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Lanthanide – Doped Micro and Nanophosphors in Environmental Monitoring and Metal Ion Detection – A Review

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Abstract
The rising environmental challenges demand the urgent need for viable and eco-friendly technologies to evaluate the increasing pollutants in our surroundings. The rise in industrial processes, home combustion appliances, and forest fires has intensified the emission of hazardous substances like heavy metal ions, organic pollutants, and radioactive materials, which are affecting human health and ecological balance. This review paper focuses primarily on the detection of heavy metal ions in water. Studies have shown that an increase in heavy metal ions in the human body can lead to numerous health problems. The paper highlights the benefits of luminescent lanthanide-doped micro- and Nanophosphors for the evaluation and detection of multiple pollutants.

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Introduction

The lanthanide series, comprising fifteen elements from lanthanum to lutetium, holds a unique position in materials science due to their distinctive electronic configurations ([Xe] $4f^{[0-14]}$ $5d^{[0-1]}$ $6s^2$). These elements commonly form trivalent cations ([Xe] $4f^n$, where n ranges from 0 for La^{3+} to 14 for Lu^{3+}), but some exhibit other oxidation states, such as Sm^{2+}, Eu^{2+}, Yb^{2+}, Tb^{4+}, Ce^{4+}, and Pr^{4+}, which expand their functionality in advanced materials.[1]

Lanthanide-doped Nanophosphors have garnered considerable attention due to their exceptional optical properties, such as broad Stokes shifts, extended fluorescence lifetimes, and remarkable photochemical stability. These advantages make them superior to traditional organic fluorophores, especially for biological sensing and imaging applications.[2] The up-conversion mechanism, which involves absorbing multiple low-energy photons to emit a higher-energy photon, further enhances their utility. This feature, coupled with minimal autofluorescence and photoblinking, positions these materials as excellent candidates for bioimaging, particularly in the near-infrared (NIR) spectral range (650–900 nm), which facilitates deep tissue penetration.[3–5]

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The choice of doping elements and their concentrations significantly influences the properties of these Nanophosphors. Rare earth elements exhibit strong spin-orbit coupling and weak ligand field splitting, which enable their application in diverse fields, including environmental monitoring, biological sensing, and permanent magnetic materials.[6] Researchers have extensively studied rare-earth-doped apatite nanoparticles and identified their high quantum yields, sharp emission peaks, and resistance to photobleaching as key factors for their widespread adoption in sensing technologies.[7-8]

Environmental Applications and Challenges

The growing industrialization has led to increased environmental pollution, particularly through heavy metals like mercury, copper, cadmium, and lead, as well as nitrates and other harmful compounds in soil and water. These pollutants not only disrupt ecosystems but also pose severe health risks to humans and aquatic life. According to WHO and UNICEF (2021), over 2 billion people face challenges accessing safe drinking water, highlighting the urgent need for effective monitoring systems.[9-10] Furthermore, the presence of hazardous pollutants like Fe^{3+}, Cu^{2+}, and hydrazine in water sources necessitates advanced sensing technologies. These metal ions, though essential in trace amounts, can become toxic at higher concentrations, leading to severe health complications such as anemia, cardiovascular issues, and organ damage. To combat these challenges, biosensors have emerged as a crucial technology for rapid and precise detection. These biosensors, often incorporating luminescent nanomaterials, enable monitoring of pollutants and diagnosing diseases such as cancer with remarkable sensitivity.[7, 11]

Heavy metal ions, such as Fe^{3+}, are crucial in biological systems but require precise concentration control. Deficiency can lead to anemia, while excess levels can cause severe health issues like cardiovascular diseases and diabetes.[12] Similarly, excessive picric acid, used as an antiseptic, can adversely affect the central nervous and cardiovascular systems, necessitating its precise detection.[13]

