

Review

Iron Sulfide Minerals as Potential Active Capping Materials for Mercury-Contaminated Sediment Remediation: A Minireview

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Abstract: Several innovative approaches have been proposed in recent years to remediate contaminated sediment to reduce human health and environmental risk. One of the challenges of sediment remediation stems from its unfeasible high cost, especially when ex situ strategies are selected. Therefore, in situ methods such as active capping have been emerging as possible options for solving sediment problems. Active capping methods have been extensively tested in field-scale sediment remediation for organic pollutants (e.g., PCBs, PAHs, DDT) contamination with good sequestration efficiency; however, these methods have not been widely tested for control of heavy metal pollutants, such as mercury (Hg). In this review, the potentials of using iron sulfide minerals to sequester Hg were discussed. Iron sulfide minerals are common in the natural environment and have shown good effectiveness in sequestering Hg by adsorption or precipitation. Iron sulfides can also be synthesized in a laboratory and modified to enhance their sequestration ability for Hg. Some of the potential advantages of iron sulfides are pointed out here. Additional tests to understand the possibility of applying iron sulfides as active caps to remediate complicated environment systems should be conducted.

Keywords: iron sulfide; mercury; sediment remediation; active capping

1. Mercury Risk and Global Management Efforts

Mercury (Hg) has been recognized as one of the most hazardous heavy metals due to its high volatility, persistence, and bioaccumulation in human beings and natural biota [1]. Long-term, high-dosage exposure of humans to Hg may pose risk to the brain, heart, kidneys, lungs, and the immune system by causing various neurodegenerative diseases, such as Minamata disease, Alzheimer's disease, and Parkinson's disease [1–5]. Hg can be released from both natural or anthropogenic sources. With elevating human industrial activities, much Hg has been mined out of the earth and discharged to the environment as waste. Major anthropogenic Hg sources include gold mining, coal combustion, and metal production [1,6–9], which have created a huge amount of Hg-containing wastewater to be discharged into wetlands, rivers, and seawater. Amos et al. [10] estimated that around 5500 ± 2700 Mg of aquatic Hg is released into rivers annually across the globe. Excluding natural mobilization of Hg from terrestrial ecosystems, Kocman et al. [11] estimated riverine discharge of Hg with a smaller value of around 800 to 2200 Mg/year. Street et al. [12] suggested that approximately 40% of combined Hg releases to land and water are sequestered rather than travel in rivers to the ocean.

Global efforts on Hg management have been put together through the Minamata Convention on Mercury, a global treaty to protect human health and the environment from adverse effects of

Hg. Updated on August 2018, 128 nations have signed the treaty with requirements including banning of primary Hg mining, reductions in Hg release to the environment (air or water), exposure through products and consumptions, remediation of contaminated regions, monitoring, outreach, and other means.

As Hg is discharged into aquatic environment (i.e., wetlands, lakes, and coasts), sediments may serve as the sink for Hg [13], which is mainly in the form of mobile divalent mercury (Hg^{2+}) and may transform to monomethyl mercury (MeHg) and gaseous mercury (Hg^0) through resuspension and diffusion [14,15]. Sediment suspension can be induced by natural physical processes (e.g., waves and storms) [16,17], anthropogenic activities (e.g., dredging, trawling, and boating) [18] and biological activities (e.g., bioturbation) [19].

Hg in natural waters occurs in forms including Hg^0 , ionic mercury (e.g., Hg^+ and Hg^{2+}), and methylated mercury (e.g., CH_3Hg^+ , $(\text{CH}_3)_2\text{Hg}$) [20]. While all forms of Hg are toxic, its methylated form, MeHg, is specifically problematic. MeHg has high bioaccumulation and biomagnified ability and poses a threat to biota and human [21]. MeHg usually occurs in minor proportions, at around 0.1–1.5% of total Hg [22–25] in anoxic sediment environments, but can pose a high risk to human health. Typically, preliminary remediation goals (PRGs) can be set as 2.6 ng/L in surface water, as this concentration represents the lowest observed adverse effect level (LOAEL) of the river otter [26].

