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Review Article

Green Synthesis of Nano-Azo Dyes: Emerging Colorimetric Sensors for Heavy Metal Ion Detection

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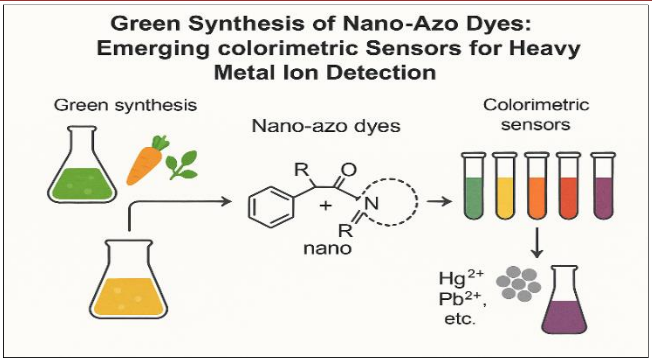

Abstract: Azo dyes are really good at helping us detect heavy metal ions in a way. We have made a lot of progress in making Azo dyes in a natural way and using tiny particles, which is called nanotechnology. This has led to the creation of Nano-azo dye products that're easier to use and give faster results. The main points of this review talk about how we can make Nano-azo dyes using things like plant extracts and natural solvents and how we can use techniques like microwaves and sound waves to save energy. Nano-azo dyes are very good at finding metals like iron, copper and lead because they have a big surface area and can be adjusted to work well with light. This is very important. Even though we have gotten some results there are still some problems to solve like making sure the Nano-azo dyes are stable and do not get confused by other ions and finding a way to make them on a big scale so we can use them commercially. We need to make the sensors stronger and find ways to use them with tiny materials and we want to use them to monitor the environment and help people's health. Using Nano-azo dyes that are made in a way is a great idea for the future because it will help us detect heavy metal ions in a way that is good, for the planet, efficient and affordable.

Keywords: Azo dyes, heavy metal detection, nanotechnology, green synthesis, Nano-azo dyes, plant extracts, environmental monitoring, biosensors.

OVERVIEW

Organic compounds containing at least one azo or R-N=N-R group are called azo compounds, and having azo compounds as their repeating units are known as azo polymers [1]. Having high extinction coefficients, facile substitution of the core structure and strong electron delocalization these compounds are also used as efficient color producing and color indicating compounds [2]. In analytical chemistry, a number of molecules containing azo groups have been utilized for the detection of metal ions, because such molecules exhibit dramatic color change upon complexation, high molar absorptivity and specific chemical reactivity [3, 4].

The importance of green chemistry principles, along with progress in molecular design, has considerably increased in recent years. Green synthetic methods are focused on the reduction or elimination of hazardous substances in the process of the preparation of the functional materials [5]. This leads to both environmental responsibility and employing safer methodologies, as has been well expressed by Anastas & Warner since 1998. The combination of these methods with nanotechnology gives Nano-azo dyes. Due to these dyes have enhanced surface area-to-volume ratios, higher reactivities and faster reaction rates [6]. Therefore, they are considered as the potential candidates for colorimetric sensing applications [7]. They are especially applicable for sensing toxic heavy metals such as Pb^{2+}, Hg^{2+}, Cd^{2+} and Ni^{2+} which has been verified by [8, 9].

This study intends to answer the current needs on the developments in eco-friendly synthesis of Nano-sized azo dyes with a critical analysis of their ever-growing application as colorimetric probes for the detection of heavy metal ions Moreover, the studies will address different routes for their preparation, the techniques to sense such ions, and the comparison among these techniques, as well as possible future trends for the development of sensing schemes exhibiting high performance, economic and at the same time green chemistry features for such detection [10].

