



## MINI REVIEW

### Gold Nanoparticles: Synthesis, Mechanism and Applications as Colorimetric Metal Ions Sensor

SK BASIRUDDIN<sup>✉</sup>

Department of Chemistry, Maulana Azad College, 8 Rafi Ahmed Kidwai Road, Kolkata-700013, India

Corresponding author: E-mail: [sk.basi@gmail.com](mailto:sk.basi@gmail.com)

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The environment, consist of the lithosphere, atmosphere, hydrosphere and biosphere, is gradually threatened by various pollutants, among which heavy metal ions pose significant ecological and health risks. Natural phenomena in everyday life contribute to metal ion contamination in the environment. Prolonged exposure to heavy metals can cause severe health issues, including neurological disorders, organ damage and increased cancer risk. Hence, the development of sensitive, selective and efficient detection probes for metal ions, especially in aqueous media, is essential. Gold nanomaterials have gained importance as promising sensors due to their unique size-dependent optical, electronic and chemical properties. Their strong localized surface plasmon resonance (LSPR), high surface-to-volume ratio and ease of functionalization make them excellent candidates for colorimetric of metal ions. This review emphasize on the application of functionalized and label-free gold nanoparticles for metal ion detection. Recent advances and examples from the literature show that these nanoprobe are effective for environmental monitoring, and we cover their production, surface modification strategies and sensing processes.

**Keywords:** Gold nanoparticles, Plasmonic nanomaterials, Surface plasmon resonance, Aggregation, Red shift, Detection.

## INTRODUCTION

The environment is composed of the land (lithosphere), the Earth's atmosphere and the water (hydrosphere) where humans, plants, animals and microorganisms live or work (biosphere) in harmony. This precious environment has been polluting by several pollutants such as inorganic ions, organic pollutants, organometallic compounds, radioactive isotopes, gaseous pollutants and nanoparticles [1]. However, we will focus only heavy metal ion detection in this review. Before dive into this topic we should know the source of metal ion in the environment. Metal ion pollution occurs when the concentration of these elements exceeds the expected levels in the environment [1-3]. Main reason of metal ion contamination is due to fast growing of industrialization and various anthropogenic activities like mining of metal, smelting, foundries, leaching of metals from different sources such as landfills, waste dumps, excretion, livestock and chicken manure, runoffs, automobiles and roadworks. Heavy metals used in pesticides, insecticides in agriculture, are the secondary source of heavy metal pollution. Some natural phenomenon also increase heavy metal pollution such as volc-

anic activity, metal corrosion and metal evaporation from soil and water and sediment re-suspension, soil erosion and geological weathering, *etc.* [1-5]. Heavy metals can cause neurological impairments, leading to the cognitive decline and memory loss. Their accumulation in the kidneys and liver can result in organ dysfunction. Prolonged exposure to heavy metal ions increases the risk of cancer, hypertension and cardiovascular issues [6,7]. Furthermore, chronic exposure weakens the immune system, making the body more vulnerable to infections. Therefore, it is necessary to engineering some suitable tool to detect the existence of presence of such metal ions in the environment especially in aqueous medium as much as in the low concentration. It was observed that nanomaterial based probe is emerging tool to detect such metal ions in the low concentration.

Gold has been valued for centuries for its stability, conductivity and elegance. However, with the rise of nanotechnology, gold has transformed from a bulk metal into tiny nanomaterials with unique properties. This shift began in 1857, when Michael Faraday first developed colloidal gold and noticed its special optical behaviour [8]. This discovery laid the foundation for gold nanomaterial research. Over time, scientists developed

advanced techniques to control gold at the nanoscale, leading to different forms such as gold nanoparticles (AuNP), gold nanorod (AuNR) and gold nanocluster. These tiny structures have unique properties based on their size and shape, especially their ability to interact with light through localized surface plasmon resonance (LSPR) [9]. By modifying their surface; researchers have expanded their use in biosensing, imaging, drug delivery and environmental monitoring.

