

1 **Elemental metals for environmental remediation: lessons from hydrometallurgy**

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7 **Abstract**

8 In the mining industry, the separation of economically valuable metals from gangue materials
9 is a well established process. As part of this field, hydrometallurgy uses chemical fluids
10 (leachates) of acidic or basic pH to dissolve the target metal(s) for subsequent concentration,
11 purification and recovery. The type and concentration of the leach solution is typically
12 controlled to allow selective dissolution of the target metal(s), and other parameters such as
13 oxidation potential, temperature and the presence of complexing/chelating agents. In the
14 remediation industry the use of elemental metals (M^0) for the removal of aqueous
15 contaminant species is also a well established process. Removal is achieved by the oxidative
16 corrosion of the M^0 and associated pH and/or redox potential change. Whilst the two
17 processes are directly opposed and mutually exclusive they both stem from the same
18 theoretical background: metal dissolution/precipitation reactions. In the mining industry, with
19 each prospective ore deposit physically and chemically unique, a robust series of tests are
20 performed at each mine site to determine optimal hydrometallurgical fluid composition and
21 treatment conditions (e.g. fluid temperature, flow rate) for target metal dissolution/yield. In
22 comparison, within the remediation industry not all such variables are typically considered. In
23 the present communication a comparison of the processes adopted in both industries are
24 presented. The consequent need for a more robust empirical framework within the
25 remediation industry is outlined.

26 **Keywords:** Environmental remediation, Extractive metallurgy, Intrinsic reactivity, Metal
27 dissolution, Zerovalent Metal.

28 **1 Introduction**

29 The primary mechanisms of contaminant removal by elemental metals (M^0) are considered to
30 be adsorption, chemical reduction, complexation, co-precipitation, incorporation and size-
31 exclusion [1-6]. Chemical reduction at the M^0 surface has often been cited as a key
32 mechanism of contaminant removal. However, it should be recognised that although chemical
33 reduction can have a profound effect on contaminant aqueous stability (e.g. solubility,
34 degradation), it does not explicitly imply contaminant removal [7]. Chemically reduced
35 contaminants can be removed via surface mediated accumulation/precipitation, but unless
36 structurally entrapped/incorporated within the M^0 corrosion product, the pollutant species is
37 always available for re-release/re-dissolution. It can therefore be stated that when determining
38 the mechanisms which govern the efficacy of a M^0 material for aqueous contaminant
39 remediation, key focus must be applied to: (i) the mechanism of aqueous M^0 corrosion; and
40 (ii) the type, concentration and distribution of corrosion products formed.

41 When testing M^0 (typically Fe^0 and Zn^0) for environmental remediation, the influence of
42 several operational parameters on the materials solubility can be investigated including
43 solution pH, particle size, and the type and intensity groundwater flow [8-10]. A similar
44 approach is applied in hydrometallurgical investigations. It has been shown that metal
45 dissolution increases with (i) increasing stirring speed, (ii) acidic or basic pH value, and, (iii)
46 decreasing particle size [11]. When testing M^0 for remedial applications, the extent of
47 contaminant removal is nominally defined by measuring the aqueous concentration of the
48 pollutant species at any given time. The dissolution of the M^0 is generally also measured;
49 however little comparative emphasis is typically placed on this variable [8]. With the
50 solubility of M^0 a key factor with regard to the materials performance for contaminant
51 removal, this seems counterintuitive. For example, at $pH < 4.5$ the significantly high
52 solubility of both Fe^{II} and Fe^{III} dictates that minimal contaminant removal will be achieved
53 [12].

54 The objective of the present communication is to highlight the fundamental importance of
55 tailoring the physical and chemical composition of M^0 , and mechanism of
56 application/deployment, for environmental applications. In order to aid discussion, the
57 procedures performed in the hydrometallurgy industry are comparatively examined.

58 **2 Hydrometallurgy**

59 Hydrometallurgy is a metallurgical process by which metals are extracted from an ore body
60 using chemical reagents. To optimize the extraction efficiency, the process can be operated at
61 a high temperature and under pressure [13]. Extracted metals are then separated to produce a
62 concentrate or an intermediary product [13-15]. Another common metallurgical process is
63 pyrometallurgy. This method, however, is typically considered as significantly more energy
64 intensive and less versatile than hydrometallurgy [13,14-17]. For example, Zn was produced
65 for over several hundred years via pyrometallurgy, however, in the 1980's, a fully
66 hydrometallurgical process was invented (Sherritt autoclave process) and production plants
67 are now in abundant operation using this technology. Pyrometallurgy will not be further
68 considered in this work as the focus is on processes occurring in aqueous solutions (for ore
69 processing and metal corrosion).

