
28     assessed. In addition to predicting the longevity of these media in large-scale filters, potential  
29     risks associated with their use were also examined. The results indicate that both CC and Al-  
30     WTR show promise for use in removing P from forestry runoff, however the raised pH of  
31     effluent from CC filters may pose an environmental concern. Al-WTR showed greater promise  
32     than CC for the treatment of DW due to its higher adsorption capacity at high concentrations.  
33     Small releases of aluminum (13.63-96.17  $\mu\text{g g}^{-1}$ ) and copper (5.25-31.9  $\mu\text{g g}^{-1}$ ) were observed  
34     from both media when treating forestry runoff, and Al-WTR also released a small amount of  
35     nickel (0.16  $\mu\text{g g}^{-1}$ ). Approximately 50% of total metal loss occurred during the first 25% of  
36     total filter loading, indicating that pre-washing of the media would help prevent metal release.  
37     These results indicate that field-scale tests are warranted for the treatment of both wastewaters  
38     with Al-WTR; CC is likely to be unsuitable for either forestry runoff or DW due to its effects  
39     on pH and its short lifespan.

40     **Keywords:** Low-cost adsorbents; adsorption; phosphorus; wastewater treatment

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## 42 Nomenclature

$a^{**}$	Time constant in Eqn. 8
A	Constant of proportionality in Eqn. 12 ( $\text{mg g}^{-1} \text{L}^{-(1/B)}$ )
B	Constant of system heterogeneity in Eqn. 12
C	Sorbate concentration in bulk solution ( $\text{mg L}^{-1}$ )
$C_b$	Breakthrough concentration ( $\text{mg L}^{-1}$ )
$C_e$	Sorbate concentration of filter effluent ( $\text{mg L}^{-1}$ )
$C_o$	Sorbate concentration of filter influent ( $\text{mg L}^{-1}$ )
$k_{BA}$	Bohart-Adams rate constant ( $\text{L mg}^{-1} \text{min}^{-1}$ )
M	Mass of adsorbent (g)
$N_t$	Time dependent sorption capacity of bed ( $\text{mg L}^{-1}$ )
$q_t$	Time dependent sorbate concentration per unit mass of adsorbent ( $\text{mg g}^{-1}$ )
t	Empty bed contact time (min)
$t_b$	Service time/operating time of bed at breakthrough (i.e. when $C_e = C_b$ ) (min)
U	Flow velocity of solution past adsorbent ( $\text{cm min}^{-1}$ )
V	Volume of solution filtered (L)
$V_x$	Volume of filter bed (L)
Z	Filter bed depth (cm)

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## 46        **1. Introduction**

47 Excessive nutrient enrichment of surface waters results in optimal conditions for the  
48 overgrowth of algae and many species of noxious aquatic plants. The proliferation of these  
49 nuisance species causes serious damage to aquatic ecosystems, both in terms of long-term  
50 losses of biodiversity, as well as the more immediate concern posed by the sudden influx of  
51 large volumes of biomass into aquatic ecosystems. Sources of nutrient pollution may be divided  
52 into two categories: point sources and nonpoint sources. Discreet, easily identifiable point  
53 sources such as outlets from municipal wastewater treatment plants and drains from livestock  
54 housing and farmyards are usually comparatively low-volume, high-concentration nutrient  
55 waste streams (Rodgers et al., 2005). By comparison, nonpoint sources such as runoff from  
56 pastures, arable lands, and forestry plantations represent high-volume, low-concentration  
57 nutrient pollution streams (Acreman, 2012). Nonpoint sources are particularly difficult to  
58 control for a number of reasons, though the primary challenge is simply that the pollution  
59 source is spread across such a large area, making it extremely challenging to implement  
60 effective runoff treatment strategies (Rao et al., 2009).

61 A multitude of technologies and management practices have been developed to curtail the loss  
62 of nutrients, primarily phosphorus (P) and nitrogen (N), from point and nonpoint sources,  
63 though the focus has been largely on P, as it is the limiting nutrient in freshwater environments  
64 (Blomqvist et al., 2004; Correll, 1999; Hilton et al., 2006; Schoumans et al., 2014). There is  
65 evidence to suggest that controlling N inputs alone can have a deleterious effect by favoring a  
66 shift in algal communities towards more objectionable nitrogen fixing cyanobacteria (blue-  
67 green algae) (Schindler et al., 2008), further supporting the assertion that P control is of primary  
68 importance to the prevention of eutrophication (Schindler et al., 2016; Sharpley et al., 2003).

69 In the case of point sources, P is usually removed at centralized wastewater treatment facilities  
70 using well advanced technologies such as metal precipitation and adsorption, enhanced  
71 biological P removal, and, more recently, struvite crystallization (de-Bashan and Bashan, 2004;  
72 Schoumans et al., 2015). Phosphorus losses from nonpoint sources are usually addressed at  
73 source through the implementation of sound management practices: balancing fertilizer  
74 application in relation to crop requirements, matching animal feed P inputs to the nutritional  
75 requirements of livestock, reducing particulate P losses by minimizing erosion, and, where P  
76 must be applied, timing applications to minimize losses in runoff. Many mitigation strategies  
77 targeting nonpoint source P losses take advantage of the fact that a large portion of total P is  
78 present as particulate bound P, and hence trapping P laden sediments in settling ponds,  
79 constructed wetlands, and riparian buffer zones is often an effective pollution control strategy.

80 There are still, however, many instances where conventional techniques and management  
81 practices such as these fail to produce desired results (i.e. mitigation of pollution), or where  
82 their implementation is not practicable (e.g. because of excessive costs). In the case of nonpoint  
83 source pollution, a problematic example is P losses from forestry harvesting on blanket peat  
84 soils. Riparian buffer zones are the recommended best management practice to control such P  
85 losses, but these have been shown to be largely ineffective at removing dissolved P released  
86 during clearfelling due to low P retention capacity of low-mineral soils and insufficient uptake  
87 by vegetation (Rodgers et al., 2010). Reducing inputs of P is not a feasible option either, given  
88 that much of the P lost during forest harvesting originates from the brash (tree harvesting  
89 residues) materials, and hence is already present on site (Finnegan et al., 2014). In the case of  
90 point source pollution, a potentially troublesome source of nutrients is improperly managed  
91 dairy wastewater (DW) (Dunne et al., 2005). Farms often lack the prohibitive amount of space  
92 required for treatment with a constructed wetland, and climatic/soil conditions often preclude

93 land spreading (Ruane et al., 2011), as to do so would simply transmute a point source problem  
94 to a nonpoint source problem.

95 In cases such as these, where source control is neither feasible nor effective and/or where  
96 traditional onsite treatment methods are not possible, alternative remedial strategies must be  
97 implemented in order to prevent unacceptable P losses. Phosphorus-sorbing materials may  
98 offer a solution, and there has been a great deal of recent interest in identifying low-cost  
99 sorbents suitable for use in on-site wastewater treatment systems (Cucarella and Renman, 2009;  
100 De Gisi et al., 2016). More recently, sorbents are also being used to remove dissolved P from  
101 surface and ground waters (Buda et al., 2012), and numerous technologies are being  
102 investigated, using sorbents (1) applied as soil amendments (Stout et al., 2000) (2) as substrates  
103 in constructed wetlands (Vohla et al., 2005), or (3) as filter media in permeable reactive barriers  
104 (Baker et al., 1997) and in-drain filters (Penn et al., 2007).

