COMPARISON OF ARSENIC SPECIATION TECHNIQUES: FOCUS ON CHEMICAL AND ELECTROCHEMICAL METHODS

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ABSTRACT

Traditional chemical methods, such as HPLC and AAS, are sensitive and selective for arsenic speciation but require complex sample preparation and are costly. In contrast, electrochemical methods, like voltammetry, offer a faster, more sensitive, and cost-effective analysis with a simpler sample preparation, highlighting their sample size and portability. The choice between both approaches depends on the specific needs of the analysis and the available resources.

Keywords: Electrochemistry, Sensors, Arsenic, Speciation, Environment.

INTRODUCTION

Arsenic, a ubiquitous element in nature, has attracted attention and concern due to its potential toxicity to humans and the environment. Its presence in water, soil, and food, along with its ability to accumulate in biological tissues, makes it an environmental contaminant and a public health risk [1-5].

Arsenic exists in various chemical forms known as species or oxidation states, among which inorganic arsenic and organic arsenic stand out. These chemical forms vary in their toxicity and mobility in the environment. For example, inorganic arsenic, especially in its trivalent form (As(III)), is considered more toxic and water-soluble than arsenic pentavalent (As(V)) [6-12].

The toxicity of arsenic is closely linked to its oxidation state. Inorganic arsenic, especially As(III), can interfere with crucial metabolic processes in organisms, such as enzyme inhibition and the generation of reactive oxygen species, which can lead to a wide range of adverse health effects, from acute gastrointestinal disorders to chronic diseases like cancer and cardiovascular disorders [13-15].

It is crucial to understand the distribution and specific toxicity of the various forms of arsenic due to its omnipresence in the environment and its impact on human health and the ecosystem in general. Exposure to arsenic, especially in its more toxic forms, poses a serious health risk, including neurological problems, skin cancer, cardiovascular disorders, and damage to the immune system.

The concept of arsenic speciation refers to the identification and quantification of the different chemical forms in which this element can be found in environmental and biological samples. This distinction is essential since the different forms of arsenic have different physical and chemical properties as well as varying levels of toxicity and mobility in the environment. For example, while inorganic trivalent arsenic (As(III)) is generally more soluble and toxic than pentavalent arsenic (As(V)), organic arsenic tends to be less toxic and more bioavailable in certain biological contexts.

In terms of regulation, the World Health Organization (WHO) sets maximum permissible limits for the concentration of arsenic in various environmental matrices. These limits are established to protect human health and reduce the risks associated with arsenic exposure. For example, in the case of drinking water, WHO has set a maximum permissible limit of 10 micrograms per liter (μ g/L) of total inorganic arsenic. Regarding soil, WHO has not established a specific limit, but it recommends evaluating concentrations based on risks to human health and the environment. For air, WHO has not set specific limits, but it monitors air quality and issues guidelines and recommendations to limit exposure to various atmospheric pollutants, including arsenic [2].

In summary, arsenic speciation and the establishment of maximum permissible limits by regulatory agencies, such as WHO, are fundamental aspects to understand, prevent, and mitigate the risks associated with arsenic exposure in the environment, protecting human health and the ecosystem in general. The effective implementation of precise analytical techniques and compliance with regulatory standards are critical steps towards sustainable management and the reduction of arsenic contamination globally. Arsenic speciation refers to the separation and quantification of its different chemical forms in a sample. This analytical approach is crucial for understanding the mobility, bioaccumulation, and specific toxicological impact of each form of arsenic in the environment and in living organisms. Additionally, arsenic speciation is essential for evaluating the effectiveness of environmental remediation strategies and establishing regulatory limits for water and food quality [16-20].

The present study aims to conduct a comprehensive comparison of arsenic speciation techniques, with a particular focus on chemical and electrochemical methods. These techniques have proven to be fundamental in the precise identification and quantification of different forms of arsenic, providing valuable information for environmental risk assessment and public health protection.

The principles, advantages, and limitations of chemical and electrochemical methods in arsenic speciation will be explored throughout this study, with the goal of providing a comprehensive overview of the available analytical tools and their application in research and environmental management.

