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Review

"Gold rush" in modern science: Fabrication strategies and typical advanced applications of gold nanoparticles in sensing

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ABSTRACT

Gold nanoparticles (AuNPs), the huge potential functional nanoparticles in scientific research, have attained tremendous interest for their unique physical–optical property since they have been discovered. They have been very popularly used as labels in diagnostics, sensors, etc. The use of AuNPs in sensor fabrication is widespread, and so many papers have been reported over the past years. The development of AuNPs used in sensor has been reviewed in some previous papers. Most of them tend to review a certain kind of analyte using one kind of technique. However, few of these reviews include the detection of inorganic and organic contaminant, as well as biomolecules at the same time. Besides, the development for AuNPs application is very fast in modern science. Therefore, in this review we summarize some recent progresses made in the field of AuNPs research. We summarize the typical synthetic strategies of AuNPs, classify the mechanism analysis of AuNPs-based sensors, and expound the role of AuNPs in these sensors. Typical advanced examples of the newly developed AuNPs-based colorimetric and electrochemical sensors and their applications in detection of various analytes, including biomolecules, metal ions, and organic environmental hormones are presented and discussed.

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1. Introduction

Recently, nanomaterials are emerging as revolutionizing materials of research with multiple disciplines of science like physics, chemistry, materials, medical science, and biology [1–8]. Gold nanoparticles (AuNPs), one kind of promising functional nanomaterial, have gained increasing scientific interests since the potable gold was found and believed to have excellent medicinal properties by Paracelsus in 4th century A.D [9,10]. However, the real scientific research on gold sol did not start until the gold hydrosols have been discovered by Michael Faraday in 1857 [11]. Later, people showed great interest in investigating the color, shape, size, synthetic strategies, optical property, and applications of AuNPs [12–15]. It can be viewed as a "gold rush" in modern science.

AuNPs can be the remarkable scaffold for novel biological and chemical sensors owing to the fascinating electrical, chemical, optical, and catalytic properties like easy synthesis, high surface-to-volume ratio, high extinction coefficients, strong distance dependent optical features, and excellent biocompatibility, as well as the characteristic of suitable surface functionalization [16–20]. Number of papers concerning the keywords "gold nanoparticles + detection" on indexed journals are increasing year by year and account for approximately one-fifth of all "gold nanoparticles" in the past twenty years (Fig. 1). Many excellent reviews have been published on the use of AuNPs as sensor. For example, Kim et al. reported a review about the fluorescent and colorimetric sensors for detection of three heavy metal ions and mentioned some typical sensors based on AuNPs [21]. Pingarron et al. reported a paper concerning AuNPs-based electrochemical biosensors, which discussed the application of AuNPs-based electrochemical enzyme biosensors, immunosensors, and DNA biosensors [22]. Recently, some papers reviewed the application of AuNPs on metal ions, anions, small molecules, proteins, and nucleic acids using various techniques [23–28]. However, most of these reviews tend to describe a certain kind of material or use one kind of technique. Few of them describe the inorganic and organic contaminants, as well as biomolecules at the same time. In addition, the new review of AuNPs is very necessary for its rapid development in recent years.

As the development of nanotechnology, more and more papers focus their attention on detecting different kinds of substances simultaneously, as well as using not only one analytical technique [29,30]. In addition, the development for the application of AuNPs is very fast and the reports of AuNPs-based sensors are increasing enormously [31]. Therefore, this review presents a brief overview on the recent advances of AuNPs in relation to the fabricating strategies and typical advanced applications on the development of chemo- and bio-sensors for metal ions, anions, biomolecules, and organic environmental hormones using colorimetric and electrochemical techniques. Because of the explosion of papers published in this field, we could not mention all of the published reports, but rather a description and summary of these methods are discussed. On the other hand, the application of AuNPs-based sensor on colorimetric detection is a relative independent part, which uses the optical properties of collective oscillation of electrons on surfaces for AuNPs (known as the "surface plasmon resonance", SPR or "localized surface plasmon resonance", LSPR) and their color change [32–34]. The typical application using the properties of good catalysis, huge specific surface area, and wonderful electron transfer is located in electrochemical sensor [35,36]. Other applications of AuNPs-based sensors such as fluorescence sensor and micro-electronic balance sensor are not mentioned here.

