

A brief review on nanoparticle based mercury sensing by optical method

Swapnadip Roy and Swadesh Mandal*

Department of Chemistry, Bankura Sammilani College, Kenduadihi, Bankura – 722102, India

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The distribution of mercury and its compounds has an adverse effect to the living system. However, among all the mercury species organic mercury is the most hazardous to the living beings. In this review article we are trying to canvass the article from the last two decades on sensitive and cost effective method to detect and/or sensing trace amount of mercury using optically active nanomaterials by colorimetric, fluorimetric and surface enhanced Raman spectrometric method.

Key words: Mercury, sensing, colorimetric, fluorescence, nanomaterials

1. Introduction

The contamination of heavy metal ions are considered to be interesting zone from the last few decades [1]. Considering the position among the heavy metal, Hg stands with sixth most toxic chemicals in the list of hazardous compounds [2–6]. The contamination of environment is more affected by anthropogenic sources of Hg due to steady rate of expansion of industrial revolution.



Figure 1. Biogeochemical cycle of mercury

However, specific source of mercury deposition was quite difficult to identify [6]. In atmosphere, Hg^{2+} is stable one among the others. Natural and anthropogenic emission of mercury is in the form of elemental mercury (Hg^0) which has an atmospheric lifetime of several years (0.8–1.7 years) and photochemically transformed into Hg^{2+} and Hg_2^{2+} . Then inorganic mercury has been transferred to the earth surface by the rain and deposited in soil and water body. Due to methylation of inorganic mercury it is converted to

methylmercury by biotic or abiotic medium. The cycles of mercury in environment is undergoing with complex physicochemical reaction via land, air, and water [6]. As one of the systemic toxicants, mercury rank at the top of priority metals for the ability to induce multiple organ failures, even at nanomolar level of exposure. Biogeochemical cycle of mercury from atmosphere to aquatic system was presented by the Figure 1.

The Minamata disease has been opened a new door to the scientific field to think about the level of toxicity of mercury. Therefore, World Health Organization (WHO) and the U.S. Environmental Protection Agency (EPA) have declared that the maximum limit of Hg^{2+} in water should be 6 and $2 \mu gL^{-1}$ [7]. Therefore, it is necessary to determine Hg^{2+} at very low levels down to $nmolL^{-1}$ [8], and removal of mercury is also important to the analytical science. There are several kinds of technologies available which have already been developed to remove mercury, for example precipitation method, redox method, ion exchange method, filtration method, electrochemical treatment, reverse osmosis, and solvent extraction [9–16]. However, presently there are several nanomaterials based separation and extraction techniques are applied [17–20]. Nanoselenium sponge has been employed to detect and removal of mercury with LOD $0.2 ngL^{-1}$, which was far less than the acceptable limit of mercury in drinking water [21]. Oxidation reduction and physicochemical adsorption method have also been applied to removal of mercury using MoS_2 nanomaterials [22]. Modified $Fe_3O_4@SiO_2-SH$ sorbents exhibited high adsorption capacity for mercury and also it has good reusability with the maximum adsorption capacity was $148.8 mgg^{-1}$ at pH 6.5 [23], the same nanocomposites has been employed

to aqueous Hg^{2+} removal with a wide pH range of 1.0–8.0, and this method showed that at $\text{pH} > 3$ it has maximum efficiency with 132 mgg^{-1} [24]. Fe_3O_4 @polyaniline has also been applied to remove mercury [25]. Ag nanoparticles (Ag NPs) used as removal of mercury with 95% and remaining mercury was found 0.75 mgL^{-1} which was below the permissible limit prescribed by WHO [26]. DNA functionalized graphene oxide was used as synthetic receptor of mercury and this bio functionalized Fe_3O_4 nanomaterials has been applied to remove the mercury [27]. Magnetic self assembled zeolite was also used for the removal of mercury species [28].

The common analytical methods have been employed to detect mercury ion with sophisticated instrument, e.g., atomic absorption spectroscopy (AAS) [29], cold vapor atomic fluorescence spectrometry (CV-AFS) [30], inductively coupled plasma mass spectrometry (ICP-MS) [31], atomic fluorescent spectroscopy (AFS) [32], electrochemical methods, gas chromatography (GC) [33], and high-performance liquid chromatography (HPLC) [34]. However, these techniques are very costly and preconcentration is required sometime for quantitative detection. The determination of mercury by the sophisticated instrument was tabulated in Table 1. Therefore, it was necessary to develop a simple, sensitive and cost effective method to determine the presence of mercury and its compounds to the environmental sample.

Optical sensors have been introduced as an alternative method for the determination of mercury with high sensitivity and selectivity. The sensitive techniques are based on application of nanomaterials due to their cost-effectiveness, simplicity and rapidity. For specific nanomaterials with their unique optical properties give a new platform for the optical sensors, the optical properties of nanomaterials depends on chemical environment, size, shape, composition, functionalized materials play a lead role in emerging application in diverse area of pollutant sensing [49–52]. These optical sensors are categorically defined by colorimetric, fluorescence and surface-enhanced Raman scattering (SERS) sensors based on their origin of the optical signals and experimental strategy. The categories of optical sensor have been discussed in this review very briefly. Nanotechnology based colorimetric sensor of mercury have been discussed on Au NPs, Ag NPs, CNPs and other nanoparticles to the analysis of real samples. However, we have also touched the flavor of paper based spot sensing of mercury using the android application.