Summary of the Journal

The following organization of this review is as follows: at first, Section [2], provides a brief introduction of green synthetic approaches for the production of Nano-azo dyes. The section highlights contemporary green-chemistry principles in comparison to traditional routes. Afterwards, in Section 3, we discuss the physicochemical features and the analytical advantages of Nano-azo dyes. This includes a study to the influence on important factors such as sensitivity, selectivity and response speed. Secondly, real applications are reviewed in Section 4. The article proffers an overview of the manner in which Nano-azo dyes are applied in the colorimetric detection of significant metallic ions, supported by the relevant results and performance vectors in contemporary research. Finally, in Section 5, challenges in the current research field would be summarized. It also describes possible future directions and opportunities to further the development of sensors based on Nano-azo dyes.

The Creation of Nano-Azo Colorings

Green chemistry provides a responsible and green-washed conventional form of by-product [11]. The latter conventional forms often require hazardous chemicals, high energy input and produce hazardous materials. Here is presented a short introduction related to state of the art green conflation methods. These include the use of bio-based reducing agents, green surfactants, and energy-efficient activation methods such as ultrasound and microwave irradiation [12].

Application of Work Excerpts and Natural Reductants

Phytochemicals derived from shops such as flavonoids, polyphenols and reducing sugars have been widely used as reducing and stabilizing agents in the synthesis of azo colorings. These naturally occurring molecules act as electron donors, which makes them biocompatible and facilitates the generation of azo bonds and nanostructures in situ, without the need of harmful solvents.

Salah Eddine Laouini and colleagues' research published in 2021 offers a potent illustration [13]. Their research group successfully synthesized Ag/ Ag_2O nanoparticles (NPs) via a facile waterless $AgNO_3$ route [14]. This process utilized a dry extract obtained from the leaves of Phoenix Dactyliferous L. The research fully investigates the effect of changing the (v/v) concentration potential (concentration of bio-extract against the precursor on the morphologies of Ag/ Ag_2O NPs [15]. Similarly, they characterize both optical and catalytic properties of the as-prepared NPs, and their activity on the degradation of dyes CR (Congo Red) and MB (Methylene Blue) [13].

Green Detergents and Waterless Media

The substitution of traditional organic detergents with more sustainable options, such as water and ethanol, is a key area in green chemistry. Application of waterless detergents presents impressive merits including diminished environmental risk along with better biocompatibility; this would be of utmost importance while the seeing techniques are to be implemented in environmental or natural frames.

A new investigation proposal that was recently put forward by Sharma *et al.*, (2022) illustrated synthesis of azo- azo-azo-azo-azo-azo-azo-monohybrids under waterless conditions. Their method was a one-pot diazotization–coupling reaction carried out at room temperature. The methodology was successful in terms of high product yield and appreciable solubilization.

Microwave Oven- and Ultrasound-Supported Conflation

The microwave oven has been introduced to speed up many chemical reactions. Frequently, reactions that are already carried out in a matter of seconds using microwaves would in fact take hours if not days when performed by conventional methods. Similarly, heat products are a well-known agent responsible for intensifying chemical reactions in microwaves. Molecular vibration frequency enhancement induced by the microwave oven irradiation is thought to be one of the factors that trigger these transformations. Rana and associates have contributed to the understanding of this miracle [15].

Microwave or solar heating have been found to speed up the rate of chemical reactions. The main reason for this is that the oven irradiation allows a larger increase in temperature for the reaction. El-Apaser M. A. and his associates [16], has gone into details regarding the experimenters similar to. They have demonstrated that the microwave oven radiation optimization probability of response could be predicted by simple thoughts involving activation energy, enthalpy and entropy values [17]. One of the good points in the oven use energy is that it is possible to get quick heating to high temperature under the atmosphere or in sealed container. This allows for easier reactions, and also reduces, if not eliminates the use of washing agents, a factor that is quite relevant when it comes to green chemistry principles [18, 19].

Comparison between Traditional and Green Synthesis Approaches

Factor	Outmoded Approaches	Green Approaches
Solvent Usage	Poisonous (e.g., chloroform, DMF)	H2O, ethanol, glycerol
Vitality Basis	Reflux, high-temperature furnaces	Microwave, ultrasound, room temperature
Tumbling Agents	Sodium borohydride, hydrazine	Plant extracts, ascorbic acid
Poisonous Spin-offs	Frequently produced	Nominal to none
Biocompatibility	Narrow	Improved
Conservational Effect	In height	Low
Response Period	Extended (hours to days)	Short (minutes to few hours)
Sustainability	Humble	Brilliant

At the same time, these styles meet today's requirements of green nanotechnologies [20]. Such merging makes them to be easily usable to large-scale and eco-friendly applications related to colorimetric sensing.