Challenges of High Arsenic Concentrations in the Environment

The challenge of high arsenic concentrations in the environment is a topic of great relevance nowadays due to its multiple implications for human health and the integrity of aquatic ecosystems. Arsenic, a chemical element naturally found in the Earth's crust, can be released into the environment through geological processes, as well as anthropogenic activities, such as mining, industry, and agriculture [21-27].

In bodies of water, arsenic can reach dangerous levels due to various sources and processes. One of the main natural sources is the release of arsenic from minerals present in soils and rocks, which can be transported to water bodies through soil erosion and surface runoff. This natural erosion can be exacerbated by human activities like deforestation and urbanization, increasing the risk of arsenic contamination in water bodies [28-30].

In addition to natural sources, human activities represent a significant source of arsenic contamination in water bodies. Mining is one of the main activities that contribute to the release of arsenic into the environment. Processes involving the release of arsenic contained in minerals are used in many mining operations, especially in the extraction of gold, copper, and other metals. The resulting waste from these operations, such as mine tailings, can contaminate nearby water bodies and spread arsenic along the hydrological network.

Other industrial activities, such as chemical production, wood treatment, and the manufacture of electronic products, can also contribute to the release of arsenic into the environment through atmospheric emissions and industrial effluents. Furthermore, intensive agriculture, especially the use of pesticides and herbicides containing arsenic, can result in soil and groundwater contamination, eventually affecting water quality in surface bodies.

The impact of arsenic on the environment is significant and multifaceted. High arsenic concentrations can have adverse effects on aquatic ecosystems, including In addition to ecological impacts, the presence of high arsenic concentrations in water poses a serious threat to human health. Chronic exposure to arsenic through the consumption of contaminated water or foods grown in contaminated soils can have serious consequences for health, including an increased risk of developing skin, lung, bladder, and other organ cancers. Also, arsenic exposure has been associated with a range of long-term health problems such as cardiovascular diseases, diabetes, nervous system disorders, and adverse effects on child development.

It requires a comprehensive approach to address the arsenic challenge in the environment that involves regulating and controlling industrial and mining activities, implementing effective water treatment technologies, as well as promoting sustainable agricultural practices to reduce soil and groundwater contamination with arsenic. Furthermore, it is essential to raise public awareness about the risks associated with arsenic exposure and promote community engagement in the protection and conservation of water resources.

Arsenic speciation is an important aspect to consider when addressing arsenic contamination in the environment and particularly in water bodies. Arsenic can be found in different chemical forms, both organic and inorganic, which have distinct properties and behaviors that influence its mobility, toxicity, and persistence in the environment.

In water, arsenic can be found in forms such as arsenite (AsIII) and arsenate (AsV), among others. Arsenite tends to be more mobile and soluble in low pH waters, while arsenate is more prevalent in high pH waters. This difference in speciation can influence how arsenic disperses and accumulates in the environment, affecting the exposure of living organisms and the effectiveness of water treatment technologies.

Arsenic speciation is also crucial in terms of toxicity. For example, arsenite is considered more toxic to aquatic organisms than arsenate. Furthermore, arsenic's ability to transform from one form to another under changing environmental conditions can lead to prolonged exposure and unknown risks for human health and the ecosystem.

Arsenic speciation is relevant in assessing water quality as regulations and contamination standards often set specific limits for certain chemical forms of arsenic. Therefore, detailed speciation analyses are essential to better understand the distribution and behavior of arsenic in water and to take appropriate measures to mitigate its impact.

In the case of water quality management and environmental protection, arsenic speciation plays a crucial role in the design of strategies for monitoring and control, as well as developing effective and sustainable water treatment technologies. It is essential to understand the chemistry of arsenic and how it interacts with other elements in water to ensure the effectiveness of mitigation measures and the protection of aquatic ecosystems and human health.

To summarize, arsenic speciation is a key aspect to consider in managing arsenic contamination in water bodies as it influences its mobility, toxicity, and persistence in the environment. The application of advanced analytical techniques and the integration of arsenic speciation into water monitoring and treatment strategies are critical to effectively address this issue and protect both the environment and public health.