105 Once a prospective sorbent has been identified, its suitability for an in-field trial must be fully  
106 assessed. Commonly, this involves laboratory-based evaluation of the medium's adsorption  
107 capacity using batch tests, though there is a growing body of research which indicates that  
108 results of these tests are not suitable for the purposes of estimating a medium's lifespan (Drizo  
109 et al., 2002; Guo et al., 2017; Penn and McGrath, 2011; Pratt and Shilton, 2009; Seo et al.,  
110 2005; Søvik and Kløve, 2005). To determine filter lifespan, flow-through experiments are often  
111 performed (Ali and Gupta, 2007), and in conjunction with predictive modeling, these can give  
112 at least an indicative estimate of potential media longevity (Shiue et al., 2011). This is an  
113 obviously important first step before costly and time consuming field studies are performed,  
114 though many flow-through methodologies can be time consuming in their own right, lasting  
115 many weeks (Razali et al., 2007), months (Bowden et al., 2009; Heal et al., 2003), or even  
116 years (Baker et al., 1998).

117 The aim of this study was to assess the potential usefulness of two low-cost adsorbents, namely  
118 aluminum drinking water treatment residual (Al-WTR) and crushed concrete (CC), as filter  
119 media intended to remove P from DW and forestry runoff. These wastewaters are  
120 representative sources of point and nonpoint P losses, respectively, and the two low-cost media  
121 have shown past promise as P-sorbing materials (Babatunde et al., 2009; Callery et al., 2015;  
122 Egemose et al., 2012). A major concern with novel low-cost adsorbents is that they may be  
123 potential sources of metals (Velghe et al., 2012). To address this concern, the final effluent  
124 from both filter media were also analyzed for metals. The potential of the materials for use as  
125 filter media was assessed using a recently developed methodology which uses a combination  
126 of rapid small-scale filter experiments and modeling techniques to make predictive estimates  
127 as to the performance and longevity of hypothetical large-scale filters (Callery et al., 2016;  
128 Callery and Healy, 2017). This novel methodology assesses the media under flow-through  
129 conditions similar to those experienced in-field, but produces results in as little as 24 hours,  
130 similar to batch tests. These tests may serve as a useful precursor to large-scale, in-field tests  
131 which are required to assess the hydraulic performance of individual filter configurations, and  
132 to examine concerns including filter clogging and wash-out of reactive materials.

## 133 **2. Materials and Methods**

### 134 **2.1 Sample collection and preparation**

135 The two low-cost adsorbents investigated in this study were Al-WTR and CC. The Al-WTR,  
136 which had an initial dry solids content of approximately 20%, was first passed through a 1 mm  
137 mesh to remove any coarse particles. The strained sludge was then oven dried at 105°C for 24  
138 hr, before being ground with a mortar and pestle and sieved; the fraction which was retained  
139 by a 0.5 mm sieve after passing a 1 mm sieve was stored in airtight, high-density polyethylene  
140 (HDPE) containers for use in the adsorption columns. The concrete was pulverized using a  
141 mortar and pestle, and dried in an oven at 105°C for 24 hr before being sieved; similarly, the  
142 fraction which was retained by a 0.5 mm sieve after passing a 1 mm sieve was stored in airtight  
143 HDPE containers prior to use in the adsorption columns.

### 144 **2.2 Preparation of filter columns**

145 For each filter medium, filter column sets, comprising four columns with lengths of 0.4 m, 0.3  
146 m, 0.2 m, and 0.1 m, were prepared using low density polyethylene (LDPE) tubing with an  
147 internal diameter of 9.5 mm. The filter columns were packed with filter media, and syringe  
148 barrels (i.e. syringes with the plungers removed), packed with a small quantity of glass wool,  
149 were fastened to the top and bottom of each filter column. The columns were affixed to a stand  
150 to maintain a stable vertical orientation throughout the experiment, and silicone tubing with an  
151 internal diameter of 3.1 mm was attached to the syringe ends at the bottom and top of the filter  
152 columns to provide influent and effluent lines. A schematic of this experimental setup may be  
153 found as Figure S1 of the supplementary material.

### 154 **2.3 Operation of filter columns**

155 Coarse straining filters, comprising a syringe barrel packed with a small quantity of glass wool,  
156 were attached to the ends of the influent lines, and these were submerged in a feed tank filled  
157 with either forestry runoff or DW. A Masterflex peristaltic pump was used to supply influent  
158 the base of each filter column at flow rates of 105-205 mL hr<sup>-1</sup>, corresponding to hydraulic  
159 loading rates (HLRs) of 1.47-2.88 m hr<sup>-1</sup>, rates typical of activated carbon adsorption filters  
160 (Chowdhury, 2013) and tricking filters (Spellman, 2013), and HLRs that have been used in  
161 reactive filters for P removal (Erickson et al., 2012). The columns were mounted vertically and  
162 operated in “up-flow” mode (as shown in schematic included as Figure S1 of the supplementary  
163 material) to preclude any bypass flow. The effluent from each filter column was collected in  
164 2 hr aliquots using an autosampler. The filter columns were operated in 12 hr on/off cycles to  
165 replicate the intermittent loading conditions that would be expected on site, thus allowing time  
166 for intra-particle diffusion of adsorbate molecules and associated rejuvenation of the sorbent  
167 surface. The collected aliquots were weighed to determine the volume of solution filtered, and  
168 subsamples of each aliquot were passed through a 0.45 µm filter before being analyzed for  
169 dissolved reactive phosphorus concentrations after APHA 4500-P E (Eaton et al., 1998).  
170 Subsamples of the aliquots were acidified to a pH<2 with nitric acid and metal concentrations  
171 were determined by ICP-MS after U.S. EPA method 6020A (U. S. Environmental Protection  
172 Agency, 2007). Metal analyses were only performed on effluent samples from the 0.4 m filter  
173 columns, as testing samples from all columns was cost-prohibitive.

#### 174 **2.4 Data collection and analyses**

175 For each filter column, after any filter loading, V (L), the mass of P removed per gram of filter  
176 medium (mg g<sup>-1</sup>), was calculated by:

$$177 \quad q_t = \sum_{i=1}^n \frac{(C_o - C_e)V_i}{m} \quad (1)$$

178 Where  $q_t$  is the mass of P retained per gram of filter medium (dependent on the contact time  
 179 between the solution and the filter medium) ( $\text{mg g}^{-1}$ ),  $C_o$  is the P concentration of the influent  
 180 ( $\text{mg L}^{-1}$ ),  $C_e$  is the P concentration of the  $i^{\text{th}}$  aliquot of filter effluent ( $\text{mg L}^{-1}$ ),  $V_i$  is the volume  
 181 of the  $i^{\text{th}}$  aliquot (of a total number of aliquots,  $n$ , whose combined volume is  $V$ ) (L), and  $m$  is  
 182 the mass of sorbent in the adsorption column (g).

183 In a recent paper, Callery and Healy (2017) proposed that the performance of multiple  
 184 adsorption columns could be described with one of two models. The first of these, which is  
 185 best suited to the description of sigmoidal breakthrough curves, is based on a modification of  
 186 the popular Bohart-Adams model (Bohart and Adams, 1920):

$$187 \quad \ln\left(\frac{C_o}{C_b} - 1\right) = kN_t \frac{Z}{U} - kC_o t_b \quad (2)$$

188 Where  $k$  is a model constant (with units of  $\text{L mg}^{-1} \text{min}^{-1}$ ),  $Z$  is the filter-bed depth (m),  $U$  is the  
 189 linear flow velocity ( $\text{cm min}^{-1}$ ),  $t_b$  (min) is the filter-bed service time at which the concentration  
 190  $C_b$  occurs, and  $N_t$  is the time-dependent bed capacity ( $\text{mg L}^{-1}$ ), defined as follows:

$$191 \quad N_t = N_o \frac{t}{t + a^{**}} \quad (3)$$

192 Where  $t$  is the filter empty bed contact time (EBCT) (min),  $N_o$  is the maximum adsorption  
 193 capacity of the filter bed per unit volume of filter medium ( $\text{mg L}^{-1}$ ), and  $a^{**}$  is a model constant  
 194 sometimes referred to as the ‘time of relaxation’, i.e. the time taken for the sorbent to reach  
 195 half of its adsorption capacity.