Thus, the purpose of this review is to investigate the synthetic strategies for AuNPs and to derive some general strategies with the basic understanding of sensing application, to summarize the results of their basic properties in a vast variety of applications on chemical, environmental, and medical science, and finally, also

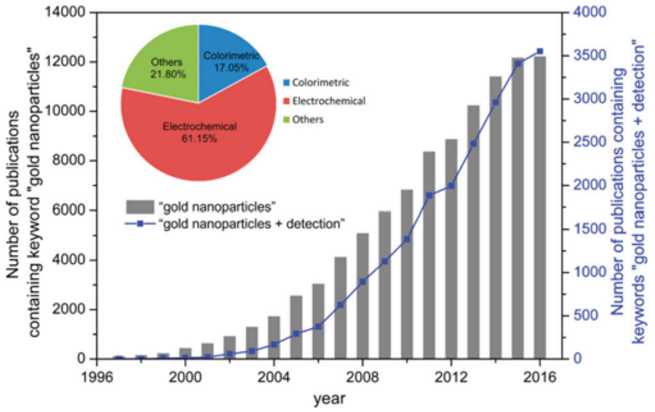

Fig. 1. Evolution of the number of publications concerning the keywords "gold nanoparticles" and "gold nanoparticles + detection" on indexed journals between 1997 and 2016. The insert pie graph exhibits the percentage of the available scientific reports which concerned the AuNPs-based sensors using different techniques from 1997 to 2016. The data comes from the research on "web of science".

