Metallic Iron for Environmental Remediation

Metallic iron (Fe^0) used as a reactive material in subsurface permeable reactive barriers is comparable to iron pipes with three major differences: (i) corrosion is welcome because it is a rather useful process [7][8][9], (ii) a reactive wall is ideally permanently water saturated, and (iii) the length of used particles (< 5 cm) is tiny compared to pipes which are up to 12 m in length. On the one hand, Fe^0 specimens used in water treatment comprise steel wool with thickness varying between 25 and 90 µm [10][11]. On the other hand, the length of these particles is comparable to the wall thickness of iron pipes (2–4 mm). There has been no real system analysis for remediation Fe^0 materials with the aim to outline the differences making their peculiar characteristics. In addition, traceably deriving the longevity of remediation Fe^0 specimens from Fe^0 pipes is impossible because of the differences highlighted.

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1. Introduction

Metal corrosion is one of the most important problems in industry, transport and agriculture [1][2][3][4]. Understanding metal corrosion comprises its detection (e.g., analytical, visual), its monitoring (e.g., mass loss, H_2 evolution) and its long-term characterization under various field conditions [4][5]. The corrosion research aims at determining the durability of metallic structures under operational conditions (e.g., oil and gas pipelines, tanks) and revealing the mechanisms of corrosion process [5][6]. This mechanism can be chemical, electrochemical or mixed [1]. Various tools have been used to characterize the corrosion resistance of different metals under various application conditions [4]. The overall result is the availability of integrated approaches to assess and predict the corrosion processes and thus the longevity of metallic structures (e.g., buried pipes) [3][4]. However, the frequency and sudden nature of metallic pipe failures worldwide indicate the inadequacy of current knowledge related to the longevity of buried metallic pipes.

Metallic iron (Fe^0) used as a reactive material in subsurface permeable reactive barriers is comparable to iron pipes with three major differences: (i) corrosion is welcome because it is a rather useful process [7][8][9], (ii) a reactive wall is ideally permanently water saturated, and (iii) the length of used particles (< 5 cm) is tiny compared to pipes which are up to 12 m in length. On the one hand, Fe^0 specimens used in water treatment comprise steel wool with thickness varying between 25 and 90 µm [10][11]. On the other hand, the length of these particles is comparable to the wall thickness of iron pipes (2–4 mm). There has been no real system analysis for remediation Fe^0 materials with the aim to outline the differences making their peculiar characteristics. In addition, traceably deriving the longevity of remediation Fe^0 specimens from Fe^0 pipes is impossible because of the differences highlighted.

The remediation Fe^0 was termed as zero-valent iron (ZVI). This acronym is perhaps the first problem of this still innovative technology. A literature research with "zero-valent iron" as keyword would never reveal the ancient literature on Fe^0 for water treatment [12][13][14][15][16]. Indeed, no research group until 2017 has referenced a single article from the ancient use of Fe^0 in water treatment [17][18]. In chemistry, metallic elements are characterized by their oxidation state, the one of Fe^0 is zero (0). Upon oxidation, Fe^0 is transformed to Fe^{II}, Fe^{III} or Fe^{IV} species. Under environmental conditions, only Fe^{II} and Fe^{III} species are stable. The oxidation of Fe^0 to Fe^{II} is a redox process characterized by an electrode potential whose value is –0.44 V [1]. According to the first principle of chemical thermodynamics, Fe^0 can be oxidized by oxidizing agents from each redox couple having a higher electrode potential (E^0 > –0.44 V). Water (H_2O or H^+) is a relevant oxidizing agent for Fe^0 under environmental conditions. The electrode potential for the redox couple H^+/H_2 is 0.00 V. Fe^0 immersed in (contaminated or polluted) water is corroded to form H/H_2 and Fe^{II} (and mixed Fe^{II}/Fe^{III}) species which are stand-alone reducing agents [19][20][21][22][23][24][25][26]. Clearly, it is not surprising that selected species undergo reductive transformations in an Fe^0/H_2O system [19][26][27][28][29].

2. The Fe^0/H$_2$O System

2.1. Overview of Fundamental Aspects

There is a transfer of electrons from the Fe^0 body (solid state) to the Fe^0/H$_2$O interface whenever a piece of a reactive Fe^0 specimen is immersed in an aqueous solution (Fe^0/H$_2$O system) [1][5][6][30]. This occurs because Fe^0 is not stable under environmental conditions or because the redox couple H$^+$/H$_2$ (E^0 = 0.00) is higher than that of Fe^{II}/Fe^0 (E^0 = –0.44) in the electrochemical series [1][22][23][24][25][26].

