Article

Assessment of the Stabilization of Mercury Contaminated Soil Using Starfish

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Abstract: Approximately 50% of the 2600 decommissioned mines in South Korea are implicated in toxic metal/metalloid releases. One of the problems experienced in orchards situated near abandoned mines is the transport of heavy metals including mercury (Hg) into the plants. Due to high levels of Hg observed in orchard soils, heavy metal remediation is needed. The stabilization process is one of the widely used techniques to immobilize heavy metals in contaminated soil and waste. In this study, two types of starfish, Asterias amurensis (ASF) and Asterina pectinifera (PSF), were considered as stabilizing materials for remediating Hg-contaminated soil. In addition to natural starfish, the Hg immobilization effectiveness of calcined forms (CASF and CPSF) was also evaluated comparatively. The effect of particle size reduction on Hg immobilization was assessed for the ASF treatment. Total dosages of less than 10 wt% of ASF and PSF and less than 5 wt% of CASF and CPSF were applied to the Hg contaminated soil. Following treatment and curing for 28 days, the effectiveness of the stabilization process was evaluated using 1N HCl extraction tests. Overall, the stabilization results showed a decrease in Hg leachability with increasing dosages of ASF, PSF, CASF and CPSF. Generally, ASF outperformed the PSF treatments and calcined forms (CASF, CPSF) were more effective than natural forms (ASF, PSF). A reduction of approximately 79% was attained in Hg leachability for the 10 wt% ASF treatments. The -#20 mesh materials were more effective on Hg immobilization than the -#10 mesh materials. The Hg immobilization effectiveness exhibited the following increasing order: PSF (-#10 mesh) < ASF (-#10 mesh) < CPSF (-#10 mesh) < ASF (-#20 mesh) < CASF (-#10 mesh). It was found that effective Hg immobilization was most probably associated with the existing sulfur content in the starfish. The results of scanning electron microscopy-energy dispersive X-ray spectroscopy (SEM-EDX) indicated that a HgS compound and pozzolanic reaction products were responsible for effective Hg immobilization.

Keywords: mercury; stabilization; immobilization; starfish; Hg; soil

1. Introduction

Elemental mercury (Hg) contamination is a serious problem in orchards near abandoned mine sites in South Korea because the Hg can be transported into plants. Mercury has been used at mining sites to amalgamate with precious metals such as gold and silver. In addition, mercury is often used to extract precious metals from other heavy minerals. As a result, higher levels of Hg are often observed in the soils of orchards near mining sites. Mercury is known to cause adverse human health effects including muscle weakness, poor coordination, skin rashes, memory problems, eyesight/hearing loss, etc. Chronic human Hg exposure can lead to kidney disease. To prevent the entry of elemental mercury into the food chain remedial action is warranted for Hg contaminated soil.

There are various remediation technologies including soil washing, electrokinetics, phytoremediation, stabilization, etc. Among them, stabilization is a cost-effective process...
capable of high remedial effectiveness in the short term. The stabilization technique involves low-solubility chemical complexes that significantly reduce speciation and exposure to the toxic form [1–4]. Reportedly, solidification/stabilization technologies are applicable for Hg levels lower than 260 mg/kg, while thermal technologies are preferred when the Hg levels in the contaminated soil exceed 260 mg/kg [5]. Therefore, since the Hg concentration (23.1 mg/kg) was lower than the applicability level (260 mg/kg), the stabilization process was used in this study to remediate the Hg-contaminated soil. It has been reported that sulfur sources can contribute to the formation of virtually insoluble HgS in contaminated soil [6]. It has also been reported that HgS is stable and has a low solubility product of $2.0 \times 10^{-49}$ in water [7]. Sodium sulfide has been used to immobilize Hg contaminated surrogates (sand spiked with mercuric nitrate) by Piao and Bishop [3]. Accordingly, the optimal conditions for stabilization at a complexing efficiency of 99% were attained at a S/Hg molar ratio of 1 to 1 and a pH of 6. At higher pH values (>10), the Hg complexing efficiency stayed at high levels (~98%) [8] suggesting that sulfide stabilization is exceptionally effective. It also has been reported that the formation of Hg-CSH phase could be possible for effective Hg immobilization in an alkali-activated slag (AAS) matrix [9]. Consequently, starfish, known to contain S, were considered in this study as stabilizing materials. Moreover, in order to investigate pozzolanic reaction products such as calcium silicate hydrates (CSHs)/calcium aluminum hydrates (CAHs), which are responsible for Hg immobilization, calcined starfish was also utilized. Two types of starfish, namely, Asterias amurensis (ASF) and Asterina pectinifera (PSF), were used to evaluate their effectiveness on Hg immobilization. To the best of the authors’ knowledge, studies on the stabilization of Hg-contaminated soil using any type of starfish have not been reported in the literature to date. In addition, particle size and calcination effects on Hg immobilization have not been previously researched. Investigating the applicability of starfish for Hg immobilization may lead to the beneficial use of captured starfish since uncontrolled disposal is known to be an environmental nuisance for coastal areas around the globe. Starfish is known to be invasive species that can devastate natural marine ecosystems. The triton shellfish, a natural predator of starfish, exist in limited populations, which restricts their ability to control starfish proliferation. Therefore, large numbers of starfish are often captured by fishermen to control outbreaks of this invasive species. Thus, the beneficial reuse of captured starfish is worthy of exploration as a sustainable means of preventing environmental degradation of coastal areas.