196 Rearranging Eqn. 2, we get an expression for  $C_b$  at any filter loading,  $V$ :

$$197 \quad C_b = \frac{C_o}{1 + e^{-k(C_o t_b - N_t \frac{Z}{U})}} \quad (4)$$

198 Eqn. 4 may also be rearranged to determine the time at which a given breakthrough  
 199 concentration will occur:

$$200 \quad t_b = \frac{N_t Z}{C_0 U} - \frac{1}{k C_0} \ln \left( \frac{C_0}{C_b} - 1 \right) \quad (5)$$

201 Assuming that the influent concentration remains constant, the total mass of P loaded onto the  
 202 adsorption column can be defined as follows:

$$203 \quad \text{mass loading} = \int_0^V C_o dV \quad (6)$$

204 The total mass of P lost in filter effluent can be defined as the integral from 0 to  $t_b$  of Eqn. 4:

$$205 \quad \int_0^{t_b} C_b dV = \frac{\ln \left( e^{C_o k t_b + e^{\frac{k N_t Z}{U}}} \right)}{k} - \frac{\ln \left( 1 + e^{\frac{k N_t Z}{U}} \right)}{k} \quad (7)$$

206 Given that, at the time of breakthrough, the volume treated can be defined as  $V = t_b * Q$  (where  
 207  $Q$  is the loading rate in  $L \text{ hr}^{-1}$ ). The total mass retained by the filter medium,  $q_t m$ , can therefore  
 208 be described by making this substitution and subtracting Eqn. 7 from Eqn. 6:

$$209 \quad q_t m = C_o t_b Q - \frac{\ln \left( e^{C_o k t_b + e^{\frac{k N_t Z}{U}}} \right)}{k} + \frac{\ln \left( 1 + e^{\frac{k N_t Z}{U}} \right)}{k} \quad (8)$$

210  $q_t$  can be determined dividing both sides of Eqn. 8 by the filter medium mass,  $m$ , which after  
 211 some simplification yields:

$$212 \quad q_t = \frac{C_o t_b Q}{m} - \frac{\ln \left( \left( e^{C_o k t_b + e^{\frac{k N_t Z}{U}}} \right) \left( 1 + e^{\frac{k N_t Z}{U}} \right) \right)}{m k} \quad (9)$$

214 The second model, proposed by Callery and Healy (2017), is best suited to the description of  
 215 non-sigmoidal, convex to linear breakthrough curves of the type often observed in flow-  
 216 through studies using low-cost adsorbents. This model is as follows:

$$217 \quad C_b = C_o - \frac{q_t m}{V B} \quad (10)$$

218 Where B is a model constant and  $q_t$  is an EBCT-dependent term for the mass of P adsorbed at  
 219 a filter loading of V per unit mass of filter bed, described by:

$$220 \quad q_t = A V_b \left(\frac{1}{B}\right) \frac{t}{t+a^{**}} \quad (11)$$

221 Where A is a model constant of proportionality (with units  $\text{mg g}^{-1} \text{L}^{-(1/B)}$ ) and  $V_b$  is the number  
 222 of empty bed volumes filtered. As with Eqn. 9, Eqn. 11 may be rearranged to find the filter  
 223 loading, V, at which any given breakthrough concentration occurs:

$$224 \quad V = \frac{q_t M}{B(C_o - C_t)} \quad (12)$$

225 Substituting Eqn. 11 for  $q_t$  in Eqn 12, we obtain the following:

$$226 \quad V = \left( \frac{B(C_o - C_e)(t+a^{**})}{V_x^{-\left(\frac{1}{B}\right)} A M t} \right)^{\frac{B}{1-B}} \quad (13)$$

227 Where  $V_x$  is the volume of the filter bed.

228 With experimental values for  $q_t$  determined from Eqn 1, an attempt was made to fit both Eqn.  
 229 9 and Eqn. 11 to the data using nonlinear regression. Values for the model were obtained using  
 230 the Solver tool in Microsoft Excel by minimizing the value obtained by the sum of the squared  
 231 errors (ERRSQ) function:

$$232 \quad ERRSQ = \sum_{i=1}^n (q_{t,i,meas} - q_{t,i,calc})^2 \quad (14)$$

233 Where  $q_{t,i,meas}$  is the measured value of  $q_t$  obtained using Eqn. 9, and  $q_{t,i,calc}$  is the value of  $q_t$   
234 predicted by either Eqn. 9 or Eqn. 11.

235 The mean percentage error (MPE), which can be negative or positive depending on whether  
236 the model over- or underestimated experimental values respectively, was also calculated by:

$$237 \quad MPE = \frac{100\%}{n} \sum_{i=1}^n \frac{q_{e,i,meas} - q_{e,i,calc}}{q_{e,i,meas}} \quad (15)$$

238 The MPE provides a more an intuitive metric of goodness of model fit, providing information  
239 both on how closely a model fits experimental data, as well as whether modeled values over-  
240 or underestimate actual values. An Excel template is provided in the Supplementary Material.

241 Using the model coefficients obtained from the small-scale columns, the performance of full-  
242 scale pilot filters could be estimated with either Eqn. 5 or Eqn.13, depending on which of  
243 Eqn. 9 or Eqn. 11 best fit the small-scale data. If using Eqn. 5, the volume at which a given  
244 breakthrough concentration,  $C_b$ , occurs may be found by multiplying  $t_b$  by the volumetric  
245 flow through the large-scale filter. If using Eqn. 13, the volume at breakthrough is found by  
246 using the mass of adsorbent in the large-scale column in the place of 'M', and the volume of  
247 the large-scale column in the place of  $V_x$ .

248

### 249 **3. Results and Discussion**

#### 250 **3.1 Phosphorus retention**

251 Graphs of P retention against filter column loading can be seen in Figure 1. When filtering  
252 forestry runoff, the maximum amount of P retained by Al-WTR and CC was 0.349 and 0.142  
253 mg g<sup>-1</sup>, respectively, and when filtering DW, the maximum amount of P removed by Al-WTR  
254 and crushed concrete was 3.673 and 1.333 mg g<sup>-1</sup>, respectively. These values do not represent  
255 saturated adsorption capacities, and had the columns been loaded for longer, further adsorption  
256 would almost certainly have taken place; however, 24-36 hr of loading was sufficient to fit  
257 Eqns. 9 and 11 to experimental data (Callery et al., 2016). Though Al-WTR displayed a higher  
258 overall adsorption capacity when filtering forestry runoff, it also displayed a faster  
259 breakthrough (assuming breakthrough to be when the column effluent reaches 10% of the  
260 influent concentration (Ahmad and Hameed, 2010; Netpradit et al., 2004)). Crushed concrete's  
261 high performance upon initial loading of the filters would seem to suggest that processes other  
262 than only adsorption (e.g. precipitation of phosphate with metals) may be playing an important  
263 role early in the reaction, even though the material had a lesser adsorption capacity overall.  
264 Similar observations were made by Molle et al. (2003), who found that rapid dissolution of  
265 lime in CC and associated increases in pH and increased Ca<sup>2+</sup> concentrations resulted in strong  
266 P precipitation in CC filters, followed by a sudden decrease in P removal rates once dissolution  
267 decreased. While P may bind to both Al and Ca surfaces in Al-WTR (with multiple sorption  
268 mechanisms possible even at low solution concentrations (Zohar et al., 2018)), at the observed  
269 pH range observed while treating both forestry runoff and DW (7.31±0.36 and 7.87±0.23  
270 respectively), sorption onto amorphous Al hydroxides, which has previously been observed to  
271 be the dominant removal mechanism at a pH of ~7.5 (Massey et al., 2018), was likely  
272 responsible for the majority of P removal. The slightly alkaline pH of the solution may,

273 however, potentially have negatively affected the sorption capacity of these Al surfaces (Zohar  
274 et al., 2018).