Traditional Techniques for Arsenic Quantification

Arsenic quantification in environmental samples and various materials is crucial due to the risks this element poses to human health and the environment. The presence of arsenic, both in total form and in its different chemical species, can have serious consequences for public health and aquatic and terrestrial ecosystems. As a result, having reliable and accurate analytical techniques for arsenic quantification is essential for monitoring and mitigating the risks associated with its presence. In this context, various traditional techniques have been developed that allow for the determination of arsenic in environmental samples with different levels of sensitivity, selectivity, and analytical complexity. These techniques are valuable tools for researchers, health professionals, and environmental managers in assessing and managing arsenic contamination. It is important to explore the traditional techniques used for arsenic quantification and understand their principles of operation, advantages, and limitations.

Atomic Absorption Spectrophotometry (AAS) for Total Arsenic:

Atomic Absorption Spectrophotometry (AAS) is an analytical technique used for the determination of total arsenic and other elements in liquid samples. It works by measuring the absorption of electromagnetic radiation by arsenic atoms in a liquid sample, allowing the quantification of the element of interest.

Operation

Sample preparation: The liquid sample is prepared using digestion or dilution techniques to convert the arsenic compounds present in the sample into a form that can be easily absorbed by the radiation beam.

Atomization: The prepared sample is introduced into an atomizer, where it is converted into a fine aerosol.

Radiation absorption: The sample aerosol is exposed to an electromagnetic radiation source of specific wavelength. Arsenic atoms in the sample absorb some of this radiation in an amount proportional to their concentration in the sample.

Detection: A detector measures the amount of radiation absorbed at a wavelength of around 193.7 nm by arsenic atoms and provides a reading that correlates with the concentration of arsenic in the sample.

Applications

AAS is widely used in industry, research laboratories, and quality control for the determination of arsenic in various environmental samples, including drinking water, wastewater, soils, sediments, foods, and pharmaceutical products.

It is especially useful in environmental monitoring studies, human health risk assessment, and scientific research related to the presence and distribution of arsenic in the environment.

Limitations

Sensitivity: The sensitivity of AAS can be limited compared to more advanced techniques, especially for samples with very low arsenic concentrations.

Selectivity: AAS may lack selectivity when analyzing samples with a complex matrix, leading to interferences and overestimation or underestimation of arsenic concentration.

Sample complexity: The presence of other chemical compounds in the sample, such as heavy metals or organic substances, can complicate the accurate determination of arsenic and require additional sample preparation to remove interferences.

In summary, Atomic Absorption Spectrophotometry is a widely used and robust analytical technique for the determination of total arsenic in liquid samples. However, its sensitivity, selectivity, and sample complexity can be limited compared to more advanced techniques, which should be considered when selecting the most suitable technique for specific arsenic analysis applications.

High-Performance Liquid Chromatography (HPLC) with Mass Spectrometry (MS) Detection for Arsenic Speciation

It is a powerful analytical technique used for arsenic speciation, i.e., the separation and quantification of different chemical forms of arsenic present in a sample. Below, its operation, applications, and limitations in terms of sensitivity, selectivity, and sample complexity will be described accordingly:

Operation

High-Performance Liquid Chromatography (HPLC): In HPLC, the sample is separated into its individual components using a chromatographic column and a mobile solvent. The separation is based on the specific interactions between the sample components and the stationary phase of the column. HPLC consists of the following steps:

- Sample preparation: The liquid sample to be analyzed is prepared, including its dilution or extraction of the components of interest.
- Sample injection into an HPLC system: The prepared sample is introduced into the HPLC system for analysis.
- Sample passing through a column with stationary material: The sample flows through a chromatographic column containing specialized stationary material.
- Separation and detection of components: The components of the sample are separated as they pass through the column, and then detected by a sensitive detector.

Analysis of the amount of each component: The peaks in the resulting chromatogram provide information about the amount of each component present in the sample.

This process allows for the precise and sensitive identification and quantification of the different components of a liquid sample, commonly used in various scientific and industrial fields.

Mass Spectrometry (MS): Individual analytes, different chemical forms of arsenic in this case, are introduced into a mass spectrometer after separation by HPLC. In the mass spectrometer, ions generated from the analytes are separated based on their mass-to-charge ratio and detected. It operates as follows:

- 1. Ionization: In this stage, the sample components are converted into electrically charged ions. This is achieved through various ionization methods, such as electrospray ionization (ESI), electron impact ionization (EI), or matrix-assisted laser desorption/ionization (MALDI), among others.
- 2. Ion separation: The generated ions are introduced into a mass analyzer, where they are separated based on their mass-to-charge ratio (m/z). Common mass analyzers include time-of-flight analyzer (TOF), ion trap analyzer, and quadrupole mass spectrometer.
- 3. Detection: The separated ions are detected and recorded based on their m/z. This detection is generally performed using an electron or ion detector, which converts the ion signal into a mass spectrum.
- Data analysis: The obtained data is analyzed using specialized software that identifies the compounds present in the sample and determines their relative concentrations.

