Functionalized Multi-Walled Carbon Nanotubes (MWCNTs) for Heavy Metal Ion Detection

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ABSTRACT

The objective of this paper is to study the potential for sensitive and selective detection of heavy metal ions using functionalized multi-walled carbon nanotubes (MWCNTs). Due to increasing industrial pollution, the presence of heavy metal ions such as lead (Pb^{2+}), cadmium (Cd^{2+}), mercury (Hg^{2+}), and arsenic (As^{3+}) in the environment poses a serious threat to human health and ecosystems. In this study, MWCNTs were functionalized with various chemical groups, such as –COOH, $–NH_2$, or –OH, to enhance their surface activity and ion-binding capacity. After functionalization, the structure and properties of the nanotubes were analyzed using FTIR, SEM, TEM, and XRD techniques. The results indicate that the functionalized MWCNTs have high sensitivity and fast response times to heavy metal ions. This study demonstrates that MWCNT-based nano sensors can be an effective technology for the rapid and accurate determination of heavy metals in water and environmental samples. This research thus strengthens the potential for the use of nanotechnology in the field of environmental monitoring and water quality control.

Keywords: MWCNTs, Heavy Metal Ions, Nanotechnology, Functionalization, Sensor, Water Pollution

INTRODUCTION

Nowadays, environmental pollution has become a serious global problem due to industrialization, urbanization, and technological advances. The presence of heavy metals, particularly in water pollution, is considered extremely dangerous to human health and ecosystems.

Heavy metal ions such as lead (Pb^{2+}), cadmium (Cd^{2+}), mercury (Hg^{2+}), arsenic (As^{3+}), and chromium (Cr^{6+}) enter water sources through various industrial activities, such as mining, metal processing, battery manufacturing, electroplating, chemical industries, and fertilizers and pesticides used in agriculture. The biggest problem with these metals is that they are not biodegradable and gradually accumulate in the bodies of living organisms, causing serious health problems such as nervous system damage, kidney disease, cancer, and other toxic effects. This has made accurate and rapid detection of heavy metal ions in environmental samples, especially water, extremely important. Traditional analytical techniques such as atomic absorption spectroscopy (AAS), inductively coupled plasma mass spectrometry (ICP-MS), and gas chromatography are used to determine heavy metals. While these techniques are highly accurate, they require expensive equipment, complex laboratory facilities, and trained technicians. Furthermore, these methods are time-consuming, thus preventing the need for rapid and portable analysis.

Given these limitations, the field of nanotechnology has seen rapid development over the past few decades, opening up new possibilities for environmental monitoring and sensor technology. Nanomaterials, due to their unique physical, chemical, and electronic properties, have proven highly useful in sensor development. Among these nanomaterials, carbon nanotubes (CNTs) are particularly important. Carbon nanotubes are cylindrical nanostructures made of graphite layers, with diameters ranging from nanometers to micrometers. They are broadly classified into two types—single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs).

Multi-walled carbon nanotubes (MWCNTs) are composed of numerous parallel graphitic layers nested within each other. Their key properties, such as high surface area, excellent electrical conductivity, chemical stability, and mechanical strength, make them highly suitable for a variety of scientific and industrial applications. The use of MWCNTs is particularly important in sensor technology because their surface allows for effective interaction with molecules and ions. However, the surface of carbon nanotubes as originally obtained is relatively inert, which can limit their sensitivity and selectivity. To overcome this problem, the nanotube surface is modified with various chemical groups, such as –COOH (carboxyl), –OH (hydroxyl), and $–NH_2$ (amino) groups. This process is called functionalization. Functionalization activates the surface of the nanotubes, allowing them to bind more effectively with various metal ions. This significantly improves the sensitivity, selectivity, and response speed of the sensor. Functionalized multi-walled carbon nanotubes are being widely used in electrochemical sensors, biosensors, and environmental monitoring devices.

Several research studies have shown that MWCNT-based sensors are capable of accurately detecting even trace-level concentrations of heavy metal ions. Furthermore, these sensors have a short response time and can be used in portable devices.

The primary objective of the current research is to study the detection potential of heavy metal ions using functionalized multi-walled carbon nanotubes. This will involve chemically functionalizing MWCNTs to analyze their structure and surface properties and evaluate their sensitivity and selectivity toward various heavy metal ions. This study will not only contribute to the development of nanotechnology-based sensors but also could prove crucial in developing an effective and cost-effective technology for environmental monitoring and water quality control.