Today, gold nanomaterials play a crucial role in science and technology, evolving from a simple metal into a multifunctional material due to having unique size dependent optical properties. Their tunable properties continue to drive innovations in nanotechnology, materials science and medicine, especially in biological science such as in photo-thermal therapy, as dark field imaging contrast agents and as surface enhanced Raman spectroscopic (SERS) substrates [10-14]. Gold nanomaterials have emerged as highly effective tools for metal ion detection, owing to their unique optical, electronic and chemical properties. Their high surface-to-volume ratio, ease of functionalization, strong localized surface plasmon resonance (LSPR) characteristics make them excellent probes for sensing applications due to their ability to exhibit distinct colorimetric and electrochemical responses upon interaction with metal ions [15,16].

Here in this review, we concentrate use of functionalized or label free gold nanoparticles for application in metal ions detection. The functionalizations of gold nanoparticles with various molecules are explored and evaluate the efficiency of metal ion detection based on affinity-based interactions and change of plasmonic property of gold nanoparticles upon interaction with metal ions. Our focus will be on the literature survey of gold nanoparticles functionalized with different affinity molecules or label free gold nanoparticles for metal ions detection.

**Interaction of light (i.e. electric field) with plasmons of gold nanoparticles:** Gold nanoparticles have large collection of electrons, which is known as plasmon. These plasmons have surface confinement within the gold nanoparticles or plasmons are said to be localized within the nanomaterials. When electromagnetic wave or light is irradiated on the gold nanoparticles, charge dipoles are resulted on the nanoparticles due light and nanomaterials interaction. Orientation of these charge dipoles depends on the geometry and shape of the nanomaterials. Direction of these charge dipoles depends on the on the direction of electromagnetic wave or light and therefore it changes with the changing of direction of light. These charge dipoles or plasmons vibrate with irradiated light against ionic metallic core of nanomaterials. When this frequency or wavelength of vibration is matched with the frequency or wavelength of light, is called localized surface plasmon resonance (LSPR) [17]. Wavelength of LSPR depends on the size, shape and dielectric constant of the medium surrounding to the nanomaterials.

Gold nanoparticle are spherical in shape, therefore, it is isotropic in shape. Gold nanoparticle only shows one LSPR. Dipole oscillation of gold nanoparticle is called transverse oscillation or transverse LSPR. Greater is the charge separation of gold nanoparticle, greater will be the restoring force. Hence greater will be its frequency of oscillation. Wavelength of LSPR of spherical AuNP changes from 515-600 nm with size chang-

ing from 10-100 nm. Gold nanoparticles have tremendous application in sensing based application due to shifting nature of LSPR wavelength with changing of size, shape and dielectric constant value at the vicinity of gold nanoparticles.

**Gold nanomaterials and their optical properties for metal ion detection:** Gold nanomaterials (AuNPs) with various size and shape have unique optical property. Spherical gold nanoparticles show localized surface plasmon resonance (LSPR). Gold nanoparticles exhibit unique plasmonic properties due to the collective oscillation of conduction electrons on their surface on interaction with radiation of light. This is called localized surface plasmon resonance (LSPR) as a result gold nanoparticle solution appears as vibrant colours depending on the size of the particles. However, when AuNPs aggregate, their plasmonic properties change due to interparticle coupling effects. Aggregation leads to a redshift in the LSPR peak and can result in changes in the intensity and width of the absorption band. This phenomenon manifests in the colloidal solution on changing the colour of the solution. Changes in the local refractive index near the nanoparticle surface also cause shifts in the LSPR wavelength. These are the basis for many sensing applications.