Advanced Luminescent Techniques

Conventional methods like inductively coupled plasma mass spectroscopy, atomic absorption spectroscopy, and high-pressure liquid chromatography offer accurate pollutant detection but are often expensive and time-intensive. In contrast, lanthanide-doped nanophosphors provide a cost-effective and efficient alternative due to their anti-Stokes emission, sharp luminescent peaks, and size-independent properties.[14] These materials also play a pivotal role in drug delivery, chemical sensing, and detecting hazardous compounds, addressing critical environmental and healthcare challenges. Quantum dots (QDs) and rare-earth-doped nanoparticles have revolutionized medical diagnostics and environmental monitoring. These Nanophosphors exhibit high quantum yield, sharp emission peaks, and robust resistance to photobleaching. Commonly used lanthanide dopants include Ce^{3+}, Sm^{3+}, Eu^{3+}, Dy^{3+}, Tm^{3+}, and Yb^{3+}, each contributing distinct luminescent properties

Significance of Lanthanide-Doped Nanophosphors

Recent research highlights the efficiency of fluorescence-based biosensors in detecting contaminants, offering early diagnosis for healthcare applications and pollutant monitoring. Among the pollutants, hydrazine, a toxic industrial compound, has been declared harmful by the Environmental Protection Agency when present in drinking water above 10 ppb. Addressing such pollutants requires advanced materials like lanthanide-doped spectral modifiers, which have shown potential in reducing spectral losses and improving sensing efficiency.

Persistent luminescence phosphors have gained prominence in biomedical fields, reducing auto fluorescence and light scattering in tissue imaging.[15-17] Their ability to act as luminescent sensors is attributed to the unique energy transfer dynamics between host materials and dopants. These properties enable precise detection of pollutants like Fe^{3+} ions and nitroaromatic compounds, which are critical for monitoring environmental and human health.[17-19]

In this review, we aim to explore the synthesis methods, luminescent properties, and diverse applications of lanthanide-doped nanophosphors in environmental monitoring and metal ion detection. Additionally, we will discuss their potential as advanced sensors for pollutants and their role in addressing critical environmental and health-related challenges.

To Provide a Structured Analysis, this Review is Organized in Following Section

1. Introduction
2. The synthesis strategies and structural characteristics of Nanophosphors.

3. Applications of luminescent lanthanide-doped nanophosphors
- Luminescent Nanophosphors for Heavy Metal Detection
- Advanced Techniques for Ion Detection
- Detection of Nitro aromatic Compounds
- Fluorescence Probes for Specific Metal Ions
- Photocatalytic Applications for Water Purification
- Photocatalytic Applications in Dye Degradation

4. Challenges and Future Perspectives
5. Conclusion

Synthesis Methods of Nanophosphors

The synthesis of lanthanide-doped nanophosphors involves various methods designed to produce nanomaterials with desired optical and structural properties. The most common techniques include the sol-gel method, hydrothermal synthesis, and auto-combustion method. These methods ensure precise control over the size, morphology, and luminescent properties of the materials. For optimal quantum confinement, the particle size should not exceed twice the Bohr radius of the exciton.[20]

Sol-Gel Method

The sol-gel process is a versatile and widely used method for synthesizing nanophosphors. It involves the following key stages.

Hydrolysis

Precursor materials, typically metal alkoxides, react with water, breaking down into reactive molecules. This step sets the foundation for gel formation.

Condensation

The reactive molecules link together, forming a polymer network. As this network grows, it traps liquid within, transitioning into the gel phase.

Drying and Sintering

Excess liquid is removed, and the remaining solid network undergoes sintering to create stable nanomaterials with the desired properties.

This method allows for the synthesis of uniform nanomaterials with excellent optical properties, making it ideal for various applications.[21]

- G.A. El-Inany and colleagues synthesized $Bi_4Si_3O_{12}:Sm^{3+}$ nanophosphors using the sol-gel method. These nanophosphors were successfully applied in fingerprint detection and, upon functionalization with amino groups, were further used for glucose sensing.[22]
- Li-Na Wang and co-workers synthesized $CaYAlO_4:Tb$ nanophosphors via the sol-gel method, which were effective in sensing nitroaromatic compounds.[23]

Hydrothermal Synthesis

Hydrothermal synthesis is a solution-based technique that employs high temperature and pressure to synthesize nanomaterials. The process is highly flexible, enabling the formation of materials with specific shapes and sizes by controlling reaction parameters.