Characteristics and Benefits of Nano-Azo Dyes

From normal performances to nanomaterials, Nano-azo colorants provide an exceptional value leap. This is largely attributable to the combination with nanoscale feature, which greatly enhance their application in analysis. The better performance is due to unique physical and chemical properties related to the Nano scale and stronger interaction with materials as well as more efficiently signal transduction.

Physicochemical Properties

Increased Face Area

[21]. An important feature of the nanomaterial is the large surface area in proportion to its volume. Such a characteristic enables stronger interactions, including adsorption and desorption, between the surface of the detector and the analyte ions of interest. Hence, fewer azocolor dye molecules are accessible to the formation of complexes; and the sensitivity of detection is enhanced, even for ultra-low concentrations of analyte.

In the 2022 research, Xu, Huang *et al*., fabricated N-CDs@mMIPs and applied them successfully for the rapid and accurate detection of OTC in food samples [22]. Mesoporous armored N-CDs@mMIPs have been developed and they showed an enhanced performance compared to the traditional silicon-based MIPs luminescence detectors. This structure had a higher specific face area. This augmented face area improved particularly the light response of the N-CD, hence strong enhancement on the response time of the N-CDs@mMIPs was obtained in the existence of OTC [23, 24].

Tunable Optical and Electronic Properties

When measured in nonmetric scale, azo dyes always exhibit confinement effects. These influences also lead to differences within the optic immersion bands, inducing variations of the molar absorptivity and, more specifically, colorimetric alterations. The position and the intensity of the maximum wavelength immersion (λmax) can be tuned by modifications in the confines of the patches, and their face charge parcels, and most important, the girding chemical terrain.

As an illustration, research by Liu *et al*., (2019) highlighted the efficacy of gold nanoparticles. These patches, composed from azo linkers, exhibit a significantly redshift in their immersion range, accompanied by improved resolution when it comes Hg^{2+}. The effect has been to improve the visibility of the presence of this element, with detection certainly feasible at very low attention, falling beneath the sub-micromolar range [25].

Structural Versatility and Functionalization

The origin of heavy element and its related compounds in the soil can be physical altered and natural process throughout terrain. These critical soil and ground water accumulations are heavily influenced by colorful anthropogenic processing (such as but not limited to: mining operations, smelting processing, electroplating, etc.) in addition to natural weathering of their sourcing mineral-source accoutrements [26].

Popular their 2021 work, A. Rossi *et al*., was concentrated on the integration and explanation of Nano management tableware, the final functionalization by resources of 11MUA, and – prominently – the construction of a colorimetric sensor based on 11MUA-modifiedAgNPs. An examine for the recognition of Ni^{+2} ions in the presence of the biologically relevant ions: Mn_2, Co^{2+}, Zn^{2+}, Hg^{2+}, Fe^{2+}, Cd^{2+}, Pb^{2+}, and Cr^{3+} was designed for this sensor [18].

Advanced Selectivity

Face- position revision enables Nano-azo detectors production; hence, Nano-azo detectors can be modified with certain functional units (face- correction) to differentiate between essence ions with similar size and charge, by this way achieving higher selectivity [27].

Relying on a porous polymer imbued with an azo color, Wang *et al.*, 2020 achieved excellent selectivity for Fe^{3+} in the presence of $FeFe^{2}$ and Al^{3+}. The positive growth was attributed to the steric and electronic complementarity of the receptor and the target ion.

Rapid Response Time

Because of their upward prolificacy boom and the incessant chatter in the nanoscale shaking phenomenon, Nano-azo detectors responses are typically fast in a matter of seconds or a couple of twinkles. This speed makes them ideal for observing effects live, doing tests in the field [29].

