

























demonstrated to increase Hg methylation by inducing microbial activity [72]. Organic Hg and total organic carbon, as well as organic Hg and elemental Hg content, were found to have positive relations. These findings indicate that elemental Hg in the soil can be converted to reactive  $\text{Hg}^{\text{II}}$ , primarily by oxidation, and then methylated by microbial activity [73]. However, the formation of MeHg in the ecosystem will be governed by a variety of factors regulating microbial activity and/or the geochemical speciation of inorganic  $\text{Hg}^{\text{II}}$  [87]. According to Dang et al. [93], Selenium (Se) has also an impact on MeHg production via HgSe formation in soil and sediments.

Demethylation of MeHg is a reverse method of methylation of Hg. It may also proceed by biotic pathways or abiotic pathways, like methylation of Hg [73,90]. The major pathway of Hg demethylation in sediments and periphyton has been proposed to be the biotic process [94]. In summer, MeHg demethylation in sediments typically increases. Significant parameters that also control the processes of Hg demethylation are sediment's redox potential and often its association with Hg concentration [72]. Additionally, a laboratory incubation analysis of surface lake sediments in China found faster demethylation and slower methylation of Hg at higher concentrations of solid-phase Se and that pH effect was variable due to the availability of Hg and microbial activity [87].

Degradation of MeHg by bacteria primarily requires reduction of  $\text{Hg}^{2+}$  to HgO and under aerobic conditions seems to be preferred. Natural demethylation is normally activated by either microbial activity or photoreduction of light. Two forms of microbial demethylation reactions typically exist: reductive and oxidative. Reductive demethylation degrades MeHg into HgO and methane ( $\text{CH}_4$ ), and HgO and carbon dioxide ( $\text{CO}_2$ ) are formed by oxidative demethylation [29].

The deposition of Hg in surface soil under anaerobic and low pH conditions can be affected the formation of net MeHg and Hg methylation rate in soil may therefore also be a result of the concentration and deposition processes of atmospheric Hg [31]. Boszke et al. [34] indicate that low pH promotes the release of mercury from bottom sediments, whereas others argue that low pH enhances mercury sorption on the sediments.

## 5. Analysis of THg and MeHg in Soil and Sediments

The analysis is a sequential process involving extraction/pre-concentration, separation, and detection. Extraction/pre-concentration methods are liquid phase microextraction, purge and trap, solid-phase extraction, and solid-phase microextraction. Separation techniques are Chromatographic techniques and non-chromatographic techniques [54].

Most of the methods for the measurement of THg in solid samples require preliminary digestion. In an acidic medium to release Hg from the sample, nitric acid ( $\text{HNO}_3$ ), hydrochloric acid (HCl), bromine monochloride (BrCl), sulfuric acid ( $\text{H}_2\text{SO}_4$ ), perchloric acid ( $\text{HClO}_4$ ), hydrogen peroxide ( $\text{H}_2\text{O}_2$ ), vanadium pentoxide ( $\text{V}_2\text{O}_5$ ), potassium permanganate ( $\text{KMnO}_4$ ), potassium dichromate ( $\text{K}_2\text{Cr}_2\text{O}_7$ ) are the most common reagents which have been used. Samples are normally digested at high temperatures, such as max. 90–100°C, in closed, semi-closed, or sealed containers. At high digestion temperatures, careful attention should be paid to preventing Hg loss. Thus, closed, or sealed digestion containers should be applied [29]. Soil samples are digested with aqua regia (HCl/ $\text{HNO}_3$ ) using microwave-assisted digestion for the extraction of pseudo-THg, due to its capacity to dissolve HgS. Soil samples can be digested in  $\text{HNO}_3/\text{H}_2\text{SO}_4$  containing large quantities of organic material and then diluted with BrCl solution to eliminate any remaining organic material [31].

For solid samples such as soil and sediments, lower and inconsistent results are provided by acid digestion using aqua regia. The use of acid digestion, including hydrofluoric acid (HF), is highly recommended to fully extract Hg from the inorganic matrix [29].

It is important to isolate MeHg from the sample before analysis to prevent matrix interference during sample processing [95]. Acid extraction (mostly combined with solvent extraction), distillation, and alkaline extraction are the most widely used methods for removing organomercury species from environmental samples [59]. The derivatization (chemical changes) steps needed for gas chromatography (GC) analysis are especially sensitive to matrix suppression. To remove MeHg from complex matrices, distillation methods are commonly used. This technique is based on a simple distillation of atmospheric pressure and does not require complex reagents for accurate results to be obtained. However, the use of distillation may lead to the formation of an artifact of MeHg [95].

Extraction of soil samples for MeHg analysis is more difficult. Extraction can include digestion to extract organic Hg from inorganic complexes with acidic potassium bromide (KBr) and copper sulfate ( $\text{CuSO}_4$ ) solution, followed by dichloromethane (DCM) to extract MeHg, and then back-extraction by argon (Ar) purging into an aqueous solution. In a purge vessel using sodium tetraethyl borate, aqueous solutions are ethylated to convert MeHg to volatile methylethyl-Hg species [31].