275 The sorbent saturation,  $q_t$ , for each sorbent-wastewater combination was modeled using Eqn.  
276 9 (the modified Bohart-Adams equation) and Eqn. 11 (the Callery-Healy model). When used  
277 to treat forestry runoff, it was found that crushed concrete's performance could be best modeled  
278 by Eqn. 9, which fit the experimental data with an MPE of 1.02% (Table 1). Eqn. 11 offered a  
279 better fit to experimental data from Al-WTR used to treat forestry runoff, concrete used to treat  
280 DW, and Al-WTR used to treat DW, with modeled values having MPEs of -4.03%, -0.27%,  
281 and -2.69%, respectively (Table 1). The negative MPEs obtained when fitting Eqn. 11 to  
282 experimental data indicate that model predictions were, on average, slightly higher than  
283 observed values, and the positive MPE obtained when fitting Eqn. 9 to experimental data  
284 indicated that model predictions were, on average, slightly higher; this indicates that both  
285 models tended to slightly under- or overestimate the adsorption capacity of each medium,  
286 though not significantly.

287

### 288 **3.2 Metals release**

289 In general, there was no significant release of metals from either media when filtering DW.  
290 However, both media released small amounts copper (Cu) and aluminum (Al) when filtering  
291 forestry runoff, and Al-WTR also released a very small amount of nickel ( $0.16 \mu\text{g g}^{-1}$ ). Figure  
292 2 shows the relationship between cumulative metals release from the 0.4 m columns and filter  
293 loading for each filter medium/wastewater combination studied. The total cumulative metal  
294 loss from 0.4 m filter columns of each filter medium/wastewater combination investigated is  
295 summarized in Table 2, and Table 3 shows metal concentrations in the influent to the filter

296 columns alongside maximum metal concentrations measured in the effluent from the 0.4 m  
297 filter columns. Of the metals tested, Mn, Cu and Fe were above the guide values for surface  
298 water intended for the abstraction of drinking water (S.L.549.21, 2002)

299 When CC was used to filter forestry runoff, there was an initial small release of chromium (Cr)  
300 and lead (Pb) - though there was subsequent uptake of these metals, resulting in net removal  
301 over the course of the experiment; manganese (Mn), zinc (Zn), and iron (Fe) were removed  
302 from the influent. There was a net removal of all metals tested when CC was used to filter DW,  
303 though there was an initial period where concentrations of Cu and Cr were slightly elevated.  
304 When filtering forestry water, Al-WTR released small amounts of Mn, though subsequent  
305 uptake resulted in net removal of Mn from the influent. Al-WTR also removed Cr, Zn, Pb, and  
306 Fe from the forestry runoff influent, and though Al was released initially, filter effluent  
307 concentrations quickly leveled off, indicating that a state of equilibrium had been reached, and  
308 further release was unlikely. Al-WTR removed all of the metals tested from DW, though there  
309 was an initial small release of nickel prior to this uptake.

310 A commonly observed phenomenon for all media was that there was a brief initial period of  
311 metal release for many metals. This was often followed either by a cessation in further release  
312 or often even subsequent uptake by the media, resulting in a net removal of metals over the  
313 duration of filter loading. This suggests that an initial washing of the filter media would be  
314 highly advisable to rinse off any loosely bound media particles and easily solubilized metals.  
315 This would likely help to prevent or significantly reduce any release of metals associated with  
316 extreme pH values and loss of particulate matter.

### 317 **3.3 Filter effluent pH**

318 Differences between filter influents and effluents were more pronounced for filters treating  
319 forestry runoff compared to filters treating DW (Figure 3). This was unsurprising, as the

320 forestry runoff was collected from a blanket peat catchment in an area which is known to have  
321 surface waters with low alkalinity and poor buffering capacities (Johnson et al., 2008). Crushed  
322 concrete initially raised the pH of the forestry runoff from an average of  $6.62 \pm 0.11$  to a  
323 maximum value of 11.18. The magnitude of this increase in pH depended greatly on the contact  
324 time between the runoff and the CC, with shorter columns showing slightly higher changes and  
325 a quicker leveling off of pH values. After the full duration of filter loading had elapsed, the  
326 effluent from the 0.1 m, 0.2 m and 0.3 m filter columns had dropped below the upper  
327 recommended surface water environmental quality standard (EQS) for pH (EPA, 2001) of 9,  
328 and the pH of the effluent from the 0.4 m column was only slightly above this level. This  
329 indicates that approximately 240 bed volumes of wastewater had to pass through the crushed  
330 concrete filter medium prior to pH reaching acceptable levels ( $\text{pH} \leq 9$ ); this requirement may  
331 preclude its use in the treatment of forestry runoff.

332 Al-WTR, by comparison, had a much smaller impact on the pH of the forestry runoff. Filter  
333 effluents showed an initial decrease in pH compared to that of the filter influent, with a  
334 minimum pH of 5.45 observed - slightly below the lower recommended surface water pH EQS  
335 of 6 (EPA, 2001). The contact time between the Al-WTR and the forestry runoff had a much  
336 less marked effect on observed changes in pH and, excluding the pH measurements from the  
337 first effluent aliquots, the effluent from all columns quickly stabilized to an average pH of  
338  $7.31 \pm 0.36$ .

339 When treating DW, effluent from both crushed concrete and Al-WTR stabilized very quickly  
340 at pHs of  $8.00 \pm 0.10$  and  $7.87 \pm 0.23$ , respectively (excluding the pH values from the first  
341 aliquot). All effluent from filters treating DW was within the recommended surface water EQS  
342 range of 6 to 9, though still raised compared to the influent pH of the DW which was measured  
343 to be  $6.96 \pm 0.04$ .

### 344 3.4 Potential for use in full-scale filters

345 Figure 4 shows indicative lifespans of full-scale filters to a breakthrough concentration of 10%  
346 filter influent concentration; these were prepared using Eqns. 5 and 12, depending on which  
347 best modeled filter medium performance in the small-scale column tests. The charts show  
348 design curves for filters of bed depths ranging from 0.5 m to 2 m, although curves for  
349 intermediate bed depths may be interpolated, if desired. The purpose of these charts is to give  
350 the user the ability to roughly estimate filter medium longevity and filter performance over the  
351 course of a hypothetical filter's lifespan, thus aiding with the decision of proceeding to field-  
352 scale trials. As would be expected, the charts indicated that increases in filter depth could be  
353 expected to result in increases in filter life span. The high P adsorption affinity of CC led to  
354 greater adsorption at low concentrations, meaning CC filters would, in theory, have a longer  
355 lifespan when treating forestry runoff.

356 In general, lower loading rates could be expected to result in longer filter lifespans due to  
357 increased EBCTs, allowing for more complete treatment. The high adsorption affinity of CC  
358 led to rapid adsorption of P, implying that the HLR applied to CC filters was of less importance  
359 in determining filter lifespan. This is evidenced in the less pronounced curvature of the lifespan  
360 curves for CC in relation to HLR (Figure 4d). Similarly, the high concentration of DW drove  
361 more rapid adsorption of P by Al-WTR, as shown by the less pronounced curvature of the  
362 lifespan curves in Figure 4d.