**Change in optical property due to aggregation of gold nanomaterials:** Gold nanoparticles are widely used for detecting toxic metal ions. Gold nanoparticles show various LSPR, which is the key optical property for detection of toxic metal ion through UV-visible absorption-based technique. Wavelength of the localized surface plasmon resonance (LSPR) of the gold nanoparticles changes upon interaction with various analyte. These wavelength shifts arise due to modifications in the dielectric constant of the surrounding environment and changes in interparticle spacing caused by nanomaterials aggregation or assembly. Wavelength of LSPR isolated or dispersed AuNPs in a medium are totally different than that of aggregated or close contacted AuNPs. When AuNPs are isolated or well disperse in solvent, wavelength of LSPR does not change. However, when it aggregated or get close to one another its LSPR wavelength gets shifted to higher wavelength (red shift) in appreciate amount. In Fig. 1, it has been shown that how LSPR wavelength changes upon interaction among Au nanomaterials in presence of light.

However, AuNPs do not come close together or aggregate by themselves and made to come close together or aggregated. Gold nanomaterials need to be functionalized in such a way that these should be highly colloiddally stable in absence of analyte and however, in presence of analytic molecules or ions they are made to come close or aggregated (red shifting of LSPR wavelength) or aggregated AuNPs are brought to the stable colloidal solution in presence of analytic ions or molecules (blue shifting of LSPR wavelength).

**Design of functionalized AuNPs based sensor:** Synthesis of AuNPs based colorimetric metal ions sensor is a multi-steps process which includes (i) synthesis of AuNPs and (ii) properly surface modification of AuNPs with appropriate functionality especially, which selectively binds with targeted metal ions. Here, some of the conventional synthesis of AuNPs have been described briefly:

