



Potential leaching of mercury from stable mercury sulphide

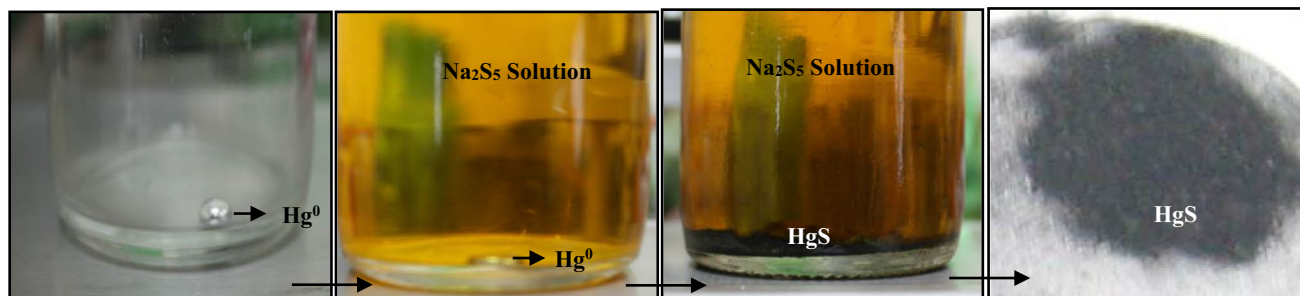
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Received: 4 April 2020 / Revised: 27 October 2020 / Accepted: 4 December 2020 / Published online: 17 May 2021
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Abstract

The study aims to stabilize elemental mercury to mercury sulphide and investigate the effect of various groundwater interferences like chloride, nitrate, sulphate and bicarbonate on the dissolution of mercury from mercury sulphide and elemental mercury. Elemental mercury was stabilized using sodium polysulphide solution in a one-step batch experiment in a period of 96 h. Mercury sulphide was formed as a black fine powder with average particle size of 10–500 nm. Mercury sulphide was tested under different pH conditions and under different concentrations of Cl^- , NO_3^- , SO_4^{2-} , and HCO_3^- for dissolution of mercury. At $\text{pH} \leq 4$ and $\text{pH} \geq 12$, dissolved mercury concentrations from mercury sulphide were 70.47 $\mu\text{g/L}$ and 41.81 $\mu\text{g/L}$, respectively. At pH 10, mercury dissolution was the lowest with dissolved mercury as 8 $\mu\text{g/L}$ proving that mild alkaline pH is necessary for stability of mercury. At low concentration of the anions, interfering effects were in the order of $\text{NO}_3^- > \text{Cl}^- > \text{SO}_4^{2-} > \text{HCO}_3^-$, whereas under high concentrations, the order was $\text{Cl}^- > \text{NO}_3^- > \text{SO}_4^{2-} > \text{HCO}_3^-$. Unlike NO_3^- and Cl^- , SO_4^{2-} and HCO_3^- ions were reported to cause potential leaching only if they are present in significantly high concentrations. Nevertheless, mercury sulphide was found to be the preferred chemical state for permanent storage of mercury in the subsurface when compared to elemental mercury.

Graphic abstract



Keywords Elemental mercury · Groundwater · Interferences · Mercury sulphide · Stability

Editorial responsibility: Hari Pant.

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Introduction

Global usage of mercury, though curtailed, is significant enough to cause risk and threat to the environment. It is estimated that approximately 2220 metric tons of mercury is emitted to the global environment per year (UNEP 2018). Mercury contamination occurs in the form of elemental mercury, inorganic mercury, organometallic mercury and particulate mercury. When elemental mercury (Hg^0) enters the soil, some mercury will volatilize and serve as a continuous source of vapour phase



contamination while the remaining mercury gets trapped in the void spaces of the soil. Once trapped in the soil, Hg° may be subjected to oxidation to soluble inorganic mercury which in turn may be subjected to biological activity leading to methyl mercury (CH_3Hg) which is highly toxic, mobile and easily bioavailable (Dranguet et al. 2014; Grégoire and Poulain 2018). Hg° is prone to oxidation in the presence of oxygen and other strong oxidants like chlorine, hypochlorite and hydrogen peroxide. In soils low in organic matter, mercury is found as mercuric chloride (HgCl_2) or mercuric hydroxide ($\text{Hg}(\text{OH})_2$) which is later transformed into CH_3Hg (Zhou and Dreisinger 2017).

When mercury is trapped in the soil as residual Hg° , it is essential to stabilize Hg° to contain its transformation to other forms of mercury and curb the vapour phase emission. Transforming Hg° to less toxic form or removal of Hg° from contaminated sites involves onsite technologies such as adsorption, amalgamation, oxidation and reduction. Though amalgamation is a successful technology, the final product is subjected to volatilization and hydrolysis. Thermal treatments like vitrification and incineration cause significant air emissions. Electrokinetic remediation target mercury only if it is in ionic form. Soil flushing with water or surfactant may not effectively mobilize the trapped Hg° (Devasena and Nambi 2013). Recent technologies like photocatalysis and microbial treatment suffer from usage in large-scale field applications (Zhang et al. 2015). Nanomaterials such as carbon nanotubes and graphene nanoparticles are gaining much interest either as stand-alone materials or through functionalization with amine, carbonyl or thiol groups where the removal efficiency of mercury is increased (Wang et al. 2020).