Summary Table: Nano vs. Conventional Azo Dyes

Possessions	Conventional Azo Dyes	Nano-Azo Dyes
Apparent Region	Low	High
Sensitivity	Moderate (μM–mM range)	High (nM–μM range)
Selectivity	Often limited	Tunable via functionalization
Response Time	Slow (minutes–hours)	Fast (seconds–minutes)
Functional Modifiability	Limited	Broad (ligands, surfactants, polymers)
Stability in Media	May degrade	Improved colloidal and thermal stability

Utilizations of Nano-Azo Dyes for Heavy Metal Ion Detection

Increased concerns about the contamination of our water supply and ecosystem due to heavy metal ions like Cd^{2+}, Fe^{3+}, Hg^{2+}, Pb^{2+}, and Cu^{2+} have generated a heightened need for analytical methods that can produce highly sensitive and highly selective data with a quick turnaround time. One of the new promising avenues for detection purposes in an analytical setting is the use of nano-azo dyes. These dyes can produce a high degree of color change in the presence of these metals and are capable of being easily synthesized and modified to fit the specific needs of each user.

Detection of Iron (Fe^{3+})

Although essential to our health, water-born excess of Fe^{3+} ions can lead to formation of radicals, which may be detrimental. Nano-azo dyes functionalized with chelating groups are also frequently used to detect Fe^{3+}. This methodology allows for both ultralow detection limits and excellent accuracy. For example, azo 4c of Dana Akram and co-workers (2021) showed a perceptible affinity toward Fe^{3+} ions with a association constant of 4.63 × 108 M^{-1}. Furthermore, this isolable azo derivative was also found to exhibit excellent sensitivity to Fe^{3+} ions, with the limit of detection being 5.14 μM. By the application of both the molar ratio method as well as the Benesi-Hildebrand method the complexation of azo 4c with Fe3+ was confirmed and the binding stoichiometry ruled as 1:2. Hence, azo 4c proved to be a promising candidate to develop potent chemical tools for sensing Fe3+ ions Figure 1 [32].

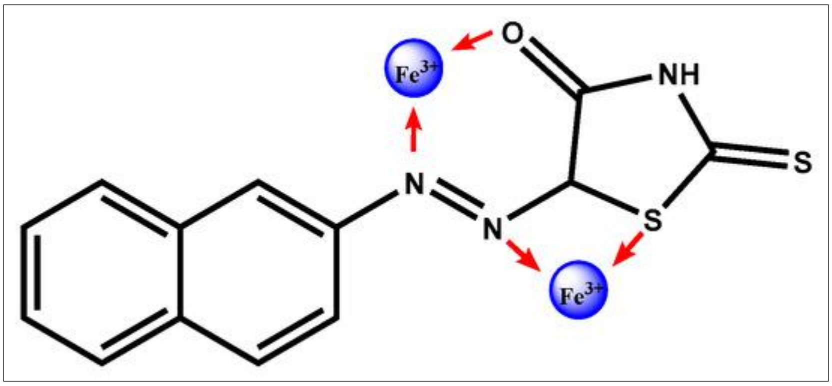

Figure 1: Chemical form for 4c azo with Fe^{3+}

Detection of Copper (Cu^{2+})

Copper (II) ions are essential for biological activities and are known to be toxic where in high amounts. Nanoscale azo dyes have been functionalized to selectively chelate Cu2+ ions using N and O as binding sites.

For example, see Homa Shafiekhani *et al.*, (2024). Thisspecific ligand functioned as a selective probe for the detection of trace amounts of Cu2+ in aqueous media. The color of the ligand changed upon addition of Cu^{2+} and turned from red to light orange under the ambient light. Under optimized conditions, LOD and LOQ are given as 0.13 and 0.44 µM, respectively. A linear working range from 1.6 to 17.5 µM was found with a correlation coefficient of 0.9964. Furthermore, the sensor was reversible and the response time was estimated to be ∼ 6 min. The stoichiometric binding established for the chemo sensor's bis-azo dye Cu2+ [BAD-Cu2+] complex was found to be 1:1 using Job's plot as a result. Figure 2