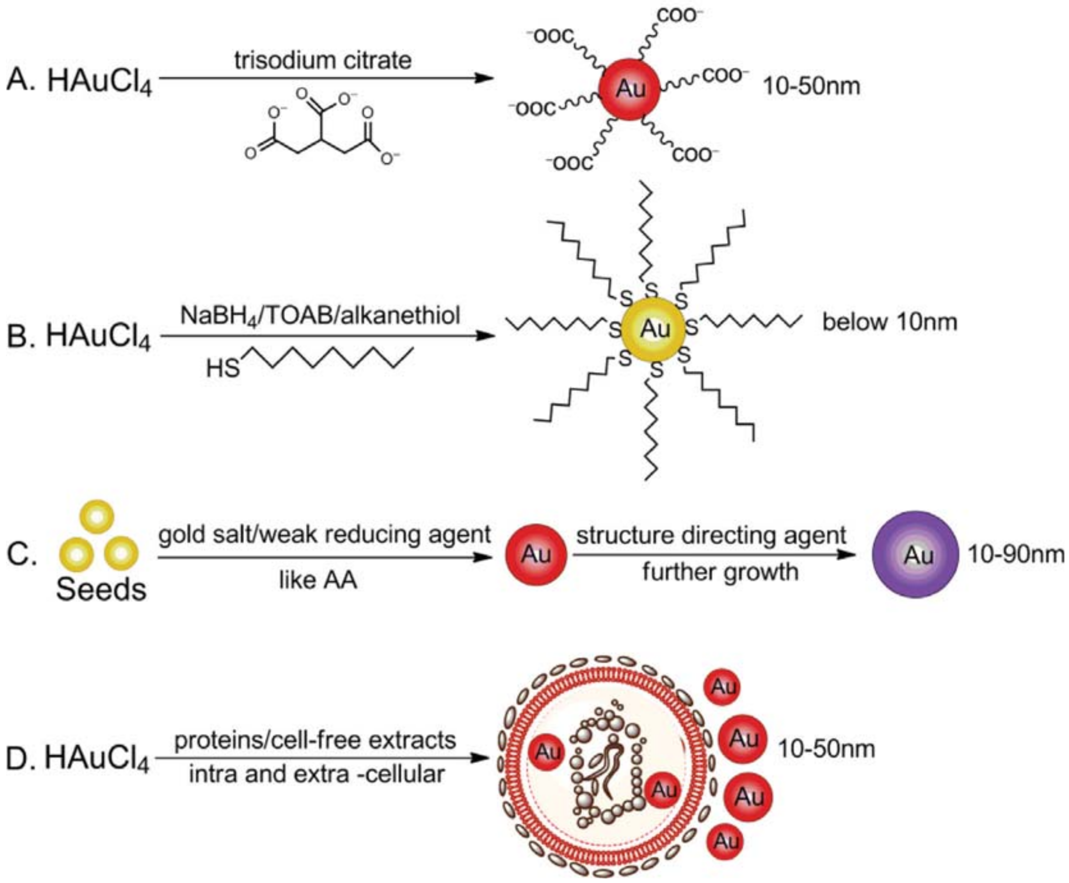

Fig. 2. (A) Turkevich–Frens method for synthesis of AuNPs by reduction of gold salts in the presence of trisodium citrate; (B) Brust–Schiffrin strategy for two-phase fabrication of small-size AuNPs by reduction of gold salts in the presence of external thiol ligands; (C) seed-mediated growth method for AuNPs synthesis by the process of nucleation, growth, and stability; (D) green synthesis of non-toxic AuNPs through intra- and extra-cellular biosynthesis in the presence of proteins or cell-free extracts.

the ultimate purpose, to open up the new insights via multi-approaches to many promising applications from synthetic routes and theoretical perspectives. From this review, the readers will understand the significant role of AuNPs in modern science and can see this highly rewarding and inherently fascinating field of investigation for advanced materials. We hope that readers can gain some inspiration from this review and push further development of AuNPs application. We believe that the outlook of AuNPs-based materials is bright in the present situation of research.

2. Strategies to fabricate gold nanoparticles

Following the discovery of AuNPs, the research has been inundated with reports on relations among the parameters, including diameter, absorption maximum, and concentration of reactants and strategies for fabrication [37–39]. In this case, numerous methods, such as reduction, photochemical reduction, seed-growth, for the fabrication of AuNPs have been reported [40,41]. Conventional approaches, including Turkevich–Frens (Fig. 2A) and Brust–Schiffrin (Fig. 2B) method, are still the most popular methods. Also, some new methods such as seed-mediated growth method (Fig. 2C) and green synthesis (Fig. 2D) have been developed. In addition, considerable researchers have paid their attention to the fabrication of AuNPs with controllable size, conditions, stability, and applications [42]. In this part, we will introduce some regular and new methods and review the development of that, as well as the advantages or disadvantages.

2.1. Turkevich–Frens method

The Turkevich–Frens method is also the called sodium citrate reduction method or classical-citrate method. It was the first and comparatively systematic method to fabricate AuNPs which was described by Turkevich in 1951 [43]. It was developed based on many reported studies (Table S1). This method allows sodium citrate add into the boiling chloroauric acid solution with thoroughly stirring, which leads to a series of color change from light yellow to purple or black and finally to ruby red. The main principle is that the gold ions (Au^{3+}) of chloroauric acid were reduced to gold atoms (Au^0) by the reducing and capping agents such as citrate [44]. There were evidences demonstrated that the mechanism for stepwise formation of AuNPs was based on three stages: nucleation, growth, and possible agglomeration [45]. However, this approach was partly restricted by narrow range of synthesis size and many studies were committed to solving this weakness. Excitingly, Frens fixed this problem by changing the ratio of sodium citrate and gold chloride and achieved a wide range from 16 to 147 nm [46]. After that, the Turkevich–Frens method was formed, and widely applied to biology, medicine, chemistry, and environment. Later, some researches introduced several new methods for further development. It can be divided into three channels: (1) vary the size and stability by controlling the pH of the reducing agent or the whole mixed reagents; (2) simplify the steps of preparation via changing the reducing agent or adding another stabilizing agent; (3) other new methods [47]. Shiba et al. reported a size control method of monodisperse AuNPs through the citrate reduction process related to a pH-shifting procedure. This report proposed an approach for different mean sizes of AuNPs ranging from 6 to 15 nm by changing the interval time between the addition of NaOH solutions and citric acid into the boiling chloroauric acid [48]. This procedure induced a pH shifting and was presented without any growth modifiers. Interestingly, Schulz et al. proposed a protocol of controlling the pH by adding a citrate buffer instead of sodium citrate solution as the stabilizer or reducing agent [49]. Recently, a paper introduced a facile approach with the size around 5 nm encapsulated with citrate and polythiophene sulfonate poly [2-(3-thienyl)ethyloxy-4-butylsulfonate]sodium salt, which both helped to stabilize the AuNPs [50]. Furthermore, the stability of AuNPs at high chloroauric acid concentrations can also be enhanced by reversing the order of addition for the citrate solution and chloroauric acid [51].