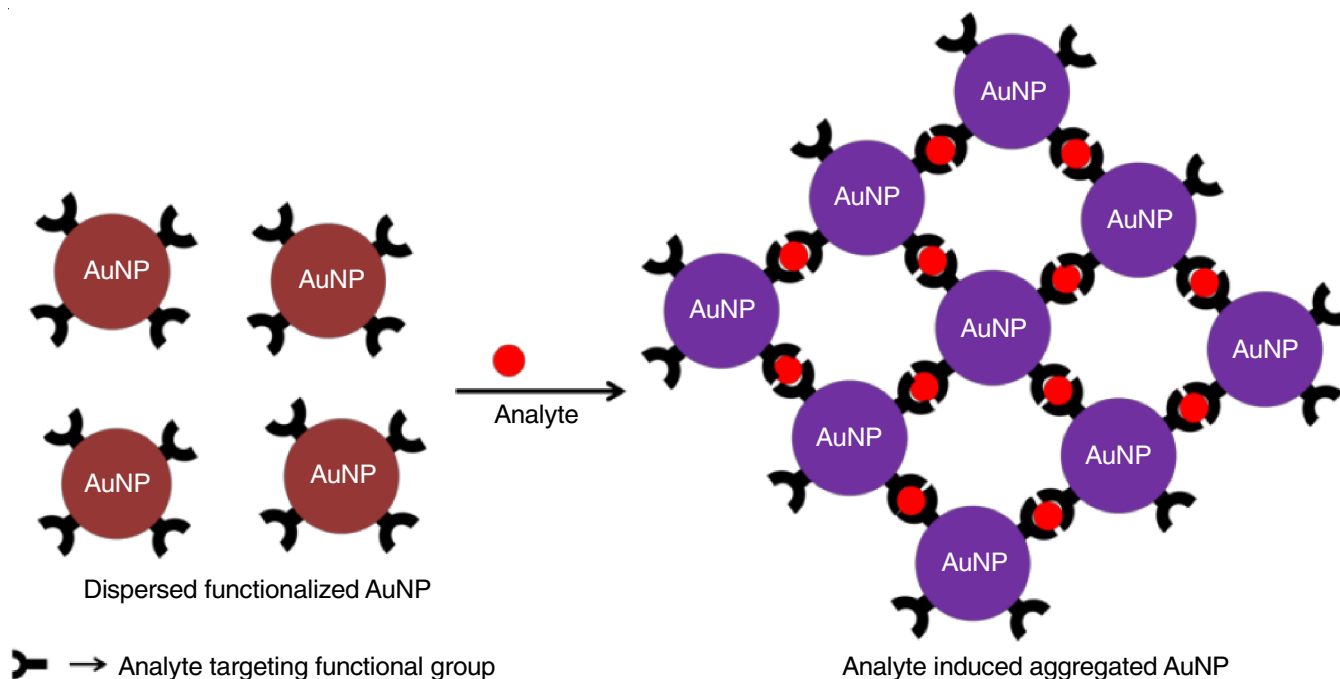


Fig. 1. Analyte induced aggregation of gold nanoparticle

#### (A) Au nanoparticles synthesis:

**I) Turkevich method:** The Turkevich method, first described in 1951 [18], is one of the most commonly used techniques for synthesizing spherical AuNPs in the size range of 10-20 nm. This method is based on the reduction of gold ions ( $\text{Au}^{3+}$ ) to gold atoms ( $\text{Au}^0$ ) in the presence of reducing agents such as citrate [18-20], amino acids, ascorbic acid or UV light [21-24]. To enhance stability, various capping or stabilizing agents are used to prevent aggregation. Initially, the Turkevich method was limited to a narrow size range of AuNPs. However, advancements in the original technique have enabled researchers to expand the achievable particle sizes. In 1973, Frens demonstrated that varying the ratio of reducing to stabilizing agents allows for the synthesis of AuNPs ranging from 16 nm to 147 nm. Further studies revealed the influence of factors such as pH, temperature and sodium citrate concentration, leading to the development of a particle growth model. These improvements have significantly broadened the applicability of the Turkevich method in nanoparticle synthesis.

**(II) Brust method:** The Brust method, first described in 1994, is a two-phase process for synthesizing AuNPs ranging from 1.5 nm to 5.2 nm in size [25]. This method, inspired by Faraday's two-phase system, uses organic solvents and allows for size control by varying the thiol-to-gold ratio. The process begins with the transfer of gold salt from an aqueous solution into an organic solvent, such as toluene, using a phase transfer agent like tetraoctylammonium bromide (TOAB). The gold ions are then reduced with sodium borohydride in the presence of an alkanethiols, which stabilizes the resulting AuNPs [26]. This reduction reaction causes a distinct colour change from orange to brown. Further purification of dodecanethiol stabilized AuNPs from TOAB was later reported by Schriffin [27]. The ability of Brust method to produce stable, monodisperse

AuNPs in organic solvents has made it widely used in various applications including biomedical research and nanomaterials engineering.

**(III) Miscellaneous methods:** Digestive ripening is an effective approach for converting polydispersed AuNPs into monodispersed ones using excessive ligands, known as digestive ripening agents [28]. The process involves heating a colloidal suspension at approximately 138 °C for 2 min, followed by further heating at 110 °C for 5 h in the presence of alkanethiols. Temperature plays a crucial role in controlling the size distribution of the resulting gold colloids. Various ligands, including thiols, amines, silanes and phosphines, have been used in the digestive ripening process [29-32]. Moreover, several alternative methods have been explored for synthesizing AuNPs. These include ultrasonic assisted synthesis [33-35], microwave assisted synthesis [36,37], laser ablation [38,39], solvothermal techniques [40] and electrochemical or photochemical reduction methods [41,42]. These techniques provide additional flexibility in tailoring AuNP properties for specific applications.

**(IV) Biological synthesis of AuNPs:** Traditional chemical methods for synthesizing AuNPs pose challenges, particularly regarding biocompatibility and environmental impact. Some chemical reagents used in synthesis can be toxic, making them unsuitable for the biological applications. To overcome these limitations, biological synthesis methods have been developed, offering eco-friendly, sustainable and reliable alternatives [43]. Microorganisms, such as bacteria and fungi, have proven to be effective in synthesizing AuNPs, either extracellularly or intracellularly. The negatively charged bacterial cell wall interacts electrostatically with positively charged  $\text{Au(III)}$  ions, facilitating AuNP formation. In intracellular synthesis, gold ions enter bacterial cells, where enzymes and biomolecules reduce