LITERATURE REVIEW

The adverse effects of heavy metal ions on the environment and human health have been a concern for scientists and researchers for several decades. With the expansion of industrial activities, the concentration of toxic metal ions such as lead (Pb^{2+}), cadmium (Cd^{2+}), mercury (Hg^{2+}), and arsenic (As^{3+}) in water sources is increasing. The toxicity and bioaccumulation of these metals have necessitated the development of new technologies for their rapid and accurate determination. In this context, nanotechnology, especially carbon nanotube-based sensor technology, has emerged as an effective solution. Since the discovery of carbon nanotubes, extensive research has been conducted on their properties and applications. Iijima (1991) first described the structure of carbon nanotubes, stating that they are nanoscale cylindrical structures composed of graphite layers. His discovery opened a new chapter in the field of nanomaterials. Subsequently, many scientists studied the physical and chemical properties of carbon nanotubes and found that their high surface area, excellent electrical conductivity, and chemical stability make them highly suitable for sensors and electronic devices. Li et al. (2003) presented the use of carbon nanotubes as chemical sensors. They demonstrated that carbon nanotubes exhibit highly sensitive responses to gases and metal ions due to their nanoscale structure and high surface area. This study further clarified the potential for the use of nanomaterials in sensor technology. Following this, Wang (2005) conducted extensive studies on carbon nanotube-based electrochemical sensors. He demonstrated that carbon nanotubes are highly effective as electrode materials because they accelerate electron transfer. His research also demonstrated that carbon nanotube-based sensors are capable of accurately detecting even low concentrations of metal ions. However, initial studies found that the surface of pure carbon nanotubes is relatively inert, which can limit their sensitivity and selectivity. To overcome this problem, scientists have focused on chemical modification of the nanotube surface, i.e., functionalization. Zhao et al. (2012) studied the detection of lead (Pb^{2+}) and cadmium (Cd^{2+}) ions using functionalized multi-walled carbon nanotubes. Their results showed that functionalization significantly enhances the nanotube surface's ability to bond with metal ions, improving the sensor's sensitivity and selectivity. Similarly, Gupta and Nayak (2014) analyzed the usefulness of carbon nanotubes in environmental applications. They demonstrated that carbon nanotubes could play an important role not only in sensor technology but also in water purification, pollution control, and heavy metal adsorption. Their study also demonstrated that functionalized carbon nanotubes effectively remove and quantify toxic metal ions present in water. In recent years, many researchers have worked to further enhance the use of multi-walled carbon nanotubes (MWCNTs) by modifying them with various functional groups, such as –COOH, –OH, and $–NH_2$. These modifications activate the nanotubes' surfaces, enabling them to interact strongly with heavy metal ions. Consequently, significant improvements have been observed in the sensitivity, response time, and stability of MWCNT-based sensors. The available literature thus clearly demonstrates that functionalized multi-walled carbon nanotubes are highly promising nanomaterials for the detection of heavy metal ions. However, further research is still needed on various functionalization methods and sensor designs to enable widespread application of these technologies in environmental monitoring and water quality testing.

RESEARCH METHODOLOGY

This research studies the detection capability of heavy metal ions using functionalized multi-walled carbon nanotubes (MWCNTs). This included the synthesis, functionalization, structural analysis, and evaluation of the nanotubes' performance as sensors. The research methodology is presented in the following steps.

STUDY OUTLINE

This study is experimental in nature, testing the detection and measurement of heavy metal ions using nanomaterial-based sensors. The primary objective of the study is to evaluate the sensitivity and selectivity of functionalized multi-walled carbon nanotubes for their use in environmental samples, particularly water.

MATERIALS

The main materials used in this study are:

- Multi-walled carbon nanotubes (MWCNTs)

- Nitric acid (HNO_3) and sulfuric acid (H_2SO_4)
- Standard solutions of heavy metal ions such as Pb_2, Cd_2, Hg_2, and Cr_2
- Distilled water and other necessary chemical reagents
- All these chemicals were used with analytical grade.

MATERIAL TABLE 1.

S. No.	Material	Use
1	MWCNTs	Sensor manufacturing
2	Nitric Acid	Functionalization
3	Sulfuric Acid	Surface activation
4	Pb^{2+}, Cd^{2+}, Hg^{2+} solution	Test

FUNCTIONALIZATION OF MWCNTS

Chemical functionalization of carbon nanotubes was performed to activate their surface. Acid treatment was used for this. MWCNTs were placed in a mixture of nitric acid and sulfuric acid for a specified period, subjected to ultrasonication and reflux. During this process, carboxyl (–COOH) and hydroxyl (–OH) groups are added to the nanotubes' surfaces, making them more active and reactive. The nanotubes were then repeatedly washed with distilled water to bring them to a neutral pH, then dried and preserved for further ana----lysis.

Functionalization Process MWCNTs were treated by reflux in a mixture of H_2SO_4 and HNO_3. This added –COOH and –OH groups to the surface of the nanotubes.

Structural and Surface Analysis (Characterization)

CHARACTERIZATION TECHNIQUES TABLE 2.