Usually, extremely sensitive atomic absorption and atomic emission techniques assess only the total quantity of metal in the sample and must therefore be combined with a chromatographic or other separation technique until it is possible to classify individual species [96]. By using different methods, the organo-Hg material can then be detected [31]. High-performance liquid chromatography-chemiluminescence (HPLC-CL), electron capture gas chromatography (GC-ECD), and gas chromatography-atomic fluorescence spectrometry (GC-AFS), for these methods, MeHg results for all soil and sediments samples returned comparable. Although, it was reported that the HPLC-CL method performed poorly in analyzing high sulfur content samples [65]. Also, Due to its speed, simplicity, relative freedom from interference, low operating costs, and high sensitivity, especially when Hg vapor is pre-concentrated on gold by amalgamation, cold vapor atomic absorption spectrometry (CV-AAS) has been used. Many published methods have relied on plasma mass spectrometry that is inductively coupled (ICP-MS) [97].

Denmark et al. [98] developed a new method for extracting MeHg selectively from severely contaminated soil and sediment samples contaminated with Hg for analysis using chemical vapor generation inductively coupled plasma mass spectrometry (CVG-ICP-MS).  $\text{HNO}_3$  was shown to be the most effective for selective extraction of MeHg from soils when compared to HCl. Fast extraction of MeHg was achieved using ultrasonic agitation in  $\text{HNO}_3$  at room temperature. Using a dilute ammonium sulfide solution, soil extracts in  $\text{HNO}_3$  were used to precipitate  $\text{HgS}$ , which resulted in the elimination of all residual  $\text{Hg}^{2+}$  without affecting MeHg levels. Compared to chromatographic separation and speciation methods, the procedure is simple and quick, and it also has distinct advantages for determining trace levels of very dangerous MeHg from severely Hg-contaminated sediments and soil. In another recent study, Saniewska and Beldowska [99], tried to develop a simple thermo-desorption method for mercury fractionation in soil and sediment samples using a direct mercury analyzer and they used soil, beach sand and marine sediment in this study. Then, the temperature range in which mercury species were released was used to identify them. Despite some limitations, the results suggest that temperature fractionation can be used as a screening approach for determining the percentage contribution groups of Hg compounds with

similar properties in solid materials. This approach could be used on solid samples with low levels of Hg in the environment. Lastly, in 2013, Kodamatani and Tomiyasu [89], developed simultaneous determination of MeHg and ethylmercury (EtHg). The approach involves extracting MeHg and EtHg into toluene as chlorides after eluting mercury species from soil/sediment samples with HCl containing  $\text{Cu}^{2+}$  and  $\text{Pd}^{2+}$ . These alkylmercury chlorides were then back-extracted into an aqueous EDTA solution, forming EDTA complexes, which were subsequently separated using reverse-phase HPLC and identified using a tris ruthenium chemiluminescence process.

## 6. Conclusion

Anthropogenic emissions of Hg have a vast effect on the environment and therefore, pose an elevated risk to human beings. This overview aims to disclose sampling methods, use and storage; it also focuses on Hg forms, mobilization and analysis in soil and sediment.

The findings of this study are listed below:

- In the sampling method, different approaches depend on the chosen area, such as two-stage, cluster, judgmental, random, stratified random, systematic grid, search and transect. Tools made of polymer, glass, stainless steel, or aluminum are required during the collecting, pre-treatment, and storage phases of environmental samples. The best technique for preserving MeHg is to freeze soil and sediment samples immediately after collection, followed by freeze-drying, grinding, homogenization, and storing the dry material in cold, dark conditions until analysis. In most of the papers, it is not specified what type of sampling or drying methods were applied.
- Clay soils can absorb Hg and lead the creation of HgS.
- Organically rich soils, such as forest soils, peaty soils, or rice paddy fields are typically connected to higher THg concentrations.
- Hg binding to humic substrates is the major process in Hg sorption.
- Plants can play an important role in Hg transport and accumulation. MeHg concentrations in biomass were found to be higher in *Solanum nigrum* (BR3) and *Cynodon dactylon* (BR2).
- It appeared that the adsorptive capacity of Hg is higher than Cr, Cd, Cu, Zn and As but lower than Pb.
- Hg can be affected by most of the conditions in the environment, which can determine the final form in soil or sediment, such as pH, redox potential, and light.
- The soil profile, production of volatile Hg species, physical movement of Hg species, and physical and chemical sorption of Hg vapor all influence the depth of the soil layers that contribute to evaporation. When compared to unplanted regions, applying sulfur to the soil and growing a plant cover reduces Hg flow by around 70% to 80%.
- *Shewanella oneidensis* MR-1 and *Geobacter spp* are two bacteria that can mediate the biotic reduction of  $\text{Hg}^{2+}$  to HgO.
- The most common organic and toxic form of Hg in the environment is accepted as MeHg. Soil moisture highly impacts MeHg, through sulfate and iron-reducing bacteria.
- A laboratory incubation analysis of surface lake sediments revealed that higher levels of solid-phase Se resulted in rapid demethylation and slower methylation of Hg and that the pH impact was varied owing to Hg availability and microbial activity.
- Climate change and permafrost thawing have the potential to increase MeHg production. Arctic

permafrost represents an important source of Hg in case warming will not decrease in the future.

- In 2018, a simple and quick approach for analyzing MeHg utilizing chemical vapor generation inductively coupled plasma mass spectrometry was introduced. When compared to HCl, HNO<sub>3</sub> has proven to be the most effective for selective extraction of MeHg from soils. Ultrasonic agitation helped to produce rapid MeHg extraction.

### Conflict of Interest

All authors declare no conflicts of interest in this paper.

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