them to metallic AuNPs. In extracellular synthesis,  $\text{Au}^{3+}$  ions are trapped by bacterial membranes or surface reductase enzymes, leading to the formation of AuNPs outside the cell. Extracellular synthesis is generally preferred, as it simplifies the extraction process by eliminating the need to separate AuNPs from the intracellular matrix [44]. Microbial proteins, enzymes and organic compounds act as natural stabilizing agents, preventing nanoparticle aggregation. Furthermore, modifying growth parameters allows control over the size and shape of the nanoparticles. However, bacterial synthesis can be time-consuming and labor-intensive, limiting its scalability [43].

Fungi have also been widely explored for AuNPs synthesis due to their ability to secrete biomolecules such as enzymes and metabolites. Both unicellular and multicellular fungi can mediate intracellular or extracellular nanoparticle synthesis. Compared to bacteria, fungi produce a greater variety of proteins and reactive compounds, making the process faster and more efficient. However, genetically modifying fungi to optimize nanoparticle production remains a challenge [43-45]. Plant-mediated synthesis of AuNPs is a sustainable, cost-effective and rapid approach that utilizes naturally occurring plant biomolecules such as flavonoids, phytosterols and quinones as reducing and stabilizing agents [46]. All parts of a plant including roots, fruits and stems can be used for AuNPs synthesis. However, leaves are often preferred due to their high phenolic content, which accelerates the reduction process. Differences in biochemical composition among plant species and tissues can influence nanoparticle characteristics, including size and shape. Despite its efficiency, plant-based synthesis presents challenges in identifying the specific bioactive compounds responsible for the nanoparticle formation due to the complex nature of plant extracts [43]. Several species of algae have also

demonstrated the ability to synthesize AuNPs. Algal biomass contains hydroxyl and carbonyl groups, which act as natural reducing and capping agents [47]. While algal synthesis is relatively straightforward, the long cultivation period required for algae growth can make the process less practical for large-scale applications [43].

Various biomolecules, including amino acids, proteins, lipids and carbohydrates, have functional groups that facilitate AuNPs synthesis. For example, chitosan, which acts as both a reducing and stabilizing agent, similarly, starch, which, in an alkaline environment, decomposes into carboxyl groups. The hydroxyl (-OH) groups of carboxylic acids in starch reduce  $\text{Au}^{3+}$  ions to  $\text{Au}^0$ , leading to AuNP formation. Since different biomolecules exhibit varying reducing capacities, they must be carefully assessed before use in nanoparticle synthesis [43]. Biological synthesis methods effectively address the biosafety concerns associated with conventional chemical approaches. By utilizing microorganisms, plants, algae and biomolecules, researchers can achieve biocompatible and eco-friendly AuNPs production.

**(B) Surface modification of AuNPs:** As-synthesized AuNPs are capped by either weakly adsorbed ligands or hydrophobic ligands depending on their process of synthesizing AuNPs as discussed in the previous section. These are the limitations of AuNPs using in sensing based applications, however, researchers have modified the surface of AuNPs according to their need or interest in application. Therefore, ligands of the as-synthesized AuNPs are exchanged with various ligands for further sensing based applications (Table-1). Various ligands have been used to modify the AuNP surface most of them are thiols (-SH) based ligands with affinity molecules or groups on the other side of the ligands for designing AuNPs sensor for metal ions.