2.2. Oxide Scale on Fe^0 and the Decontamination Process

A look at the mechanism of oxide scale formation reveals that it cannot be electronically conductive. In fact, the initial scale is very porous and cannot transfer electrons because air and water are not electronically conductive (an aqueous solution can be ionic conductive—electrolyte). In subsequent stages, available pores are filled with nascent FeCPs, but they are never uniform and the oxide scale is a mixture of iron hydroxides and oxides [5]. An oxide scale made up of Fe$_3$O$_4$ alone would have been electronically conductive. However, such an Fe$_3$O$_4$ scale cannot exist under natural conditions (immersed Fe^0). All other FeCPs are at best semi-conductors and cannot relay electrons from Fe^0 under natural conditions. Clearly, reports justifying the reductive efficiency of Fe^0/H$_2$O systems using the semi-conductive nature of FeCPs are mistaken [31]. This assertion encompasses the Fe^0/pyrite/H$_2$O system, whose efficiency is mainly justified by the semi-conductive nature of FeS species [32][33][34].

The oxide scale on Fe^0 is definitively a diffusion barrier for all dissolved species, including the pollutants. It is also the contaminant scavenger such that electrochemical corrosion of immersed Fe^0 induces the generation of contaminant scavengers and other reducing agents. Thus the generation of solid FeCPs is a necessary process which has the (perceived negative) side effect of being expansive. Thus, designing an efficient and sustainable system requires answering the question: how long can FeCPs be generated to satisfactorily treat water while keeping a reasonable hydraulic conductivity (permeability)?

2.3. Chemical Aspects

Fe^{2+} from Equation 1 is transformed to ferrous hydroxide (Fe(OH)$_2$) and ferric hydroxide (Fe(OH)$_3$) which have a strong tendency to form colloids of particles that normally carry a positive charge [35][36][37]. These minerals are further transformed to other Fe^{II}/Fe^{III} minerals (e.g., Fe$_2$O$_3$, FeOOH, green rust) exhibiting different affinities to dissolved species. The nature of oxides in each individual system depends on the intrinsic reactivity of the used Fe^0 material and the environmental conditions [5][38]. For example, two different Fe^0 specimens corroding under the same environment will not necessarily produce the same iron oxides, because the composition of the oxide scale depends on the relative kinetics of Fe^0 dissolution and Fe hydroxide precipitation, which in turn depends on the solution chemistry, including the pH value, dissolved ions and the salinity [5][6][39]. On the other hand, in-situ generated free Fe^{2+} are adsorbed to the surface of available minerals to form the so-called structural Fe^{II} with a reducing power far larger than that of the free Fe^{2+} (E^0 < 0.77 V) and sometimes stronger than Fe^0 (E^0 < –0.44 V) [40]. The availability of several reducing agents in the Fe^0/H$_2$O system, and especially from structural Fe^{II}, partly stronger than Fe^0 implies that the electrochemical series of metal alone cannot predict the chemistry of the system.

2.4. Physical Aspects

The volumetric expansive nature of iron corrosion is the most important physical phenomenon occurring in Fe^0/H$_2$O systems [41]. There is expansion because the parent metal (Fe^0) produces in-situ both: (i) H$_2$, occupying a volume about 3100 times larger [26][42], and (ii) each solid oxide and hydroxide is at least twice larger in volume than Fe^0 (V$_{oxide}$ > V$_{iron}$) [43][44]. For example, the specific density of magnetite (Fe$_3$O$_4$) is about one half that of iron (Fe^0). It implies that after corrosion a space twice larger than the initial space is occupied [45][46][47]. While it can be assumed that H$_2$ escapes from each open system, no free expansion of oxides in porous systems (e.g., water filters, reactive walls) can be assumed [26]. External or internal free expansion occurs in metallic pipes [48][49] and on the walls of steel canister for radioactive waste repositories [47][50]. On the contrary, free expansion cannot be expected in steel-reinforced concrete structures [45][46]. Accordingly, considering expansive iron corrosion, which culminates into permeability loss is an essential design parameter for porous Fe^0/H$_2$O systems (Fe^0 filters). For each Fe^0 filter, the temporal production of both H$_2$ and oxide is decisive for the long-term efficiency and the permeability of the system [42][44][51][52].