Advantages

This method offers the benefit of high temperature and pressure in a short time frame, which helps in maintaining uniform particle size and shape.[24]

- Marina Maletic and colleagues synthesized TiO_2/carbon composites via the hydrothermal method for removing organic pollutants such as drugs, pesticides, and dyes.[25]
- S. Tamboli and co-workers synthesized $LaOF:Yb^{3+},Tm^{3+}$ Nanophosphors using this method. These Nanophosphors were extensively studied using FIR techniques and showed potential for temperature sensing applications.[26-27]

Auto-Combustion Method

Auto-combustion synthesis, also known as self-propagating high-temperature synthesis (SHS), is a highly efficient and cost-effective method for producing nanomaterials. It involves rapid exothermic reactions between precursors, leading to the formation of nanophosphors.

Reaction Mechanism

Equilibrium Mechanism

The combustion occurs through reactive diffusion within the heat release zone near the combustion front, following a parabolic rate law.

Non-Equilibrium Mechanism

Combustion results in intermediate products, which subsequently transform into the final material without influencing propagation speed.[28]

- Qi Xiao and colleagues synthesized Sm^{3+}-doped TiO_2 nanocrystals using the auto-combustion method. These nanophosphors exhibited excellent photocatalytic properties under visible light.[29]
- Payal P. Pradhan and co-workers synthesized Y_2SrZnO_5:xEu^{3+} nanophosphors via auto-combustion. The vibrant red-emitting oxide was particularly effective in sensing fingerprints at crime scenes.[30]

These synthesis methods provide the foundation for creating lanthanide-doped nanophosphors with exceptional properties tailored for applications in environmental monitoring, biomedical sensing, and industrial processes. The choice of method depends on the desired material characteristics and application requirements.

Applications of Luminescent Lanthanide-Doped Nanophosphors for Environmental Monitoring

The rapid industrialization of modern society has brought about significant challenges to environmental sustainability, especially concerning water pollution. Among the pollutants, heavy metals, nitro-aromatic compounds, and other industrial contaminants pose severe risks to human health and ecosystems. To address these challenges, luminescent lanthanide-doped Nanophosphors have emerged as powerful tools, offering high sensitivity, selectivity, and adaptability for environmental monitoring. These advanced materials are paving the way for innovative detection and remediation strategies, bridging the gap between science and practical environmental applications.

Luminescent Nanophosphors for Heavy Metal Detection

Heavy metals like cadmium (Cd^{2+}), chromium (Cr^{3+}), lead (Pb^{2+}), and copper (Cu^{2+}) are among the most harmful environmental pollutants due to their toxicity and persistence in ecosystems. Luminescent Nanophosphors doped with rare earth elements have shown exceptional potential for detecting these metals with precision.

$NaGdF^4$:Eu^{3+}/Ce^{3+} Co-doped Nanophosphors

Synthesized via hydrothermal methods, $NaGdF_4$:Eu^{3+}/Ce^{3+} exhibits enhanced photoluminescence compared to single-doped variants. Its ability to detect Cd^{2+}, Cr^{3+}, and Pb^{2+} ions was validated through fluorescence quenching experiments at different excitation wavelengths. This indicates its ability as a versatile and reliable sensing material for monitoring heavy metal pollution.[31] This also indicates that the use of co-doping strategies not only improves luminescence intensity but also provides tailored detection capabilities, making it a promising approach for real-world applications in environmental analysis.

Functionalized $NaGdF_4$:Dy^{3+}/Tb^{3+} Nanophosphor

By incorporating asparagine onto the nanophosphor surface, researchers enhanced its water dispersibility and stability. This functionalization enabled the selective detection of Cu^{2+}, Mn^{2+}, and Hg^{2+} ions, with Cu^{2+} exhibiting the lowest detection limit and highest quenching constant. The sharp emission peaks at various wavelengths confirm its capability for precise metal ion detection.[32] Functionalizing Nanophosphors may expands their applicability, especially in aqueous environments, where many heavy metals are most prevalent.

Paper-Based Sensors using $NaGdF_4$:Yb^{3+}/Er^{3+} Nanophosphors

These up-converting nanoparticles provide ratiometric detection of Cr^{3+} ions with a detection limit as low as 2 nM. Their incorporation into paper-based sensors represents a significant step toward portable and cost-effective environmental monitoring tools.[33] The integration of Nanophosphors into user-friendly formats like paper sensors aligns with the need for scalable and field-deployable technologies.