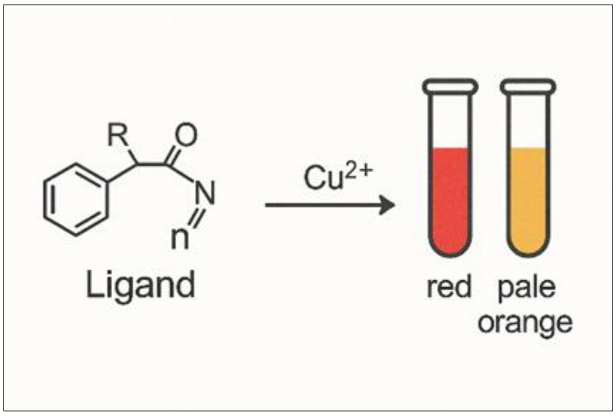

Figure 2: Bis-azo dye Cu^{2+} [BAD-Cu^{2+}] complex

Detection of Lead (Pb^{2+})

Lead requires meticulous detection, particularly in the realm of environmental surveillance, due to its neurotoxic properties. Nano- azo dyes, especially those encapsulated in mesoporous structures, have a pronounced affinity for Pb^{2+}.

Marco Zannotti and his platoon (2024) utilized a green system for the production of Gold nanoparticles (AuNPs). This procedure turned out to be a fruit shell submarine extract (OPE, from Citrus sinensis) as a sinking go- between. (32) The colorimetric assay was also subjected to evaluation at different time intervals, at the presence of Pb2 ions. This method exhibited variable Limits of Discovery (LOD) values: 13.31 µM after 15 twinkles and 0.05 µM after 90 twinkles. The feasibility of the proposed colorimetric assay, on the use of AuNPs@OPE for real samples, was validated through the analysis of drinking water samples for the presence of Pb2 [34, 35] Figure 3.

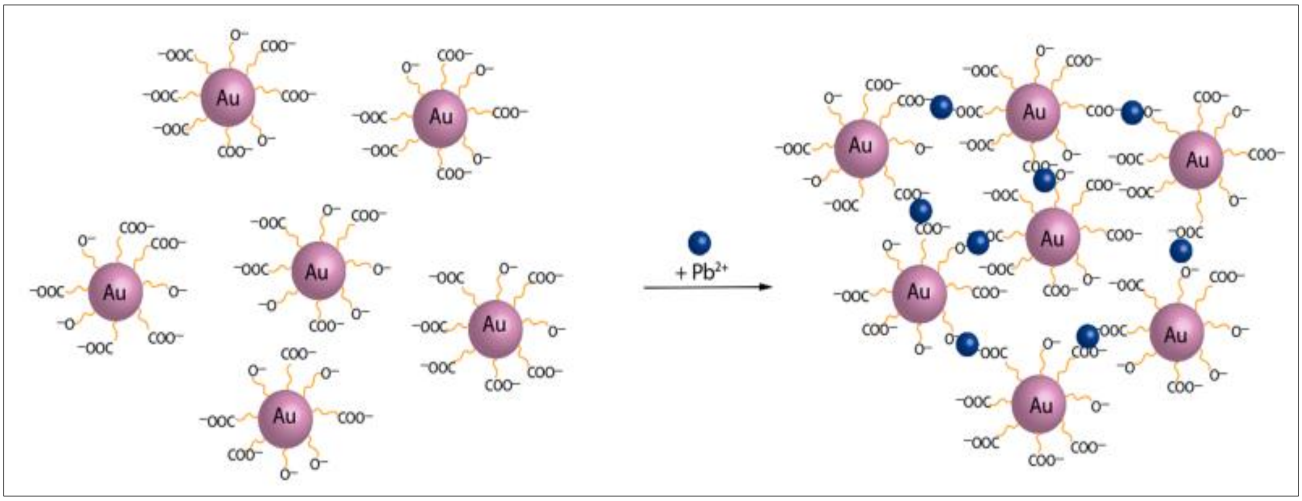

Figure 3: Visual depiction of the colorimetric mechanism employed by AuNPs@OPE for sensing Pb^{2+}

Other Metal Ions (Hg^{2+}, Cd^{2+}, Ni^{2+})

Nano-engineered azo dyes showcase significant potential in detecting mercury, cadmium, and nickel ions, delivering valuable analytical data.