Much effort on Hg control has been put in water treatment and sediment remediation. For wastewater treatment of Hg, lime softening, chemical precipitation, coagulation, reverse osmosis, ion exchange, and membrane filtration have been considered [27]. As for sediment and groundwater Hg remediation, many techniques have been employed, such as adsorption, biosorption, ion exchange, chemical precipitation, reduction, and stabilization/fixation. Among these methods, adsorption and stabilization/fixation have been proved to be the most practical techniques [8,28,29]. Dredging followed by ex situ methods has been commonly executed due to its long-term effectiveness and relatively short remediation period [30]. However, dredging can be preventatively costly. Also, dredging may cause remobilization of contaminants during remediation, and may inevitably retain contaminants, posing a long-term threat [31].

2. In Situ Remediation and the Need for Alternative Mechanisms

One of the main advantages of in situ technologies is that they used toward reducing environmental impacts, avoiding the huge cost of dredging, and leading to remobilization of contaminants. The main challenges are related to the difficulties in ensuring that the contaminants are effectively reduced by the technologies due to the heterogeneity of sediment and the uncertainty in the distribution of contaminants. In situ technologies require a treatability test to evaluate their efficiency at the specific site [32].

A relatively new term of “active capping”, which involves the use of chemically reactive materials to sequester contaminants to reduce their mobility, toxicity, and bioavailability, could immobilize contaminants in sediment [33]. The remediation cost of active capping is likely to be smaller than for conventional strategies (e.g., dredging or sand capping). Additionally, active capping may further enhance or improve habitat recolonization by benthic organisms [34]. Active materials, such as activated carbon, have been shown to reduce the toxicity of hazardous organic compounds (HOCs), polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), and pesticides in laboratory or field studies [35–44]. Since 2015, more than 25 field studies have been conducted by active capping approaches [45].

For the application of active capping on Hg-contaminated sites, laboratory studies have been conducted using activated carbon [45–49], biochar [46,50], or surface-modified black carbon [49,51] to immobilize Hg. Some studies have shown that the amendment of black carbon may successfully reduce bioavailable Hg and MeHg to benthic organisms [47,48]. Nevertheless, the sorption capacity of activated carbon toward Hg is still limited due to the nonpolar characteristics of activated carbon, which hinder interactions between charged metal species and the solid surface [29]. The other main

concern of applying black carbon to sediment remediation is its possible adverse effects on the benthic organisms itself.

To date, a promising active material in remediating Hg-contaminated sites is yet to be proven with good adsorption efficiency, stability, and eco-friendliness. Consequently, further research is needed in several areas, including (1) fabricating novel capping materials with good adsorption affinity for Hg or other heavy metals; (2) establishing competitive adsorption models of amendments in sediment condition to evaluate real adsorption outcomes in nature disturbance; (3) introducing amendments with biological tests to prove the efficiency of reducing Hg bioavailability; (4) developing efficient, low-impact capping delivery systems; (5) assessing long-term stability and ecological recovery of placing active caps; (6) conducting life-cycle analysis for active capping remediation.