The objective of this study was to assess the efficacy of starfish as stabilization materials for Hg contaminated soil. The effects of starfish type, particle size, and calcination on the effectiveness of Hg immobilization in contaminated soil were investigated.

2. Materials and Methods

2.1. Contaminated Soil Collection

Mercury (Hg) contaminated soil was obtained from orchards located at Cheonan-si, Chungcheongnam-do, South Korea. This area is close to the Ilbo mine, which was operated for the extraction of gold, silver, and copper and closed in 1982. The contaminated soil was sampled at a depth of 30 cm from the surface with a shovel. The collected soil samples were air-dried and passed using the No. 10 sieve to obtain a uniform particle size (2 mm materials). The total Hg concentration as measured by aqua regia (1 mL of HNO$_3$ and 3 mL of HCl) was approximately 23.1 mg/kg. Physico-chemical and mineralogical results for the Hg contaminated are presented in Table 1, while bulk chemistry results determined by X-ray fluorescence (XRF, ZSX100e, Rigaku, Japan) are shown in Table 2.
Table 1. Contaminated soil characterization results.

| Soil Properties | Hg Contaminated Soil | Regulatory Limit |
|-----------------|----------------------|------------------|
| pH(1:5)         | 8.23                 |                  |
| CEC(cmol/kg)    | 41.05                |                  |
| EC(dS/m)        | 2.14                 |                  |
| Composition (%)  |                      |                  |
| Sand            | 7.6                  |                  |
| Silt            | 66.7                 |                  |
| Clay            | 15.7                 |                  |
| Texture         | Silty loam           |                  |
| Hg(mg/kg)       | 23.1                 |                  |
| Major mineral compositions | | 4(Korean warning standard 1) |
|                 | Quartz, Muscovite    |                  |
|                 | Illite, Albite       |                  |
|                 | Clinochlore          |                  |

1 Korean warning standards for soils in residential area; 2 Soil classification determined by particle size analysis (PSA); Sand, 20–2000 µm; silt, 2–20 µm; clay, <2 µm; 3 Soil texture based on the United States Department of Agriculture (USDA) classification; 4 Mineral composition obtained by the Jade software [5].

Table 2. Major chemical composition of Hg contaminated soil, ASF, PSF, CASF and CPSF.

| Major Chemical Composition (%) | Hg Contaminated Soil | ASF | PSF | CASF | CPSF |
|-------------------------------|----------------------|-----|-----|------|------|
| SiO₂                          | 71.66                | 0.26| 0.31| 0.05 | 0.28 |
| Al₂O₃                         | 15.17                | 0.0963| 0.144| 0.02 | 0.06 |
| Na₂O                          | 1.14                 | 2.10 | 1.37 | 3.22 | 1.29 |
| MgO                           | 1.42                 | 7.04 | 7.24 | 14.85| 11.38|
| K₂O                           | 4.52                 | 0.356| 0.165| 0.36 | 0.038|
| CaO                           | 1.07                 | 81.34| 86.00| 76.13| 84.26|
| Fe₂O₃                         | 3.6                  | 0.128| 0.0593| 0.03 | 0.025|
| SO₃                           | 0.193                | 4.13 | 2.68 | 2.46 | 1.66 |
| MnO                           | 0.0784               | -    | 0.0242| 0.004| 0.012|
| pH(1:5)                       | 8.23                 | 7.35 | 7.22 | 12.5 | 12.49|

2.2. Stabilizing Agents

Two types of natural starfish, *Asterias amurensis* (ASF) and *Asterina pectinifera* (PSF), were used as stabilizing agents (Figure 1).