Stabilization is a proven technology where different chemical reagents are employed for controlling the release of vapour phase mercury or dissolved mercury. As stabilized mercury is the ideal form to contain the insidious hazard posed by trapped Hg° , the best stabilizing agents and its robustness in stabilization have to be investigated. Chemical stabilization approaches include the use of sulphur compounds such as elemental sulphur, thiosulphate and sodium sulphide that are allowed to react with Hg° to form mercury sulphide (HgS). However, while using the above agents, pre-treatment may be essential to prevent dissolution of mercury (He et al. 2015). Traditional cement-based stabilization techniques suffer from the limitations of maintaining high pH and undesirable mercury release from cement (Rachman et al. 2018). Zhang and Bishop (2002) used powdered activated carbon (PAC) in combination with Portland cement for treating 1000 mg/Kg of mercuric nitrate $\text{Hg}(\text{NO}_3)_2$. They suggested pre-treatment of PAC with carbon disulphide (CS_2) is essential for the improved results with an optimum pH of 5 to 5.5. However, a high risk of mercury vapour along with generation of hydrogen sulphide gas prevails.

Zhang et al. (2015) utilized natural zeolite and thiol functionalized zeolite which exhibited effective stabilization of mercury even with lower dosage of zeolites (0.35 g/g of mercury) and thiol functionalized zeolite (0.5 g/g of mercury). Sulphur polymer cement (SPC), a mixture of 95 wt% elemental sulphur and 5 wt% organic modifiers were also used to stabilize mercury. In a study by Fuhrmann et al. (2002), Hg° was heated at 104 °C with powdered SPC to form a molten liquid and later set to form a monolithic waste. Lopez-Delgado et al. (2012) encapsulated liquid mercury using sulphur polymer stabilization where Hg° was mixed with sulphur powder and the resultant HgS was mixed with the inert sulphur containing polymer and heated to 145 °C while stirring for 30 min. The resultant mixture was solidified into high strength construction blocks showing superior mechanical properties. Weisener et al. (2005) demonstrated the removal of dissolved mercury from groundwater using zerovalent iron as the reactive media. Dissolved mercury removal occurred as a result of reductive precipitation of zero valent iron and sulphate present in ground water under alkaline conditions with moderate Eh values. The above-reported stabilizing agents have limitations with pH, generation of toxic gases, addition of heat and efficiency of stabilization.

Mercury forms complexes with soft ligands including chlorides, sulphides, and hydroxides and generates insoluble compounds. Among the complexes, HgS is the most thermodynamically stable form which is formed as a result of the chemical reaction between elemental mercury and sulphur or sulphur compounds (Devasena and Nambi 2013). HgS has the potential to serve as a long-term undisturbed form of mercury in the subsurface. Mercury exhibits a very high affinity for sulphide in mildly reducing environments (100 mV to – 100 mV) such as in stream and lake sediments forming insoluble HgS while HgCl_2 and $\text{Hg}(\text{OH})_2$ are dominant under highly oxidizing conditions (> 400 mV) (Wang and Driscoll 1995). According to Barnett et al. (2001), investigations on mercury leaching from HgCl_2 and HgS samples revealed that HgCl_2 is highly soluble with high bioavailability potential of 100% when compared to HgS which exhibited bioavailability of less than 1%. Study by Svensson et al. (2006) ensured permanent storage of mercury as HgS in groundwater saturated repository in granite bedrock. They explored various conditions for formation of HgS using sulphur and iron sulphide as sulphur sources and found that HgS would be formed under anaerobic, alkaline conditions with elemental sulphur as the suitable sulphur source. Anaerobic condition with pH 10.5–11 and sulphur/mercury molar ratio of 1–1.3 was found to be the optimum conditions for the deep permanent storage of HgS .