70 **2.1 Dissolving metals in hydrometallurgy**

71 The dissolution of metals (metal leaching) from minerals can occur either through biological,
72 chemical or electrochemical processes. Due to its low-cost, simple application and high metal
73 yield, the most common method employed to date in the hydrometallurgy industry has been
74 the use of chemical leachates.

75 Leaching involves the use of aqueous solutions containing a lixiviant, the type of which
76 leachate selected is typically dependent on the type of target metal, however, the type and
77 concentration of any gangue material is generally also considered [11,18-21]. For example,
78 chemical reducing agents within the gangue material (iron, organics, sulphur, etc.) can
79 provide adsorptive sites for any redox-amenable contaminant species, such as uranium,

80 decreasing the efficiency of in-situ uranium leaching [19]. In such cases, complexing agents
81 (such as dissolved carbonate), high temperatures and/or oxidising agents can be incorporated
82 into the lixiviant to significantly enhance the uranium extraction yield [19].

83 **2.2 Lessons from hydrometallurgy**

84 The myriad different factors to consider when assessing the metal/H₂O system is an area of
85 research that transcends both the hydrometallurgy and remediation industries.

86 Similar to ore bodies, the reactivity of engineered metals depends primarily on three factors:
87 (i) the nature and proportion of any associated, impurity or alloying elements (or gangue
88 materials), (ii) the method of manufacture (or diagenesis), and (iii) the stability of the surface
89 oxide layer when immersed in an aqueous environment. Until recently, materials have
90 typically been characterised for the removal extent of selected contaminants without
91 addressing the intrinsic reactivity which typically correlates with the metal's tendency to
92 dissolve [8-10].

93 The importance of this issue was recently presented for Fe⁰ [7,8]. Because Zn⁰ is currently
94 investigated as an alternative to Fe⁰, Table 1 summarises the experimental conditions of
95 recent work by Salter-Blanc and Tratnyek [6] and selected references therein. Table 1
96 highlights that, when testing different Zn⁰ materials for environmental remediation, mass
97 loadings from 25 to 250 g/L were tested under various mixing intensities and various
98 experimental durations. The arising question is how to compare the obtained results? The
99 electrochemical reactivity of Zn⁰ for contaminant reduction is given by the standard redox
100 potential of the couple Zn^{II}/Zn⁰ (-0.763 V), which is considered to be constant for Zn⁰
101 regardless from the used particle size. However, numerous other factors must be constant in
102 order to successfully compare different experiments, including: (i) the M⁰ mass loading; (ii)
103 the presence of any associated, impurity or alloying elements; (iii) the solution pH; (iv) the
104 solution temperature; (v) the particle size (surface area); and (vi) the solution stirring/agitation
105 speed.

106 **3 The metal/H₂O system**

107 The aqueous solubility of metal ions (Mⁿ⁺) from elemental metal (M⁰) at pH > 4.5 (natural
108 waters) is strongly influenced by physical and chemical composition of the initial surface
109 oxide layer [12,25,26]. Because contaminated natural waters contain target pollutants in trace
110 amounts, the solvent (H₂O) is typically present in large stoichiometric abundance. Therefore,
111 the metal/H₂O system in natural waters should be regarded as the primary domain for aqueous
112 metal corrosion and precipitation reactions. The corrosion process may also be significantly
113 influenced by the presence of the contaminants and other ubiquitous water species (including
114 dissolved CO₂ and O₂). It is therefore not unexpected, that pure M⁰ oxide/hydroxides are
115 typically the most abundant aqueous corrosion products identified [27-29].

116 **4 Hydrometallurgy versus contaminant removal in metal/H₂O systems**

117 In the hydrometallurgy industry, numerous different physico-chemical parameters are
118 typically modified in order to improve the technique, including: (i) leachate composition (pH,
119 presence of complexing/chelating agents, etc.); (ii) leachate temperature; and (iii) subsurface
120 permeability. Depending on the specific chemistry of the target metal, the following
121 respective alterations can be applied: (i) the leachate pH is buffered to either strongly acidic or
122 basic and/or complexing/chelating agents are used; (ii) the leachate temperature is raised; and
123 (iii) secondary subsurface fracturing techniques are employed to significantly enhance the
124 subsurface permeability. In the present section the potential use of such processes in the
125 remediation industry is comparatively discussed. The influence of M⁰ (or ore) particle
126 size/surface area and mixing intensity/agitation is also included for discussion.