TABLE-1  
Au NANOMATERIALS BASED NANOPROBE FOR METAL ION DETECTION

Metal ions/ analyte	Functionalized molecules with Au nanomaterial (Au-NP)	Limit of detection (LOD)	Mode of sensing	Ref.
$\text{Cr}^{3+}$	Ingenious aspartic acid	0.6 nM	Naked eye/UV-vis	[48]
$\text{Cd}^{2+}$	Guanidine thiocyanate (GT)	10 nM	Naked eye/UV-vis	[49]
$\text{Cu}^{2+}$	Glycine-histidine dipeptide	0.04 $\mu\text{g/L}$	Naked eye/UV-vis	[50]
$\text{Cu}^{2+}$	Crown ether/carboxylic acid (L cystein)	150 nM	Colorimetric/UV-vis	[51]
$\text{Cr}^{3+}$	11-Mercaptoundecanoic acid (MUA)	0.1 nM	Naked eye/UV-vis	[52]
$\text{Cu}^{2+}$	Indigo-carmin (IC)	0.021 $\mu\text{M}$	Naked eye	[53]
$\text{Cr}^{3+}$	4-Mercaptobenzoic acid (MBA)	5 $\mu\text{M}$	Naked eye/UV-vis	[54]
$\text{Pb}^{2+}$	N-Decanoyltromethamine (NDTM)	10 $\mu\text{M}$ /0.35 $\mu\text{M}$	Naked eye/UV-vis	[55]
$\text{Mn}^{2+}$ , $\text{Pt}^{2+}$	Proteins of <i>P. stutzeri</i>	100 & 190 ppm	Naked eye/UV-vis	[56]
$\text{Hg}^{2+}$	Citric acid trisodium salt/ascorbic acid	0.1 nM	Naked eye & SERS	[57]
$\text{As}^{3+}$	Sucrose	4 $\mu\text{g/L}$	Naked eye/UV-vis	[58]
$\text{Ag}^+$	Thiamazole	0.042 nM	Naked eye/UV-vis	[59]
$\text{Al}^{3+}$ , $\text{Fe}^{3+}$ , $\text{Cr}^{3+}$	Diaminodiphenyl sulfone	0.62, 0.69 & 0.78 $\mu\text{M}$	Naked eye/UV-vis	[60]
$\text{Hg}^{2+}$	DNA-gold nanozyme-modified paper device	10 nM	Naked eye/UV-vis	[61]
$\text{As}^{3+}$	Glutathione (GT)	~ 0.11 ppb	Naked eye/UV-Vis & SERS	[62]
$\text{Ba}^{2+}$	2-Sulfanylethanesulfonate (MSA) and 11-mercaptoundecanoic acid (MUA)	1.25 $\mu\text{M}$	UV-vis	[63]
$\text{Fe}^{3+}$	Lactose/alginate	0.8 $\mu\text{M}$	Naked eye/UV-vis	[64]
$\text{Cd}^{2+}$	Aptamer	1.12 $\mu\text{g/L}$	Naked eye/UV-vis	[65]
$\text{Hg}^{2+}$	Ractopamine	0.30 $\mu\text{M}$	UV-vis	[66]
$\text{As}^{3+}$	Glutathione	0.12 ppb	Naked eye/UV-vis	[67]
$\text{Al}^{3+}$	Xylenol orange	12 ppb	Naked eye/UV-vis	[68]
$\text{Cu}^{2+}$	Methanobactin	3.26 nM	Naked eye/UV-vis	[69]



**AuNPs as metal ion detecting probe:** AuNPs-based sensors are widely used in various sensing applications due to their unique optical (*i.e.* localized surface plasmon resonance), electrical and chemical properties. AuNPs have a high surface-to-volume ratio, which enhances sensitivity by providing a large surface for analyte binding. Moreover, their chemical stability and biocompatibility make them suitable for biosensing and other sensing applications. AuNPs are primarily used in (i) colorimetric detection (aggregation-based mechanism); (ii) surface-enhanced Raman spectroscopy (SERS) where AuNPs amplify the Raman signals of molecules (analyte) bound to AuNPs surface through specific interaction, allowing for ultra-sensitive detection of analyte molecules; (iii) electrochemical sensors where AuNPs enhance the electrochemical response when used in sensors for detecting metal ions; and (iv) fluorescence-based detection.