The ASF and PSF were collected from Jeju Island and Yeosu City, South Korea, respectively. The collected materials were washed in tap water for 24-h desalting and removal of and various impurities. Subsequently, the ASF and PSF were then rinsed several times with deionized (DI) water. After that, the cleaned ASF and PSF were air-dried in the sun for 7 days and then ground with a crushing blender to obtain a fine, homogenized powder clearing the US standard #10 sieve (2 mm). Moreover, the ASF was passed through the #20 sieve (0.85 mm) in order to evaluate the particle size effect on Hg immobilization. The calcined ASF (CASF) and PSF (CPSF) were prepared through the calcination process of natural ASF and PSF at 900 °C for 2 h in an electric furnace (J-FM3, JISICO, South Korea). Chemical characterization using XRF for the ASF, PSF, CASF, and CPSF is presented in Table 2.

2.3. Stabilizing Experiments

The mercury (Hg) contaminated soil was treated with ASF and PSF at 2 wt%, 4 wt%, 6 wt%, 8 wt%, and 10 wt% and CASF and CPSF at 1 wt%, 2 wt%, 3 wt%, 4 wt%, and 5 wt%. The control treatment with a 0 wt% mixing ratio was used for establishing benchmark levels. Water content of 20% was applied to provide the full sample hydration. Following stabilization treatment, a 4-week curing period was allowed in a sealable plastic container at ambient conditions (20 °C, 25% humidity). A severe leaching test involving 1N HCl
extraction was used to assess the effectiveness of Hg stabilization in contaminated soil. The treatability matrix is presented in Table 3.

**Figure 1.** Pictures of ASF (a) and PSF (b).

**Table 3.** Treatability matrix for Hg contaminated soil.

| Sample ID   | Contaminated Soil (wt%) | ASF/PSF (wt%) | CASF/CPSF (wt%) | Liquid to Solid (L:S) Mass Ratio |
|-------------|--------------------------|---------------|-----------------|---------------------------------|
| Control     | 100                      | 0             | 0               | 20:1                            |
| 2 wt% ASF/PSF | 100                      | 2             | 0               | 20:1                            |
| 4 wt% ASF/PSF | 100                      | 4             | 0               | 20:1                            |
| 6 wt% ASF/PSF | 100                      | 6             | 0               | 20:1                            |
| 8 wt% ASF/PSF | 100                      | 8             | 0               | 20:1                            |
| 10 wt% ASF/PSF | 100                      | 10            | 0               | 20:1                            |
| 1 wt% CASF/CPSF | 100                      | 0             | 1               | 20:1                            |
| 2 wt% CASF/CPSF | 100                      | 0             | 2               | 20:1                            |
| 3 wt% CASF/CPSF | 100                      | 0             | 3               | 20:1                            |
| 4 wt% CASF/CPSF | 100                      | 0             | 4               | 20:1                            |
| 5 wt% CASF/CPSF | 100                      | 0             | 5               | 20:1                            |
2.4. X-ray Powder Diffraction (XRPD) Analyses

The X-ray powder diffraction (XRPD) analyses were conducted to investigate the mineralogical content in the Hg contaminated soil and stabilizing agents (ASF, PSF, CASF, and CPSF). The XRPD samples were prepared by pulverizing the sample into a particulate residue clearing the US-standard #200 sieve (0.075 mm). The collected step-scanned X-ray diffraction patterns were analyzed by an X-ray diffractometer (XRD) (X’Pert PRO MPD, PANalytical, Almelo, The Netherlands) equipped with a diffracted beam graphite-monochromator with Cu radiation at 40 kV and 40 mA. The XRPD patterns were collected in the 2θ range of 5–65°, step size of 0.02°, and count time of 3 s/step. Mineral phases were characterized using the Jade software v. 7.1 [10] and the PDF-2 reference database [11].