3. Iron Sulfide Minerals: Potential Alternatives

3.1. Introduction of Iron Sulfide Minerals

Iron sulfide minerals (Fe_xS_y) have been found to have good immobilizing ability to double-valent metals, such as Mn^{2+} , Ca^{2+} , Mg^{2+} , Ni^{2+} , Cu^{2+} , Cd^{2+} , Co^{2+} , Zn^{2+} , Pb^{2+} , and Hg^{2+} [52–57], basically due to their adsorption or precipitation properties (mechanisms are further described in Section 3.2). In addition, iron sulfides have also been reported to degrade organic contaminants by reduction mechanisms. The overall reaction mechanisms of iron sulfides to different contaminants are illustrated in Figure 1 [58]. The common forms of iron sulfide minerals include mackinawite (FeS), greigite (Fe_3S_4), pyrite (FeS_2), and pyrrhotite (Fe_{1-x}S) [59]. In earlier research, mackinawite, pyrite, and pyrrhotite have been shown to have high potential to sequester Hg [60,61]. In more recent studies, scientists tend to use lab-synthesized nano-FeS (mackinawite) for adsorption due to its high surface area. Strategies for synthesizing nano-FeS were often proceeded by mixing S^{2-} solution and Fe^{2+} solution with equivalent molar of Fe and S under an anoxic environment. The mixture of Fe and S would react immediately and precipitate into FeS (mackinawite). The precipitant was often subsequently treated by aging, centrifuging, rinsing, and finally storing in a nitrogen environment as nano-FeS final products. The S^{2-} solution was often prepared by $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$ crystal [62,63]. As for Fe^{2+} solution, $\text{FeSO}_4\cdot(\text{NH}_4)_2(\text{SO}_4)_2\cdot 6\text{H}_2\text{O}$ (Mohr's salt), $\text{FeSO}_4\cdot 7\text{H}_2\text{O}$, or $\text{FeCl}_2\cdot 6\text{H}_2\text{O}$ [62,63] were often used. The synthesized FeS was reported with a specific surface area ranging between 7 and $100\text{ m}^2/\text{g}$ [53,64], with a particle size within the range of few nanometers to 400 nm, depending on the aging time [65].

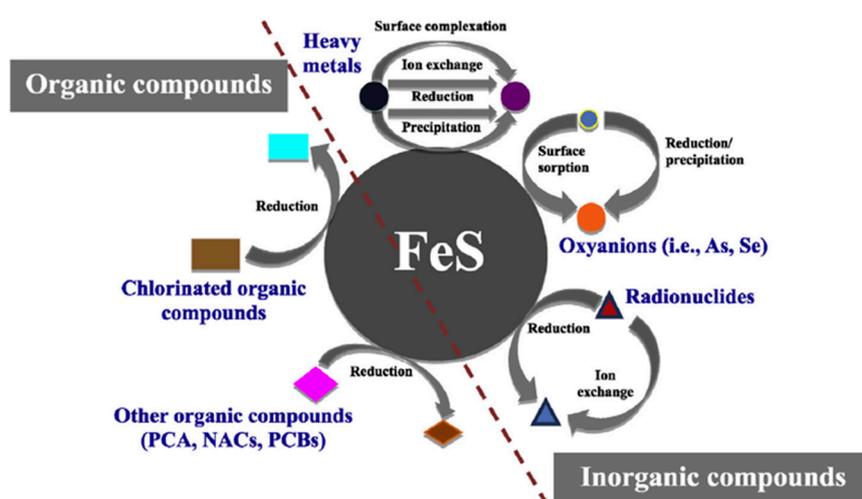


Figure 1. Schematic diagram of the reaction mechanisms of iron sulfide with various contaminants (Gong et al. [58]).

Some of the pioneering studies have been conducted using FeS to sequester Hg (Table 1). Liu et al. [59] reported FeS with adsorption capacity up to 1700 mg/g (by calculation) at pH = 5.6; more than

99% Hg was removed at Hg/FeS ratio < 1000, with 77% of the removal by precipitation and 23% by adsorption (Hg/FeS = 0.22). Sun et al. [64] found that commercial pyrite had lower Hg sorption capacity (9.9 mg/g) as compared to laboratory-synthesized mackinawite (769.2 mg/g), probably due to the smooth surface of pyrite. Skyllberg and Drott [66] conducted a slurry batches study, discovering that the dosage of 2% FeS in 5000 µg-Hg/g organic-rich soil may outcompete O/N ligands in sediment for Hg sorption, resulting in 50% Hg sorbed on FeS to form HgS₄ (metacinnabar). As the ratio of FeS increased to up to 20%, a complete outcompete with 100% Hg sorbed on FeS was observed.