Table 1: Mercury Detection by Sophisticated Instrumental Techniques

Species	Instrument	Analytical Application	LOD	Ref
Hg^{2+} , CH_3Hg^+	AAS	Fish samples	0.35 ngmL^{-1} (Hg^{2+}) and 0.54 ngmL^{-1} (CH_3Hg^+)	[35]
Hg	CV-AAS	Road Dust sample	0.3 ngg^{-1}	[36]
CH_3Hg^+ , PhHg^+ , Hg^{2+}	ET-AAS	Water sample	$0.06 \text{ } \mu\text{g L}^{-1}$	[37]
Hg^{2+}	ICP-OES	Water sample	0.04 ng mL^{-1}	[38]
CH_3Hg	CE-ICP-MS	Water sample	0.084 pgmL^{-1}	[39]
Hg^{2+} , CH_3Hg^+	HPLC-ICP-MS	Environmental sample	0.0076 ngmL^{-1} (CH_3Hg^+) and 0.0014 ngmL^{-1} (Hg^{2+})	[40]
CH_3Hg^+ , $\text{C}_2\text{H}_5\text{Hg}^+$, In-Hg	HPLC-ICP-MS		0.78 ng L^{-1} (In-Hg), 0.63 ng L^{-1} (CH_3Hg^+) and 0.49 ng L^{-1} ($\text{C}_2\text{H}_5\text{Hg}^+$)	[41]
Hg^{2+}	HPLC-VWD	Water sample	$0.04 \text{ } \mu\text{g L}^{-1}$	[42]
CH_3Hg^+ , PhHg^+ , Hg^{2+}	HPLC-ICP-MS	Water, soil and rice sample	0.40 (Hg^{2+}), 0.49 (CH_3Hg^+) and 1.4 (PhHg^+) ng L^{-1}	[43]
Total Hg	CV-ICP-MS	Water sample	0.7 ng L^{-1}	[44]
Hg^{2+}	CV-AFS	Human saliva sample	2.5 ng L^{-1}	[45]
Hg^0	AFS	White vinigers sample	0.08 ng mL^{-1}	[46]
Hg^{2+}	UV-Vis	Water sample	$0.12 \text{ } \mu\text{gL}^{-1}$	[47]
Hg^{2+}	CV-AFS	Water sample	$0.02 \text{ } \mu\text{gL}^{-1}$	[48]

AAS - Atomic Absorption Spectroscopy

CV-AAS - Cold vapor-Atomic Absorption Spectroscopy

ET-AAS - Electrothermal-Atomic Absorption Spectroscopy

ICP-OES - Inductively Coupled Plasma-Optical Emission Spectroscopy

CE-ICP-OES - Capillary Electrophoresis—Inductively Coupled Plasma-Optical Emission Spectroscopy

HPLC - High Performance Liquid Chromatography

VWD - Variable Wavelength Ultraviolet Detector

ICP-MS - Inductively Coupled Plasma-Mass Spectroscopy

AFS - Atomic Fluorescence Spectroscopy

UV-Vis - Ultra Violet-Visible

LOD - Limit of Detection

2. Colorimetric sensors

Colorimetric assay is the simple analytical method in which detection can be made by even naked-eye due to colour change. The colorimetric method

has been extensively studied to the biological and chemical analysis using UV-Vis spectrophotometer. In this technique, generally Ag NPs, Au NPs, CNT, and their functionalized derivatives are widely used due to their optical property. The surface plasmon of these nanomaterials depends on their dielectric medium, size, and distance between the particle and this phenomena is the main basis for the colorimetric sensor, and with changing any parameters of the nanomaterials have an effect on the change of color formation of the solution. However, the colloidal stability and compatibility with biomolecule also enhances the sensitivity to colorimetric assay [50,54].

2.1 Au NPs based colorimetric assay

Among all the nanomaterials gold is widely used in colorimetric determination method due to its excellent optical, electrochemical and other properties [55]. The colorimetric sensor of Hg^{2+} as per literature has two division, in first category which is based on Hg^{2+} -induced aggregation of gold nanoparticles (Au NPs) and other category is based on the Hg^{2+} -inhibited aggregation of Au NPs. Hg^{2+} -inhibited aggregation has relatively more selective in nature and it also prevent false-positive results [56]. Whether, in aggregation inhibition, mercury plays a competitive role with the other chemicals which are responsible to the aggregation of Au NPs.

Recently android application has been used to detect the mercury ion the sample with the help of paper based colorimetric method using AuNPs functionalized with Papain and 2,6-pyridinedicarboxylic acid. The real-time android application has been observed by the color change within the concentration range 0.1 gL^{-1} to 0.001 mgL^{-1} of mercury [57]. Another paper based colorimetric detection has been employed with the smart phone, and the experiment showed the detection performance with 50 nM mercury into the pond water by utilizing thymine- Hg^{2+} -thymine coordination chemistry. The color variation was performed due to the different degrees of aggregation of AuNPs [58]. Another work where nanoenzyme based AuNPs (AuNZ-PAD) has been applied. Onsite determination of mercury was found 0.06 ng and 0.012 ng detection limit [59]. The disaggregation was studied on fabricated gold nanorods using L-cysteine through chemisorptions interaction. Addition of Hg^{2+} returned the color blue green from gray dark. This method successfully applied to determine of Hg^{2+} in aqueous solution between concentration range 0.5 to

250 μM Hg^{2+} [60]. Synthesized 3,5-Dimethyl-1-thiocarboxamidepyrazole (Pzl) was introduced to aggregate Au NPS which showed the color change from red to blue, in presence of Hg^{2+} color was remain same whether absence of mercury it formed blue which indicating the anti aggregation of Au NPS. The nanomolar level of mercury from water sample was determined by this method [61]. Hexadecyl trimethyl ammonium bromide (CTAB) also introduced color change from red to blue by the aggregation of citrate capped Au NPs. In presence of Hg^{2+} , inhibition of Au NPs aggregation was performed with Hg-Au alloy formation. In this method, the detection limit was found to be 11.9 nM and method was successfully applied to measure the mercury in tap water sample [62]. The anti aggregation was performed by thymine and mercury, mercury bonded with thymine and then Au NPs were dispersed in the solution [63]. Biothiols, like glutathione (GSH) and cysteine (cys) were included to anti aggregation of Au NPs to detect mercury colorimetrically, the detection limits of this assay for GSH, Cys, and Hcys are 17, 9, and 18 nM, respectively [64]. The synthesized Au NPs by poly(diallyldimethylammonium) chloride (PDDA) as reducer and stabilizer has been employed to determine mercury by anti aggregation. In presence of cysteine, gold aggregated and color has changed to ruby red to royal purple, addition of Hg^{2+} it formed complex with cysteine and color has been came back to purple to red. The detection limit was observed method was observed visually to with a detection limit of $2.5 \times 10^{-8} \text{ M}$ [65]. Colorimetric determination of mercury with ppt level was investigated by the Rex and his co-workers in 2006. The maximum blue shift absorption wavelength of the longitudinal mode (Figure 2) band of gold nanorods was observed at traces of Hg [66]. Chitosan functionalized AuNPs used as probe for the colorimetric detection, chitosan and mercury forms a complex through chelation and therefore aggregation was formed. The color change has been produced in this method and detection was carried out within the limit $1.35 \mu\text{M}$ [67]. Without an effective competitor in presence of mercaptophenylboronic acid (MPBA), Hg^{2+} inhibited the aggregation of AuNPs by the Au-S bond through self dehydration condensation of boronic acid group. Colorimetric study was performed through visible color change of the AuNPs solution from red to blue with detection limit 8 nM [68]. The mechanism of the detection of mercury was presented by the Figure 3a and the re-