In a word, these methods described above are all developed on the basis of the Turkevich–Frens method and promote the further development. Afterward, the Turkevich–Frens method can prepare AuNPs with the wide diameters ranging from 5 to 250 nm [52–54]. In general, this method is simple since the size can be controlled by altering the molar ratio of $HAuCl_4$ and sodium citrate [55]. In addition, the citrate ions stabilizer can be easily exchanged by other ligands with high affinity, especially thiols, which allows for widespread application in sensors. However, there are still some drawbacks needing to overcome: (1) the size of relatively stable AuNPs is between 10 and 50 nm, which will aggregate and be nonspherical when go beyond this range; (2) the degree of stability for AuNPs largely depends on the stabilizer and is susceptible to environmentally induced aggregation, such as pH, ion strength, and salt concentration, which render them not ideal structures for further investigations on cells; (3) whether the resulting AuNPs are poisonous or not is closely related to the stabilizer, which may be harmful to the environment. Although it has something unsatisfactory, the Turkevich–Frens method is still one of the most common ways to fabricate AuNPs until now.

2.2. Brust–Schiffrin method

AuNPs can be synthesized by Turkevich–Frens method in aqueous phase. It can be also generated in organic phase or a two-phase liquid–liquid system, which was reported by Brust and Schiffrin in 1994 and called the Brust–Schiffrin method [56]. This method was inspired from the two phase system of Faraday and achieved a significant breakthrough in the realm of AuNPs fabrication since the size ranging from 2 to 5 nm of AuNPs can be obtained, which can remedy one of the defects of Turkevich–Frens method [53]. In this procedure, a two-phase (water–toluene) system was designed to reduce the $AuCl_4^-$ by sodium borohydride ($NaBH_4$) to Au^0 (AuNPs) in the presence of an alkanethiol. The tetraoctylammonium bromide (TOAB) was used as the phase-transfer reagent. $AuCl_4^-$ was transferred from aqueous-phase to toluene-phase and reduced by $NaBH_4$. When $NaBH_4$ added, the color of organic phase shifted from orange to deep brown rapidly. The specific preparing process of this technique was described by Brust and Schiffrin clearly. The prepared AuNPs are capped by densely packed monolayers and quite stable. Moreover, the size of AuNPs can be tuned by the reaction conditions, like temperature, thiol/gold ratio, and reaction rate [57]. Maye and Zhong found that the temperature determined noticeably change in size of AuNPs and the chain length could influence the reactivity of AuNPs [58]. Teranishi et al. also used the Brust–Schiffrin method to prepare AuNPs with TOAB as a phase transfer agent and an alkanethiol as a capping agent [14]. To control the size, AuNPs were further aged in TOAB, which was melted at 150–250 °C. Besides, owing to the nucleation–growth-passiva tion kinetics model, larger mole ratio of S/Au can lead to smaller average core size. Furthermore, fast addition of $NaBH_4$ and immediate quenching of solutions give smaller, more monodispersed AuNPs [59–61]. Compared with other methods, Brust and Schiffrin method has some apparent advantages: (1) it introduces a simple way to fabricate the surface-functionalized AuNPs directly; (2) it can be repeatedly isolated and dispersed with monolayer in organic solvents without irreversible aggregation; (3) the prepared AuNPs are soluble in non-polar solvents and can be easily purified by gel-filtration chromatography, which means they can be handled like a simple chemical compound without any apparent changes in properties; (4) the size of AuNPs can be adjusted by various conditions not only the thiol/gold ratio.

Due to the above advantages, Brust–Schiffrin method is often used for the synthesis and application of thiol-ligand AuNPs [62,63]. Recently, some thiolate-stabilized AuNPs are fabricated via a modified Brust–Schiffrin method. Wieckowska and Dzwonek introduced an ultrasmall AuNPs preparing method functionalized with hexanethiol and anthraquinone/hexanethiol monolayer by a modified Brust–Schiffrin method [64]. Other thiolate ligands such as ω-functionalized alkylthiolate [65,66], arenethiolate [67,68], bifunctional alkanethiolate [69], and others have been used to fabricate functionalized AuNPs.