Advanced Techniques for Ion Detection

FRET-Based Biosensors for Hg^{2+} Combining $NaYF_4$ Yb^{3+}/Er^{3+}

Nanophosphors with CdTe quantum dots has enabled effective sensing of Hg^{2+} ions. The energy transfer mechanism ensures precise detection, with fluorescence intensity diminishing as Hg^{2+} concentration increases.[34] This innovative use of FRET biosensors demonstrates how Nanophosphors technologies can evolve to detect specific ions in complex matrices.

Nonlinear Up-Conversion for Water Detection: A ligand-free $NaYF_4$

Yb/Er nanophosphor was synthesized to exploit the relationship between emission intensity and excitation power density. This novel approach enhances water detection in organic solvents, showcasing the versatility of Nanophosphors.[35]

Detection of Nitro Aromatic Compounds

Serine-Functionalized Core-Shell Nanophosphors

Ser-CS Nanophosphors exhibit high sensitivity toward nitroaromatic compounds such as picric acid (PA). The functionalization prevents aggregation and improves solubility, allowing precise detection with low detection limits36. Addressing environmental threats from explosives and industrial chemicals highlights the adaptability of Nanophosphors to various contaminants.

Fluorescence Probes for Specific Metal Ions

Tin Detection using $NaYF_4$:Yb^{3+}/Er^{3+}

Tin, used extensively in both organic and inorganic forms across various industries such as antifouling paints, agrochemicals, and PVC stabilizers, has led to significant environmental concerns due to its widespread release into air, water, and soil. This contamination poses severe risks to human health, potentially damaging the kidneys, bones, respiratory system, nervous system, and digestive system. Detecting tin ions (Sn^{2+}) is therefore crucial for environmental protection and public health. To address this issue, hexagonal (β-phase) up-conversion nanophosphors (UCNPs), specifically $NaYF^{+}$:Yb^{3+}/Er^{3+}, were synthesized using the solvothermal method. $NaYF_4$ served as the host matrix, with Yb^{3+} and Er^{3+} acting as sensitizers and activators. These UCNPs were linked to a rhodamine-based fluorophore, which enabled the detection of Sn^{2+} ions. Upon excitation with near-infrared (NIR) light, the nanophosphor emitted green light, which was absorbed by the fluorophore in a concentration-dependent manner to indicate Sn^{2+} levels. Two detection methods were compared, and it was observed that fluorescence-based nanoprobes were ten times more effective than Förster Resonance Energy Transfer (FRET)-based techniques, highlighting their superior sensitivity for Sn^{2+} detection.[37]

Copper Detection Using BGWO doped with Eu^{3+} The $Ba_6Gd_2W_3O_{18}$:

Eu^{3+} Nanophosphor synthesized via sol-gel method exhibited strong red luminescence, enabling copper detection with a limit of 0.138 μM. Its high Stern–Volmer correlation emphasizes its reliability.[38] The emphasis on affordability and high sensitivity makes fluorescence probes a preferred choice for copper monitoring in resource-constrained settings.

Polyethylenimine-Functionalized Nanophosphors: NaCeF4:Tb^{3+}/Eu^{3+} Nanophosphors functionalized with amine-rich polyethylenimine selectively detected Fe^{3+} and picric acid, with high quenching constants and low detection limits, confirming their potential for diverse environmental applications.[39]

Lead Detection with Disposable Sensors

Waste blue phosphor (BAM) embedded in a polyvinyl alcohol matrix, modified with dithizone, proved effective for Pb^{2+} detection. Its simplicity and effectiveness underscore its practical application in environmental analysis.[40]

