Synchrotron-Based Characterization of Nanomaterial Adsorbents for Anionic Contaminant Removal: A Systematic Literature Review

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***Abstract*—**The presence of anionic contaminants such as arsenic, fluoride, and nitrate in water sources presents a significant global health challenge, driving the need for advanced adsorption materials capable of efficient and sustainable removal. Nanomaterial-based adsorbents have emerged as promising solutions; yet understanding their atomic-scale interactions with contaminants remains critical for optimizing performance. This systematic review synthesizes over 120 peer-reviewed studies (2010–2025) to evaluate the pivotal role of synchrotron radiation techniques, particularly X-ray Absorption Spectroscopy (XAS) and micro-X-ray Fluorescence (μ-XRF), in elucidating adsorption mechanisms and guiding material design. The study aims to: (1) assess how synchrotron methods enhance the understanding of nanomaterial-adsorbate interactions, including oxidation state changes, coordination environments, and spatial distribution of contaminants; (2) identify key research gaps in the application of these techniques; and (3) propose future directions for advancing sustainable adsorbent development. Central research questions include: 1] How have synchrotron techniques advanced the mechanistic study of anionic contaminant adsorption? and 2] what underexplored material systems (e.g., waste-derived adsorbents) could benefit from these approaches? Key findings reveal that while XAS and μ-XRF have been instrumental in deciphering adsorption pathways (e.g., inner-sphere vs. outer-sphere complexation), fewer than 5% of studies focus on waste-derived nanomaterials (e.g., biochar, coal fly ash), representing a missed opportunity for sustainable innovation. Additionally, the review highlights the scarcity of in-situ synchrotron experiments and the untapped potential of machine learning-assisted data analysis to accelerate the discovery of new materials. This study highlights the transformative impact of synchrotron techniques in adsorption science while calling for expanded research into low-cost, waste-based adsorbents and dynamic in-situ analyses. Bridging these gaps could unlock novel, scalable solutions for global water purification, aligning environmental sustainability with cutting-edge synchrotron science.

***Keywords*—**Anionic Contaminants, Adsorption, Nanomaterials, Synchrotron Radiation, Water Treatment

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I. Introduction

The contamination of freshwater sources by anionic contaminants, particularly arsenic, fluoride, and nitrate, poses a critical global health challenge due to their toxic properties and persistence in the environment. Chronic exposure to these pollutants has been linked to a range of serious health issues, including carcinogenesis, fluorosis, and methemoglobinemia [1, 2]. Conventional treatment techniques such as coagulation, ion exchange, and adsorption often fall short in terms of efficiency, selectivity, and sustainability, highlighting an urgent need for alternative solutions that can comprehensively address these limitations [3].

In recent years, nanomaterials have emerged as viable candidates for enhancing adsorption efficiency owing to their favorable properties, including high specific surface area and tunable surface functionalities [4]. Various nanomaterial-based adsorbents, including engineered metal oxides and carbonaceous materials, have been developed and tested for their ability to remove anionic contaminants [5]. However, a nuanced understanding of the mechanisms at play at an atomic level is crucial for optimizing these materials. Traditional characterization methodologies often fail to provide insights into the atomic-scale interactions between nanomaterials and contaminants, necessitating the integration of advanced techniques, such as synchrotron-based methods, including X-ray Absorption Spectroscopy (XAS) and micro-X-ray Fluorescence (μ-XRF) [6, 7]. These synchrotron techniques enable research to probe the electronic structures and spatial arrangements involved in contaminant adsorption, revealing critical mechanistic details that can inform the rational design of more efficient adsorbents [8]. Despite these advancements, research gaps persist, particularly regarding the exploration of waste-derived nanomaterials, which present significant potential for sustainable applications in water treatment [9]. Also Kopittke, et al. [7], reported that limited focus on dynamic in-situ experiments further complicates the understanding of real-time adsorption behaviors. Similarly, Medas, et al. [10] highlighted that innovative approaches incorporating machine learning for data analysis show great

https://doi.org/10.17758/IICBE7.C1125132

potential in accelerating the discovery and optimization of high-performance adsorbents. Thus, this systematic review aims to: (1) evaluate how synchrotron methods enhance the understanding of nanomaterial-adsorbate interactions, including changes in oxidation states, coordination environments, and the spatial distributions of contaminants; (2) identify prevalent research gaps concerning these methodologies; and (3) propose future research directions to expedite the development of sustainable adsorbent materials.