363

364 As evident from comparison of Figure 4c and Figure 4d, the lifespans of filters utilizing Al-  
365 WTR to treat DW could be expected to be much greater than those of filters containing CC.  
366 This indicates that field-scale testing of Al-WTR could be expected to yield much better results  
367 than field-scale testing of CC.

368

369 Characterization methodologies like the one described in this paper are a critical first step in  
370 evaluating novel P-sorbing materials; however, subsequent full-scale in-field testing is still  
371 critical to investigate potential issues concomitant with in-field use, e.g. the formation of  
372 preferential flow pathways, surface clogging, effects of animal activity and vegetation etc.  
373 (Buda et al., 2012).

#### 374 **4. Conclusions**

375 Crushed concrete and aluminum water treatment residual (Al-WTR) were investigated as  
376 potential filter media for use in filters intended to remove dissolved phosphorus from forestry  
377 runoff and dairy wastewater (DW). The primary study findings were as follows:

- 378 • The performance of small-scale adsorption columns could be described with great  
379 accuracy using two recently developed models.
- 380 • Model extrapolations to full-scale filters imply that, for a treatment standard of 90%  
381 phosphorus removal,
  - 382 ○ both crushed concrete and Al-WTR show promise for the treatment of  
383 forestry runoff, though crushed concrete may have slightly greater longevity  
384 as a filter medium due to its higher phosphorus adsorption affinity at low  
385 concentrations;
  - 386 ○ for the treatment of DW, filters containing Al-WTR would, due to its higher  
387 phosphorus adsorption capacity, likely have much greater longevity than  
388 filters containing crushed concrete.
- 389 • When utilized in small-scale filters, crushed concrete and Al-WTR both released  
390 small quantities of aluminum and copper when filtering forestry runoff; filters  
391 containing Al-WTR also released a small amount of nickel. There was no  
392 cumulative metal loss from either media when filtering DW.

393           • Effluent from small-scale filters utilizing crushed concrete to treat forestry runoff  
394           was above recommended EPA environmental quality standards.

395   **Acknowledgement**

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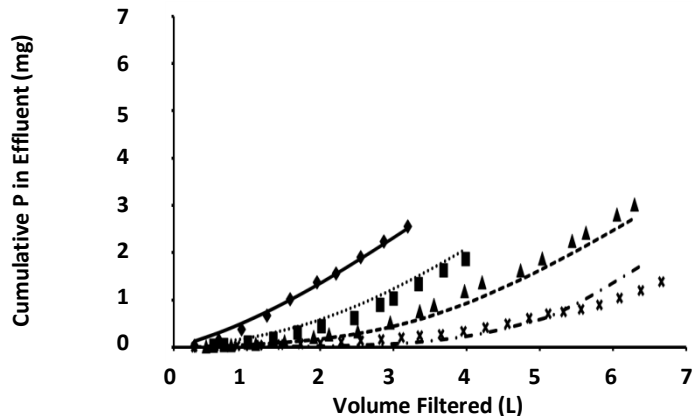
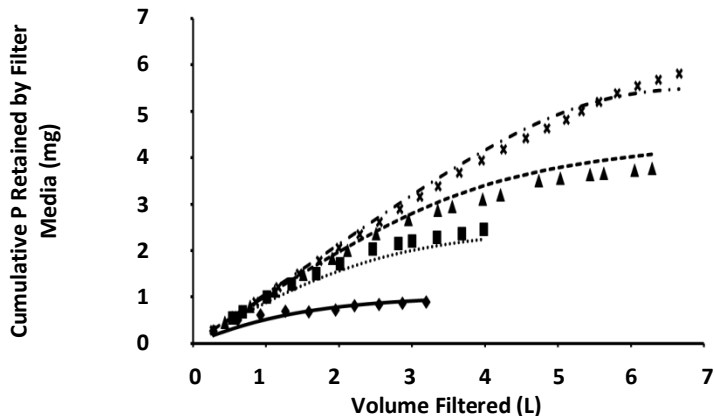
## Figures

**Figure 1.** Phosphorus removal and cumulative phosphorus lost in filter column effluent for various filter depths and wastewater/filter media combinations.

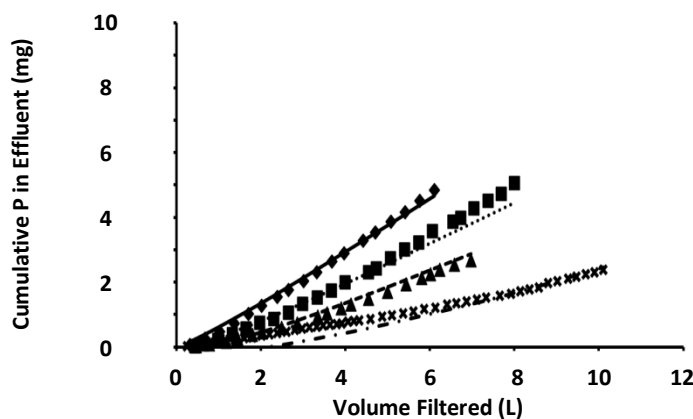
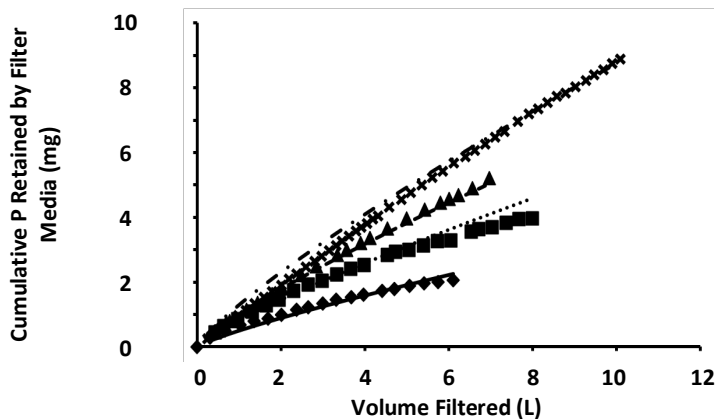
**Figure 2.** Cumulative metal release/uptake by 0.4 m filter columns.

**Figure 3.** Filter column influent and effluent pH and their relationship to EPA recommended environmental quality standards (EPA 2001).

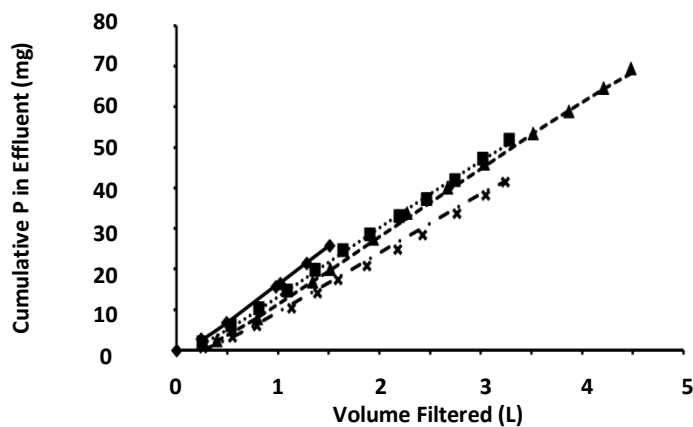
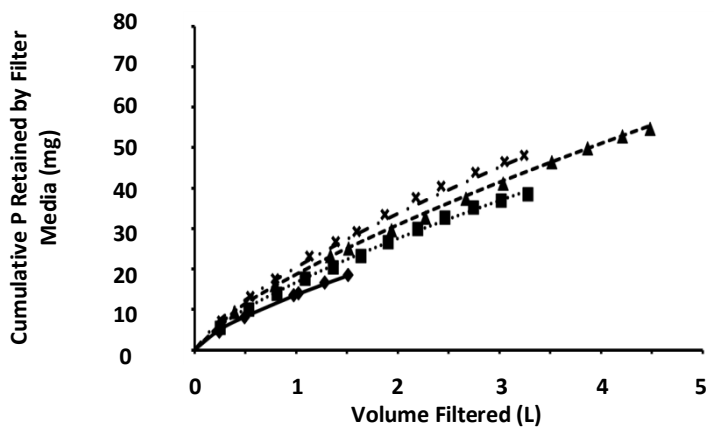
**Figure 4.** Indicative full-scale filter lifespan to effluent breakthrough concentration of 10% filter influent concentration for filter-beds of various depths subject to HLRs of 1-3 m hr<sup>-1</sup>.