3.2. Hg Sequestration Mechanisms by Iron Sulfides

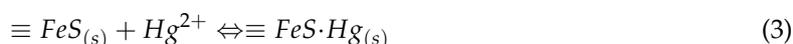
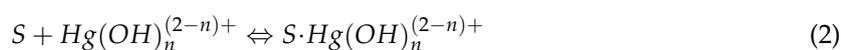
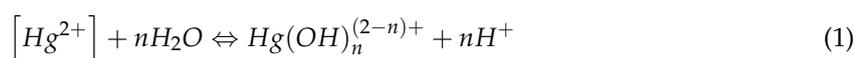
Hg is a soft Lewis acid, therefore it shows high affinity to sulfur, a Lewis base, to form HgS_(s). HgS_(s) has two polymorphs: red cinnabar (α-HgS) and black metacinnabar (β-HgS), both are extremely stable, with K_{sp} = 2 × 10⁻⁵⁴ [67] and 2 × 10⁻⁵⁴ [68] for cinnabar and metacinnabar, respectively, and even more stable than complexes with organic thiol (K_{sp} = 10^{-22~--28}) [20]. Therefore, sulfur is generally recognized as a primary sink for Hg in the environment [69,70].

FeS can sequester Hg by adsorption (Equation (3)), substitution (Equation (4)), and precipitation (Equation (7)). Adsorption includes complexation of Hg with reactive sites on FeS. Jean and Bancroft [60] suggested that the adsorption of Hg can be achieved by two steps: (1) hydrolysis of Hg ions to initiate adsorption behavior (i.e., Equations (1) and (2)) or (2) monolayer adsorption by a specific adsorption site (i.e., Equation (3)).

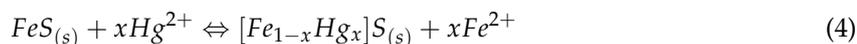
Substitution of FeS to HgS occurs by fitting Hg²⁺ molecules into Fe²⁺ sites of FeS_(s). Therefore, the feasibility of substituting FeS to HgS can be assessed by comparing the coordination number and bonding length. The Fe–S bond of mackinawite has the structure of four-fold coordination and bonding length of 2.24 Å [71]. Although metacinnabar has four-fold coordination, the bonding length of β-HgS is 2.53 Å (~0.29 Å exceeded) [72]; therefore, mackinawite's surface is unsuited for Hg substitution.

Precipitation (Equation (7)) occurs after partial dissolution of FeS_(s) (Equation (6)). Deonarine and Hsu-Kim [73] demonstrated nanoparticle β-HgS_(s) formation and suggested that the bioavailable neutral HgS species may be nanoparticles rather than the neutral HOHgSH⁰ previously proposed. These nano-HgS_(s) particles were found to be more structurally disordered than micro-HgS_(s) [74] and had a great contribution to Hg methylation [75]. Moreover, nano-HgS_(s) was found to have decreasing methylation potential with aging time [76] as nano-HgS_(s) aggregated [73]. As there is limited scientific research for FeS in biological tests, whether FeS decreases Hg bioavailability is still unclear.

Adsorption [72,77]



Substitution [72,77]



Precipitation (after partial dissolution of FeS) [66,72,77]



Table 1. Adsorption of Hg by various iron sulfide materials.