To illustrate, A. Balamurugan and his group developed and studied Azo-2. This compound emerged as a novel dual colorimetric chemosensor, capable of detecting both Hg^{2+} and F− ions. The specific colorimetric reactions of Azo-2 toward Hg^{2+} ions depended on the specific dehydration process catalyzed by Hg^{2+}, ultimately yielding a strong electron-attracting nitrile group within a DMSO/water mixture at a pH of 5 [34]. Azo-2 additionally proved to be an outstanding and selective chemosensor for F^- ions [35] Figure 4.

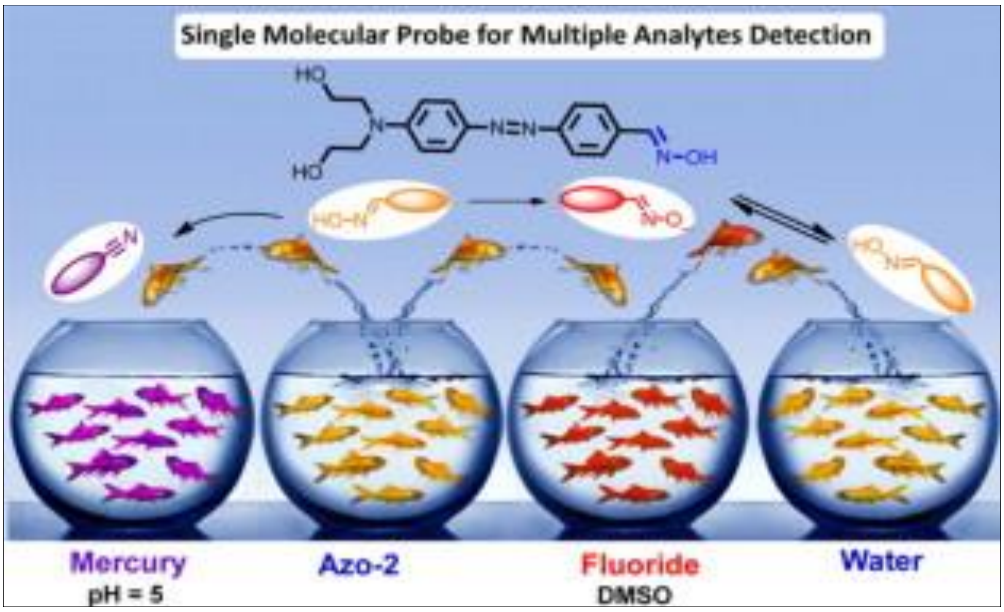

Figure 4: A molecular probe built around azobenzene and featuring aldoxime groups was created and put together. This probe's design enables it to perform double-duty, identifying both Hg^{2+} and F^{-} ions.

[36], different approach is based on creating complexes of heavy metals and detecting their optical properties [37]. An example of such a method was based on visual detection using dye-loaded membranes. Several ions, including (Hg2+, Cu2+, Ag+, Ni2+, Co3+ Pd2+, Zn2+), and Fe2+ were detected using various aromatic dyes and the detection limits were in the sub-ppm range.

Practical Applications

Due to their exceptional analytical properties, nano-azo dyes have been utilized in various operational systems and instruments:

Portable test equipment and dipsticks containing Nano-azo dyes are utilized for precise identification of heavy metal pollutants within potable water networks and industrial effluent in the field, which are now routinely monitored through monitoring monitoring drinking water. Water quality in aquatic ecosystems like lakes and rivers is continuously monitored through the use of paper-based sensors and smartphone data analysis with fluorescent dyes.

Nano-azo dyes are utilized in polymer membranes and electrodes to enhance electrochemical detection, resulting in faster responses and more efficient sensors for electrochemistry detection [38, 39].