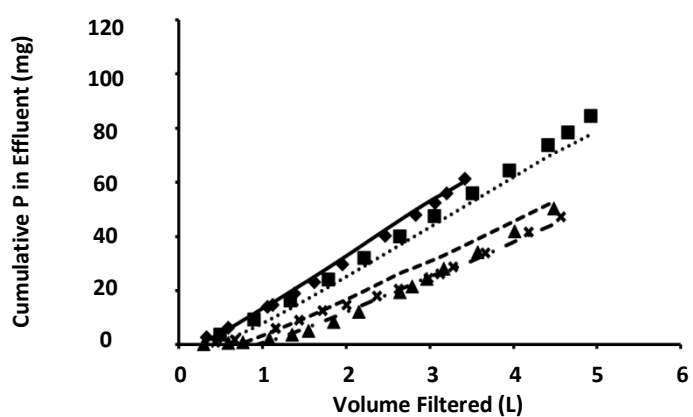
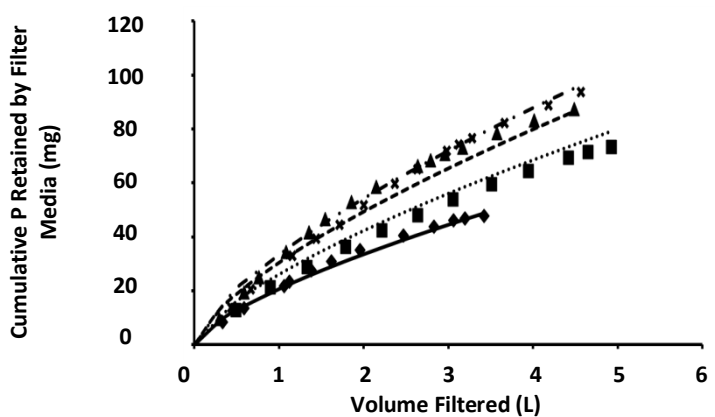
(b) AI-WTR treating Forestry Runoff



(c) Crushed Concrete treating DW



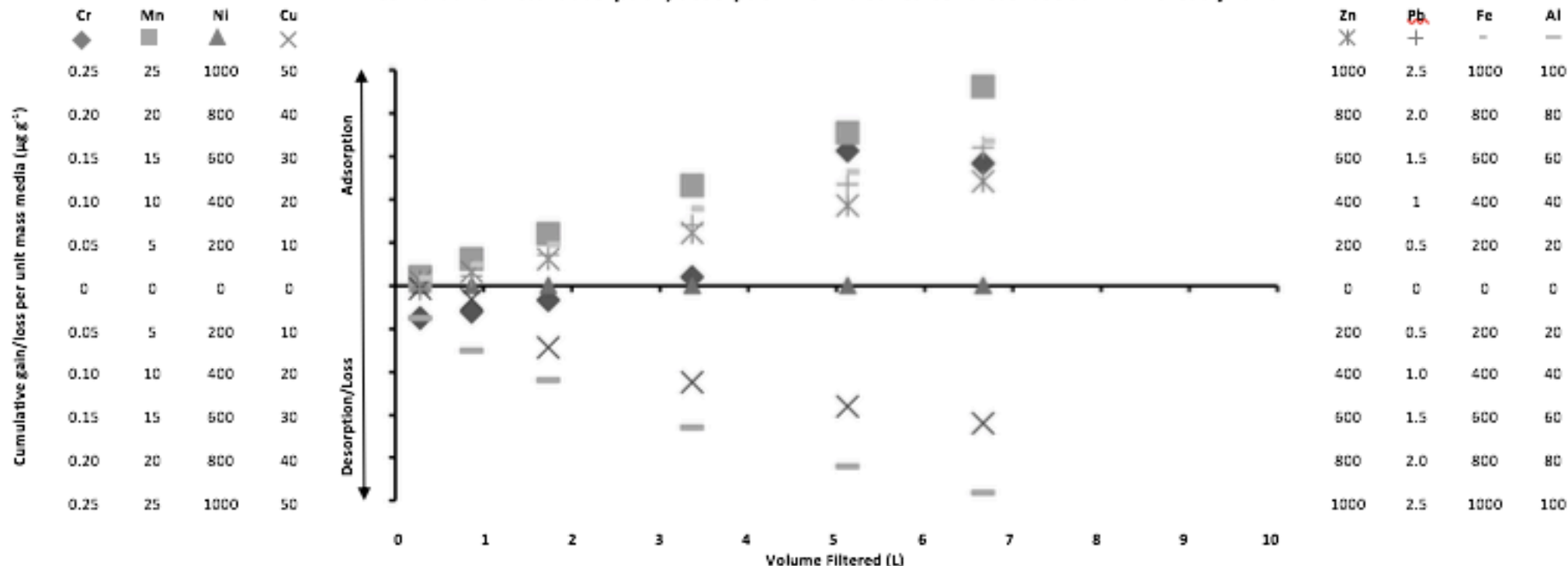
(d) AI-WTR treating DW



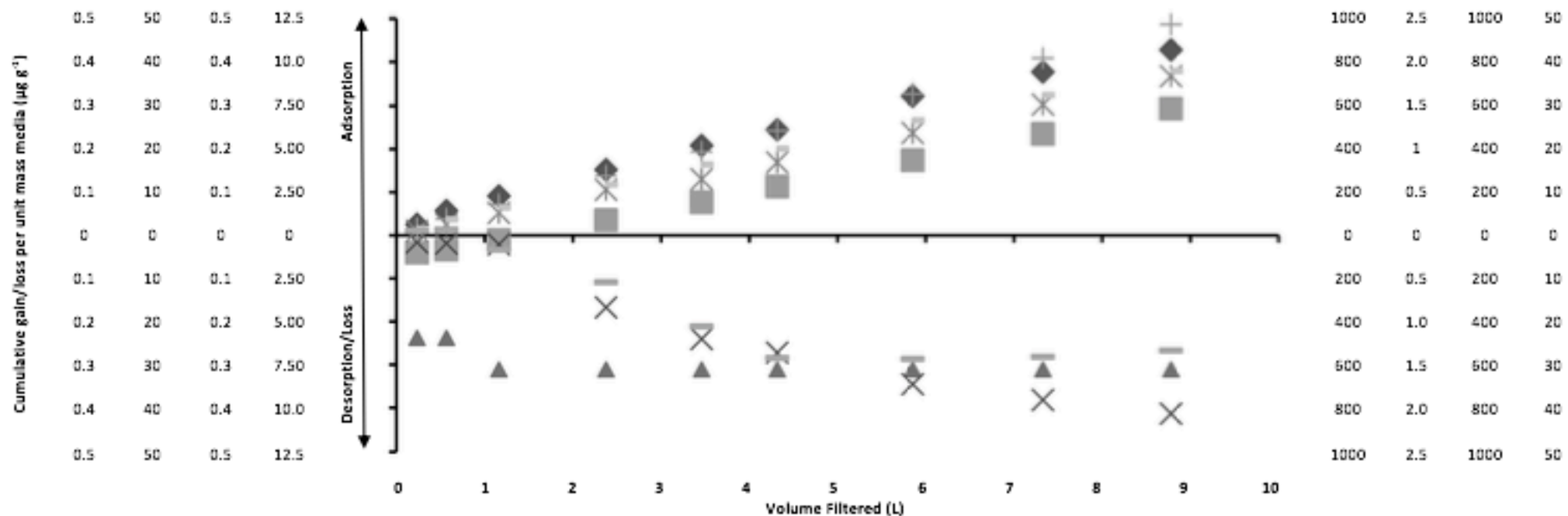
Filter depth:  
Observed:  
Modelled:



