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Laboratory Comparison of Four Iron-Based Filter Materials for Water Treatment of Trace Element Contaminants

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45 ABSTRACT

46 A laboratory investigation was conducted to evaluate four iron-based filter materials for
47 trace element contaminant water treatment. The iron-based filter materials evaluated were zero
48 valent iron (ZVI), porous iron composite (PIC), sulfur modified iron (SMI), and iron
49 oxide/hydroxide (IOH). Only filter material retained on a 100 mesh sieve (> 0.15 mm) was used
50 for testing. Tested trace element contaminant solutions combined As, Cr, and Se (added as
51 AsO_4^{3-} , CrO_4^{2-} , and SeO_4^{2-} , respectively) or combined Cd^{2+} , Cu^{2+} , and Pb^{2+} . The laboratory
52 investigation included saturated falling-head hydraulic conductivity tests, contaminant removal
53 batch tests, and low-to-high flow rate saturated solute transport column tests. Hydraulic
54 conductivity test results indicate that all four iron-based filter materials have sufficient water
55 flow capacity as indicated by saturated hydraulic conductivity values greater than 1×10^{-2} cm/s.
56 Essentially 100% of each trace element contaminant (As, Cd, Cr, Cu, Pb, and Se) was removed
57 by SMI during batch and column tests, while IOH exhibited good removal of each trace element
58 except Se. The combined batch and column test results showed ZVI and PIC were most capable
59 of treating Cd, Cr, Cu, and Pb. Consequently, ZVI, PIC, SMI, and IOH all have substantial
60 potential for trace element contaminant water treatment.

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62 **Keywords:** Trace Elements, Iron-Based Filter Materials, Water Treatment

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68 **1. Introduction**

69 Natural process along with agricultural, mining, and manufacturing activities can result in
70 the environmental release of trace element contaminants which degrade surface and ground
71 waters [1]. Often, more than one trace element contaminant is found to be present in these
72 degraded waters. For example, waste disposal sites for fly ash generated by coal-burning power
73 plants can leach arsenic and selenium into the environment [2-4]. High levels of both arsenic
74 and selenium can also be found in evaporation ponds that collect agricultural drainage waters in
75 the San Joaquin Valley in California, U.S.A [5-7]. Chromated copper arsenate (CCA) wood
76 treatment facilities or landfills where discarded CCA treated wood is deposited can pose arsenic,
77 chromium, and copper contamination risks to underlying ground waters [8-10]. Mobile,
78 leachable forms of arsenic, chromium, and selenium are found in wildfire affected soils [11].
79 Acid mine drainage from mining locations can lead to heavy metal pollution of surface waters.
80 As a case in point, during the early 1980's, acid mine drainage from the Yak tunnel at the
81 California Gulch mining site in Colorado, U.S.A. discharged a combined total of 210 tons per
82 year of cadmium, copper, iron, lead, manganese, and zinc into California Gulch, which is a
83 tributary to Arkansas River [12].

84 This study focused on the trace element contaminants; arsenic (As), cadmium (Cd),
85 chromium (Cr), copper (Cu), lead (Pb), and selenium (Se). The most mobile types of As found
86 in the environment are forms of arsenate ($H_nAsO_4^{3-n}$) and arsenite ($H_nAsO_3^{3-n}$) [13,14]. With
87 regard to Cr in natural waters, the chromate forms, $HCrO_4^-$ and CrO_4^{2-} , are some of the most
88 common mobile species [13,14]. The environmentally mobile types of Se are forms of selenate
89 ($HSeO_4^-$ and SeO_4^{2-}) and selenite ($H_nSeO_3^{2-n}$) [13]. Within soils, surface waters, and ground

90 waters, mobile Cd, Cu, and Pb are often present as divalent cations (i.e. Cd^{2+} , Cu^{2+} , and Pb^{2+})
91 [13,14].

92 Iron-based materials, such as elemental iron (i.e. zero-valent iron) and iron
93 oxides/hydroxides, have been evaluated for water treatment of trace element contaminants.
94 Zero-valent iron (ZVI) has been employed within permeable reactive barriers to remove trace
95 element contaminants from ground water [15]. Iron oxides/hydroxides (IOH) have been used by
96 municipalities and in the home for filtration water treatment of trace element contaminants,
97 particularly arsenic [16,17]. The mechanisms by which ZVI removes trace element
98 contaminants from water are complex, and depending on the contaminant species involved, can
99 include either individually or in combination; electrochemical reduction in association with iron
100 oxidation [18-22], chemical precipitation [18, 23, 24], and inner-sphere or outer-sphere surface
101 complexation on iron corrosion products (iron oxides/hydroxides) [19-22,25]. The mechanisms
102 by which IOH removes trace element contaminants from water are likewise complex, and again
103 depending on the contaminant species involved, can include either individually or in
104 combination; electrochemical reduction in association with oxidation of ferrous iron to ferric iron
105 [26] and inner-sphere or outer-sphere surface complexation on IOH particles [8, 26-29]. Two
106 recently developed iron-based materials, porous iron composite (PIC) and sulfur modified iron
107 (SMI), may also have filter treatment potential for waters contaminated with trace elements.
108 Research on PIC and SMI water treatment capabilities have to date been very limited. The trace
109 element contaminant removal mechanisms for PIC and SMI have not yet been identified, but are
110 likely similar to the ZVI and IOH trace element contaminant removal mechanisms.

111 There has been little direct trace element filter treatment comparison of ZVI versus IOH,
112 and especially, ZVI or IOH versus PIC or SMI. Consequently, for the purpose of providing a

113 preliminary comparison of ZVI, PIC, SMI, and IOH in regard to trace element contaminant filter
114 treatment potential, a laboratory investigation was carried out with saturated falling-head
115 hydraulic conductivity tests, contaminant removal batch tests, and low-to-high flow rate
116 saturated solute transport column tests. In accordance with previous studies indicating the
117 common occurrence of mixed trace element contaminant release into the environment [2-12],
118 solutions with As, Cr, and Se species combined together and solutions with Cd, Cu, and Pb
119 species combined together were both utilized in the batch tests and the column tests. The stated
120 research objective was therefore to conduct a preliminary laboratory comparison of zero-valent
121 iron, porous iron composite, sulfur modified iron, and iron oxide/hydroxide filter materials with
122 respect to hydraulic efficiency and mixed trace element contaminant water treatment capabilities.