2.5. Kinetic Aspects

Aqueous corrosion of Fe^0 materials under environmental conditions (pH > 4.5) is an electrochemical process involving iron dissolution at the anode and H_2 evolution at the cathode (Equation 1). This electrochemical reaction is accompanied by the formation of an oxide scale on Fe^0 which is not protective as a rule [6][53][54][55]. In general, oxide scale growth and its protectiveness depend primarily on the precipitation rate of iron hydroxides [5][39]. As the Fe^0 surface corrodes under the initial scale, corrosion continuously undermines the scale. Voids are created and are progressively filled up by the ongoing hydroxide precipitation. The relative rate of (i) Fe^0 oxidative dissolution and (ii) hydroxide precipitation in the Fe^0 vicinity determine the protectiveness of the oxide scale. According to Nesic [5], when the rate of hydroxide precipitation exceeds the rate of Fe^0 dissolution, a dense protective oxide scale is formed. Conversely, when Fe^0 dissolution undermines the new oxide scale faster than hydroxide precipitation can fill in the voids, a porous and non-protective scale forms.

2.6. Investigating the Fe^0/H_2O System

A Fe^0/H_2O system is made up of two interfaces: (i) Fe^0/oxide and (ii) oxide/H_2O. Because the oxide scale is never electronically conductive and is a diffusion barrier to many species, only water can quantitatively reach the Fe^0 surface. The net result is that Fe^0 oxidative dissolution is an electrochemical reaction (water is reduced) but all other observed/reported chemical reduction occur within the oxide film or at the oxide/H_2O interface [56][57][58]. For the Fe^0/H_2O remediation system, it means that contaminants are not reduced by electrons from Fe^0. This has important implications on the operating principles of Fe^0/H_2O systems: first, using the electrochemical series to predict the reductive transformation of any species has been a mistake, and second, using the stoichiometry of any electrochemical reaction involving Fe^0 is also a mistake. Accordingly, contaminants are 'just' dissolved species, capable of modifying: (i) the conductive properties of the electrolyte (H_2O), (ii) the ion conductivity of the oxide scale, and (iii) formation and the transformation of the oxide scale. Experiments pertaining to filtration systems must be performed under conditions enabling the formation of an oxide scale on the vicinity of Fe0 (e.g. quiescent batch experiments). For all other systems (e.g. fluidized systems), field dynamics (e.g. shaking intensity) must be reproduced during testing.

3. Selection and Characterization of Fe^0 Materials

As discussed above, the scientific reason for using all Fe^0 materials (e.g., granular iron and bimetallics, iron filings, iron nails, iron wire, nano-Fe^0, scrap iron, steel wool) in water treatment is the electrode potential of the redox electrode Fe^{II}/Fe^0: $E^0 = -0.44$ V. Clearly, all reactive Fe^0 materials have the same redox potential. The intrinsic reactivity of individual Fe^0 specimens depends on a myriad of factors from which some are not readily accessible to the researcher. Relevant influencing factors include: alloying elements, Fe^0 form, manufacturing processes, metallography, Fe^0 grain size, surface area, and surface oxidation state. Accordingly, each Fe^0 material has its own intrinsic reactivity which should be characterized in order to better understand how it is influenced by operational conditions to induce the intended remediation goal.

The long history of using Fe^0 for technical chemical applications, including water treatment reveals that there has always been efforts to select appropriate materials for individual applications. For example, the porous "spongy iron" (sponge iron or direct reduced iron) was proven more suitable in filtration systems than dense materials [12][13][59]. Similarly, multi-metallic systems and nanoscale materials were recently developed to address (recalcitrant) contaminants that were less sensitive to treatment with granular materials [60]. For completeness, it should be stated that there is no Fe^0 material for all situations such that one should rationally select appropriate materials for each specific application. For example, while treating water in fluidized systems (Anderson Process), dense materials were better than sponge iron [13][59]. Similarly, Hildebrant et al. [61] reported that Fe^0 materials of low reactivity according to the EDTA test exhibited a better efficiency for fluoride removal. The question arises how to select the right Fe^0 specimen for a given application? This calls for the development of standardized protocols for selection and characterization of Fe^0 materials for various applications.

4. Conclusion

The corrosion of iron in remediation Fe^0/H_2O systems is an electrochemical process, coupling Fe^0 oxidative dissolution to the reduction of water (protons) and to no other available oxidizing agent, including dissolved O_2. This is because the universal oxide scale on Fe^0 acts as diffusion barrier to dissolved species and a conduction barrier to electrons from the metal body. In other words, water is the sole chemical which can remove electrons from the Fe^0 surface. Fe^0 oxidation and water reduction must not necessarily occur at the same locality. The spatial separation of oxidative (anodic) and

reductive (cathodic) reactions is possible as the metal body allows the free flow of electrons from anodic to cathodic sites. The tendency of Fe^0 to give off electrons (Equation 1) is the same for all Fe^0-based materials ($E^0 = –0.44$ V). This makes material selection and characterization critical in designing sustainable Fe^0/H_2O systems.

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