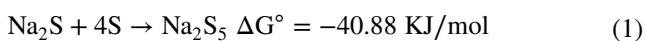
Han et al. (2006) studied the distribution of mercury in soil spiked with various forms of mercury such as HgS , $\text{Hg}(\text{NO}_3)_2$ and HgCl_2 . It was found that soil contaminated

with HgS showed insignificant mercury solubility compared with $\text{Hg}(\text{NO}_3)_2$ and HgCl_2 contaminated soil. Oxidation of HgS was tested by adding 30% hydrogen peroxide to the contaminated soil residue in agitated condition. The authors did not encounter HgS oxidation, and therefore, it has been reported that HgS has high relative binding intensity of mercury compared to other soluble mercury compounds such as $\text{Hg}(\text{NO}_3)_2$ and HgCl_2 . HgS is classified as non-hazardous and stable of all forms of mercury compounds. It is the most insoluble metallic sulphide compound with $\log K_{\text{sp}}$ value of $2 \times 10^{-52.7}$ (Devasena and Nambi 2013). As the leaching of mercury in the subsurface is no longer considered trivial, it becomes essential to investigate the leaching potential of mercury from stabilized HgS under the influence of groundwater interferences such as chloride (Cl^-), sulphate (SO_4^{2-}), nitrate (NO_3^-) and bicarbonate (HCO_3^-) ions and under different pH conditions. The results are then compared with dissolution of mercury from Hg° present in unstable form.

Materials and methods

Batch experiments were conducted (i) to find the optimum condition that promotes HgS formation, (ii) to find the time required for complete HgS formation, (iii) to investigate the dissolution of mercury from stable HgS and (iv) to compare the results with dissolution of mercury from unstable Hg° . Elemental sulphur (S°), sodium sulphide nanohydrate ($\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$) and Hg° were obtained from M/s Rankem Chemicals. As per Eq. 1, stock solution of Na_2S_5 was prepared by mixing $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$ and S° in distilled deionized water at 24 °C in an orbital shaker at 100 rpm until Na_2S_5 formation was established. According to Eq. 2, known weight of Hg° was taken in acid-cleaned serum bottles and freshly prepared Na_2S_5 solution was added to it by varying the molar ratio of $\text{Na}_2\text{S}_5/\text{Hg}^\circ$ from 0.5 to 3.5. pH of the mix was found to be 10.5. The bottles were shaken by continuous stirring at 200 rpm in an orbital shaker and were monitored for HgS formation at the end of 3, 6, 9, 12, 24, 48, 72 and 96 h.

Once HgS was formed, samples were filtered using glass microfiber filters of size 0.45 μm . The amount of Hg° precipitated as HgS was found out gravimetrically. Filtrates were tested for dissolved mercury if any. Similar batch experiments were conducted independently with S° and Na_2S in order to find the best sulphur source for stable HgS formation.



PerkinElmer Analyst 700 Atomic Absorption Spectroscopy (AAS) equipped with a flow injection automated system (FIAS) 100 cold vapour analyser was used to analyse dissolved mercury. Absorbance was measured at 253.7 nm wavelength as a function of mercury concentration. The detection limit was 0.2 $\mu\text{g/L}$. Surface morphologies and crystalline sizes of HgS were found using scanning electron microscopy (SEM) analysis (JEOL, JSM-6380, Japan). To obtain a much higher spatial resolution, the characterization was also supplemented by high-resolution transmission electron microscopy (HR-TEM) analysis (JEOL 3010 UHR instrument). HgS precipitates were ultrasonically dispersed in isopropyl alcohol and a small aliquot was spotted on a carbon-coated copper grid and was allowed to dry. It was then observed under HR-TEM. Sample solutions were measured for size distribution with particle size analyser (Wet particle size analyzer, Brookhaven 90 plus).

Following stable HgS formation and its size characterization, dissolution of mercury from HgS precipitates was investigated in the presence of different anions. Concentrations of 5, 10, 50, 100, 250, 500 and 1000 mg/L of Cl^- , SO_4^{2-} , NO_3^- and HCO_3^- ions were prepared independently with distilled deionized water to which 0.5 mg of stable HgS particles were added. The reaction mixtures were shaken for 24 h at 200 rpm in an orbital shaker. After 24 h, the mixtures were filtered and the filtrates were analysed for mercury. The same procedure was adopted for testing the dissolution of mercury from Hg° . Dissolution of mercury from Hg° and HgS was tested under different pH values of 2, 4, 6, 8, 10 and 12. pH was adjusted using 0.1 N NaOH solution and 0.1 N HCl. All batch experiments were repeated in duplicates.

Results and discussion

The effect of pH and the interferences of Cl^- , SO_4^{2-} , NO_3^- and HCO_3^- ions on mercury stabilization were investigated by introducing the salts at different concentrations to stable HgS and unstable Hg° and by subjecting HgS and Hg° under different pH conditions. Stable HgS precipitates were produced with Na_2S_5 solution in batch experiments with $\text{Na}_2\text{S}_5/\text{Hg}^\circ$ molar ratio of 1 in a period of 96 h (Fig. 1). Na_2S_5 solution was formed as a one-step reaction with elemental sulphur and sodium sulphide. The molar ratio of $\text{Na}_2\text{S}_5/\text{Hg}^\circ$ was fixed as 1 since it resulted in complete stabilization in shortest time period (Devasena and Nambi 2013). Independent batch experiments with other sulphur sources such as S° and Na_2S with the same molar ratio of 1 did not yield HgS precipitates. Sulphur particles were adsorbed only on the surface of Hg° and were unsuccessful to disrupt the cohesive forces within Hg° demonstrating the treatment to be a surface phenomenon. The reaction of mercury with