127 **4.1 Effect of solution pH**

128 The aqueous concentration of H⁺ (pH = -log[H⁺]) is arguably the important parameter for both
129 the hydrometallurgy and remediation industries. Indeed, both metal solubility and metal
130 speciation are strongly pH dependent (Figure 1). In aqueous systems, hydroxides are the most
131 soluble phase of metals. Fig.1 compares the solubility of Al^{III}, Fe^{II}, Fe^{III} and Zn^{II} hydroxides

132 and shows that in the pH range of natural waters (pH 5.0 to 9.5), Al^{III} and Fe^{III} hydroxides are
133 relatively low soluble while Fe^{II} and Zn^{II} hydroxides are relatively soluble [30].
134 In the hydrometallurgy industry, the pH can be shifted to more acidic or more basic ranges to
135 optimize the extraction yield. For example, in-situ uranium leaching can be achieved by
136 carbonate solutions (pH > 6.0) or by sulphuric acidic solutions (pH < 3.0) [19]. In the
137 remediation industry, the solution pH must be maintained at a value which is favourable for
138 metal oxide precipitation (e.g. pH > 4.5 for Fe^0) [7,12]. This corresponds to the pH range of
139 natural waters wherein the metal surface is considered bound by a ubiquitous oxide layer
140 [12,25,29,31]. The initial oxide layer is considered porous and non protective, with
141 subsequent transformations dependent on the reactivity of metal with the surrounding
142 environment.

143 **4.2 The effect of temperature**

144 In general, increasing the solution temperature significantly accelerates the dissolution rate in
145 both processes. In hydrometallurgy, temperatures can be discretionary elevated to optimize
146 the extraction yield [11]. In remediation metal/ H_2O systems, ambient (room) temperatures
147 (20.0 to 24.0 °C) are typically too high to accurately represent the subsurface environment,
148 which is generally between 10.0 to 15.0 °C.

149 **4.3 The effect of subsurface permeability**

150 In the hydrometallurgy industry the permeability of the subsurface can be significantly
151 improved by the use of secondary subsurface fracturing techniques, such as hydraulic,
152 pneumatic or explosive processes. Such processes can be employed in the remediation
153 industry to: (i) improve the flow rate of a contaminant plume which is passing through a
154 permeable reaction barrier; or (ii) to facilitate the movement of nanoscale M^0 into soil pores
155 for in-situ aqueous pollutant treatment. Logistical factors such as workforce safety and the
156 potential for accidental aqueous contaminant or nanoscale M^0 release into the local
157 groundwater systems however must be considered.

158 **4.4 The effect of particle size**

159 Assuming the composition and shape of a M^0 material remains constant; its tendency and rate
160 of dissolution typically increases as particle size decreases. The specific surface area (SSA)
161 and the particle diameter (d) are inversely proportional ($SSA = 6/\pi d$) [32]. Therefore, surface
162 area exposure can be regarded as a useful indicator of: (i) the solubility of an ore body in the
163 hydrometallurgy industry; or (ii) the aqueous reactivity and associated contaminant removal
164 efficacy of the M^0 in the remediation industry. However, this respectively assumes that: (i) the
165 ore body is soluble in the leachate solution; and (ii) the M^0 is a stronger chemical reducing
166 agent than the aqueous contaminant specie(s). As a consequence, the intrinsic reactivity of the
167 M^0 should be characterised prior to testing different particle sizes and surface area.

168 This is routinely performed in the hydrometallurgy industry, wherein numerous factors are
169 typically considered, including: (i) the concentration of the leaching agent, (ii) the ore surface
170 area, and (iii) the duration of the leaching process. However, in remediation industry such
171 factors are often overlooked. In addition, a single M^0 particle size and a single method of
172 application is typically selected for contaminant remediation, whilst in the hydrometallurgy
173 industry, several different application processes and lixiviant compositions are simultaneously
174 employed to maximise metal dissolution and recovery [15,19].