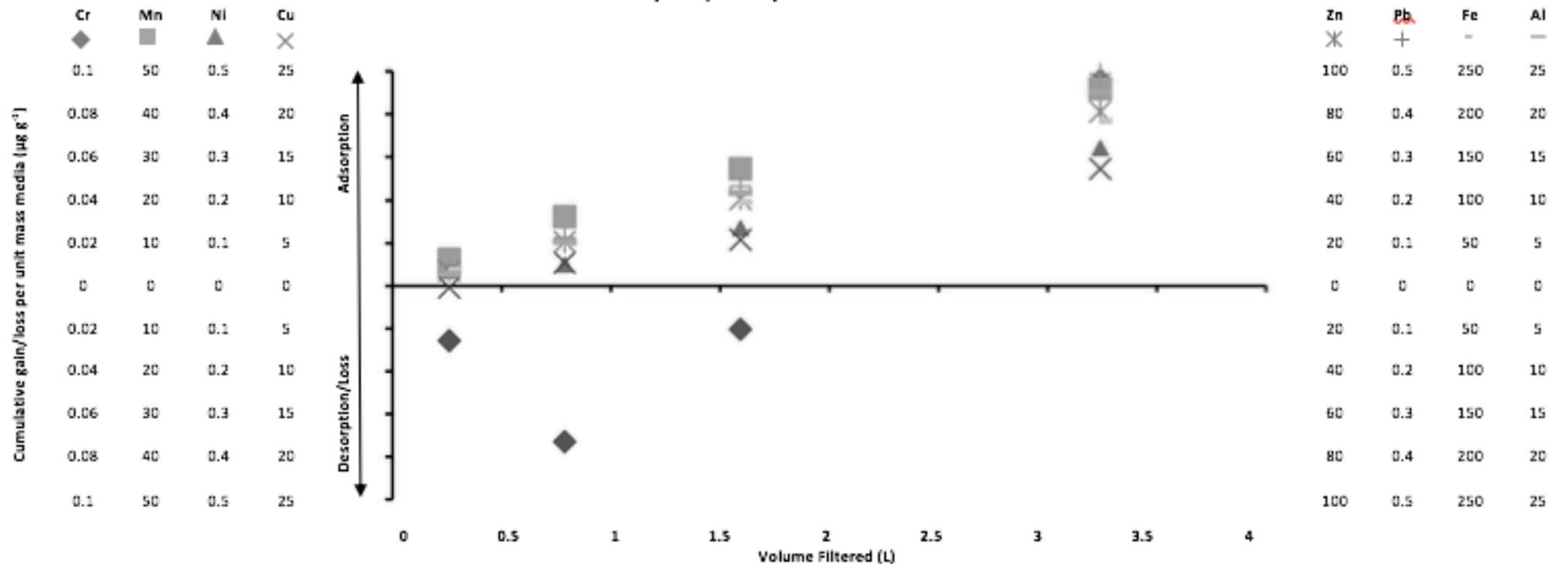
Cumulative metal adsorption/desorption from crushed concrete loaded with forestry runoff



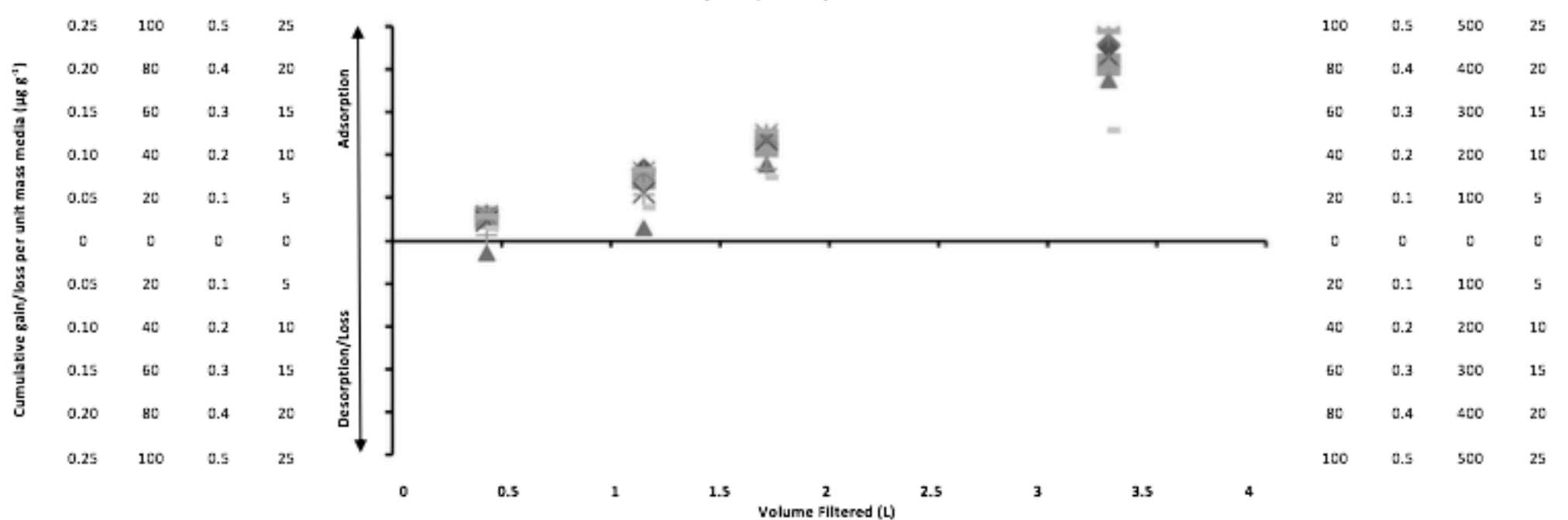
Cumulative metal adsorption/desorption from Al-WTR loaded with forestry runoff