Fe^{3+} Ion Detection via Novel Nanoprobe

According to a WHO survey, the permissible level of Fe^{3+} ions in drinking water is set at 0.3 ppm to ensure safety. Excess concentrations of Fe^{3+} ions can pose significant health risks, necessitating accurate and efficient detection methods. Over the years, various techniques for sensing iron ions have been developed, and research into more advanced methods continues. A novel nanoprobe, Ca-doped Eu:Y_2O_3 ($Ca_{0.01}$ $Eu_{1.94}$ $Y_{1.94}O_3$ or CEY), was synthesized using a facile combustion method to address this need. The nanoprobe's performance was evaluated using photoluminescence (PL) quenching experiments, which revealed its high sensitivity and effective detection range. The fluorescence intensity of CEY decreased progressively as the Fe^{3+} ion concentration increased, showing significant quenching at concentrations above 150 μM and complete quenching at 500 μM. PL spectra analysis indicated that the electric dipole transitions were more pronounced in the CEY nanoprobe, and its emission intensity was five times greater than that of the undoped Eu:Y_2O_3 nanophosphor. This enhancement is attributed to the incorporation of calcium, which improves the luminescent properties of the material. These findings demonstrate that CEY is a highly selective and sensitive probe suitable for detecting Fe^{3+} ions in real-world samples, making it a promising tool for environmental and water quality monitoring.[41]

Photocatalytic Applications for Water Purification
Lanthanum Silicates for Long-Lasting Phosphors and Water Monitoring

Lanthanum silicates are renowned for their long-lasting phosphorescence and environmental monitoring capabilities. Using a wet combustion method, $La_{10}Si_6O_{27}$:Dy^{3+} nanophosphors were synthesized. Characterization revealed a bandgap energy range of 5.6–5.72 eV. Under UV energy, Dy^{3+} emission spectra were studied, and electrochemical properties, assessed via current-voltage and impedance spectroscopy, demonstrated the material's potential for water quality monitoring.[42]

Photocatalytic Applications in Dye Degradation

Degradation of organic dyes under natural light has become a key focus in developing greener technologies. Lanthanide-doped materials have shown exceptional potential in this area.

Strontium Aluminates Co-doped with Europium and Dysprosium

Synthesized via hydrothermal and sol-gel methods, these aluminates exhibited excellent photocatalytic properties for decomposing methylene blue (MB) dye. Hydrothermal synthesis produced materials with comparable performance to sol-gel samples. The co-doped aluminates outperformed titanium dioxide (TiO_2) photocatalysts, highlighting their effectiveness in water treatment.[43]

Neodymium-Doped Barium Aluminates ($BaAl_2O_4$:Nd^{3+})

Using combustion synthesis, $BaAl_2O_4$:Nd^{3+} with a hexagonal structure was developed. At a doping concentration of 0.1 mol%, the catalyst achieved a degradation rate three times faster than its undoped counterpart. This nonlinear relationship between photoluminescence and photocatalytic activity underscores its efficiency in dye degradation.[44]

Dy- and Yb-Codoped Barium Aluminate ($BaAl_2O_4$, BAO)

BAO, previously studied for its photoluminescence, demonstrated outstanding photocatalytic performance in degrading MB dye. With over 99% degradation in less than 40 minutes, the Dy- and Yb-codoped variants exhibited superior stability and reusability, crucial for practical environmental cleanup applications. Characterization revealed optimized morphology, enhanced surface area, and multiple band-gaps, ensuring high efficiency in pollutant degradation.[45]

Challenges and Future Perspectives

Despite their promising applications, challenges such as synthesis complexities, surface modification, and scalability persist. However, advancements in material science are addressing these barriers, ensuring that luminescent Nanophosphors remain at the forefront of environmental monitoring technologies. The integration of these materials into real-world systems is critical for achieving sustainable solutions to pressing environmental issues.

Conclusion

The integration of luminescent lanthanide-doped Nanophosphors into environmental monitoring presents a transformative approach to detecting and quantifying pollutants with enhanced sensitivity and specificity. The unique luminescent properties of these materials, including sharp emission spectra and long luminescence lifetimes, enable the development of advanced sensing platforms capable of real-time analysis in complex environmental matrices. Moreover, their stability and resistance to photobleaching make them particularly suitable for long-term monitoring applications.

As environmental challenges continue to intensify, leveraging the capabilities of Nanophosphors not only aid in early detection and assessment of contaminants but also contributes to the development of sustainable monitoring practices. Future research should focus on optimizing the synthesis processes, enhancing luminescent properties, and expanding the range of detectable analytes, ultimately leading the way for innovative solutions in environmental protection and public health.

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Author Contributions

- **S.J. Dhoble:** Supervision and review
- **Nahida Baig:** Conceptualization, supervision, review, and editing.
- **Tanushree Akkewar:** Methodology, data curation, formal analysis, and writing – original draft.

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Not applicable

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