Applications of HPLC-MS:

HPLC-MS for arsenic speciation has a wide range of applications in environmental research, toxicology, food science, and clinical analysis. It allows for the identification and quantification of different arsenic forms in environmental samples, such as surface and groundwater, soils, sediments, and food products. It is especially useful in studies of human arsenic exposure and in the health risk assessment associated with different arsenic chemical species.

Limitations

HPLC-MS is highly sensitive and can detect and quantify arsenic species at very low concentrations (in the range of parts per billion).

Selectivity: HPLC-MS is highly selective and can differentiate between different arsenic chemical forms with high precision, allowing for accurate speciation.

Sample complexity: Despite its high sensitivity and selectivity, sample preparation can be more complex compared to other analytical techniques. Careful sample preparation is required to avoid interferences and ensure result accuracy.

Cost and expertise: HPLC-MS is a sophisticated technique that requires specialized equipment and personnel with experience in its operation and data interpretation. The cost of acquiring and maintaining the equipment can be very high.

Therefore, HPLC-MS is a powerful and versatile analytical technique for arsenic speciation that offers high sensitivity and selectivity. However, its application can be limited by the complexity of sample preparation and the need for technical expertise to operate it effectively. Transmission Electron Microscopy (TEM) for Arsenic Particle Characterization

TEM is a technique used for particle characterization at a nanometric scale. It operates as follows:

Operation

Sample preparation: The sample is prepared by cutting very thin sections (usually less than 100 nanometers) to allow electrons to pass through it.

Image generation: An electron beam is directed towards the prepared sample. Some electrons pass through the sample, while others scatter or are absorbed by it. The electrons passing through the sample form an enlarged image on a detector, which is then visualized on a screen.

Sample analysis: The resulting image allows for the visualization of details of the sample at a nanometric level, such as morphology, crystal structure, and composition of arsenic particles present in it.

Applications

TEM is used in a wide range of applications, including research in nanotechnology, materials science, cell biology, and environmental science. In the specific case of arsenic, TEM is used to study the presence and distribution of arsenic particles in environmental samples, such as soils, sediments, atmospheric aerosols, and surface waters. It also allows the comprehension of the morphology and crystal structure of arsenic particles, crucial for evaluating their toxicity and mobility in the environment.

Limitations

Sensitivity: TEM has high sensitivity and can detect arsenic particles at a nanometric scale. However, the detection of very small particles may be limited by the microscope's resolution.

Selectivity: TEM offers high selectivity by providing detailed images of arsenic particles in a sample. However, precise identification of arsenic particles may require complementary techniques to confirm their composition.

Sample complexity: Sample preparation for TEM can be laborious and require specialized technical skills. Additionally, some samples may be sensitive to the electron beam, affecting the quality of the obtained images.

To summarize, Transmission Electron Microscopy is a powerful tool for the characterization of arsenic particles at a nanometric scale, offering high sensitivity and selectivity. However, its application may be limited by the complexity of sample preparation and the interpretation of the obtained images.

Finally, the determination of total arsenic and arsenic speciation in environmental samples requires the use of a combination of specific analytical techniques that provide accurate and reliable data. Each technique has its own advantages and limitations in terms of sensitivity, selectivity, and response time, so it is important to select the most appropriate technique based on the objectives of the study, sample nature, and available resources. Careful selection of analytical techniques is fundamental to ensure the quality and reliability of the obtained results, which in turn contributes to effective management of arsenic contamination and protection of human health and the environment.

Electrochemical sensors.

Electrochemical sensors are devices used to detect and quantify the presence of chemical substances in a sample, based on converting a chemical signal into an electrical signal. In their case, the electrochemical reaction occurs at the interface between the electrode and the sample, generating an electrical current proportional to the concentration of the analyte of interest.