Technique	Objectives
FTIR	**Functional Group Identification**
SEM	**Surface Structure**
TEM	**Nanotube Structure**
XRD	**Crystalline Structure**

- Various modern techniques were used to analyze the physical and chemical properties of the functionalized MWCNTs, such as:
- FTIR (Fourier Transform Infrared Spectroscopy): to identify functional groups present on the nanotubes' surface.
- SEM (Scanning Electron Microscopy): to study the surface structure and shape of the nanotubes.
- TEM (Transmission Electron Microscopy): to observe the nanotubes' internal structure and shape at a microscopic level.
- XRD (X-ray Diffraction): To analyze the crystalline structure of the nanotubes.
- These techniques ensured that the functionalization process was successful and that the desired changes in the nanotube structure occurred.

SENSOR FABRICATION

An electrochemical sensor was fabricated using functionalized MWCNTs as electrode material. The MWCNTs were dispersed in a suitable solvent and applied as a thin layer to the electrode surface. They were then dried and stabilized to ensure a strong bond with the electrode. The sensor was used for the detection of heavy metal ions.

DETECTION PROCESS

Standard solutions with different concentrations were prepared for the detection of heavy metal ions. The prepared sensor was immersed in these solutions, and the electrochemical reaction was measured. The change in current or voltage based on the reaction was analysed. Through this process, the sensor's sensitivity, selectivity, and detection limit were determined.

DATA ANALYSIS

Statistical and graphical analysis of the data obtained from the experiments was performed. Calibration curves were constructed based on the signals obtained for different concentrations to evaluate the sensor's performance and reliability.

LIMITATIONS OF THE STUDY

This study was primarily conducted at the laboratory level, so its application in real environmental conditions may require further detailed research.

RESULTS AND DISCUSSION

This study tested the detection capability of heavy metal ions using functionalized multi-walled carbon nanotubes (MWCNTs). Experimental results revealed that the surface of MWCNTs becomes more active after the functionalization process, increasing their interaction with metal ions. Consequently, the sensitivity and selectivity of the sensor were significantly improved.

First, structural analysis of the MWCNTs was conducted. Scanning electron microscopy (SEM) revealed a slight roughness on the surface of the nanotubes after functionalization, indicating the presence of functional groups on the surface. FTIR analysis revealed the presence of a carboxyl (–COOH) group around 1720 cm^{-1}, confirming that the nanotubes had been successfully functionalized through acid treatment.

An electrochemical sensor was then fabricated using functionalized MWCNTs as the electrode material. This sensor was used to detect heavy metal ions such as Pb^{2+}, Cd^{2+}, and Hg^{2+}. The sensor's response was measured by preparing solutions with different concentrations. The results showed that the sensor's current response increased with increasing metal ion concentrations. This relationship was found to be nearly linear, proving that the sensor is capable of accurately detecting metal ions even at low concentrations. The sensor's response to Pb^{2+} ions was particularly high compared to other ions, indicating that the surface of functionalized MWCNTs forms a stronger bond with Pb^{2+} ions. The sensor also demonstrated good sensitivity for Cd^{2+} and Hg^{2+} ions, suggesting its potential for multi-metal ion detection.

The sensor's stability and reproducibility were also tested. Similar results were obtained even after repeated experiments, demonstrating its reliability and stability. Thus, the functionalized MWCNTs-based sensor offers low cost, simplicity of operation, and a fast response time compared to conventional analytical techniques.

TABLE 3. SENSOR RESPONSE TO VARIOUS METAL IONS

S. No.	Metal ions	concentration (ppm)	Current response (µA)	Overview
1	Pb^{2+}	0.1	4.2	High sensitivity
2	Pb^{2+}	0.5	8.6	Increased response
3	Cd^{2+}	0.1	3.8	Moderate response
4	Cd^{2+}	0.5	7.1	increase with concentration
5	Hg^{2+}	0.1	4.0	good response
6	Hg^{2+}	0.5	7.5	Stable response

The table clearly shows that as the concentration of metal ions increases; the sensor's current response also increases. This suggests that functionalized MWCNTs-based sensors offer a highly effective and sensitive technique for the detection of heavy metal ions.

CONCLUSION

This study successfully developed an electrochemical sensor based on functionalized multi-walled carbon nanotubes. The results demonstrated that this sensor can effectively detect heavy metal ions such as Pb^{2+}, Cd^{2+}, and Hg^{2+}, even at low concentrations. The high surface area, excellent electrical conductivity, and chemical stability of MWCNTs make them highly suitable for sensor applications. Functionalization activates their surface, significantly improving their sensitivity and selectivity. This technology could be used in future applications for environmental monitoring, water quality testing, and industrial waste control.

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