2.5. Scanning Electron Microscopy-Energy Dispersive X-ray Spectroscopy (SEM-EDX) Analyses

Scanning electron microscopy-energy dispersive X-ray spectroscopy (SEM-EDX) was used for getting insight into the mechanism of Hg stabilization. SEM-EDX tests involved placing sample aliquots on a double-sided Pt-coated carbon tape, using a Hitachi S-4800 SEM instrument (Tokyo, Japan) equipped with the Horiba EMAX EDX system (Tokyo, Japan). In order to evaluate the Hg immobilization mechanism, the treated samples exhibiting the greatest reduction in Hg leachability (10 wt% ASF, 10 wt% PSF, 5 wt% CASF, and 5 wt% CPSF) were subjected to SEM-EDX analysis.

2.6. Physicochemical Analyses

The soil pH values were measured in accordance with the Korean Standard Test (KST) method [12] at a liquid-to-solid (L:S) ratio of 5:1 (L/kg). The CEC and EC values were analyzed based on the National Academy of Agricultural Science (NAAS) test method [13]. The total Hg levels were determined using an aqua regia extraction agent. Soluble Hg concentrations were analyzed using an atomic absorption spectrometer hybrid vapor generator (AA-700 series HVG-1) (Shimadzu, Tokyo, Japan). All the results of AA analysis were reported as the mean (within a 10% error) of triplicates. For the sake of QA/QC, three distinct quality control standards were employed for every 10 samples analyzed.

3. Results and Discussion

3.1. XRPD Analyses

The XRPD patterns of the Hg contaminated soil and stabilizing agents such as ASF, PSF, CASF, and CPSF are presented in Figures 2 and 3, respectively. The major phases identified in the Hg contaminated soil were quartz (PDF# 46-1045), muscovite (PDF# 07-0025), albite (PDF# 41-1480), illite (PDF# 26-0911), and clinochlore (PDF# 29-0701).
Calcite ((Ca, Mg)CO$_3$, PDF# 43-0697) was observed to be the main phase in the natural stabilization materials (ASF and PSF). The detected main phases for the CASF were quicklime (CaO, PDF# 48-1467), periclase (MgO, PDF# 45-0946), and oldhamite (CaS, PDF# 08-0464). This indicated that the transformation of calcite into quicklime and periclase was facilitated by the calcination process at a high temperature (900 °C, 2 h). Moreover, a CaS phase was also clearly formed as a result of the calcination process. For the CPSF, similar to the CASF, quicklime and periclase were identified as major phases. This is indicative of the complete decomposition of calcite to quicklime and periclase. No CaS was observed in the CPSF. This indicated that high content of S in the ASF may contribute to the formation of CaS in CASF but not in CPSF.

Figure 2. XRPD pattern of Hg contaminated soil.

Figure 3. XRPD patterns of ASF, PSF, CASF and CPSF.
3.2. Effectiveness of the Stabilization Treatment

Results of the extracted Hg levels using the 1N HCl extraction solution for the ASF(-#10 mesh), ASF(-#20 mesh), PSF(-#10 mesh), CASF(-#10 mesh), and CPSF(-#10 mesh) treatments are presented in Figures 4–8. In general, the Hg leachability decreased with increasing stabilizing agent dosage.

![Figure 4](image-url)

**Figure 4.** Hg leachability of the control and ASF (-#10 mesh) treated samples after 28d of curing, upon 1N HCl extraction.

![Figure 5](image-url)

**Figure 5.** Hg leachability of the control and ASF (-#20 mesh) treated samples after 28d of curing, upon 1N HCl extraction.
**Figure 6.** Hg leachability of the control and PSF (-#10 mesh) treated samples after 28d of curing, upon 1N HCl extraction.

**Figure 7.** Hg leachability of the control and CASF (-#10 mesh) treated samples after 28d of curing, upon 1N HCl extraction.