2.3. Seed-mediated growth method

Size control of AuNPs has been investigated for ages. As for the two methods referred above, they are limited with relatively narrow size distribution or fast growth to some extent. Seed-mediated growth method, also called as step-by-step seed-mediated growth approach, is another common way to control the size of AuNPs. It can be traced back to 1989 when Wiesner et al. [70] added $HAuCl_4$ solution to the gold seeds produced by $HAuCl_4$ reduction with phosphorus, which was similar with Faraday's original colloidal synthesis [71]. In this approach, small size of AuNPs are prepared via reducing gold salts with strong reducing agent such as $NaBH_4$ or citrate first and then used as seeds for the fabrication of larger size AuNPs by adding a solution of gold salt in the presence of weak reducing agent such as ascorbic acid (AA). Finally, a structure directing agent is added to prevent further nucleation and promote the growth of AuNPs [72,73]. The physical properties of AuNPs were improved when compared with other approaches. However, this synthetic procedure was still unsatisfactory for the small population of impurities. Subsequently, Murphy improved this approach using AA as reducing regent instead of citrate in the growth solution and hexadecyltrimethylammonium bromide (CTAB) as stabilizer to fabricate uniform AuNPs up to 40 nm [74]. Later, Han group used the same protocol to synthesize AuNPs with the controlled size (10–90 nm) [75]. Recently, some researches tend to study the reaction conditions, which can control the size of prepared AuNPs. Piella et al. introduced a precise control over the size between 3.5 and 10 nm by tuning the temperature, pH, gold precursor to seed, and concentration of sodium citrate [76]. Compared with other methods, the seed-mediated growth method inhibits any secondary nucleation via providing a controlled number of seeds and growth condition. Although the seed-mediated growth method provides a broad size distribution, a work reported by Jana et al. demonstrated that the presence of seeds often led to further nucleation, rather than growth. To avoid additional nucleation, a slow addition of reducing agent step was needed [77]. In this regard, Jana et al. further reported a step by step seed-mediated growth method using the growth solution and AA, which provided the diameter size of AuNPs ranging from 5 to 40 nm with narrow size distribution [74]. As a popular technique for AuNPs synthesis, the seed-mediated growth method amplifies the size step-by-step. The shape and size can be easier to control. Therefore, this route is broadly used in the shape- and size-controlled AuNPs fabrication. However, there are some points limiting the application of such approach: (1) it is difficult to find a suitable growth condition which can inhibit the additional nucleation during the growth stage; (2) the factors which influence the process of procedure are many, such as seed concentration, reducing agents, and rate of reducing agent addition.

2.4. Green synthesis

Although the three methods mentioned above can efficiently produce AuNPs, a main shortcoming of these approaches is that the toxic byproducts may induce environmental consequences during large scale generation. Besides, the use of toxic solvents and chemicals can be problematic for biological application [78]. Recently, in response to these concerns, green synthesis methods using green materials such as prokaryotic organisms (bacteria), eukaryotic organisms (fungi and extract of plants), and others (proteins and amino acid) [79,80] and assisted techniques like photochemistry, supersonic, or microwave [81–83] to synthesize AuNPs have been developed.

The way of AuNPs fabrication using prokaryotic organisms such as algae and bacteria is mainly divided in two parts: the intracellular biosynthesis and extra-cellular biosynthesis. Konishi et al. obtained the crystalline AuNPs with particle range of 10–20 nm via using the mesophilic anaerobic bacterium, *Shewanella algae* [84]. The crystalline AuNPs presented in the periplasmic space and the intracellular recovery of that was attractive as an eco-friendly alternative. The papers reported by Husseiny et al. [85] and Singaravelu et al. [86] both introduced the extracellular synthesis of monodisperse AuNPs using the prokaryotic organisms of *Pseudomonas aeruginosa* and marine alga, respectively. The sizes of AuNPs were in the range of 15–30 nm and 8–12 nm, respectively. Another synthetic procedure is proposed by using the *Escherichia coli* K12 cells without any addition of growth media, pH changes or stabilizing agents/electron donors [87]. The way of intra- or extra-cellular biosynthesis for AuNPs are both eco-friendly and practical. But a recent paper showed that the production of AuNPs from biomass required longer time than the production of AuNPs from the cell-free extracts [88].