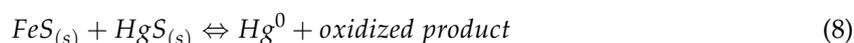
Type of Adsorbent	Nano-CMC-FeS	Nano-CMC-FeS	FeS/Al ₂ O ₃	Nano-Fes, Pyrite	Nano-FeS	Nano-FeS	Nano-FeS
Hg concentration range	21.96, 193.04, and 100.53 mg/kg Hg sediment	177 mg/kg Hg sediment	0–80 mg/L	1–100 mg/L	100, 200, and 250 mg/L	0.01–1 mM	0.05–20 mM
Hg to sorbent ratio (w/w)	0.011–0.11, 4.26×10^{-3} – 8.47×10^{-3} 0.018–0.036	0.038–0.5	0–10	0.06–0.6 (FeS) 0.2–20 (pyrite)	0.2, 0.25, 0.5	50–4000	4.4×10^{-4} –3.5
Sorption condition	pH = 7.0; T = 20 °C; t = 20 h; on rotator 30 rpm; 0.1 M NaNO ₃	pH = 7.0; T = 22 °C; t = 1 week; on rotator 30 rpm	pH = 6.0; T = 30 °C; t = 24 h; on shaker 180 rpm	pH = 7.0; T = 30 °C; t = 24 h; on shaker 180 rpm	pH = 8; t = 10–144 min; rotated	pH = 5.6; T = not stated; t = 24 h; on shaker; anoxic with constant N ₂ purging	pH = 4–11; T = 25 °C; t = 48 h [Cl] _T = 0.2 M; on shaker
Removal efficiency of Hg	Q _{max} = 2866.6 mg/g 79–96% removal in batch contacted with Hg sediment.	Distribution coefficient = 8.93×10^6 Hg _(aq) decrease > 73% in all batches contacted with Hg sediment, with the maximum reduction of 97% (Hg/sorbent = 0.5).	Q _{max} = 891 mg/g (theoretical) Q _{max} = 313 mg/g (observed) >97% removal in 24 h and >95% removal in 30 d test.	Q _{max} = 762 mg/g (FeS) Q _{max} = 9.9 mg/g (pyrite)	K _D = 1.98×10^6 More than 99% removal	Q _{max} = 1700 mg/g Hg removal >99% when Hg/FeS is smaller than 1000 at [Hg] ₀ = 1 mM	Q _{max} = 88 mg/g Removal of Hg was higher than 99% when Hg/FeS is smaller than 0.05
Reference	[63]	[78]	[79]	[64]	[62]	[59]	[72]

Jeong et al. [72] discovered that adsorption and precipitation may have different contribution proportions within different Hg/FeS ratios (molar ratio). It was reported that with Hg/FeS < 0.05, adsorption dominates sorption mechanisms; when Hg/FeS between 0.05 and 1, precipitation starts to occur; as Hg/FeS ratio rises above 1, the formation of HgCl₂ clusters may occur. Some lateral studies had similar observations [59], while others found different results with no adsorption mechanism found with Hg/FeS at around 0.002–0.012 [66]. A possible explanation is a difference in aging time of FeS materials, or different ionic medium used in the sorption test ([Cl] = 0.2 M used by Jeong et al. [72]).

3.3. Hg Reduction by Aqueous Fe²⁺

Besides sulfur, reduced ferrous ion also has a great impact on Hg sequestration. Reactions of Fe²⁺ and Hg²⁺ may result in the reduction of Hg²⁺ to Hg⁰, which is more volatile and thus reduces exposure to methylating microorganisms (Equation (8)) [80]. This hypothesis was first proposed by Barringer et al. [81], who found trends of the positive correlation between Fe concentration and Hg concentration through observation of 14 underground wells. Lamborg et al. [82] observed a Hg pollution plume underground and found a high correlation of Fe²⁺ concentration and Hg⁰ concentration. It was suggested that the direct reduction of Hg²⁺ may be caused by magnetite, goethite, or aqueous Fe²⁺ [83–85]. Hg⁰ may also be synthesized as a byproduct of Fe-reducing microorganisms' metabolism [86,87]. The study of Bone et al. [80] used Hg L_{III}-edge extended X-ray absorption fine structure spectroscopy (EXAFS) and batch experiments to provide more insight on Fe reduction. In this work, the addition of mackinawite in anoxic Hg solution may cause precipitation and form β-HgS_(s) (see Equations (5)–(7)), releasing Fe²⁺ into solution. The release of Fe²⁺ may cause β-HgS_(s) redissolution and reduce Hg²⁺ into Hg⁰ in a relatively short amount of time (i.e., 1 h). This work suggested that Hg⁰ may account for a significant fraction of Hg in FeS-rich or FeS-amended sediment, also in agreement with the finding of previous research [88].