123 **2. Materials and methods**

124 *2.1. Iron-based water filtration products*

125 Four iron-based water filtration products were evaluated in this investigation. The first
126 product, zero valent iron, designated as ZVI, was obtained from Connelly-GPM, Inc. (Chicago,
127 Illinois - Product Name: CC-1190), and can be described as a ground iron aggregate comprised
128 of shavings from cast iron borings. The second product, porous iron composite, designated as
129 PIC, was obtained from North American Höganäs, Inc. (Hollsopple, Pennsylvania - Product
130 Name: Cleanit® LC), and is manufactured with high purity elemental iron powder to which
131 functional additives are mixed. The mixture was then is processed through powder metallurgical
132 technologies to achieve an iron composite media with high internal porosity where functional
133 groups are evenly distributed in either free or partially alloyed forms. The third product, sulfur
134 modified iron, designated as SMI, was obtained from SMI_PS, Inc. (Walnut Creek, California),
135 and can be described as a hydrogen reduced, high surface area iron powder that has been

136 additionally modified through chemical reaction with pure sulfur to produce sulfur/iron
137 compound surface coatings on the iron particles. The fourth product, an iron oxide/hydroxide,
138 designated as IOH, is manufactured by LANXESS AG (Leverkusen, Germany: Product Name -
139 Bayoxide® E33), and can be described as a synthetic, high surface area goethite (Chemical
140 Formula - α -FeOOH). To improve hydraulic conductivity, fine-grained particles passing
141 through a 100 mesh sieve (particle size < 0.15 mm) were removed from the ZVI, PIC, SMI, and
142 IOH filter materials that were tested in this investigation.

143 Properties of the ZVI, PIC, SMI, and IOH filter materials are provided in Table 1. The
144 pH and oxidation/reduction potential (ORP) were measured on 1:2 by weight slurry mixtures of
145 filter material to deionized water. Table 1 shows that, with respect to pH, the ZVI and PIC
146 slurries were strongly alkaline (> 10), the IOH slurry was moderately alkaline (8 - 10), and the
147 SMI slurry was near neutral (6 - 8). The ZVI, PIC, and SMI had ORP values less than -500 mV,
148 indicating that ZVI, PIC, and SMI would likely produce very low redox conditions within a filter
149 treatment system environment. The laboratory measured specific gravity [30] of IOH was 3.62
150 g/cm³, which was much lower than the specific gravity values of 7.03, 7.13, and 5.95 g/cm³ that
151 were obtained, respectively, for ZVI, PIC, and SMI. Table 1 particle size analysis data, based
152 mechanical sieving methods [30] and a particle size classification from Perloff and Baron [31],
153 shows that the 100 mesh sieved ZVI and IOH filter materials were predominantly comprised of
154 medium sand sized particles, while the 100 mesh sieved PIC and SMI filter materials were
155 predominantly comprised of fine sand sized particles.

156 2.2. Test solutions

157 The constituent and chemical properties of the test solutions used in this study are
158 provided in Table 2. The hydraulic conductivity test solution, the As-Cr-Se batch test solution,

159 and the As-Cr-Se column test solution all had pH values near neutral, while the Cd-Cu-Pb batch
160 test solution and the Cd-Cu-Pb column test solution were strongly acidic. The ORP values were
161 well above zero for all test solutions. For the As-Cr-Se batch and column test solutions, arsenic
162 (As) was initially added as the oxyanion, AsO_4^{3-} , chromium (Cr) was initially added as the
163 oxyanion, CrO_4^{2-} , and Se was initially added as the oxyanion SeO_4^{2-} ; however, once solution
164 equilibrium was achieved, As was likely present in some form of arsenate ($\text{H}_n\text{AsO}_4^{3-n}$) or
165 arsenite ($\text{H}_n\text{AsO}_3^{3-n}$), Cr was likely present in some form of chromate (HCrO_4^- or CrO_4^{2-}), and Se
166 was likely present in some form of selenate (HSeO_4^- or SeO_4^{2-}) or selenite ($\text{H}_n\text{SeO}_3^{2-n}$) [32]. For
167 the Cd-Cu-Pb batch and column test solutions, cadmium (Cd), copper (Cu), and lead (Pb), were
168 likely present as divalent cations, Cd^{2+} , Cu^{2+} , and Pb^{2+} [32]. Trace element concentrations for
169 the batch tests (50 ppm for each contaminant) and column tests (25 ppm for each contaminant)
170 would be considered quite high for degraded surface and ground waters [1,13,14], and as a
171 consequence, serve to provide a rigorous assessment of iron-based filter material water treatment
172 capabilities.

173 *2.3. Saturated falling-head hydraulic conductivity tests*

174 Saturated hydraulic conductivity is the porous media property governing the rate at which
175 water flows through a filter material (e.g. ZVI, PIC, SMI, or IOH) under saturated conditions.
176 To be practical for water treatment use from a hydraulic standpoint, a filter material must have a
177 sufficiently high saturated hydraulic conductivity, at least equal to and preferably much greater
178 than 1.0×10^{-3} cm/s, which is a stormwater sand filter design criteria [33-35]. Standard falling-
179 head hydraulic conductivity tests [36,37] were employed for saturated hydraulic conductivity
180 measurement. Saturated falling-head hydraulic conductivity tests were carried out on 100 mesh
181 sieved ZVI, PIC, SMI, and IOH filter materials at ambient laboratory temperatures ranging from

182 20⁰ to 25⁰ C. For each individual filter material, duplicate saturated falling-head hydraulic
183 conductivity tests were conducted so as to make certain that results were consistent. That is,
184 hydraulic conductivity measurements were obtained from two separately packed columns for
185 each individual filter material. The packed ZVI, PIC, SMI, or IOH filter material columns were
186 15.2 cm in length, 4.1 cm in diameter, and were saturated with 0.01 mole/L CaSO₄ solution 24
187 hours prior to hydraulic conductivity measurement.