Fig. 1 Conversion of elemental mercury to mercury sulphide

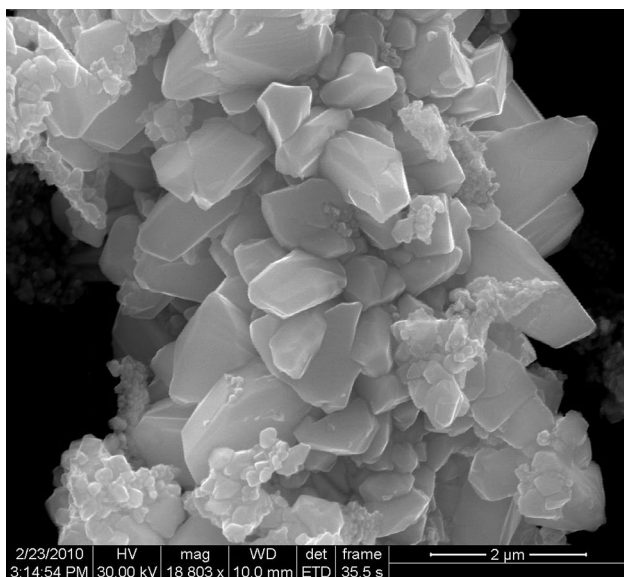
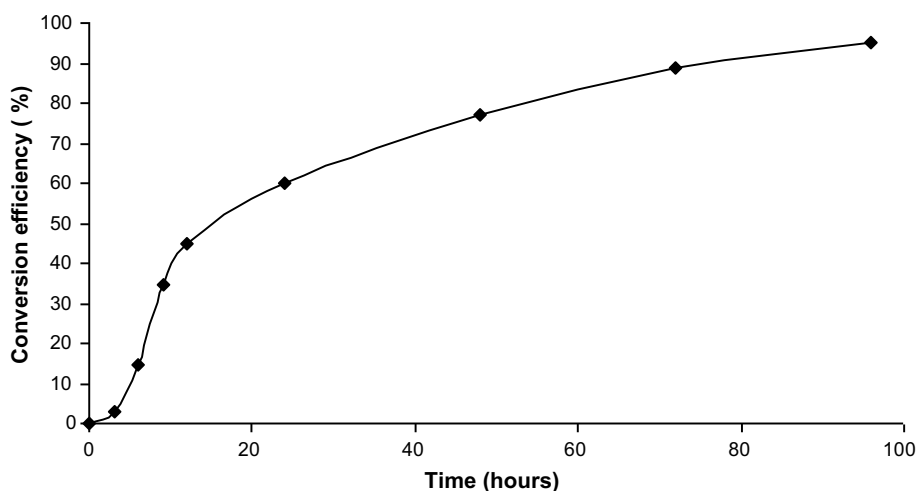


Fig. 2 SEM image of mercury sulphide particle

Na_2S_5 was thermodynamically favourable, indicating that Na_2S_5 is a suitable sulphur source under mild alkaline condition (pH 10.5) generated during the reaction.

Figure 2 shows the SEM images of the HgS precipitates where several crystallites in micron and nanorange were present. The particles were not completely homogeneous and different particles sizes in the range of 10–500 nm were observed. It is possible that HgS precipitates can agglomerate and agglomerated particles display various sizes and clusters. HR-TEM image showed an evidence of crystalline HgS precipitates and also showed particle agglomeration (Fig. 3). The particle size distribution was totally Gaussian with a mean diameter of 425 nm consisting of 10–500 nm of HgS subunits that were structurally disordered due to agglomeration (Fig. 4).