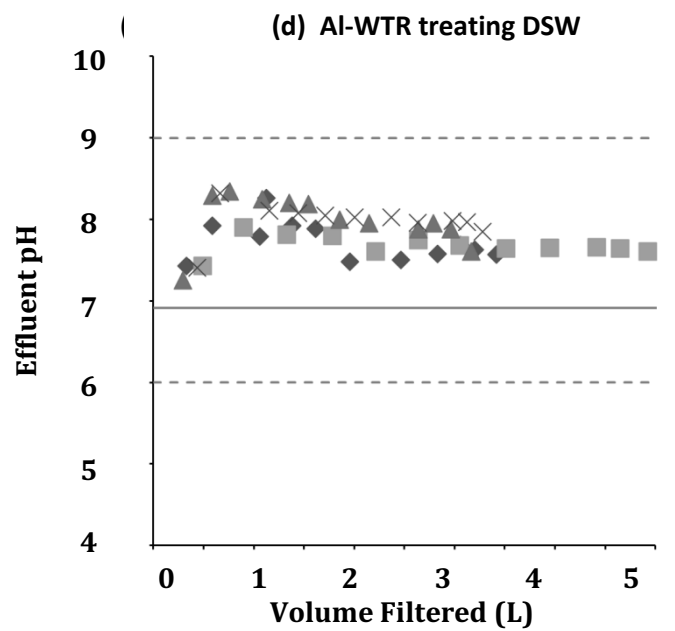
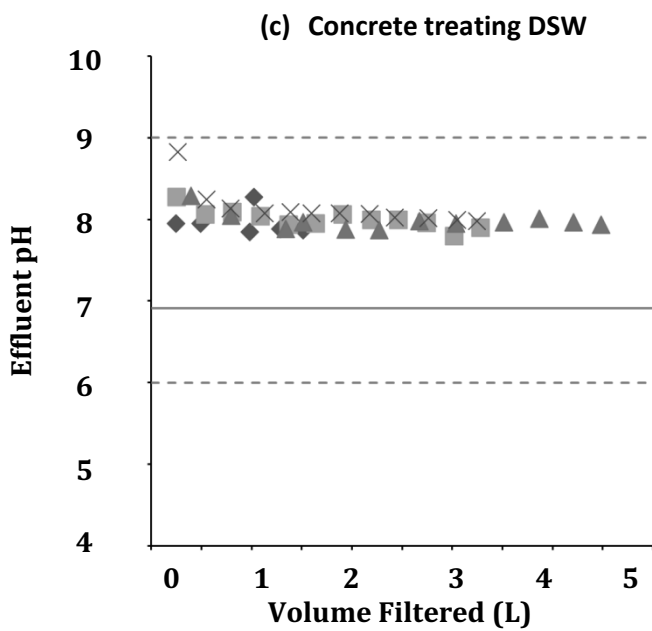
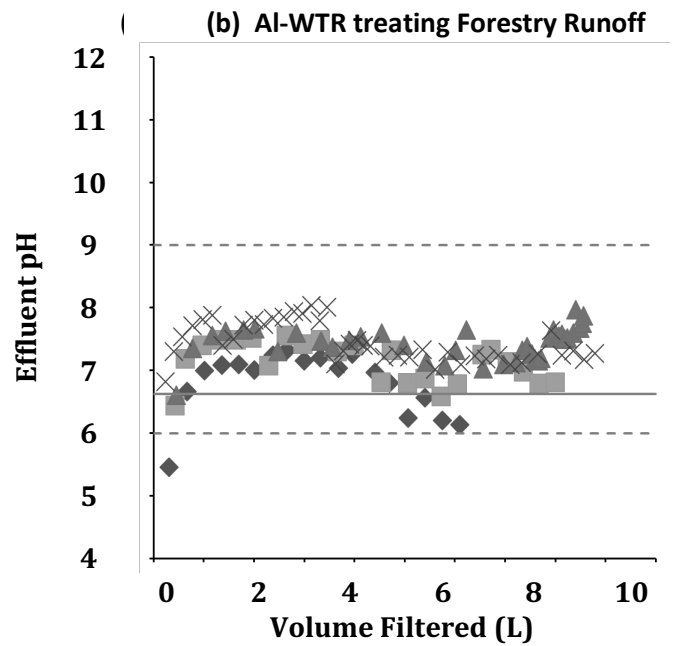
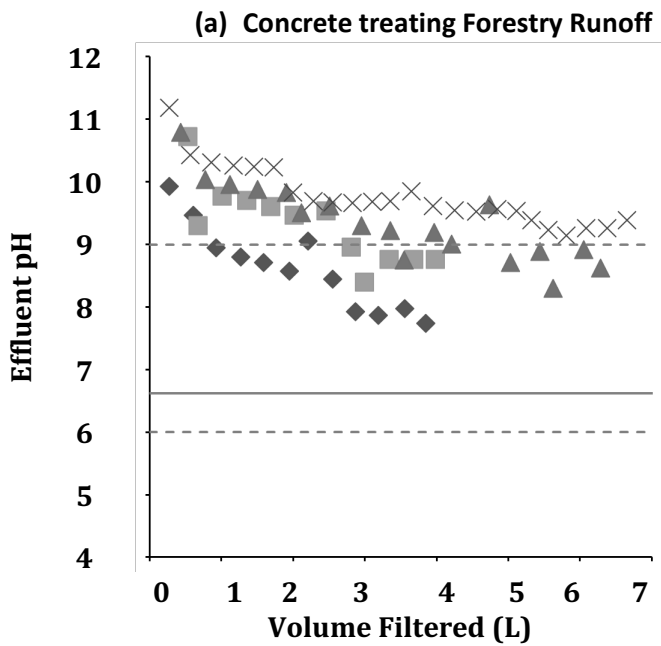


Cumulative metal adsorption/desorption from Crushed Concrete loaded with DSW



Cumulative metal adsorption/desorption from AI-WTR loaded with DSW





Filter depth:                      0.1m                      0.2m                      0.3m                      0.4m

■                      ◆                      ▲                      ×

EPA recommended EQS:    - - - - -

Influent pH:                    \_\_\_\_\_



## **Tables**

**Table 1.** Mean Percentage Errors (MPEs) obtained fitting  $q_t$  predictions obtained using Eqn. 9 and Eqn. 11 to small-scale adsorption column data using minimization of the ERRSQ function (Eqn. 14). Positive MPEs indicate that greater adsorption was predicted by the model than was observed experimentally, and negative MPEs indicate the converse.

**Table 2.** Cumulative release of metals from 0.4m filter columns.

**Table 3.** Maximum observed metal concentrations in filter effluents from 0.4m columns.

1 **Table1**

<b>MPE of modelled vs. observed data</b>				
	Forestry Runoff		DSW	
	Concrete	AI-WTR	Concrete	AI-WTR
Equation 9	<b>1.02%</b>	-14.77%	4.01%	-4.09%
Equation 11	-5.24%	<b>-4.03%</b>	<b>-0.27%</b>	<b>-2.69%</b>

Values in bold indicate lowest MPE

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39 **Table 2**

		<b>Cumulative metals release (<math>\mu\text{g g}^{-1}</math> filter media)</b>								
		Cr	Mn	Ni	Cu	Zn	Cd	Pb	Fe	Al
Forestry	CC	-	-	-	31.90	-	-	-	-	96.17
Runoff	Al-WTR	-	-	0.16	5.25	-	-	-	-	13.63
DSW	CC	-	-	-	-	-	-	-	-	-
	Al-WTR	-	-	-	-	-	-	-	-	-

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46 **Table 3**

		<b>Maximum filter influent/effluent concentrations (<math>\mu\text{g L}^{-1}</math>)</b>								
		Cr	Mn	Ni	Cu	Zn	Cd	Pb	Fe	Al
Forestry Runoff	Influent	2.1				1888.0	0.0			
		4	96.37	0.00	30.26	2	0	7.05	3230	194
	CC	<b>5.6</b>			<b>356.8</b>		0.0			
		<b>0</b>	9.72	0.00	<b>4</b>	43.76	0	<b>10.96</b>	829	<b>1560</b>
Al-WTR	1.4	<b>432.1</b>				0.0				
	2	<b>4</b>	<b>20.25</b>	<b>93.45</b>	921.24	0	1.47	1630	<b>389</b>	
DSW	Influent	3.3	671.7		326.6		0.0			
		6	4	11	6	825.05	0	5.49	2690	241
	CC	<b>5.8</b>	382.8		<b>344.3</b>		0.0			
		<b>2</b>	3	10.3	<b>1</b>	193.15	0	2.12	1220	109
Al-WTR	2.2	169.6		229.1		0.0				
	6	9	<b>12.35</b>	9	242.58	0	4.89	1270	102	

Values in bold indicate elevated metal concentrations in filter effluent

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