188 *2.4. Contaminant removal batch tests*

189 Contaminant removal batch tests were carried out with ZVI, PIC, SMI, and IOH at
190 ambient laboratory temperatures ranging from 20⁰ to 25⁰ C. Control batch tests without filter
191 material, designated NFM, were also carried out to quantify contaminant amounts adsorbed by
192 the experimental apparatus. These contaminant removal batch tests were conducted to provide a
193 preliminary assessment of the potential contaminant removal capabilities of ZVI, PIC, SMI, and
194 IOH, and in turn decide if further saturated solute transport column testing was warranted for any
195 of these filter materials. The repeatability of results for trace element contaminant removal was
196 confirmed by conducting a set of three batch test replicates for each combination of filter
197 material (or NFM) and As-Cr-Se or Cd-Cu-Pb batch test solution . One batch test within each
198 three replicate set was chosen for measurement of pH and ORP within the reaction vessel
199 immediately after batch test completion.

200 In each filter material batch test, 5 g of filter material and 40 g of either As-Cr-Se or Cd-
201 Cu-Pb batch test solution (see Table 2) were combined in a 50 mL polypropylene centrifuge tube
202 (BD Biosciences, Bedford, Massachusetts). The filter material and batch test solution solution
203 were thoroughly mixed by placing the centrifuge tube on a laboratory rotator (Mini LabRoller
204 Rotator, Labnet International, Inc., Woodbridge, New Jersey) operated at 20 rpm. Each batch

205 was then stopped after 24 hours of mixing. Procedures for the control batch tests (NFM) were
206 the same, with only As-Cr-Se or Cd-Cu-Pb batch test solution (40 g), but no filter material,
207 added to the centrifuge tube.

208 Once pH and ORP values were obtained for one batch test within each three replicate set,
209 all batch test centrifuge tubes containing filter material (or NFM) and solution were then
210 centrifuged (Beckman Model TJ-6 Centrifuge, Beckman Coulter, Inc., Fullerton, California) at
211 2500 rpm (800 g) for 15 minutes in order to separate the filter material from the solution. The
212 supernatant solution was then decanted into a second 50 mL polypropylene centrifuge. This
213 process of separating solution from filter material was done for the purpose of discontinuing
214 chemical reactions between the filter material and test solution upon batch test completion.
215 Batch test pH and ORP were measured with equipment previously described (Table 1).
216 Concentrations of As, Cr, and Se or Cd, Cu , and Pb present within the batch test supernatant
217 solutions were measured using inductively coupled plasma (ICP) methods [38] using a Perkin-
218 Elmer (Waltham, Massachusetts) Optima 3300 DV ICP - OES Spectrometer. Trace element
219 contaminant detection limits for this ICP method are As - 0.061 ppm, Cd - 0.001 ppm, Cr - 0.003
220 ppm, Cu - 0.001 ppm, Pb - 0.016 ppm, and Se - 0.054 ppm.

221 *2.5. Low-to-high flow rate saturated solute transport column tests*

222 By allowing control of solution flux and the filter material to solution exposure time,
223 saturated solute transport column tests can help better evaluate the likely trace element
224 contaminant (As, Cd, Cr, Cu, Pb, Se) removal behavior of ZVI, PIC, SMI, or IOH filter materials
225 within an actual water filter treatment system. For these column tests, continuously operated
226 variable flow rate peristaltic pumps (Model 3384 and Model 3386, Control Company,
227 Friendswood, Texas) delivered the initial influent trace element contaminant solution to the

228 bottom inlet of a vertically oriented Teflon[®] permeameter containing a packed filter material (or
229 empty) column. Ambient laboratory temperatures during testing ranged from 20^o to 25^o C.
230 Filter material columns contained within the Teflon permeameters had a diameter of 5.1 cm and
231 a length of 15.0 cm. Effluent exiting the permeameter was collected over time in glass
232 containers. Excepting a small amount of flexible 5 mm ID silicon tubing (Catalog Number 3364,
233 Control Company, Friendswood, Texas) threaded through the peristaltic pump, 6 mm ID Tygon
234 (Saint-Gobain Performance Plastics, Akron, Ohio) R-3603 plastic laboratory tubing was used to
235 deliver solution between the influent supply container, peristaltic pump, permeameter, and
236 effluent sample container. Every other effluent sample (one out of two) was centrifuged to
237 remove any filter material present and then analyzed for As, Cr, and Se or Cd, Cu, Pb using
238 previously described inductively coupled plasma methods [38]. Direct pH measurements were
239 obtained for those effluent samples not analyzed for trace element contaminants (again, one out
240 of every two samples).

241 Table 3 provides information on the ten saturated solute transport column tests conducted
242 during this study. The column test information provided in Table 3 includes filter material (or no
243 filter material), contaminant solution, filter material packing density, column pore volume, flow
244 rate, flux, contact time, and effluent amount. Column tests 1 through 5 were carried out with the
245 As-Cr-Se column test solution (Table 2), while column tests 6 through 10 were conducted with
246 the Cd-Cu-Pb column test solution (Table 2). Column tests 1 and 6 were carried out with empty
247 columns that contained no filter material (NFM). These empty column tests were conducted for
248 baseline comparison purposes in order to quantify the amounts of trace element contaminants
249 lost by the experimental apparatus and procedures. Since trace element contaminant adsorption
250 on experimental apparatus is likely to be greatest under low flow rate conditions, column tests 1

251 and 6 were therefore carried out at one relatively low flow rate of approximately 50 mL/h.
252 Column tests 2 through 5 and 7 through 10 were conducted with the Teflon permeameters
253 packed with ZVI, PIC, SMI, or IOH filter materials. These filter material column tests (2
254 through 5 and 7 through 10) were carried out in two flow rate steps, with an initial longer
255 duration lower flow rate (~ 45 to 60 mL/h) followed by a shorter duration much higher flow rate
256 (> 1500 mL/h). By conducting the filter material column tests (2 through 5 and 7 through 10)
257 with both low and high flow rates, insight was gained on the water filter treatment contact time
258 needed for effective removal of trace element contaminants.