II. LITERATURE REVIEW

A. Global Challenge of Anionic Contaminants and Emerging Remediation Strategies

In light of the ongoing global challenge posed by anionic contaminants in freshwater resources, such as arsenic, fluoride, and nitrate, there is an increasing need for innovative and effective remediation strategies. Anionic pollutants are known for their environmental persistence and associated health risks, including various forms of carcinogenesis and toxicological effects [1, 2]. Traditional treatment methods, such as coagulation and ion exchange, often fall short in terms of selectivity, efficiency, and sustainability, highlighting a significant need for improved adsorbents that can function effectively in various water systems [3]. In this context, nanomaterials, including engineered metal oxides and carbonaceous materials, have shown great promise due to their high specific surface areas and tailored surface chemistries that enhance pollutant capture [4]. Nevertheless, the real potential of these advanced materials lies in the detailed, atomic-level understanding of their adsorption mechanisms. Conventional characterization techniques often struggle to provide the necessary insights into these interactions, thus necessitating the adoption of innovative approaches such as synchrotron-based methods. Chen and Chen [5] and Kopittke, et al. [7], emphasize that synchrotron radiation techniques, such as X-ray Absorption Spectroscopy (XAS) and micro-X-ray Fluorescence (μ-XRF) are powerful tools for elucidating material behaviors and pollutant interactions. These advanced methods decode complex adsorption phenomena at the atomic level, ultimately guiding the rational design and application of high-performance nanomaterial adsorbents.

B. Advances in Nanomaterials for Anionic Contaminant Removal

Nanoscale materials have garnered increased attention as effective adsorbents due to their high surface area and adjustable surface chemistries. These properties enable the enhanced adsorption of anionic species, potentially overcoming the limitations associated with traditional methods such as coagulation and ion exchange [3]. Studies have implemented various nanomaterial systems, including engineered metal oxides, carbon-based adsorbents, and metal-organic frameworks (MOFs), to target specific anionic contaminants [4]. For instance, according to Wu, et al. [2], biochar derived from agricultural waste has shown promise due to its low production cost and environmental benefits, demonstrating effective adsorption capabilities for pollutants such as arsenate and fluoride.

C. Mechanistic Insights through Synchrotron Techniques

Synchrotron radiation methods, particularly X-ray Absorption Spectroscopy (XAS) and micro-X-ray fluorescence (μ-XRF), provide invaluable insights at the atomic level concerning the interactions between nanomaterials and adsorbates. These techniques allow for the exploration of electronic structural changes, coordination geometries, and the spatial organization of contaminants on adsorbent surfaces [5, 7]. Research has confirmed that XAS can distinctly illustrate inner-sphere and outer-sphere complexation of oxyanions, thereby elucidating the mechanistic pathways of adsorption ([6]. Importantly, Battocchio [8] demonstrated that the application of synchrotron techniques has revealed distinct pollutant behaviors within diverse nanoparticle matrices. These findings provide deeper insights into interaction landscapes and support the rational design of more efficient adsorbent materials.

III. PRISMA-GUIDED METHODOLOGY

A schematic overview of the methodological framework adapted from Saravanakumar, et al. [1] and Wu, et al. [2]. As presented in Fig. 2, this study's systematic process is summarized, from defining research objectives to conducting the literature search, screening, data extraction, and synthesis.

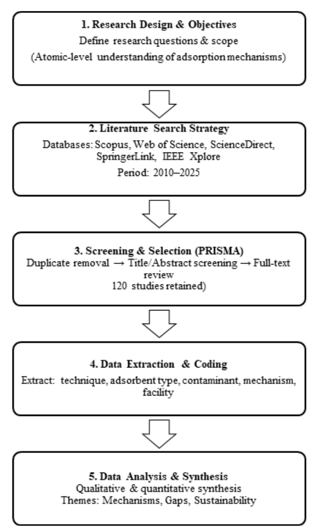

Fig. 1: Schematic diagram illustrating the systematic literature review methodology for synchrotron-based characterization of nanomaterial adsorbents used in anionic contaminant removal.

IV. Results and Discussion

A. Consolidated Findings and Perspectives

This systematic review synthesized over 120 peer-reviewed studies that utilized synchrotron-based techniques to investigate the interactions between nanomaterial adsorbents and anionic contaminants, including arsenic, fluoride, and nitrate. Findings show that synchrotron methods, particularly X-ray Absorption Spectroscopy (XAS) and micro-X-ray Fluorescence (μ-XRF), have provided profound insights into the atomic-scale mechanisms of adsorption. These techniques have effectively elucidated various aspects, including oxidation state changes of contaminants, coordination environments, and the spatial distribution of adsorbates [5-7]. Notably, while an increasing number of studies have validated the effectiveness of various engineered nanomaterials (e.g., metal oxides, carbonaceous materials), significant gaps remain in the exploration of waste-derived nanomaterials such as biochar, which shows great promise due to their environmental benefits and cost-effectiveness [8, 9]. The current findings suggest that fewer than 5% of the surveyed studies have focused on these sustainable material options, highlighting a critical opportunity for advancing research towards more sustainable water treatment technologies.