sult of spectral study was shown in Figure 3b. Another way to synthesis Au NPs by borohydride in presence of 2-[3-(2-amino-ethylsulfanyl)-propylsulfanyl]-ethylamine (AEPE) and Triton X-100. The performance of this method was quite low level of 35 nM to water sample [69]. Thioglycolic acid functionalized gold nanoparticles (TGA-AuNPs) were carried out to detect 5 nM Hg²⁺ by naked-eye and tolerate 1000-fold foreign metal cations [70]. 2-mercapto succinic acid (MSA) was applied to cape Au NPs to determine mercury in water and vegetable sample; in this method mercury has also been separated out by Alumina-coated MSA-capped AuNPs easily and separated mercury measured by spectrophotometrically with nanolevel [71]. Dithiocarbamate derivative of calixarene, Au NPs was used as colorimetric sensor to detect mercury with limit detection limit of 40 ppb [72]. 5,10,15,20-tetrakis (4-carboxyl)-21H,23H-porphyrin-based MOF (Fe-TCPP-MOF) has been successfully applied to detect Hg²⁺ ion colorimetrically by catalytic method of methylene blue (MB) with detection limit of 103 pM [73].

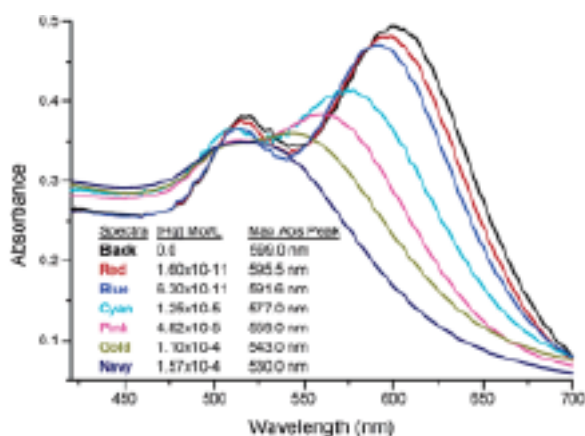


Figure 2. UV-visible absorption spectra showing the spectral shift at several Hg(II) concentrations. [Ref. 66]

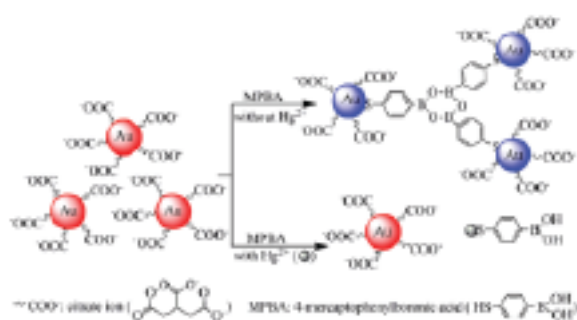


Figure 3a. Schematic presentation of the AuNPs colorimetric mechanism for Hg²⁺ [Ref. 68]

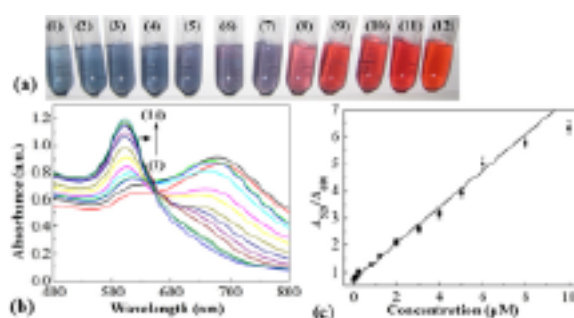


Figure 3b. (a) Photographic image, (b) UV-Vis absorbance spectra, (c) and linear plot of A₂₅₀/A₆₉₀ vs. Hg²⁺ concentration of the MPBA (10 µM)-AuNPs solution (pH 4.0) containing various concentration of Hg²⁺: (1) 0, (2) 0.01, (3) 0.05, (4) 0.1, (5) 0.2, (6) 0.8, (7) 1.2, (8) 2, (9) 3, (10) 4, (11) 5, (12) 6, (13) 8 and (14) 10 µM [Ref. 68]

2.2 Ag NPs based colorimetric assays

Paper based colorimetric determination of mercury detection with limit 10 µg L⁻¹ has been developed by Monisha et al., 2021 using smartphone and color detector app. AgNPs printed Wattman No. 1 filter paper has been used to determine the Hg²⁺ by changing the color [74]. The synthesis of PVP-AgNPs and its application to determine Hg²⁺ by smartphone was presented by Figure 4a. Figure 4b presenting the UV-Vis spectra with various metal ion and corresponding TEM images. Including colorimetric determination, Ismail et al., 2019 have also applied cellulose filter paper based silver nanoparticles to determine mercury. However, this technique was time dependent, but this method was applicable to detect the concentration below 50 µM with more than 1 hr time [75].