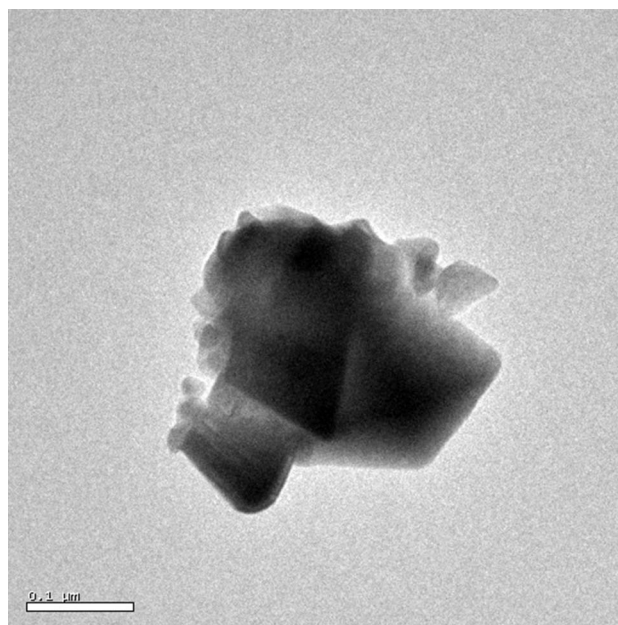
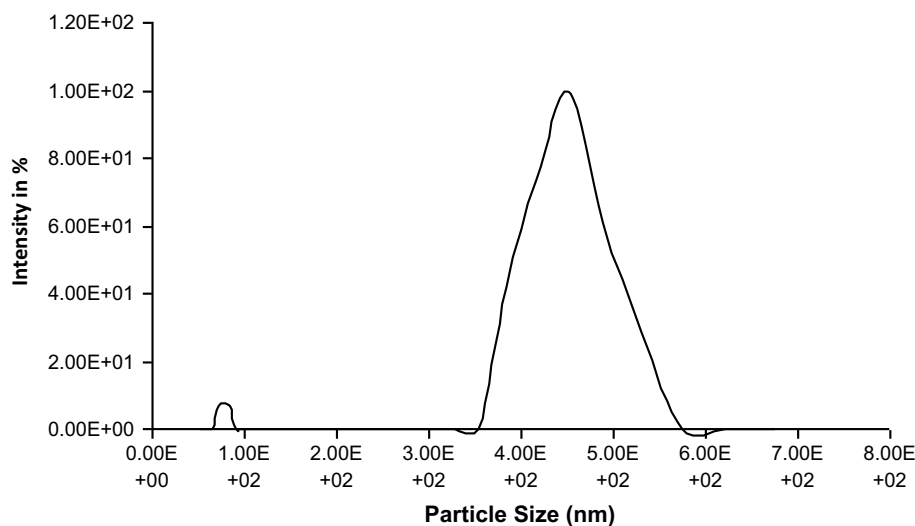
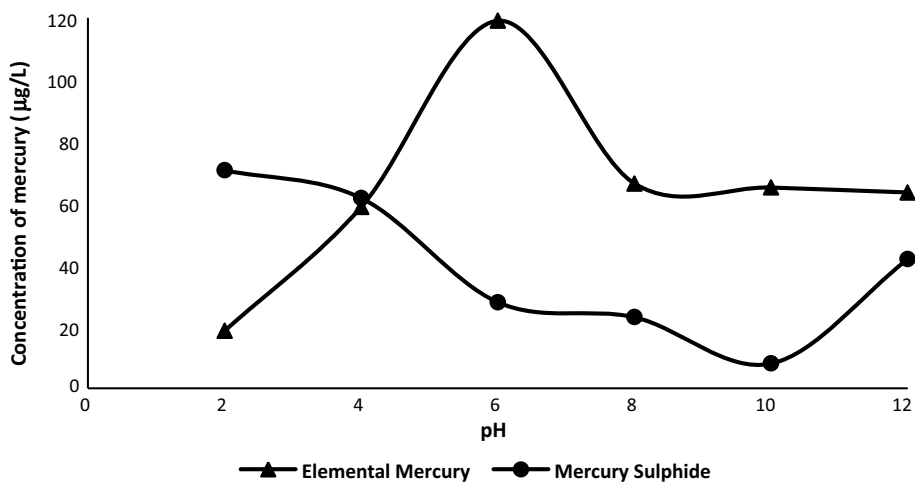


Fig. 3 TEM image of mercury sulphide particle

Following HgS formation, the effect of pH on dissolution of mercury from Hg^0 and HgS was investigated. Mercury dissolution is indicated as total mercury concentrations in the filtrate. Mercury was stable at neutral pH condition (27.85 $\mu\text{g/L}$ at pH = 6 and 23 $\mu\text{g/L}$ at pH = 8) and mild pH condition (8 $\mu\text{g/L}$ at pH = 10). Mercury dissolution was found to be 70.57 $\mu\text{g/L}$ at pH = 2 and 41.81 $\mu\text{g/L}$ at pH = 12, showing that mercury dissolution was high under highly acidic and highly basic conditions (Fig. 5). Hence, mild alkaline pH of 10 to 10.5 is the ideal condition for stable HgS. Samples containing unstable Hg^0 showed elevated mercury concentrations of 119 $\mu\text{g/L}$ at pH 6, 66.3 $\mu\text{g/L}$ at pH 8, 65 $\mu\text{g/L}$ at pH 10 and 63.4 $\mu\text{g/L}$



Fig. 4 Particle size analysis of mercury sulphide particle**Fig. 5** Mercury dissolution from elemental mercury and mercury sulphide versus pH

L at pH 12 when compared to HgS. Under acidic conditions, a potential risk of highly soluble and toxic hydrogen sulphide gas formation may exist. At the same time, higher pH can result in soluble mercury disulphide, mercurous sulphate (Hg_2SO_4) and mercuric sulphate (HgSO_4). Nonetheless, highly alkaline or highly acidic pH is not representative of natural environments.