Challenges and Future Perspectives

Although significant progress has been made in environmentally friendly synthesis and the practical application of Nano-azo dyes, which can be used as color-shifting detectors for heavy metal ions, there are still significant issues to be resolved. The complete commercial and practical potential of these technologies must be realized before these issues can be resolved.

Current Challenges

Stability Issues

Nanomaterial production with sustainable practices unlocks significant potential for enhancing green technologies and decreasing environmental impacts associated with manufacturing. Although obstacles still exist, the ongoing investigation and innovative ideas pave the way for viable/productive processes to develop as an environmentally friendly approach to synthesise nanomaterials [40]

Interference and Selectivity

The prevalence of pollution caused by heavy metal ions has gained significant attention. Both the health of humans and the natural environment are at risk due to contamination.... There are also potential solutions in nanotechnologies, which allow for molecular and atomic level manipulation, measurement of materials at about 1-100 nm (100 nanometers). The ratio of surface area to volume in these materials is significant. Due to their easily accessible nature, they are highly valuable tools, particularly for environmental cleanup. In particular, the capabilities of nanocomposites have shown to be promising for detecting and quantifying heavy metals. Water resource quality is still a top priority for modern researchers. Environmental sustainability is at risk due to the prevalence of heavy metal pollution. This has led to ongoing research in developing sensors that are both sensitive and fast-responding, as well as highly selective [41, 42].

Scalability and Commercial Production

Efforts are given due to the advantages of green synthesis over traditional chemical methods, but difficulties arise related to reproducibility and scalability, as well as cytotoxicity. In the future, it is important to standardize synthesis techniques and develop appropriate production methods for industrial applications while also exploring more comprehensive approaches to studying AgNPs' cytotoxic pathways. Plant-assisted green synthesis appears to be a promising and environmentally friendly option, with significant potential for creating AgNPs that have practical applications in biomedicine [43, 44].

Future Opportunities

Incorporation with Further Nanotechnologies

Study new materials developed for the first stage of analytical testing, which is designed to identify and pre-concentrate toxic metals in consumer goods and sustainable products.? The study of toxic metals has been increasingly centered on a distinct metallic crystalline solid, known as an organic framework, in recent years. F.D. Their inherent porosity results in large internal surface areas, high adsorption capacities and flexibility for functional modifications, while also maintaining stability. The use of MOFs as swift and potent adsorbents for extracting large metallic ions from complex models is significant in nutrition care evaluations and environmental pollution control strategies [45].

Expansion to Biomedical and Food Safety Applications

The development of Nano-sensor technology represents a significant leap forward in the fight against microorganisms that cause foodborne diseases. ". Nano-sensors have been identified as more dependable, precise and efficient than conventional methods [46], in this evaluation. By incorporating them into food safety protocols, we can significantly improve our ability to detect and control foodborne pathogens, ultimately contributing to a decrease in the incidence of food-related illnesses. But some obstacles still exist: such as improving the technology, ensuring it is affordable to consumers and complying with regulatory requirements. It is important to address these issues and enhance Nano-sensor technology as much as possible in future studies. Beyond food safety, these advancements have wider benefits for public well-being and environmental monitoring [47]. The ongoing development of Nano-sensor technology is crucial for ensuring an increasingly healthy future for our food supply [46].

DEDUCTION

Nano-azo dyes, produced using green chemistry-compliant techniques, are a potentially effective and environmentally friendly form of color sensors used to detect large metallic ions. The higher quality of analysis is largely due to the specific physical and chemical traits of these dyes, such as larger surface area, variable light properties, and greater selectivity; these colors have sensitivity that is enhanced while response times are accelerated. In addition to combining nanotechnology and environmentally friendly synthesis, we can also create cost-effective, fast-growing, and reliable sensing platforms.

Despite significant progress made, challenges such as stability, selectivity, and industrial application scaling up remain. As a result, it is imperative to continue investigating and improving these current deficiencies. Future projects focusing on long-lasting sensors, nanocomposites with multiple applications, and their implementation in fields like environmental monitoring and biomedical science will facilitate the transition from experimental models to functional, deployed systems."'.

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