3-(Trimethoxysilyl) propyl methacrylate (TMPM) functionalized silver nanoparticles has been performed to detect mercury ion by colorimetric method, where stabilized Ag NPs was aggregated by the addition of Hg²⁺ ion which was clearly demonstrated by the color change of yellow to colorless and absorbance peak shifting. The detection limit was found to be 62.3 ± 1.9 nM which was further deep-rooted with atomic absorption spectrophotometric analysis by this group [76]. Hg²⁺ has been studied colorimetrically to performed with capping agent 1-dodecanethiol (C₁₂H₂₅SH) on morphological transition of AgNPs at room temperature with detection limit 3.3 nM [77]. Another study where colorimetric method was developed for the detection of Hg²⁺ ion using silver nanocrystals (Ag NCs) modified by Tris-base

(TB), again improvement was implemented by construction of self assembled monolayer Ag NCs film at n-hexane-water interface for ultrasensitive detection of Hg^{2+} [78].

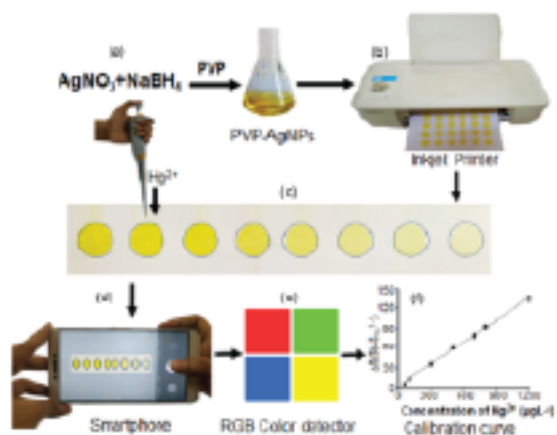


Figure 4a. Schematic diagram for synthesis of (a) PVP-AgNPs, (b) inject printing of PVP-AgNPs on paper substrate, (c) deposition of Hg^{2+} (500 $\mu g L^{-1}$) on printing paper with different concentration of Hg^{2+} by micropipette, (d) recording of image using smartphone, (e) measurement of color intensity by RGB color detector app and (f) standard calibration curve [Ref. 74]

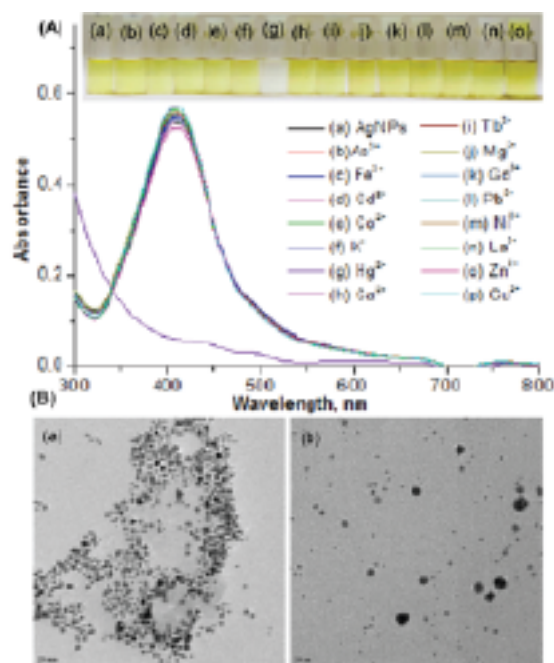


Figure 4b. (A) Glass vial containing PVP-AgNPs with different metal ions along with their UV-Vis spectra; (B) TEM image of (a) PVP-AgNPs and (b) PVP-AgNPs with Hg^{2+} (500 $\mu g L^{-1}$) [Ref. 74]

Colorimetric sensing method was carried out by gelatin functionalized AgNPs in three different phases including solution, hydrogel network and paper substrate. Aggregation was started after the incorporation of mercury ion which was determined by the color change yellow to colorless. This mechanism, therefore, applied to determine within the limit of 25 nM mercury sample [79]. Whereas, biologically synthesized AgNPs produced yellowish brown color due to high SPR which turned to colorless after addition of Hg^{2+} ion. The proposed method was successfully applied to the environmental sample with detection limit $2.2 \times 10^{-6} \text{ mol L}^{-1}$ [80]. Kokum fruit extract also used for biogenic synthesis of unmodified, monodispersed, anionic Ag NPs which was then selectively interacted with Hg^{2+} to form amalgamation through etching. Colorimetric detection estimated the detection limit was 6.2 ppb whereas memristive switching like property of Ag NPs showed 8.2 ppb detection limit [81]. In another greener approach to synthesis $Ag@AgCl$ -NPs has been proposed using *Chaetomorpha sp* green algae. The method was positioned with concentration dependent degradation of synthesized nanomaterials in presence of Hg^{2+} . The limit of detection was found to be 4.19 nM [82]. Orange (*Citrus sinensis*) peel extract was used to synthesis Ag NPs which was employed to visual colorimetric method to detect mercury and the detection limit was $1.24 \times 10^{-6} \text{ mol L}^{-1}$ [83]. Chlorophyll functionalized Ag NPs was synthesized to detect 2.7 μM and 60 μM mercury by the UV-vis spectrophotometer and the naked eye [84]. Unmodified AgNPs with average diameter size 8.3 nm showed the absorbance blue shift and decreased due to the presence of Hg^{2+} ion which was indicated oxidation of Ag^0 to Ag^+ . The detection limit was found in this method 0.06 ppm but when Cu^{2+} doped then the limit was found 0.008 ppm [85]. Again, a colorimetric method where Hg^{2+} reduced to Hg^0 by $NaBH_4$ and formed a core shell around the unmodified Ag NPs, in presence of lysine Ag NPs getting aggregation and color was changed to orange to yellow. The techniques has eligible to detect upto 1 nM [86]. Another method where displacement of calixarene moiety has been used for the mercury detection by spectrophotometry and amperometry, Hg^{2+} and Hg^0 selectively detected in aqueous phase and vapor phase by this method with detection limit 0.5 nM (0.1 ppb) and 10 nM (2 ppb) by UV-Vis and amperometry respectively [87]. The response of mercury on thiophene-functionalized calix AgNPs of reference

87 was represented by Figure 5, which was clearly advocating the change of intensity with the change of mercury concentration. Chitosan impregnated Ag NPs was synthesized by the solvent casting method to detect mercury through naked eye with detection limit 5.29 nM [88].