175 **4.5 The effect of the stirring speed**

176 The type and intensity of mixing operations is a strong driver for metal
177 dissolution/precipitation. In the hydrometallurgical industry by increasing the leachate flow-
178 rate greater metal dissolution is typically ensured. In the remediation industry, the method of
179 M^0 application depends on the physical and chemical structure of the contaminated site. Care
180 must therefore be taken during empirical tests to use mixing devices and mixing intensity that
181 are relevant for the conditions of the field. In particular, the formation and transformation of a
182 surface oxide layer in the vicinity of the metal has a strong bearing on the reactivity of the M^0 .
183 For slow mixing speeds (e.g. $< 50 \text{ min}^{-1}$) oxide formation is not generally physically disturbed

184 [8]. A large number of studies have however used considerably higher mixing intensities. For
185 example, Pang et al. [33] used a shaking intensity of 150 min^{-1} to keep nano- Fe^0 suspended,
186 while Hao et al. [34] used a mixer stirred at 500 min^{-1} to ease nitrate transport to the surface
187 of iron filings.

188 In addition, it is essential to consider contaminant transport to the metal surface as diffusion-
189 limited and discuss the nature and stability of oxide scales under relevant conditions
190 [25,29,35].

191 **5 Concluding remarks**

192 In the present work a comparison has been presented for the testing framework implemented
193 within the hydrometallurgical industry for ore characterisation and acid/alkaline leaching and
194 testing framework implemented within the remediation industry for contaminated site
195 characterisation and aqueous pollutant removal. By comparison the considerably more robust
196 empirical framework exhibited within the former industry presents a strong consequent need
197 for more stringent protocols within the latter. It has been outlined that with every
198 contaminated site chemically and physically unique there exists a strong need for empirical
199 tests that are site specific and environmentally relevant. Only once this has been achieved, can
200 a remedial material that is specific for the unique conditions of the contaminated site be
201 effectively selected.

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- 297

297 **Table 1:** The variability of operational conditions employed for batch experiments as
 298 illustrated by: specific surface area (SSA), mass loading (ρ) and redox conditions. The
 299 experimental designs also further differ in terms of mixing devices and intensities, and
 300 the type, concentration and speciation of aqueous contaminant species..

301

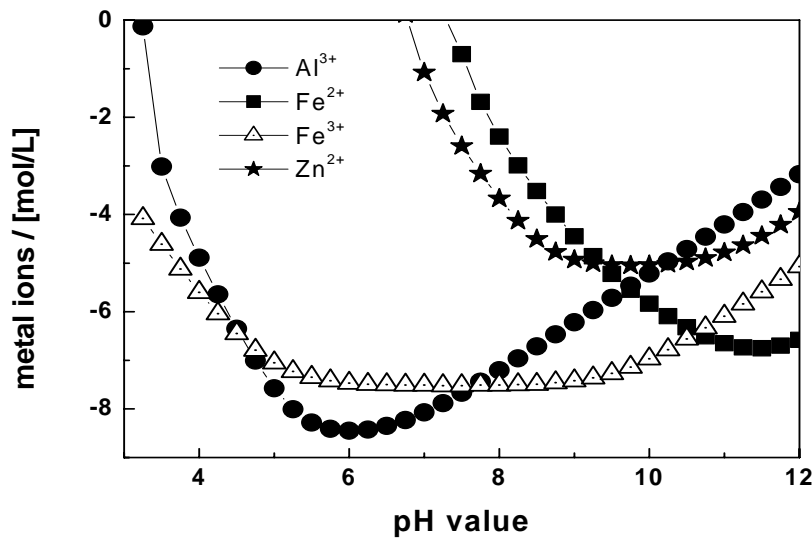
Mesh size	SSA (m ² /g)	Density (g/cm ³)	V (mL)	m_{Zn} (g)	ρ (g/L)	Redox	Ref.
30	0,035	-	1000	200	200	anoxic	[22]
30	0,035	-	125	5	40	anoxic	[23]
-10 / 50	0,023	-	120	3,0	25	anoxic	[24]
30	0,038	-	120	3,0	25	anoxic	[24]
< 325	0,350	-	120	3,0	25	anoxic	[24]
< 325	0,620	2,60	80	20	250	anoxic	[6]
20 / 60	0,016	2,34	80	20	250	anoxic	[6]
200 / 325	0,160	3,27	80	20	250	anoxic	[6]

302

303

303 **Figure 1:** The pH dependence of metal hydroxide solubility for $\text{Al}(\text{OH})_3$, $\text{Fe}(\text{OH})_2$, $\text{Fe}(\text{OH})_3$
304 and $\text{Zn}(\text{OH})_2$ [30]. It can be observed that if Zn^0 is used for environmental remediation, care
305 must be taken to control Zn^{II} species concentration in the effluent. For Fe^0 , Fe^{II} species are
306 rendered insoluble by oxidation to Fe^{III} species. At neutral pH values, the solubility of Al^{III}
307 and Fe^{III} species is less than 10^{-7} M.

308



309