Randall and Chattopadhyay (2004) investigated mercury leaching profiles of waste rocks over different pH and Eh ranges. It was found that both sulphide formation and sulphide stabilization required a mild alkaline pH (typically 10 to 10.5) and mild reducing conditions typically in the range between -100 and 100 mV. According to Thoming et al. (2000), at neutral pH, dissolution of inorganic mercury or Hg° occurs especially when the Eh is between 400 and 1000 mV. With Eh and pH less than 400 mV and 10 , respectively, mercurous chloride (Hg_2Cl_2) would be the dominant form of mercury. HgS has only a relatively narrow range of stability in the pH/redox potential field and is located at low

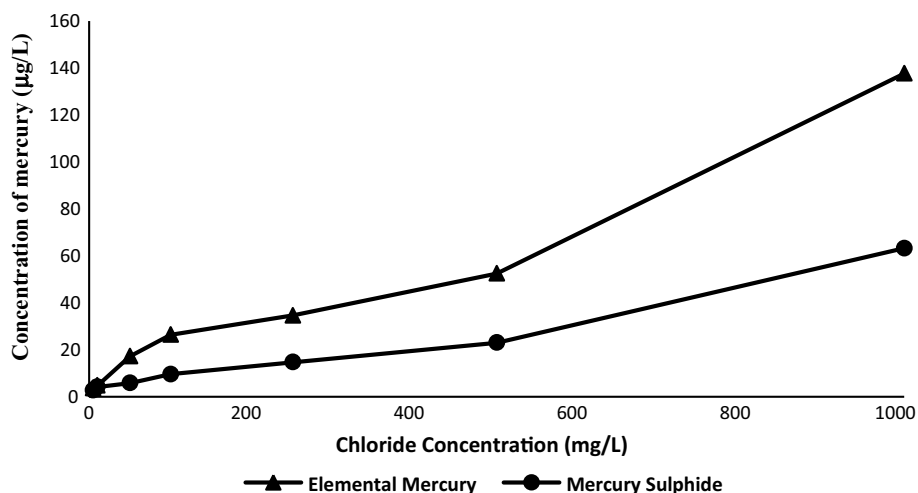
redox potentials. However, no effort was made to monitor or maintain the redox conditions in this study.

Figure 6 shows mercury dissolution of 4.83 $\mu\text{g/L}$ and 4.2 $\mu\text{g/L}$ from Hg° and HgS, respectively, at chloride concentration of 10 mg/L. With chloride concentration of 1000 mg/L (50 times that of naturally occurring chloride), mercury release was an order of magnitude higher showing 63.24 $\mu\text{g/L}$ of dissolved mercury from HgS and two orders of magnitude higher dissolving 137.6 $\mu\text{g/L}$ of mercury from Hg° . The results indicate that Cl^- ion when present at low concentration does not significantly cause mercury release both from Hg° and HgS. The formation of water-soluble HgCl_2 complex occurs only at a higher concentration of chloride ion. The presence of Cl^- at high concentrations would increase the mercury dissolution by forming more potentially leaching HgCl_2 complex and thus increase its migration in the groundwater.

Hg concentrations from HgS in the presence of HCO_3^- ions ranged from 1.2 to 15.4 $\mu\text{g/L}$ showing that



Fig. 6 Mercury dissolution from elemental mercury and mercury sulphide versus chloride concentration



the influence of HCO_3^- ions was insignificant with stable HgS. For the same HCO_3^- concentration, mercury dissolution varied between 24.2 and 75.4 $\mu\text{g/L}$ with Hg° showing that mercury(II)bicarbonate ($\text{Hg}_2(\text{HCO}_3)_2$) formation was higher in the presence of with Hg° (Fig. 7). Although the HCO_3^- ion is abundant in oxidic subsurface condition, the authors found the impact of HCO_3^- ions on mercury leaching from HgS to be relatively small. The influence of NO_3^- was evident both at lower and higher concentrations of the salt. With 10 mg/L of NO_3^- , dissolved mercury was 16.8 $\mu\text{g/L}$ and 23.8 $\mu\text{g/L}$ from HgS and Hg° , respectively. With concentration of 1000 mg/L, dissolved mercury raised to 58.7 $\mu\text{g/L}$ from HgS and 128.2 $\mu\text{g/L}$ from Hg° (Fig. 8) showing that the presence of NO_3^- ions could significantly increase mercury concentration in groundwater. Hg-nitrate precipitates forming at low concentrations can remobilize at higher nitrate concentrations and form water-soluble $\text{Hg}(\text{NO}_3)_2$ complexes.