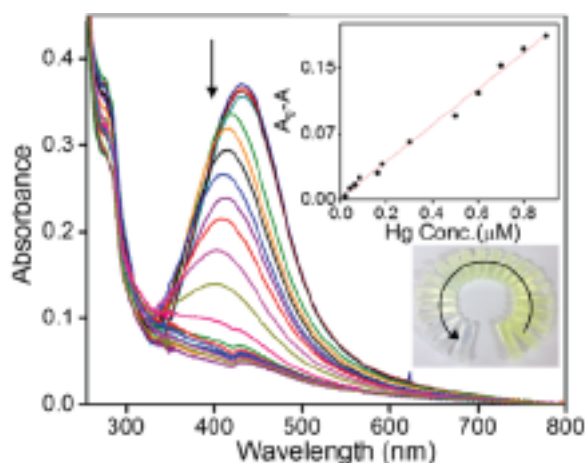


Figure 5. UV–vis spectral change for ThC-AgNPs (1 mL of stock solution) recorded after 2 m upon addition of incremental amount of Hg^{2+} (20–400 nM). (Inset: plot of the change in absorption intensity as a function of concentration of Hg^{2+} showing the linear behavior in the concentration range 20–900 nM (top) and the photographic image of the corresponding changes in color of ThC-AgNPs solution upon addition of Hg^{2+} in aqueous media (bottom))

2.3 CNPs based colorimetric assays

Carbon nanomaterials gathered a huge consideration because of their wide range of applications in daily life and specialized fields such as bioimaging, catalysis, sensors, and photoelectronics and whole slew. Carbon nanoparticles are also used for the colorimetric and fluorometric sensing method. Oxygen doped, nitrogen-rich, photoluminescent polymer carbon nanoribbon (ONPCR) fluorescent nanoprobe with superior water dispersibility, high-stability, and their individual optical properties were successfully synthesized for the Hg^{2+} and Ag^+ ion. In this process the detection limit of mercury was remarkably noticed by 0.68 nM [89]. After this nanoparticles era Carbon Dots are introduced commercially and these carbon dots are fabricated for dual channel ratiometric fluorometric determination of mercury. Carbon dots have two distinct fluorescence emissions at 500 and 678 nm at excitation at 400 nm. Green emission has reversibility within the

pH 7.0–12.0 and red emission had selective fluorescence to Hg^{2+} by formation of non emission complex with carbon dots and Hg^{2+} can be detected with limit 6.25 nM [90]. Carbon nanoparticle and rhodamine B has been applied to their dual emission at 437 and 575 nm respectively excited at 350 nm. The fluorescence was quenched by Hg^{2+} through effective electron or energy transfer process due to synergetic strong electrostatic interaction whereas that of the RhB remains constant. This method suggested astonishing detection limit of 43 nM [91]. Sulfur and nitrogen doped carbon nanoparticle synthesized to determine mercury by fluorescence quenching method with detection limit 0.05 nM [92]. Pamelopeel was employed to synthesize carbon nanoparticle which are further used as probe to determine mercury, in this method detection limit was found to be 0.23 nM [93]. Highly fluorescent carbon nanotube has been synthesized by the hydrothermal method from sodium citrate, which has high quantum yield and photostability. This technique established that 10 nM Hg^{2+} could be detected in aqueous solution [94].

Recently much attention has been paid on carbon nanotube based DNA hybrid fluorescent sensor. Thymine-thymine mismatch of duplex DNA has been enforced as an additive to enhance detectability of the biochemical sensors [95–96]. Zhang et al., 2010, studied SWCNTs and a fluorophore (6-carboxyfluorescein) labeled T-rich ss-DNA (single stranded DNA) to increase correctness of carbon nano tube based sensor. Hg^{2+} enhanced the fluorescence intensity and at the concentration greater equal to 8 μM plateaued was observed at equilibrium. The detection range for the DNA hybrid sensor is reported to be in the range 0.05–8.0 μM Hg^{2+} ions [95]. Candle light soot was applied to synthesize carbon nanoparticle which was employed to single strand DNA probe to detect mercury. Fluorescence quenching was performed due to carbon nanoparticle and T- Hg^{2+} -T induced hairpin structure did not adsorb on CNP and therefore fluorescence worked same way. A detection limit as low as 10 nM was achieved [97]. Colorimetric and fluorometric both methods were employed to detect mercury by the carbon nanodots which was synthesized by green technique. Both of the colorimetric and fluorescence probe shows excellent sensitivity and good selectivity over other transition metal ions for Hg^{2+} ions detection with detection limits of 5.7×10^{-10} mol L^{-1} and 5.5×10^{-10} mol L^{-1} , respectively [98]. Carbon nanodots as a new

generation of nanozymes, and cysteine as an anti-radical biomolecule has been used to colorimetric detection of Hg^{2+} . In this method hydrogen peroxide mediated oxidation of tetramethylbenzidine (TMB) into a blue colored cation radical was performed where cysteine was acting as on/off switch for the sensor. The limit of detection was found to be 23 nM for Hg^{2+} [99]. Figure 6 represented the Ref. 99 for the time profile and spectral profile at 260 s of color development in presence of varying concentration of Hg^{2+} .