Various electrochemical techniques are employed for the detection and quantification of analytes, including voltammetry, potentiometry, electrochemical impedance, amperometry, electrochemical chromatography, among others. Each one of these techniques has its own advantages and is suitable for different types of samples and analytes. Voltammetry is one of the most used techniques in detecting heavy metals, which plays a significant role in environmental monitoring.

Voltammetry involves applying a variable electrical potential across an electrode immersed in the solution of interest while measuring the resulting electrical current.

For heavy metals, voltammetry offers several advantages that make it a highly effective and preferred technique, for example:

- Sensitivity: Voltammetry is highly sensitive to low concentrations of heavy metals. It can detect trace and even sub-trace levels of these metals in a sample, making it ideal for environmental and quality control applications.

- Selectivity: The voltammetric technique can be selective towards a specific metal by the proper choice of the electrode and experimental conditions. This allows for the precise detection and quantification of a particular metal in the presence of other metal ions, which is crucial in the analysis of complex samples.

- Wide range of applications: Voltammetry is applicable to a wide range of sample matrices, including aqueous, biological, and soil samples, making it versatile for various areas such as environmental science, analytical chemistry, and metallurgy.

- Speed and ease of use: Voltammetry is relatively fast and simple to perform compared to other analytical techniques, making it suitable for routine and field applications.

- Low cost: Voltammetric equipment is generally more affordable than other sophisticated analytical instruments, making them accessible to laboratories with limited budgets.

- In situ studies: Voltammetry allows for measurements directly at the sampling site (in situ), which is advantageous for environmental monitoring applications and field studies.

Consequently, to overcome the disadvantages of traditional techniques, electrochemical techniques emerge as a powerful and versatile alternative for the determination of heavy metals due to their sensitivity, selectivity, wide range of applications, ease of use, low cost, and portability, making them a tool for realtime decision-making in environmental monitoring and industrial quality control.

Arsenic speciation in electrochemical sensors.

There are several electrochemical techniques used for the determination of arsenic in samples based on voltammetric methods, the most important ones are mentioned below:

- Adsorptive Stripping Voltammetry of Hydrogen (H-AdSV): This method involves the reduction of arsenic on the working electrode surface after preconcentrated accumulation through hydrogen adsorption. The technique offers high sensitivity and selectivity for the determination of arsenic in aqueous samples.

- Hanging Mercury Drop Electrode (HMDE): Hanging Mercury Drop Voltammetry is another electrochemical technique used for the determination of arsenic. Arsenic is reduced to arsenic (III) on the surface of the mercury electrode, allowing its detection and quantification. This is one of the most used techniques for arsenic speciation and determination of heavy metals. Although it is one of the most selective and sensitive techniques, it has several significant disadvantages, especially in terms of environmental impact and human health:

- Mercury contamination: Mercury is a highly toxic and bioaccumulative heavy metal. The use of mercury electrodes in electrochemical analysis can result in the release of mercury into the environment during the manufacturing, use, and disposal of the electrodes. This contributes to water, soil, and aquatic ecosystem contamination, impacting wildlife and human health.

- Risks to human health: Exposure to mercury, either through direct contact with mercury electrodes or the release of mercury vapors during their use, poses a serious risk to human health. Inhaling it can cause neurological damage, developmental disorders in children, reproductive problems, and other adverse health effects.

- Impact on aquatic ecosystems: Mercury contamination from mercury electrodes can have devastating effects on aquatic ecosystems, including the bioaccumulation of mercury in the food chain. Aquatic organisms, such as fish and shellfish, can accumulate dangerous levels of mercury, posing a risk to humans consuming these products.

- Environmental regulations: Due to the serious risks to human health and the environment associated with mercury, there are strict regulations limiting its use and release in many countries. The use of mercury electrodes may face legal restrictions and specialized handling requirements for proper disposal, which can increase the costs and complexities associated with their use.

Thus, the use of mercury electrodes in electrochemical analysis presents significant environmental and public health disadvantages due to the release of mercury and its toxic effects on ecosystems and people. For this reason, researchers have sought safer and environmentally sustainable alternatives in analytical practice.