**Figure 8.** Hg leachability of the control and CPSF (-#10 mesh) treated samples after 28 d of curing, upon 1N HCl extraction.
For the coarse ASF treatment (-#10 mesh materials), the lowest Hg leachability of 0.29 mg/kg was attained with the 10 wt% ASF treatment, resulting in a higher than 79% decrease in the Hg levels for the treated sample compared to the control sample. In the ASF treatment with -#20 mesh materials, the Hg leachability was further reduced as compared to the -#10 mesh materials (Figure 5). The lowest Hg concentration of 0.1 mg/kg (approximately 94% reduction) was obtained with the 10 wt% ASF treatment (-#20 mesh material). This indicates that stabilizing agents of high surface area (fine particle size distribution) can significantly decrease Hg leachability.

Similarly, the PSF treatment (-#10 materials) showed a decrease in Hg leachability with increasing PSF dosage. Approximately, 1 mg/kg of Hg leached (36% reduction in Hg leachability) was attained with the 10 wt% PSF treatment. However, this reduction in Hg leachability was less than the ASF treatment. This indicates that the type of starfish used influences the degree of Hg immobilization. The better Hg immobilization performance of the ASF over the PSF treatment is most probably explained by the higher SO$_3$ content of the ASF (4.13%) compared to the PSF (2.68%) (Table 2). The SO$_3$ content could contribute to the formation of HgS which is mainly responsible for effective Hg immobilization. It has been reported that Hg$^0$ and Hg$^{2+}$ prefer to complex with sulfur and sulfides to form insoluble HgS based on the following equations [14,15]:

$$\text{Hg}^0 + S \rightarrow \text{HgS} \quad (1)$$

$$\text{Hg}^{2+} + \text{H}_2\text{S} \rightarrow \text{HgS} + 2\text{H}^+ \quad (2)$$

$$\text{Hg}^0 + \text{Na}_2\text{S}_5 \rightarrow \text{HgS} + \text{Na}_2\text{S}_4 \quad (3)$$

Moreover, Piao and Bishop [8] reported that sulfide-induced stabilization was very effective in stabilizing mercury-containing wastes. In their study, the lowest TCLP Hg concentration of 4 ug/L was attained at a pH of 6 and a [S]/[Hg] molar ratio of 1.

The CASF treatments outperformed the ASF treatments (both -#10 and -#20 materials). Moreover, decreases in Hg leachability were observed with increasing CASF dosage. The lowest Hg leachability of 0.16 mg/kg was attained with the 5 wt% CASF treatment. Approximately 91% reduction in Hg leachability was realized with the 5 wt% CASF treatment. This indicates that treatment with calcified ASF can be more effective than the natural ASF treatment in immobilizing Hg in contaminated soil. These results also indicate that the added cost associated with CASF may be balanced by its reduced dosage requirements of this stabilization agent compared to the lower cost but higher dosage requirements needed for natural ASF to obtain comparable Hg immobilization in contaminated soil. Thus, a technoeconomic analysis may be required to resolve this issue.

Similar to the CASF treatment, the CPSF treatment was more effective for Hg immobilization than the PSF treatment. The lowest Hg leachability of 0.62 mg/kg was obtained with the 5 wt% CPSF treatment resulting in an approximate 64% Hg leachability reduction. The CPSF treatment was not as effective as the CASF treatment. This may be due to the lower S content of the PSF compared to the ASF treatment.

In terms of the mechanism responsible for Hg immobilization, the effective Hg immobilization obtained upon ASF, PSF, CASF, and CPSF treatments is most probably linked to the mineral phase of HgS, which is a sparingly soluble phase. It has been reported that sulfide can contribute to the formation of HgS in contaminated soil [16]. Moreover, the highest Hg immobilization performance of the calcined (CASF, CPSF) over the natural (ASF, PSF) treatments may be explained by the formation of pozzolanic reaction products such as CSHs and CAHs, which are known to play an important role in immobilizing Hg in the contaminated soil. Reportedly, effective immobilization of Hg can be achieved by the formation of insoluble mercury silica gel in the alkali-activated slag matrix [9]. The immobilization of Hg can also be attained by the incorporation of the Hg ion in the lattice structure of C-S-H compounds [4]. Application of CASF and CPSF treatments
instigates soil pH increases that cause the clay structure to break down releasing Al and Si that become available to form CSHs and CAHs. Therefore, Hg immobilization upon CASF and CPSF treatments is likely achieved by both pozzolanic reaction products and HgS compound formation. This can provide a synergistic effect for effective Hg immobilization as compared to the ASF and PSF treatments.