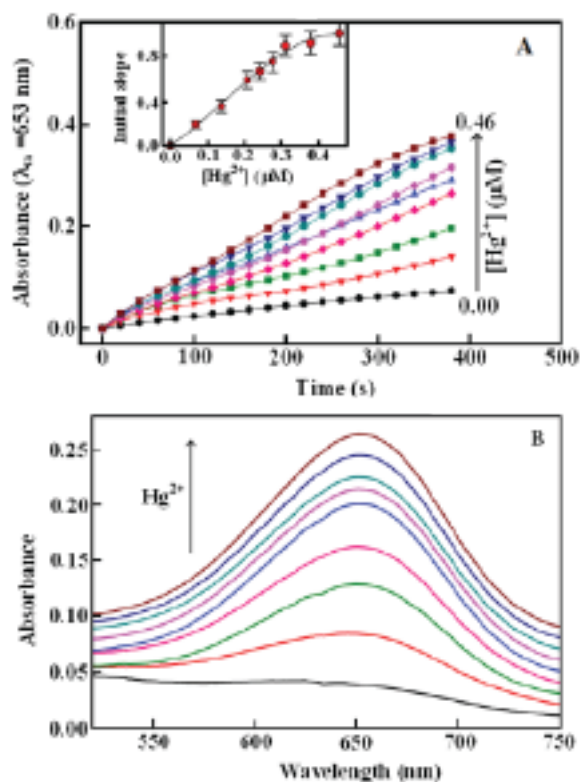


Figure 6. Time profile (A) and spectral profile at 260 s (B) of color development in the presence of varying concentrations of Hg^{2+} , followed spectrophotometrically. The reaction mixture contained TMB ($50 \mu\text{M}$), Cys ($5 \mu\text{M}$) and CDs ($80 \mu\text{g}/\text{mL}$) in sodium acetate buffer of pH 3.5 (0.1 M). The oxidation reaction was triggered by 1 mM of H_2O_2 . Inset of Fig. 6 shows the response of initial slope as a function of Hg^{2+} concentration [Ref. 99].

2.4 Others NPs based colorimetric assays

CuI was prepared by the dielectric barrier discharge on Cu wire and it has been applied to trace mercury detection by visual colorimetric method and this method has good agreement with cold vapor atomic fluorescence method. Colori-

metrically 1 ngmL^{-1} to $1 \mu\text{g mL}^{-1}$ concentration could be measured by color changing from white to reddish brown [100]. L-cysteine functionalized Cu NPs was applied to the colorimetric sensor for the determination of mercury with the detection limit of $4.3 \times 10^{-8} \text{ molL}^{-1}$ [101]. Pt NPs has also been used as paper based device to detect mercury colorimetrically, 3,3,5,5-tetramethylbenzidine (TMB) substrate has been preloaded to the detection zone of PCD and this was oxidized by the Pt NPs to form blue color. Addition of mercury to the PCD it reduced the blue coloration due to the interaction of mercury to the Pt NPs. In this method visually $0.01 \mu\text{M}$ mercury can be detected [102]. Mn_3O_4 NPs was used to detect mercury and cadmium colorimetrically with detection limit 3.8 and $2.4 \mu\text{g L}^{-1}$ respectively [103]. Zinc and cobalt grafted mesoporous silica nanoparticles (MSNs) was used to detect mercury through naked-eye based on ‘turn off’ and ‘turn on’ catalytic activity of MSNs. Chromogenic substrate (2,2'-azino-bis (3-ethylbenzothiazoline-6-sulfonic acid) diammonium salt (ABTS) was produced green colored cation radical of ABTS [104]. $\text{Fe}_3\text{O}_4@ZnO$ nanocomposite has been applied as peroxidase mimetic oxidation of tetramethylbenzidine (TMB) in the presence of H_2O_2 to form blue colored cation radical, in presence of cysteine the color has been turned back and after the addition of Hg^{2+} blue color was returned. Using this method mercury could be detected from environmental sample and water sample with detection limit of 23 nM [105]. $\text{ZnO}@ZnS$ core-shell nanoparticle as a paper sensor to detect Hg^{2+} ions in aqueous solution with detection limit as low as $10 \mu\text{M}$ [106].

3. Fluorescent Sensors

Due to $5d^{10} 6s^0$ electronic configuration Hg^{2+} do not have any intrinsic spectroscopic or magnetic behavior and it is also belongs to the so called ‘silent ions’. The fluorometric detection of such ions is forecasted in the use of small-molecule fluorescent sensors be contained in an ionophore-chromophore system. Fluorescence technique has been applied to determination of heavy metals like mercury due to its high spatial resolution, ultra sensitivity and selectivity, real-time detection, local observation, operational simplicity and ultimately no destructive in nature [107–108]. For the detection of analyte there are four categorically defined probes have been used like simple organic dye or molecular ligands, supramolecules, metal

organic framework and covalent organic framework, and nanoprobes as quantum dots, graphene oxides etc. [109].

The water soluble small molecular probes are interesting and important to detect mercury, Shu et al., reported a simple 4-hydroxynaphthalimide-derived fluorescent probe to detect both inorganic mercury and methyl mercury with detection limit 5.8 nM, the speciation study has been done by different rate of desulfurization of Hg^{2+} and CH_3Hg^+ [110]. The naphthalimide fluorophore have several advantageous physicochemical importances including a large Stokes' shift and a high quantum yield for which it become a popular fluorophore [109, 111–112]. Turn-on Hg^{2+} fluorescent probe has been reported with a naphthalimide group by Tian et al., 2020 with detection limit 4.0×10^{-8} M for Hg^{2+} . The method was work on the wide pH range and Hg^{2+} react with the 1,2 dithiolated group to produce aldehyde with green fluorescence [113]. Another method has been applied to detect Hg^{2+} in aqueous solution and living cells with the help of 4-amino-1,8-naphthalimide also with iminodiacetic acid and picolinic acid as receptors with detection limit 20.66 ppb for Hg^{2+} [114]. Morpholine moiety was introduced to the naphthalimide derivative as 'turn off' and 'turn on' fluorescence probe for Hg^{2+} and Cu^{2+} respectively. The detection limit of mercury has been found 6.11×10^{-8} M [115]. Li et al., proposed a naphthalimide method to detect mercury with 4.93×10^{-8} M detection limits. They have synthesized naphthalimide derivative with hydrophilic hexanoic acid group which was independent to the pH range of 4.0 to 8.0 and selectively determined Hg^{2+} [116]. Chen et al., 2019 recently reported a naphthalimide-based fluorescent and colorimetric probe, NAP-PS, in which diphenylphosphinothioyl group recognizes Hg^{2+} in analytical system. The ratiometric fluorescence response of NAP-PS is due to its reaction with mercury, in which the thiophosphinate P–O bond has been cleaved in the probe to form the intramolecular charge transfer (ICT) system, NAPO. The linear range of detection was within 0–12 nM, whilst the detection limit was calculated as 43 nM [117]. Rhodamine based fluorescence also used vigorously to detect mercury. Turn on detection techniques has been applied to detect ppb level mercury and fluoride with dual probe using rhodamine derivative with O-silyl protected mono- and dihydroxy benzaldehydes at pH 7.4. The mechanism was based on Hg^{2+} -induced reversible ring opening of the spiro-lactam with changing the color from colorless to