AuNPs exhibit a distinct localized surface plasmon resonance (LSPR) effect, which gives them a characteristic red colour in their dispersed state. Metal ions can cause the AuNPs to aggregate due to the electrostatic or coordination or specific interaction with the surface ligands of the AuNPs. This aggregation results in a shift of the LSPR band from the visible (red) region to the near-infrared (blue or purple) region. The colour change is easily observable by the naked eye, making this method a simple and rapid technique for detecting metal ions. We have seen that most of the articles demonstrated that some targeting analyte specific molecules or ligand have been functionalized with the surface. As a result in presence of analyte molecules such functionalized gold nanoparticles get cross linked among themselves from the stable colloidal solution. Therefore, colour of the AuNPs changes from their dispersed state. In this way, the metal ions or analyte molecules have been detected through calorimetrically. Some reports have been discussed here.

Yang *et al.* [48] reported a one-pot synthesis of aspartic acid-functionalized gold nanoparticles (Asp-AuNPs) for colorimetric detection of  $\text{Cr}^{\text{III}}$  ions. Aspartic acid acts as both a reductant and stabilizer and hence no need for the additional surface modification of the nanoparticles. They prepared various ligands like aspartic acid, maleic acid, mercupto succinic acid and citrate stabilized AuNPs. Among them only aspartic acid stabilized AuNPs show efficient  $\text{Cr}^{3+}$  detection compared to other ligands stabilized AuNPs. The Asp-AuNPs exhibit stability and optical properties comparable to others and offer superior  $\text{Cr}^{3+}$  recognition. Detection is based on aggregation-induced colour change from wine red to blue-gray, with a detection limit of 0.6 nM. The method was successfully applied to environmental water samples.

Bhamore *et al.* [49] reported a simple, selective and sensitive optical method for  $\text{Cd}^{2+}$  detection in environmental water using guanidine thiocyanate (GT)-functionalized AuNPs based on the surface plasmon resonance properties of AuNPs. The surface of the prepared citrate stabilized AuNPs was modified using ligand exchange technique with guanidine thiocyanate, which controls AuNP size and has the ability to forms stable complex with  $\text{Cd}^{2+}$  using amine groups present in guanidine thiocyanate. In this system, the amine groups will coordinate

with  $\text{Cd}^{2+}$  while the thiol group interacts with AuNPs *via* Au-S bonding. In the presence of  $\text{Cd}^{2+}$ , the AuNPs solution undergoes aggregation, leading to a visible colour change. This method offers a linear detection range of 0.025-50  $\mu\text{M}$  with a 10 nM detection limit. This report demonstrated high selectivity and effective application in  $\text{Cd}^{2+}$  of environmental water samples.

Similarly, numerous research papers have been published on gold nanoparticles (AuNPs)-based detection of metal ions, showcasing its diverse strategies for improving sensitivity, selectivity and practical applications. These studies explore various functionalization techniques using polymers, ligands, biomolecules and surfactants [48-69] to enhance AuNP interactions with target metal ions and specific as well as selective detection of metal ions in experimental condition at the same time in environmental samples. All these reports have been summarized in Table-1 categorized in specific metal ions, targeting the specific molecules, limit of detection (LOD) and their mode of sensing techniques.

## Conclusion

This review highlights the synthetic process, mechanism and the advancements in the colorimetric detection of metal ions using gold nanoparticles (AuNPs), emphasizing their unique optical properties, ease of functionalization and high sensitivity. AuNPs serve as efficient colorimetric sensors due to their distinct surface plasmon resonance (SPR), which enables a visible colour change upon interaction with target metal ions. Various detection strategies, including aggregation-based methods, functionalized AuNPs and biomolecule-assisted sensing, have been explored to enhance selectivity and sensitivity for the specific metal ions. The advantages of using AuNPs as colorimetric sensor include its simplicity, cost-effectiveness and real-time monitoring capabilities, making it valuable materials for environmental monitoring, industrial applications and biomedical diagnostics. In conclusion, the colorimetric detection of metal ions using AuNPs is a promising, efficient and scalable approach that can significantly contribute to rapid and reliable metal ion analysis. In future, these sensors will become much more useful and efficient as nanotechnology and materials science continue to improve.

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## CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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