259 **3. Results and discussion**

260 *3.1. Saturated falling-head hydraulic conductivity results.*

261 Saturated hydraulic conductivity measurements are provided in Table 4 for the 100 mesh
262 sieved zero valent iron (ZVI), porous iron composite (PIC), sulfur modified iron (SMI), and iron
263 oxide/hydroxide (IOH) filter materials. Filter material packing densities for the saturated falling-
264 head hydraulic conductivity tests are also provided in Table 4. There was a wide range in filter
265 material packing densities, from an average of 0.52 g/cm³ for IOH to 3.04 gm/cm³ for ZVI. All
266 measured saturated hydraulic conductivity measurements for the four iron-based filter materials
267 were at least an order of magnitude greater than the previously discussed 1.0 x 10⁻³ cm/s
268 hydraulic conductivity criterion used for stormwater sand filters [33-35]. The lowest two-test
269 average hydraulic conductivity values were exhibited by PIC (1.80 x 10⁻² cm/s) and SMI (1.73 x
270 10⁻² cm/s), and these results are probably due in large part to PIC and SMI having a much greater
271 amount of fine sand sized particles (> 75%) than the other two iron-based filter materials. By far
272 the highest average hydraulic conductivity was obtained with IOH (9.75 x 10⁻² cm/s) and is
273 likely the result, at least in part, of IOH having a much greater amount of medium sand sized

274 particles (> 95%) than the other iron-based filter materials tested. Consequently, overall results
275 for the saturated falling-head hydraulic conductivity tests generally indicate that ZVI, PIC, SMI,
276 and IOH all have sufficient hydraulic conductivity for use in water filter treatment.

277 3.2. Contaminant removal batch test results.

278 Results for the contaminant removal batch tests are provided in Table 5 and Table 6. The
279 pH values, ORP (oxidation/reduction potential) values, and trace element contaminant
280 concentrations for the As-Cr-Se (arsenic-chromium-selenium) batch tests are shown in Table 5.
281 Again, for the As-Cr-Se batch test solution, 50 ppm of As was originally added as the oxyanion,
282 AsO_4^{3-} (arsenate), 50 ppm of Cr was added as the oxyanion, CrO_4^{2-} (chromate), and 50 ppm of
283 Se was added as the oxyanion, SeO_4^{2-} (selenate). The pH, ORP, and trace element contaminant
284 concentrations for the Cd-Cu-Pb (cadmium-copper-lead) batch tests are shown in Table 6. For
285 the Cd-Cu-Pb batch test solution, Cd (50 ppm), Cu (50 ppm), and Pb (50 ppm) were again likely
286 initially present as divalent cations, Cd^{2+} , Cu^{2+} , and Pb^{2+} .

287 Table 5 shows that the As-Cr-Se NFM (control - no filter material) and SMI batch tests
288 had near neutral pH (6 - 8), while the As-Cr-Se batch tests with ZVI, PIC, and IOH had alkaline
289 pH values (> 8). Positive ORP values were measured for the As-Cr-Se NFM, PIC, and IOH
290 batch tests. The As-Cr-Se batch tests with ZVI and SMI had negative ORP values, indicating
291 that reducing conditions were present during these batch tests. Based on initial and final
292 contaminant levels, the As-Cr-Se NFM batch tests indicate that batch test equipment and
293 procedures alone remove ~11% of As, ~1% of Cr, and 11% of Se, which in turn must be taken
294 into account when analyzing the As-Cr-Se batch test results for ZVI, PIC, SMI, and IOH. There
295 was complete 100% removal of As, Cr, and Se during the As-Cr-Se batch tests conducted with
296 SMI, at least below detection limits, which were 0.061 ppm for As, 0.003 ppm for Cr, and 0.054

297 ppm for Se. Near 100% of As, ~96% of Cr, and ~52% of Se were removed during the As-Cr-Se
298 batch tests with IOH. The As-Cr-Se batch tests with ZVI had averaged ~41% As removal, ~83%
299 Cr removal, and ~43% Se removal. The results for the As-Cr-Se batch tests with PIC (~23% As
300 removal, ~9% Cr removal, and ~18% Se removal) were only modestly better than results
301 obtained with the As-Cr-Se NFM batch tests. Consequently, the As-Cr-Se batch tests results
302 provided in Table 5 indicate, in a preliminary manner, that SMI may work best for mixed As, Cr,
303 and Se removal, followed closely by IOH, then ZVI, and finally PIC.

304 Table 6 shows that the Cd-Cu-Pb NFM (control - no filter material), PIC, and SMI batch
305 tests had moderately acidic pH (4 - 6), while the Cd-Cu-Pb IOH batch test pH was near neutral
306 (6 - 8), and Cd-Cu-Pb ZVI batch test pH was strongly alkaline (> 10). Positive ORP values were
307 measured for the Cd-Cu-Pb NFM and IOH batch tests. The Cd-Cu-Pb batch tests with ZVI,
308 PIC, and SMI had negative ORP values, indicating that reducing conditions were present during
309 testing. Based on initial and final contaminant levels, the Cd-Cu-Pb NFM batch test results
310 show that batch test equipment and procedures remove ~14% of Cd, ~10% of Cu, and ~3% of
311 Pb. The Cd-Cu-Pb batch tests with ZVI, PIC, SMI, or IOH all had near 100% removal of Cd,
312 Cu, and Pb (detection limits: 0.001 ppm for Cd, 0.001 ppm for Cu, and 0.016 ppm for Pb).
313 Therefore, even taking into account contaminant losses due to batch test equipment and
314 procedures, Table 6 shows that ZVI, PIC, SMI, and IOH all have promise for water treatment of
315 Cd, Cu, and Pb. Preliminarily, these batch test results indicated that further investigation of ZVI,
316 PIC, SMI, and IOH with saturated solute transport column tests was warranted in order to better
317 assess the trace element contaminant water treatment effectiveness and efficiency of these four
318 iron-based filter materials.