pink. The detection limit was observed 0.6 ppb and 0.9 ppb for F^- and Hg^{2+} respectively [118]. Another rhodamine based RDV fluorophore has been synthesized and applied to detect mercury with detection limit 136 nM. This method has been applied to water sample with 10 ppm detection limit without interference of other metals [119]. Rhodamine-rhodanine-based "turn-on" fluorescent sensor (RR1) was successfully applied to detect mercury in solution and living cells with the concentration as low as a 0.5 pM of Hg^{2+} [120]. In Ref. 120, Figure 7 showed that the addition of 1 equivalent Hg^{2+} the fluorescence intensity of RR1 increased by 26 fold, emission maximum shifted from 579 to 583 nm which was indicating the ring opening reaction was induced by the mercury. However, there are several publications on rhodamine based mercury sensor with very low detection limit and high sensitivity [121–125].

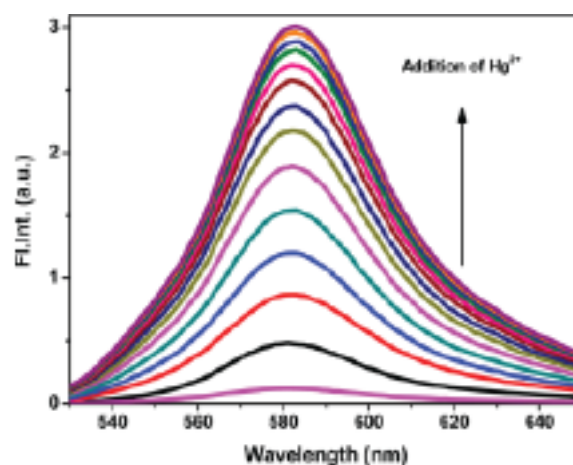


Figure 7. Fluorescence titration of RR1 ($10 \mu\text{M}$) with Hg^{2+} ($0\text{--}12 \mu\text{M}$) in the water-ACN ($60/40$ v/v) mixture. Excitation: 520 nm [Ref. 120]

The coumarins or benzo- α -pyrones is also important class of organic compounds which was employed several researchers to monitor the mercury species. Coumarin based N-[4-(2-Oxo-2H-chromen-3-yl)-thiazol-2-yl]-acrylamide (OCTAA) fluorescent monomer was successfully synthesized for determination of Hg^{2+} with detection limit $0.020 \mu\text{molL}^{-1}$ [126]. Turn on coumarin based ratiometric fluorescence probe has been synthesized, in which monoaza15-crown-5 moiety acting as an ionophore and 7-amino-4-methylcoumarin which functions as the chromophore of the system, and thiourea acting as linker to the system [127]. In another experiment, 8-(1,3-dithian-2-yl)-7-hydroxy-4-methylcoumarin (LS) has been

designed and synthesized by Ke et al., 2019. The sensor LS showed highly selective fluorescent sensing for Hg^{2+} with a low detection limit of 0.81 nM in the pH range from 6.15 to 9.96 in ethanol/water (1 : 1, v/v) solution [128]. However, there are also available fluorophores which are used to monitor the mercury using boron-dipyrromethene (BODIPY), schiff base, cinamaldehyde, anthracene, squarene, pyrene and azo dyes [129–137].

Supramolecular polymers are also came in the front to ascertain the presence of mercury, compared with other traditional polymers supramolecule have better recycling properties, however, it has owing to reversible monomer-to-polymer transition. Cheng et al., 2017 proclaimed the construction of a new supramolecular system through self-assembly of a thymine-substituted copillar[5]arene and a tetraphenylethylene (TPE) derivative in the presence of Hg^{2+} . Copillar[5]arene coordinated with Hg^{2+} tightly through T– Hg^{2+} –T pairings and the method prescribed the host-guest interaction [138]. Highly selective and sensitive chemosensor for sensing of mercury ion has been applied to the supramolecular self assembled mechanism by (E)-1-((5-(4-nitrophenyl)furan-2-yl)methylene) semicarbazone (BI) based on the 5-(4-nitrophenyl)-2-furan and semicarbazide groups [139].

Metal organic and covalent organic framework probe was also applicable for the detection of mercury. Ferrous metal-organic framework nanoparticle (Fe(II)-MOF-NPs) was prepared for the detection mercury using the colorimetric and photoluminescence of synthesized MOF. In this method Hg^{2+} can be detected within the limit of 1.17 and 1.14 nM in photoluminescence and colorimetric respectively [140]. Cadmium based organic framework has been applied to its dual emission mode by ratiometric fluorescence method. The method is unaffected in presence of other transition and heavy metals, and detection limit was found to be 2 nM [141]. Cu-MOF has also been applied to detect mercury with the detection limit 0.0633 nM by Differential Pulse Voltametry and Cyclic Voltametry [142]. The rare earth MOF has been developed by 2-aminoterephthalic acid with Sm metal ion. The fluorescence of Sm-MOF has been quenched by the Hg^{2+} ion, and method provided selectivity towards mercury and could be measured by 0.87 μM [143].