In 2014, the study conducted by J.C.M.Gamboa and collaborators investigated a vibratory printed electrode using gold nanoparticles modified with carbon nanotubes for the determination of arsenic (III), a dangerous form of arsenic in water and other environmental media. They designed and manufactured a highly sensitive and selective electrochemical electrode by combining gold nanoparticles and carbon nanotubes, known for their favorable electrochemical properties, deposited on a printed electrode substrate, allowing for the creation of a miniaturized and low-cost detection electrode. Once the electrode was fabricated, its performance in detecting arsenic (III) was evaluated through electrochemical techniques such as voltammetry. The results showed that the gold nanoparticle-modified electrode with carbon nanotubes exhibited high sensitivity and selectivity for the detection of arsenic (III), with very low detection limits and a wide range of covered concentrations. Therefore, this research work demonstrates an effective alternative and the feasibility of using a vibratory printed electrode with gold nanoparticles and carbon nanotubes for the precise detection of arsenic (III), which could have significant applications in environmental monitoring, food safety, and public health.

The study by Saha and Sarkar published in the Talanta journal focuses on the development and application of an advanced analytical technique for the specific detection of As (III) through anodic stripping voltammetry with differential pulse. In this study, a vitrified carbon electrode modified with chitosan-Fe(OH)3 is used, representing a significant advance in the detection of arsenic and, more specifically, in its speciation. The improved sensitivity of this technique is derived from the ability of the modified chitosan-Fe(OH)3 electrode to selectively concentrate As (III) on the electrode surface, facilitating its detection with high sensitivity. This enhanced sensitivity is crucial in detecting low levels of arsenic, a highly concerning environmental contaminant due to its toxicity. Regarding selectivity, the choice to modify the electrode with chitosan-Fe(OH)3 also contributes to enhance the method's ability to discern between different arsenic species, such as As (III) and As (V). This is crucial in characterizing samples containing mixtures of these arsenic forms, as each presents distinct properties and behaviors. Thus, the research by Saha and Sarkar stands out for its innovative and highly specialized approach to detecting As (III) and its potential to significantly contribute to the arsenic speciation field. The improvements in sensitivity and selectivity achieved through electrode modification with chitosan-Fe(OH)3 open new possibilities in the accurate detection and characterization of arsenic, fundamental for both laboratory research and environmental analysis.

CONCLUSION

Arsenic speciation through electrochemical techniques emerges as a superior alternative compared to traditional approaches, due to its exceptional sensitivity, selectivity, analysis speed, and more competitive operating costs.

Firstly, electrochemical techniques offer outstanding sensitivity in the detection of different arsenic species, allowing them to detect very low levels of these toxic substances. This high sensitivity is crucial in many applications where precise and reliable detection of arsenic and its forms is required, such as in environmental or public health studies.

The selectivity of electrochemical techniques is a critical advantage since it allows the distinction between various forms of arsenic present in a sample, essential for assessing its toxicity and conducting a comprehensive characterization of arsenic contamination in different contexts. This reliable differentiation capability provides detailed information that can be fundamental for decision-making related to environmental management and public health.

Another important advantage is the speed at which electrochemical techniques can provide results. In many cases, the analysis speed is a critical factor for making quick and effective decisions in emergency situations or real-time quality control. The ability to obtain real-time information or results in very short timeframes makes electrochemical techniques a valuable tool in environments where it is crucial to have accurate and timely data.

In terms of operating costs, electrochemical techniques tend to be more efficient and economical compared to traditional methods of arsenic speciation. These technologies usually require lower reagent consumption and can be automated to optimize resources and reduce long-term costs. Hence, their economic viability makes them an attractive option for a wide range of applications in various sectors, from scientific research to industry.

Furthermore, the versatility of electrochemical techniques is remarkable, as they can be adapted and customized to meet the specific needs of different fields, such as environmental research, water quality monitoring, food control, or clinical analysis in the health sector. This flexibility makes them a multidisciplinary tool that can address a variety of challenges and issues, expanding their scope and relevance in multiple contexts.

In conclusion, electrochemical techniques emerge as an advanced and effective solution for arsenic speciation, redefining the analysis landscape of this substance in terms of sensitivity, selectivity, speed, and economic efficiency. Their potential to adapt to a wide range of applications in environmental research, public health, and industry positions them as a powerful and versatile tool in the fight against arsenic contamination and other related challenges.

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