319

320 3.3. *Low-to-high flow rate saturated solute transport column test results.*

321 As previously mentioned, these column tests allow control of solution flux and the filter
322 material to solution exposure time; therefore facilitating better assessment of the likely trace
323 element contaminant water treatment capabilities of iron-based filter materials within an actual
324 water filter treatment system. Representative column tests results are displayed in Fig. 1. The
325 compiled effluent pH and effluent contaminant concentration results for all ten column tests are
326 provided in Tables 7 and 8. The influent concentration for each trace element contaminant was
327 25 ppm for the As-Cr-Se column tests and the Cd-Cu-Pb column tests. The likely initial form
328 (species) of the trace element contaminants has already been discussed.

329 Table 7 shows that the effluent pH for the As-Cr-Se SMI column test 4 was generally
330 acidic (< 6), while the As-Cr-Se NFN column test 1 effluent pH was near neutral (6 - 8). The
331 As-Cr-Se IOH column test 5 effluent had near neutral to moderately alkaline pH (6 - 10), and the
332 effluent pH values for the As-Cr-Se ZVI and PIC column tests 2 and 3 were strongly alkaline (>
333 10). With the influent As-Cr-Se column test solution having a near neutral pH of 6.46, it is
334 apparent that ZVI, PIC, SMI, and IOH can significantly impact this contaminated water's pH.
335 Consequently, additional water treatment in conjunction with ZVI, PIC, SMI, and IOH may be
336 required, if the release of alkaline or acid waters into the environment is a potential problem.

337 The Table 7 effluent contaminant levels for As-Cr-Se NFM column test 1 indicates that
338 column test equipment and procedures alone can remove ~12% of As, ~3% of Cr, and ~9% of
339 Se, and these results need to be taken into account when analyzing the As-Cr-Se column test
340 results for ZVI, PIC, SMI, and IOH. The most effective contaminant removal for the As-Cr-Se
341 column tests was exhibited by the SMI filter material. For the As-Cr-Se SMI column test 4,
342 regardless of whether flow rate was low (227 min contact time) or high (8 min contact time),

343 there was no As, Cr, or Se detected in the effluent. The IOH filter material effectively removed
344 As and Cr, but not Se at both low and high flow rates (297 and 6 min contact time, respectively).
345 The ZVI and PIC filter materials, at both high and low flow rates, worked very well removing
346 Cr; however, they were not particularly good at removing As and Se. The chemical reactions
347 between the iron-based filter materials and the As, Cr, or Se species present appear to occur quite
348 rapidly, as was inferred based on the last low flow rate effluent contaminant concentration values
349 being generally similar in magnitude to the high flow rate contaminant concentration values (Fig.
350 1b and Table 7). Furthermore, the Table 7 results indicate that with regard to waters containing a
351 mixture of As, Cr, and Se, the contaminant removal effectiveness of ZVI, PIC, SMI, and IOH is
352 determined primarily by the amount of contaminants over time to which these iron-based filter
353 materials have been exposed.

354 By test completion, the effluent pH for the Cd-Cu-Pb ZVI, PIC, and SMI column tests 7,
355 8, and 9 had acidic pH values (< 6) of 5.10, 5.59, and 5.31, respectively, which were similar the
356 acidic 4.96 pH value of the influent Cd-Cu-Pb column test solution. The final effluent pH for the
357 Cd-Cu-Pb IOH column test 10 had a value of 7.23 indicating that IOH may have the beneficial
358 attribute of being able to neutralize the acidic pH present in certain heavy metal contaminated
359 waters. The Table 8 effluent contaminant levels for Cd-Cu-Pb NFM column test 6 indicates that
360 column test equipment and procedures removed only ~3% of Cd, ~7% of Cu, and ~3% of Pb,
361 which would not be considered substantially large contaminant losses. For both low and high
362 flow rates, Table 8 shows that ZVI, PIC, SMI, and IOH removed basically all of the Cu and Pb
363 from the influent Cd-Cu-Pb column test solution. All Cd (at least below detection limits) was
364 removed by ZVI, PIC, SMI, and IOH at low flow rates; however, only SMI removed all Cd at
365 high flow rates. These results imply that, with regard to waters containing a mixture of Cd, Cu,

366 and Pb, the chemical reactions between all iron-based filter materials and Cu or Pb species
367 appear to occur quite rapidly, chemical reactions between SMI and Cd species also occur quite
368 rapidly, but chemical reactions seem to be slower between Cd species and either ZVI, PIC, or
369 IOH. Overall, Fig. 1d and Table 8 indicate that ZVI, PIC, SMI, and IOH all exhibit promise for
370 removal of Cd, Cu, and Pb when these heavy metal trace elements are present together in
371 contaminated waters.

372 **4. Conclusions**

373 A laboratory study that included saturated falling-head hydraulic conductivity tests,
374 contaminant removal batch tests, and low-to-high flow rate saturated solute transport column
375 tests was conducted to evaluate the capabilities of four iron-based filter materials to remove trace
376 element contaminants from waters containing either arsenic, chromium, and selenium (As-Cr-Se)
377 or cadmium, copper, and lead (Cd-Cu-Pb). Sulfur modified iron (SMI) was found to completely
378 remove As, Cd, Cr, Cu, Se, and Pb (at least below detection limits). An iron oxide/hydroxide
379 (IOH) was especially effective in removing As, Cd, Cr, Cu, and Pb, but not Se. Potential
380 advantages for using IOH compared to the other three iron-based filter materials include its very
381 high hydraulic conductivity and the release of treated waters that are not extremely acidic or
382 alkaline. The zero valent iron (ZVI) and porous iron composite (PIC) filter materials generally
383 worked quite well removing Cd, Cr, Cu, and Pb. These results are preliminary, and more
384 laboratory and field research is certainly needed to fully evaluate these four iron-based materials
385 for filter treatment of waters contaminated with mixtures of trace elements.