Increased sulphate concentration liberates more mercury from HgS precipitate than from Hg° (Fig. 9). Sulphate reduction might increase the sulphide levels which in turn can weaken the mercury–sulphur bonds, oxidize HgS and release free Hg. The $\log K_{sp}$ value of HgSO_4 is 6.5×10^{-7} . Once an equivalent amount of sulphide is added, there would be no free Hg in the solution. Ferrous sulphate salt can also be added to consume the excess sulphide (Piao and Bishop 2006). The difference in the dissolved mercury concentration values can be related to the solubility values of HgS, HgCl_2 , $\text{Hg}_2(\text{HCO}_3)_2$, $\text{Hg}(\text{NO}_3)_2$ complexes formed. At low concentration of the anions, interfering effects were in the order of $\text{NO}_3^- > \text{Cl}^- > \text{SO}_4^{2-} > \text{HCO}_3^-$, whereas under high concentrations the order was $\text{Cl}^- > \text{NO}_3^- > \text{SO}_4^{2-} > \text{HCO}_3^-$. Unlike NO_3^- and Cl^- ions, SO_4^{2-} and HCO_3^- ions were reported to cause potential leaching only if they are present in significantly high concentrations.

Fig. 7 Mercury dissolution from elemental mercury and mercury sulphide versus bicarbonate concentration

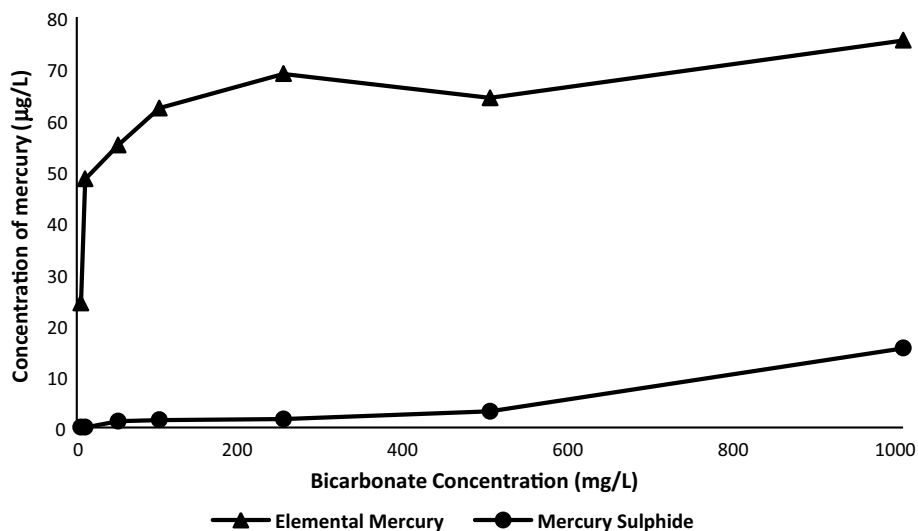


Fig. 8 Mercury dissolution from elemental mercury and mercury sulphide versus nitrate concentration

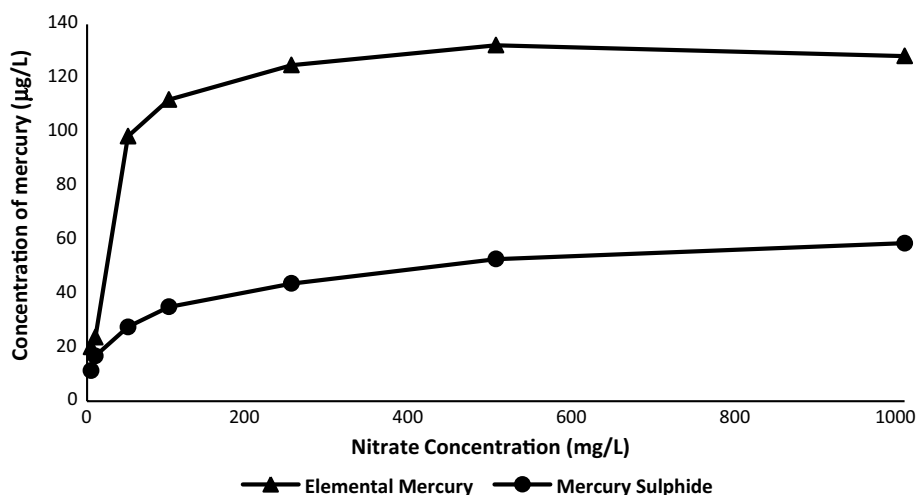
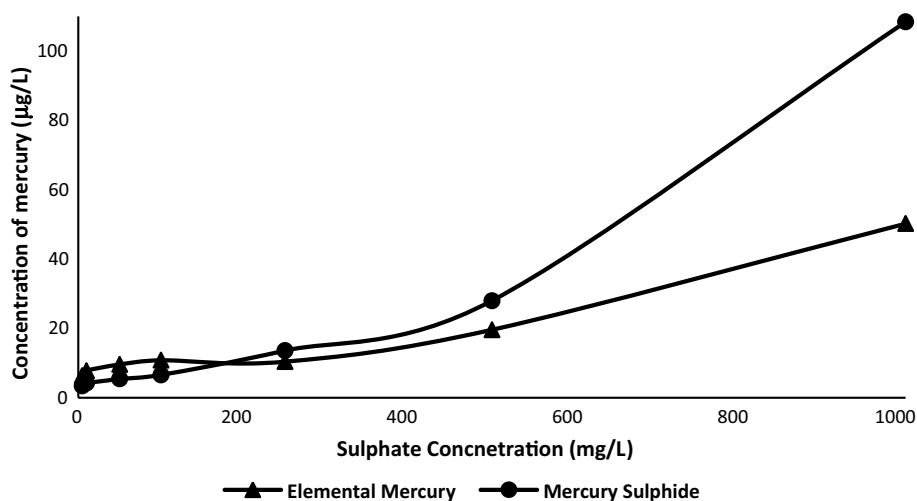