4. SERS sensors

SERS technique is also used because of its analytical sensitivity to detect the mercury using Raman

active materials, it has some advantage like, high enhancement factor (EF) up to 10^{6-9} ; ability to discriminate trace analyte; narrow and representative Raman fingerprinting spectra which enhance the detection ability of multiple target together; free from sample pretreatment; and able to monitor and detect even in ambient condition (e.g., oxygen, humidity, etc) [144–148]. Colorimetric and SERS dual mode sensing has been applied to detect Hg^{2+} based on rhodanine-stabilized gold nanobipyramids (Au NBs) by Qi et al., 2018 and schematically presented by Figure 8. Au and NBs were modified by rhodanine through gold-thiol (Au-S) affinity interactions. The sensing of mercury was monitored on the basis of red shift of longitudinal LSPR and the method showed the linear response for the concentration of Hg^{2+} from 5.0×10^{-7} to 6.0×10^{-5} M having the detection limit 2.0×10^{-7} M and measured by an absorption spectrometer [149]. The Sun et al., 2015, employed AuNPs decorated silicon nanowire array (SiNWA) as SERS substrates to perform the detection technique with the help of DNA assistance technology as T– Hg^{2+} –T. The detection limit has been suggested by this method for Hg^{2+} ions was 1 pM [144]. Ultrasensitive method for SERS detection of Hg^{2+} was developed based on the gold nanoparticles chain induced by T– Hg^{2+} –T base pairs, this method achieved the lower limit of detection of 0.45 pg mL^{-1} in the range of 0.001–0.5 ng mL^{-1} [150]. Graphene layer was structurally fabricated with triangular Au nanoarrays and Au nanoparticles like sandwich with uniform subnanometer gaps to detect Hg^{2+} in water through T– Hg^{2+} –T coordination. The detection limit was found to be 8.3×10^{-9} M [151]. Based on T– Hg^{2+} –T pairs formation and changing orientation of DNA on AuNPs shell surface has been developed quantitative detection of Hg^{2+} in aqueous solution. A wide range of concentration has been measured by this method from 1×10^{-8} to 1×10^{-3} M [152]. Again, SERS microprobe was applied for the detection of Hg^{2+} ions based on a single DNA-modified gold microshell with detection limit of 50 nM [153]. Single strand DNA has also been applied to detect mercury by SERS with the help of self assembled gold nanostar. In presence of the Hg^{2+} the electromagnetic field was increased due to the self assembly of gold nanostar. The detection limit was 0.8 pg mL^{-1} and its linear range is 0.002 to 1 ng mL^{-1} [154]. For sensitive detection and effective removal of Hg^{2+} and Ag^+ ions carried out by SERS-active platform by using the oligonucleotide-functionalized

magnetic silica sphere (MSS)@Au nanoparticles (NPs). This method was based on T-Hg-T and C-Ag-C to capture Hg^{2+} and Ag^+ ions which exhibiting excellent responses to Hg^{2+} ions in the range of 0.1–1000 nM and for Ag^+ in the range of 10–1000 nM [155].

SERS study was performed after the decorating of inner wall of capillary with 4,4'-dipyridyl (Dpy) functionalized silver nanoparticles (AgNPs). The SERS signal was decreased when Hg^{2+} coming to the surface to separate out Dpy. The detection limit of this method was detected as 0.1 ppb [156]. The schematic presentation of Ref. 156 was shown in Figure 9. Droplet based SERS method of water sample has been studied by Wang et al., 2009. The Hg^{2+} ion changes the SERS signal by interacting with AuNPs with reporter molecule as rhodamine B and this method suggested the range of detection between 100 and 500 ppt of Hg^{2+} [157]. In the direct determination of mercury was carried out by AgNPs using SERS technique with detection limit of 0.9 pM (18.2 ppt) Hg^{2+} [158].

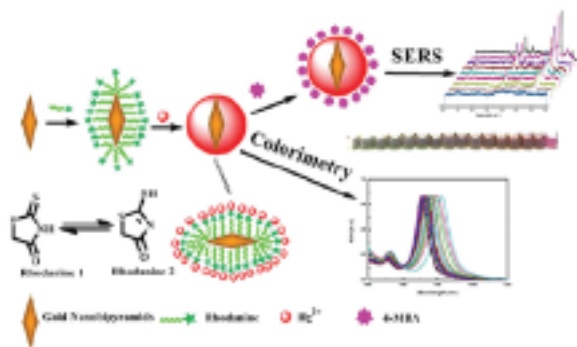


Figure 8. Schematic illustration of the formation of a partition layer on the surface of the Au NPs and the dual-modal sensing of Hg^{2+} [Ref. 149]

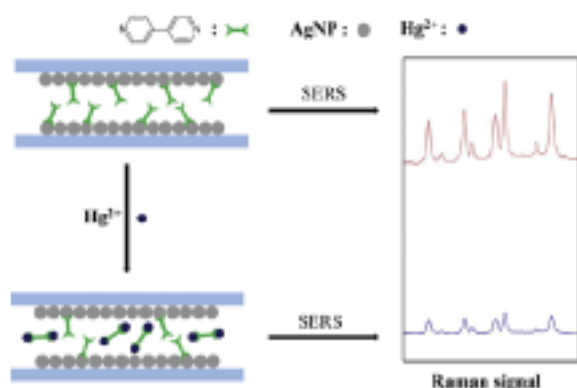


Figure 9. Schematic of the SERS sensing strategy for Hg^{2+} detection based on the prepared capillary sensor [Ref. 156]

5. Conclusion

This review is centralizing to describe the mechanisms for optical and visual detection of mercury from 2010 to till date by the optically active nanomaterials. The leading-edge activity increases the pollution to the environment and therefore the detection of heavy metal like mercury is necessary to detect selectively, sensitively and cost effectively, using the optical method like colorimetry, fluorimetry and visual detection along with SERS techniques. Paper based visual detection of mercury has also been discussed for the spot detection.

5.1 Future scope

It is considered that this review article will help to the researchers to do research on mercury detection and sensing by colorimetric and other optical method. However, keeping in our mind the simple, sensitive and cost effective optical method will contribute more effectively to develop the science and technology. Nanomaterial based optical sensor have a potential tool to determine the mercury in biological sample, food sample, water sample and other environmental sample. However, some nanomaterials have tendency to aggregate and lower self stability. Therefore, the researchers should have to pay more attention to synthesize stable optically active nanomaterials as well as to design for further modification of nanomaterials for the better result. It is also noted that android application is growing potentially day by day, and paper based sensing of mercury has high possibility in future to detect mercury easily with less than its toxic level.

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