386 **Authors Note**

387 The use of manufacturer/developer/marketer names are for informational purposes only
388 and does not imply endorsement by the authors or their organizations.

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525 **Table 1**
 526 Iron-based filter material chemical and physical properties.^a

Filter Material	pH ^b	ORP ^c mV	Specific Gravity g/cm ³	Particle Size Distribution % by wt.			
				Course Sand Size 2.0-4.75 mm	Medium Sand Size 0.43-2.0 mm	Fine Sand Size 0.075-0.43 mm	Silt/Clay Size <0.075 mm
				ZVI	10.35	-592	7.03
PIC	10.14	-538	7.13	0.00	23.50	76.48	0.02
SMI	6.75	-660	5.93	0.12	21.11	78.71	0.06
IOH	8.18	26	3.62	0.04	97.87	2.05	0.04

527 ^a Values of pH and ORP represent a three replicate average obtained from 1:2 by weight slurry
 528 mixtures of filter material to deionized water at ambient laboratory temperatures of 20⁰ to 25⁰ C.

529 ^b pH was determined using an Oakton (Vernon Hills, Illinois) pHTestr 10 BNC.

530 ^c ORP is oxidation/reduction potential measured with a YSI Inc. (Yellow Springs, Ohio),
 531 EcoSense pH100 Meter and an YSI Inc., 115-1 ORP Probe.

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547 **Table 2**
 548 Test solution constituents and chemical properties.

Solution	Contaminant Concentrations ppm	Added Chemical Compound Concentrations ppm	Solution Chemical Properties	
			pH	ORP mV
Hydraulic Conductivity Test Solution	-	CaSO ₄ - 1361 (0.01 mole/L)	7.03	193
As-Cr-Se Batch Test Solution	As - 50 Cr - 50 Se - 50	KH ₂ AsO ₄ - 120 K ₂ CrO ₄ - 187 K ₂ SeO ₄ - 140	6.78	112
Cd-Cu-Pb Batch Test Solution	Cd - 50 Cu - 50 Pb - 50	CdCl ₂ - 82 CuCl ₂ *2H ₂ O - 134 PbCl ₂ - 67	4.48	238
As-Cr-Se Column Test Solution	As - 25 Cr - 25 Se - 25	KH ₂ AsO ₄ - 60 K ₂ CrO ₄ - 93 K ₂ SeO ₄ - 70	6.46	187
Cd-Cu-Pb Column Test Solution	Cd - 25 Cu - 25 Pb - 25	CdCl ₂ - 41 CuCl ₂ *2H ₂ O - 67 PbCl ₂ - 34	4.96	255

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Table 3
Saturated solute transport column test information.

Column Test Number	Filter Material	Test Solution	Column Dry Bulk Packing Density	Column Pore Volume	Average Low Flow Rate, - Flux -, and (Contact Time)	Low Flow Rate Effluent Amount	Average High Flow Rate, - Flux -, and (Contact Time)	High Flow Rate Effluent Amount	Total Effluent Amount
			g/cm ³	cm ³	mL/h - mL/cm ² ·h - (min)	L and (# pore volumes)	mL/h - mL/cm ² ·h - (min)	L and (# pore volumes)	L and (# pore volumes)
1	Empty Column (NFM ^a)	25 mg/L As, Cr, Se	NA	NA	47.88 - NA - (NA)	8.85 (NA)	NA - NA - (NA)	NA (NA)	8.85 (NA)
2	ZVI	25 mg/L As, Cr, Se	3.23	160.84	51.29 - 2.56 - (188.15)	8.59 (53.40)	2550.31 - 127.33 - (3.78)	1.81 (11.23)	10.40 (64.63)
3	PIC	25 mg/L As, Se, Cr	2.04	214.35	55.22 - 2.76 - (232.90)	9.19 (42.90)	1722.89 - 86.02 - (7.46)	2.05 (9.54)	11.24 (52.44)
4	SMI	25 mg/L As, Cr, Se	2.06	196.16	51.76 - 2.58 - (227.39)	8.62 (43.94)	1538.79 - 76.83 - (7.65)	1.96 (9.97)	10.58 (53.91)
5	IOH	25 mg/L As, Cr, Se	0.52	256.87	51.83 - 2.59 - (297.36)	8.63 (33.58)	2615.34 - 130.57 - (5.89)	1.85 (7.21)	10.48 (40.79)
6	Empty Column (NFM ^a)	25 mg/L Cd, Cu, Pb	NA	NA	49.36 - 2.46 - (NA)	9.18 (NA)	NA - NA - (NA)	NA (NA)	9.18 (NA)
7	ZVI	25 mg/L Cd, Cu, Pb	2.73	184.00	52.61 - 2.62 - (209.85)	8.55 (46.49)	6173.00 - 308.19 - (1.79)	2.54 (13.80)	11.09 (60.29)
8	PIC	25 mg/L Cd, Cu, Pb	2.03	215.01	54.07 - 2.69 - (238.59)	9.03 (42.00)	4059.10 - 202.65 - (3.18)	2.37 (11.01)	11.40 (53.01)
9	SMI	25 mg/L Cd, Cu, Pb	2.06	195.86	53.23 - 2.65 - (220.77)	7.72 (39.42)	3477.95 - 173.64 - (3.38)	2.09 (10.66)	9.81 (50.08)
10	IOH	25 mg/L Cd, Cu, Pb	0.51	258.07	60.01 - 3.00 - (258.03)	8.48 (32.88)	3533.57 - 176.42 - (4.38)	2.45 (9.51)	10.93 (42.39)

577 ^a NFM is the notation for “no filter material”.