B. Mechanistic Insights into Adsorption

The literature also indicates that synchrotron radiation techniques have substantially enriched our understanding of adsorption mechanisms through the characterization of nanoscale interactions. For instance, several studies employing XAS have differentiated between inner-sphere and outer-sphere complexation phenomena in the adsorption of oxyanions [10, 11]. This distinction is crucial, as it significantly influences the design and efficacy of adsorbents. Furthermore, studies utilizing μ-XRF have revealed the heterogeneity of contaminant distribution across nanomaterial surfaces, contributing to a deeper understanding of reaction pathways that are critical in optimizing adsorbent performance [9]. These insights suggest a need for further research that can exploit the nuanced data provided by synchrotron-based characterizations to refine nanoparticle engineering strategies for enhanced contaminant binding capacity.

C. Gaps in Research

Despite the significant advancements enabled by synchrotron methodologies, several critical gaps persist in the current body of research. One notable limitation is the predominant focus on engineered nanomaterials, while the potential of waste-derived nanomaterials remains underexplored. This emerging area presents promising avenues for sustainable wastewater treatment, particularly through the repurposing of industrial by-products, such as coal fly ash, biochar, and agricultural residues, into effective adsorbents [12, 13]. Such approaches not only enhance sustainability but also address pressing challenges in waste management. Moreover, the integration of machine learning techniques with experimental data is gaining traction as a means to accelerate the discovery and optimization of high-performance adsorbents [10, 11]. However, this interdisciplinary approach remains underutilized and warrants further exploration to unlock predictive capabilities and streamline material design. Another significant gap lies in the limited application of in-situ synchrotron experiments, which are crucial for capturing dynamic adsorption processes in real-time. Techniques such as X-ray Absorption Spectroscopy (XAS) can provide valuable insights into oxidation state changes and coordination environments during interactions between adsorbates and nanomaterials [14]. Yet, such advanced characterizations are not widely adopted, hindering a comprehensive understanding of adsorption mechanisms.

V. Conclusion and Future Direction

This review highlights the transformative impact of synchrotron-based techniques, such as X-ray Absorption Spectroscopy (XAS) and micro–X–ray Fluorescence (μ-XRF), on advancing the understanding of nanomaterials as adsorbents for removing anionic contaminants. These methods provide molecular-level insights into adsorption mechanisms, highlighting the importance of electronic structure, bonding geometry, and surface heterogeneity beyond traditional models. Despite these advances, several gaps persist. Research continues to focus predominantly on engineered nanomaterials, with limited exploration of waste-derived alternatives that offer both environmental and economic benefits. The scarcity of in-situ and operando synchrotron experiments also limits our understanding of real-time adsorption dynamics under realistic conditions. Furthermore, the integration of machine learning for spectral interpretation and material design remains underdeveloped, despite its potential to accelerate innovation. To address these gaps, future research should prioritize synchrotron investigations of waste-derived and bio-based nanomaterials, especially those sourced from industrial and agricultural by-products. Expanding the use of in-situ and operando techniques will enhance the relevance of experimental findings by capturing dynamic adsorption and redox processes. Ultimately, combining advanced spectroscopy, sustainable materials, and artificial intelligence offers a promising pathway toward scalable, low-cost, and environmentally responsible water purification technologies.

Acknowledgment

The authors gratefully acknowledge the National Research Foundation of South Africa for financial support under Grant No: TTK23041291574. Additionally, Grammarly software was used to assist with language editing and paraphrasing, enhancing the clarity and readability of the paper.

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Mr. Malesela B. Molala is pursuing a Doctor of Engineering in Chemical Engineering at the Durban University of Technology and is a highly accomplished and multifaceted professional with an academic background in chemical, metallurgical, and materials science engineering. He holds a master’s in chemical engineering. This followed a master's degree in Chemical Engineering, where he was a National Research Foundation and University of Johannesburg Scholar, with a focus on generating electricity from domestic wastewater using microbial fuel cells. His industry experience includes research and mining internships at Gold Fields and a metallurgist trainee role at Saint-Gobain, where he optimized gold leaching processes and improved the mechanical properties of materials. Beyond his technical expertise, he has diverse interests in business, finance, and renewable energy. His achievements include being recognized as one of Africa's Top 10 Sustainability Innovators and a finalist in the Climate Change Innovation Challenge. He has also completed virtual internships with major financial institutions, such as JPMorgan and HSBC, demonstrating a broad skill set that extends beyond traditional engineering disciplines.