Reduction of Hg by Fe²⁺ [78,89]



3.4. Advantages of Using Iron Sulfides

Using FeS as an active capping material could be highly beneficial because the great performance of FeS sequestration of Hg has been reported. Although studies related to reducing Hg bioavailability of in situ investigations are still limited, some potential advantages of using FeS can be proposed here.

First, FeS has larger Hg sorption capacity and affinity as compared to black carbon materials. In general, black carbon materials have Hg sorption affinity at log(K_D) around 3–7 [46] and sorption capacity (by Langmuir definition) around 100–500 mg/g [8]. For FeS, Hg sorption affinity can be as large as log(K_D) 6–7 and the adsorption capacity can exceed 1000 mg/g [59].

Second, sorption of Hg on FeS could be highly selective among many other double-valent heavy metals. Since Hg²⁺ and S²⁻ have a high precipitation constant (log K~20.6) [90], few metals are likely to compete with Hg for S²⁻, even though Hg may be blocked by chloride complexation [90].

Third, by using FeS, a common mineral in sediment, adverse bio-effects of amendments may be less than that by using black carbon materials. One-fifth of previous studies using black carbon to sequester Hg have reported adverse bio-effects [91]. Benthic species suffered from reduced species richness, biomass loss, reduced feeding rate, organ damage, or reduced growth after carbon amendment. Although not yet fully understood, possible explanations are that black carbon may bind to nutrients, thus reducing their bioavailability to the benthic ecosystem. This may not be the case if FeS is used as amendment materials since FeS is a natural mineral. However, there is little information to support this perspective.

Fourth, Hg sorbed on FeS has high stability, thus it is unlikely to remobilize into the environment or be bioavailable for Hg-methylating microorganisms. As suggested in Section 3.2, precipitation of

Hg and FeS can be highly stable. In fact, Hg–S complexes are the dominant Hg species in the sediment environment (even with S concentration as small as 1 nM) by thermal dynamic calculation [7] and by site investigations [70,92]. In an oxic environment, Wolfenden et al. [93] suggested that Hg and S had good affinity in batch experiments. On the other hand, the release of Fe after FeS amendment may also reduce Hg mobilization and methylation. Mehrotra et al. [94] discovered that by amending $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$, net production of MeHg was decreased in *Desulfobulbus propionicus* (1pr3), a known Hg-methylating, sulfur-reducing bacteria. By the addition of $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ in 6 estuarine sediment batches, Mehrotra and Sedlak [95] found that Hg methylation increased at lower Fe concentration (0.3–3 mM), but methylation was decreased at higher Fe concentration (30–300 mM). With addition of 30 mM Fe, net methylation was reported to decrease by a factor of 2.1–6.6.

Despite many potential advantages for applying FeS to Hg-contaminated sediment, uncertainties in this approach should also be addressed. One of the uncertainties for FeS capping is that this application may cause Hg reduction to Hg^0 , as mentioned in the previous section. The strategy of active capping involves chemical binding to sequester contaminants; hence, whether a portion of Hg reemitted to the atmosphere as Hg^0 is acceptable should be further studied and discussed. The other uncertainty concerns the enhancement of Fe content in sediment, which could lead to a significant change in the oxidation-reduction conditions of applied sediment and possible release of Fe into overlying water. Microcosm or field studies should thus be conducted to verify these uncertainties before FeS capping is applied to actual sites.