Fig. 9 Mercury dissolution from elemental mercury and mercury sulphide versus sulphate concentration



Conclusion

Elemental mercury is considered to be a serious environmental contaminant because of its toxic nature, transboundary movement and ability to bioaccumulate. It is often found in the vicinity of industrial facilities such as chlor-alkali plants, thermometer manufacturing units, copper and zinc smelting facilities and pharmaceutical factories. Generation of mercury wastes, though curtailed, is prevailing in developing economies as it is still employed in many manufacturing and mining industries. US EPA has set standards and proposed different technologies for low mercury subcategory with less than 260 mg/kg total mercury and high mercury inorganic subcategory with greater than 260 mg/kg total mercury. While stabilization has been suggested to achieve the standard of 0.2 ppm for low mercury wastes, recovery or retorting has been set for high mercury wastes where mercury is recovered in a

thermal processing unit that initially volatilizes and then condenses the mercury. It has also been substantiated by Chalkidis et al. (2020) that mercury-bearing wastes need to be stabilized and solidified before their final disposal or permanent storage either by thermal treatment or by chemical treatment depending upon the concentration of mercury-laden waste.

This paper investigated mercury stabilization and found sodium polysulphide stabilization to be the major removal route. The potential dissolution of mercury from stable HgS and untreated Hg^0 under different pH conditions and under different groundwater interfering salts were analysed. Sodium polysulphide was chosen as the best sulphur source for mercury stabilization when compared to elemental sulphur and sodium sulphide. Stabilization of Hg^0 was achieved through a series of batch experiments where Hg^0 was converted to HgS in a period of 96 h under optimum mild alkaline with Na_2S_5/Hg^0 molar ratio of 1. Maintaining a pH of 10–11 was found to be ideal for mercury stabilization and long-term storage of HgS .



Stabilized mercury should be subjected to standard leaching tests, and hence, the dissolution of mercury from stable HgS was investigated under varying concentrations of each of Cl^- , SO_4^{2-} , NO_3^- and HCO_3^- ions. The results show that Cl^- has a strong influence on mercury dissolution from HgS. At low concentration of the anions, the interfering effects were in the order of $\text{NO}_3^- > \text{Cl}^- > \text{SO}_4^{2-} > \text{HCO}_3^-$ whereas under high concentrations the order was $\text{Cl}^- > \text{NO}_3^- > \text{SO}_4^{2-} > \text{HCO}_3^-$.

The investigations should be extended in future in line with the Leaching Environmental Assessment Framework (LEAF) method proposed by US EPA (2019) where the mass transfer rates, pH and liquid-to-solids ratio are to be considered while investigating the stabilization of mercury. The four LEAF test methods 1313, 1314, 1315 and 1316 would highlight the effects of significant factors affecting the leaching behaviour of metals and other inorganic constituents present in wastes. Nevertheless, Hg^0 can be converted as HgS for in situ remediation of subsurface zones for permanent storage of mercury in underground salt mines or for permanent storage of mercury in landfills as emphasized in the 2011 Basel Convention.

Further studies should be carried out in (i) finding a green route for stabilization and further minimize the environmental impacts, (ii) performing additional investigations on effects of soil minerals and interferences on HgS stability over longer time scales, (iii) full-scale demonstrations and long-term monitoring of the stability of mercury-bearing wastes, (iv) exploring microbial pathway during stabilization, (v) developing cost-effectiveness and economics of the full-scale treatment technology taking into consideration the quantity and types of mercury-bearing wastes, (vi) developing geochemical modelling especially to find out the long-term behaviour of stabilized mercury and (vii) incorporating the US EPA's LEAF framework during stabilization of mercury-laden waste streams.

Acknowledgements The authors would like to thank the SAIF facility at Indian Institute of Technology Madras, India, for SEM and TEM analysis.

Funding The authors have no relevant financial or non-financial interests to disclose. The authors declare they have no financial interests. The authors have no financial or proprietary interests in any material discussed in this article.

Compliance with ethical standards

Conflict of interest The authors have no conflict of interest to declare that are relevant to the content of this article.

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