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Table 4
Iron-based filter material hydraulic conductivities and associated packing densities.

Filter Material	Test Number	Column Dry Bulk Packing Density g/cm³	Saturated Hydraulic Conductivity cm/s
ZVI	1	3.00	3.89×10^{-2}
	2	3.08	1.73×10^{-2}
PIC	1	2.22	1.28×10^{-2}
	2	2.13	2.32×10^{-2}
SMI	1	1.98	2.15×10^{-2}
	2	1.97	1.30×10^{-2}
IOH	1	0.51	1.03×10^{-1}
	2	0.52	9.20×10^{-2}

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618 **Table 5**
 619 Batch test results for As-Cr-Se solution.

Filter Material	Final Test Conditions		Final Contaminant Solution Concentration ^a Average ^b and (Standard Deviation ^b)		
	pH	ORP ^c mV	As	Cr	Se
NFM^d	6.37	150	44.64 (0.59)	49.59 (0.68)	44.54 (0.52)
ZVI	10.31	-186	29.56 (2.46)	8.60 (0.64)	28.38 (2.44)
PIC	8.17	72	38.44 (1.31)	45.27 (2.04)	40.95 (0.85)
SMI	6.35	-150	ND (NA)	ND (NA)	ND (NA)
IOH	8.10	155	0.10 (0.00)	1.76 (0.04)	24.08 (0.44)

620 ^a Initial concentration of As = 50 ppm, Cr = 50 ppm, and Se = 50 ppm.
 621 ^b Based on three batch test replicates. ND = not detected. NA = not applicable.
 622 ^c ORP is oxidation/reduction potential.
 623 ^d NFM is the notation for “no filter material”.

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651 **Table 6**
 652 Batch test results for Cd-Cu-Pb solution.

Filter Material	Final Test Conditions		Final Contaminant Solution Concentration ^a Average ^b and (Standard Deviation ^b)		
	pH	ORP ^c mV	Cd	Cu	Pb
NFM^d	4.85	245	43.16 (3.99)	45.09 (2.99)	48.74 (3.78)
ZVI	10.21	-117	0.55 (0.48)	ND (NA)	ND (NA)
PIC	5.99	-190	ND (NA)	ND (NA)	ND (NA)
SMI	4.88	-143	ND (NA)	ND (NA)	ND (NA)
IOH	7.51	98	ND (NA)	ND (NA)	ND (NA)

653 ^a Initial concentration of Cd = 50 ppm, Cu = 50 ppm, and Pb = 50 ppm.
 654 ^b Based on three batch test replicates. ND = not detected. NA = not applicable.
 655 ^c ORP is oxidation/reduction potential.
 656 ^d NFM is the notation for “no filter material”.

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684 **Table 7**
 685 Column test results for As-Cr-Se solution.

Column Test	Filter Material	Effluent pH		Effluent Contaminant Concentrations ^a					
		Average and (Standard Deviation for Low Flow or Range for High Flow)		Average ^b , - Start to Finish Values - ^b , and (Standard Deviation for Low Flow or Range for High Flow) ^b					
		Low Flow Rate	High Flow Rate	Low Flow Rate			High Flow Rate		
				As	Cr	Se	As	Cr	Se
1	Empty Column (NFM ^c)	6.15 (0.14)	NA (NA)	21.93 -22.1 to 21.7 - (0.13)	24.31 -24.4 to 24.1 - (0.11)	22.77 -22.9 to 22.5 - (0.12)	NA -NA to NA - (NA)	NA -NA to NA - (NA)	NA -NA to NA - (NA)
2	ZVI	10.08 (0.20)	10.48 (0.14)	13.37 -9.66 to 16.6 - (2.88)	ND -ND to ND - (NA)	17.57 -16.0 to 19.3 - (1.39)	19.55 -20.3 to 18.8 - (1.57)	0.05 -ND to 0.09 - (0.09)	22.04 -23.0 to 21.1 - (1.93)
3	PIC	10.33 (0.11)	10.55 (0.07)	11.73 -10.0 to 12.9 - (1.16)	ND -ND to ND - (NA)	15.78 -17.0 to 15.2 - (1.01)	14.93 -16.7 to 13.1 - (3.58)	ND -ND to ND - (NA)	17.33 -19.7 to 14.9 - (4.82)
4	SMI	3.80 (0.24)	5.95 (0.31)	ND -ND to ND - (NA)	ND -ND to ND - (NA)	ND -ND to ND - (NA)	ND -ND to ND - (NA)	ND -ND to ND - (NA)	ND -ND to ND - (NA)
5	IOH	7.98 (0.16)	8.28 (0.13)	0.05 -0.08 to 0.06 - (0.02)	ND -ND to ND - (NA)	18.23 -ND to 27.9 - (11.57)	0.04 -0.04 to 0.03 - (0.01)	6.03 -2.67 to 9.38 - (6.71)	27.40 -28.7 to 26.1 - (2.57)

686 ^a Initial concentration of As = 25 ppm, Cr = 25 ppm, and Se = 25 ppm.

687 ^b ND = not detected. NA = not applicable.

688 ^c NFM is the notation for “no filter material”.

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711 **Table 8**
 712 Column test results for Cd-Cu-Pb solution.