3.5. Material Engineering

As demonstrated in the previous section, FeS is a material with high potential for Hg sequestration. However, limitations of FeS have been considered, such as kinetic adsorption availability, as laboratory-synthesized FeS would aggregate [77] to reduce its surface area (7–50 m^2/g) [53,59]. Some efforts have been made to stabilize (i.e., via inhibiting aggregation) nano-FeS by carboxymethyl cellulose (CMC) addition. The highly negative surface charges of CMC-FeS were found to maintain greater surface areas of FeS by inducing strong electrostatic repulsion, thereby preventing particle agglomeration [63,78]. Thus, particle size can be controlled in a range of 15–75 nm, with a mean size of 35 nm [77]. The schematic difference of FeS and CMC-FeS synthesized in a laboratory is illustrated in Figure 2 [77]. The CMC-FeS after treatment was found to have improved Hg sorption. Xiong et al. [78] investigated the effectiveness of CMC-FeS to reduce aqueous Hg in sediment. CMC-FeS demonstrated high affinity to Hg with a distribution coefficient of 8.93×10^6 L/kg (with Hg/adsorbent ratios = 0.038–0.5). The aqueous Hg in Hg sediment batch tests was reduced by >73% in all batches with a maximum reduction of 97% (Hg/sorbent = 0.5). Furthermore, the column tests showed that leachable Hg was reduced by 47%. Gong et al. [77] also used three types of soil or sediment to proceed with similar studies. The aqueous Hg in Hg-containing sediment batch tests reduced by 79–96% (with Hg/adsorbent ratios = 4.26×10^{-3} – 0.036). The column tests showed that leachable Hg was reduced by 90–93%.

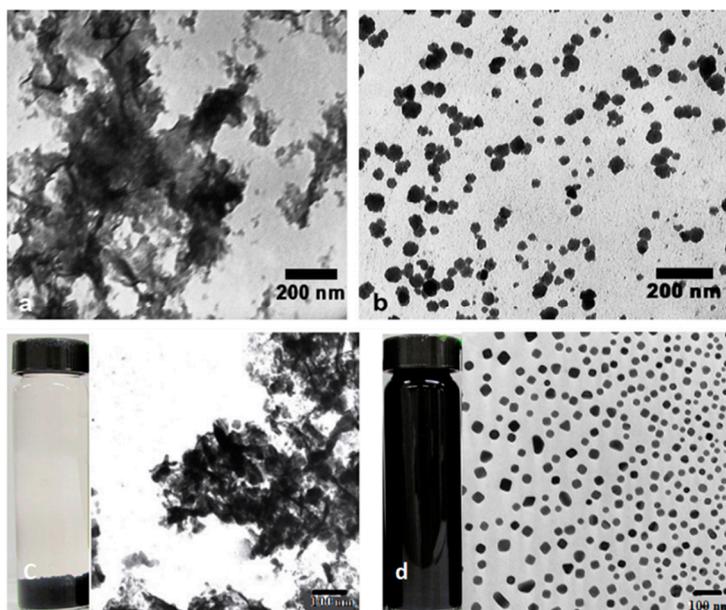


Figure 2. Schematic diagram of the lab-synthesized nano-FeS (a,c) without carboxymethyl cellulose (CMC), as compared to CMC-FeS with (b) 0.2% CMC (Xiong et al. [78]) and (d) 0.05% CMC (Gong et al. [77]). For here FeS = 0.5 g/L.

4. Conclusions

In this review, FeS has been demonstrated as a high-potential amending material in remediating Hg-polluted sediment via active capping approaches. Greater sorption capacity, sorption affinity, and stability of FeS to Hg were observed as compared to those found in black carbon studies. Adsorption and precipitation of FeS have been revealed as major mechanisms of Hg sequestration of FeS. However, many challenges of using FeS in real site remediation still remain, as FeS has not been tested in in situ studies. Information about the bioeffects of additional FeS in the sediment environment is also limited. Nonetheless, the application of FeS as an active capping material remains of high potential and thus should be further studied.

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