Overall, Hg immobilization was effective with the ASF, PSF, CASF, and CPSF treatments. The effectiveness of Hg immobilization was found to have increased in the following order:

PSF (-#10 mesh) < ASF (-#10 mesh) < CPSF (-#10 mesh) < ASF (-#20 mesh) < CASF (-#10 mesh)

This indicates that particle size reduction can lead to effective Hg immobilization without the calcination process. This indicates that when S content is present at high enough levels within the natural starfish, then effective Hg immobilization can be achieved by reducing particle size without the calcination process if soil pH is a concern for plant growth.

3.3. SEM-EDX Analyses

The SEM-EDX results for the lowest Hg leachability samples, namely the 10 wt% ASF, 10 wt% PSF, 5 wt% CASF, and 5 wt% CPSF are presented in Figure 9. In all treated samples, Hg was identified by SEM-EDX analysis along with S, indicating that HgS formation is responsible for effective Hg immobilization. It has been reported in previous research that HgS formation can be effective for Hg immobilization [16]. It has been reported that successful stabilization/solidification of elemental mercury (33.1 wt%) in waste was achieved using only sulfur (two times higher than mercury) with paraffin. In sulfur-treated waste, both HgS black (cubic phase) and HgS red (orthorhombic phase) were observed by XRPD analysis [17].

![Figure 9. Cont.](image-url)
4. Conclusions

Two different types of starfish *Asterias amurensis* (ASF) and *Asterina pectinifera* (PSF) were used effectively to immobilize Hg in contaminated soil. Calcination (CASF and CPSF) and particle size reduction effects on the stabilization process were also investigated. The applied dosage of amendments to the Hg contaminated soil ranged from 2 wt% to 10 wt% for natural (ASF, PSF) and 1 wt% to 5 wt% for the calcined forms (CASF, CPSF). All the

In the CASF and CPSF treated samples, Hg was identified by SEM-EDX analysis along with Ca, Al, Si, and O. This indicates that effective Hg immobilization is likely related to the generation of pozzolanic products such as CSHs and CAHs. This mechanism controlling Hg immobilization has also been reported in the alkali-activated slag matrix [9].

Figure 9. SEM-EDX analyses for the Hg in contaminated soil treated with 10 wt% ASF (-#10 mesh) (a), 10 wt% PSF (-#10 mesh) (b), 5 wt% CASF (-#10 mesh) (c), and 5 wt% CPSF (-#10 mesh) (d) treated samples.
treated samples were cured for 4 weeks following the treatment. After the curing period, Hg immobilization in the treated samples was assessed using the 1N HCl extraction test. The stabilization results showed that Hg leachability was reduced with increasing dosage for all stabilization agents. Higher than 90% Hg leachability reduction was attained with the 10 wt% ASF (-#20 mesh materials) and the 5 wt% CASF treatments. The most effective stabilizing agent was found to be CASF. Moreover, fine (-#20 mesh) were more effective than coarse (-#10 mesh) stabilizing materials, proving that particle size reduction of the stabilization agent can be an effective pre-treatment option for improved Hg immobilization in contaminated soils. The Hg immobilization effectiveness increased in the following order: PSF (-#10 mesh) < ASF (-#10 mesh) < CPSF (-#10 mesh) < ASF (-#20 mesh) < CASF (-#10 mesh). The SEM-EDX analyses indicated that the S inherently present in the starfish contributed to Hg immobilization. The formation of HgS was identified as the compound responsible for Hg immobilization. Moreover, pozzolanic reaction products such as CSHs/CAHs, identified in the CASF and CPSF samples, mainly contributed to the immobilization of Hg in the contaminated soil. Overall, the starfish could be used to effectively immobilize Hg in contaminated soil. Particle size reduction and calcination of the stabilization materials proved to be viable options for improving the effectiveness of Hg immobilization in contaminated soils. These options along with the type of starfish waste materials and associated S content should be carefully considered during the design stage of effective stabilization schemes for remediation of elemental Hg contaminated soil.

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