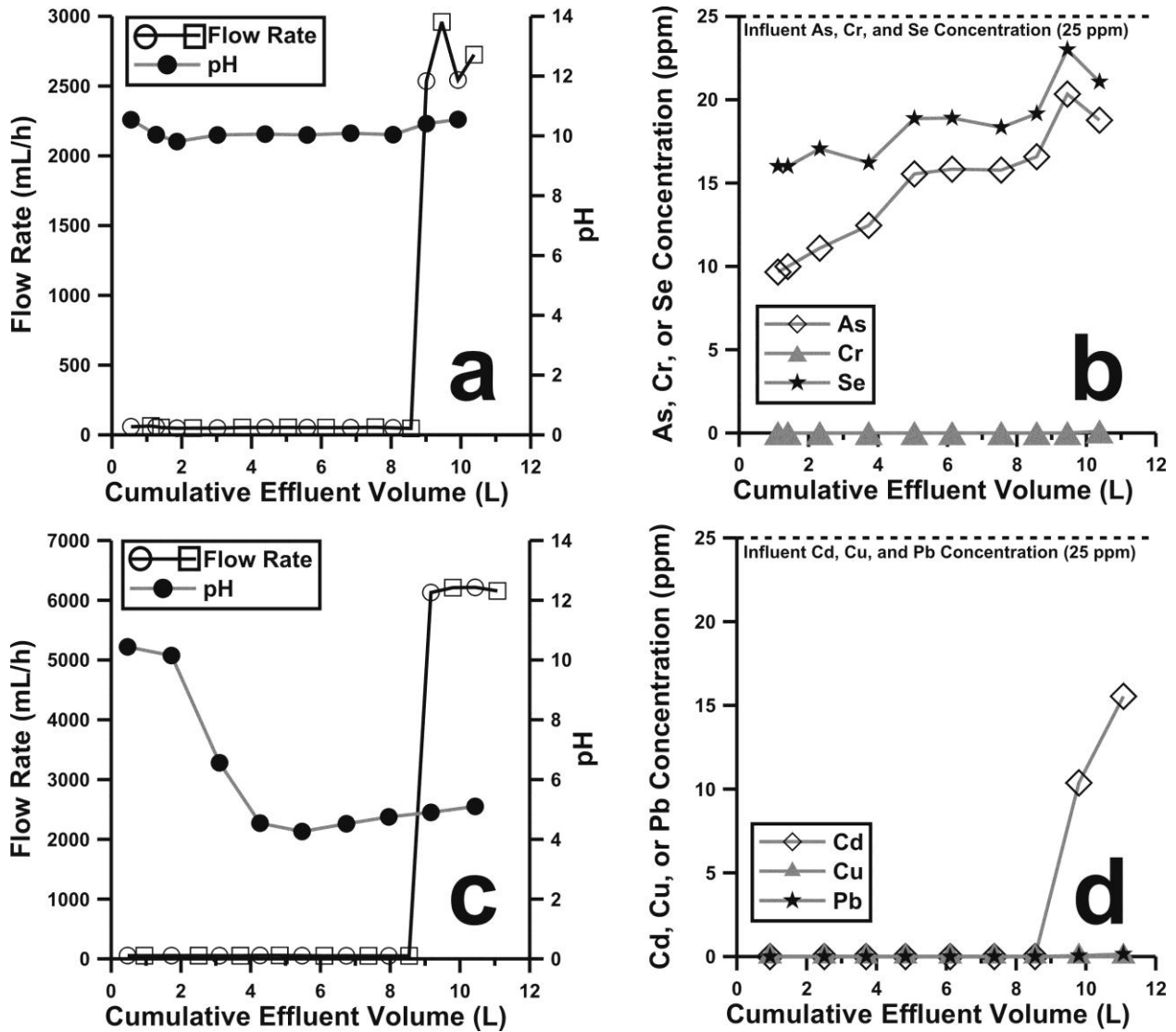
Column Test	Filter Material	Effluent pH		Effluent Contaminant Concentrations ^a					
		Average and (Standard Deviation for Low Flow or Range for High Flow)		Average ^b , - Start to Finish Values - ^b , and (Standard Deviation for Low Flow or Range for High Flow) ^b					
		Low Flow Rate	High Flow Rate	Low Flow Rate			High Flow Rate		
				Cd	Cu	Pb	Cd	Cu	Pb
6	Empty Column (NFM ^c)	4.84 (0.20)	NA (NA)	25.84 - 26.3 to 25.1 - (0.42)	23.33 - 23.8 to 22.3 - (0.47)	24.25 - 24.9 to 22.9 - (0.61)	NA - NA to NA - (NA)	NA - NA to NA - (NA)	NA - NA to NA - (NA)
7	ZVI	6.46 (2.73)	5.00 (0.20)	ND - ND to ND - (NA)	ND - ND to ND - (NA)	ND - ND to ND - (NA)	12.96 - 10.4 to 15.5 - (5.17)	ND - ND to ND - (NA)	0.10 - 0.05 to 0.15 - (0.09)
8	PIC	8.47 (1.85)	6.37 (1.56)	ND - ND to ND - (NA)	ND - ND to ND - (NA)	ND - ND to ND - (NA)	2.61 - ND to 5.21 - (5.21)	ND - ND to ND - (NA)	ND - ND to ND - (NA)
9	SMI	4.16 (0.25)	5.30 (0.02)	ND - ND to ND - (NA)	ND - ND to ND - (NA)	ND - ND to ND - (NA)	ND - ND to ND - (NA)	ND - ND to ND - (NA)	ND - ND to ND - (NA)
10	IOH	7.91 (0.11)	7.41 (0.35)	ND - ND to ND - (NA)	ND - ND to ND - (NA)	ND - ND to ND - (NA)	8.30 - ND to 16.6 - (16.60)	ND - ND to ND - (NA)	ND - ND to ND - (NA)

713 ^a Initial concentration of Cd = 25 ppm, Cu = 25 ppm, and Pb = 25 ppm.

714 ^b ND = not detected. NA = not applicable.

715 ^c NFM is the notation for “no filter material”.

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740 **Fig. 1.** Representative column test results; (a) flow rate and pH for As-Cr-Se ZVI column test 2
 741 - flow rate open circle symbols denote pH measurement samples, while open square symbols
 742 denote contaminant measurement samples, (b) measured contaminant concentrations for As-Cr-
 743 Se ZVI column test 2, (c) flow rate and pH for Cd-Cu-Pb ZVI column test 7 - flow rate open
 744 circle symbols denote pH measurement samples, while open square symbols denote contaminant
 745 measurement samples, and (d) measured contaminant concentrations for Cd-Cu-Pb ZVI column
 746 test 7.