So far, a large number of fungal species like *Neurospora crassai*, *Aspergillus foetidus*, *Fusarium semitectum*, and *Aspergillus oryzae* have been reported to fabricate AuNPs through intra- or extra-cellular manners successfully [80]. In 2001, Mukherjee et al. introduced a green chemistry approach that used the fungus, *Verticillium* sp. (AAT-TS-4), to reduce $AuCl_4^-$ ions and form AuNPs with the diameter around 20 nm [89]. For all we know, it is the first time that a eukaryotic organism was used to synthesize AuNPs. From now on, many researchers stated to studying the green method for fabricating AuNPs using eukaryotic organisms [90] such as plant leaves [91]. Many studies developed by using the extract of plant leaves, such as *Cinnamomum camphora* [92], *Tamarind* [93], and *Black tea* [92] to fabricate different sizes of AuNPs. These methods are often environment-friendly and have wide application in biosensors, photonics, and others. Admirably, some of them are not devoted to the synthesis of AuNPs, but to the toxicity and practical application of prepared AuNPs using fungi reduction. For example, Roy et al. studied the extracellular biosynthesis of AuNPs using *Aspergillus foetidus*. The cell cytotoxicity was less toxic compared with that of the normal gold salt solution on A549 cell [94]. Nune et al. obtained the tea-generated AuNPs (T-AuNPs) by using the phytochemicals in tea. T-AuNPs showed significant affinity toward breast (MCF-7) and prostate (PC-3) cancer cells, which paved the way for researching on molecular imaging and therapy with such AuNPs [95]. In general, the synthesis of AuNPs with fungi exhibits more environmental tolerance since fungi are more tolerant to higher metal concentration and can secrete amount of extracellular redox proteins to reduce metal ions.

Except for those two approaches mentioned above, some other new green methods using some assistants e.g., dextrose [96], proteins [97], or amino acids [98] have been shown up, as well as some assistive techniques. Photochemistry is a green synthesis method, which can enhance the quality of AuNPs due to the reproducibility, simplicity, and mild synthesis conditions [99,100]. The source of light is usually sunlight or ultraviolet light [101,102]. The light irradiation makes the system produce radicals function as the reducing agent that are capable of reducing Au^{3+} to Au^0 and form AuNPs [103,104]. The whole process occurs in only seconds to minutes, the reaction time is greatly reduced. Actually, this is an energy transfer process, in which the energy of light transfers to the electronic energy. Similar with this energy conversion method, ultrasound- and microwave-assisted methods are well introduced [105,106]. The electromagnetic radiation is converted into heat energy with the microwave-assisted methods [107]. The process is rapid and do not show any cytotoxicity toward practical application, creating a new field for synthesis of eco-friendly AuNPs [108,109]. Although the energy conversion methods do not need chemical reductant, it needs to introduce energy and suffers from the limitations of shape and application in some extent. Moreover, synthesizing in organism and lacking of good mechanical/regular stirring for green methods usually make them irregularity. In spite of this, to date the development of green synthesis method for AuNPs is still needed and plays an important role in biosensors.

3. Colorimetric application of AuNPs in sensing

Colorimetric application of AuNPs in sensing is one of the most promising analytical methods which provides a rapid, simple, and inexpensive way for determination of analytes [110]. The main mechanism of this method lies in the interparticle distances and interparticle surface plasmon coupling, which are very sensitive to the local medium. When the interparticle distances are less than approximately the average particle diameter of AuNPs, the color will change from red to blue, which can be easily observed by the naked eyes [111,112]. Furthermore, the readout can be recorded using a UV–vis spectrophotometer. The channel applied to sensors can be divided into two strategies, the red shift and blue shift in the absorption, leading to aggregation and disaggregation of AuNPs, which present as the color changes from red to blue and blue to red, respectively. Furthermore, when pH or ionic strength of the AuNPs surface changed, the process of aggregation and redispersion can lead to a circle. Owing to the advantages of easy to observe and record, the colorimetric sensor based on AuNPs has been widely applied for detection of biomolecules, inorganic ions, and organic environmental hormones.

3.1. Detection of biomolecules

3.1.1. Amino acids

Colorimetric detection of typical amino acids based on AuNPs is a vital application of AuNPs in sensing. The group of Mirkin introduced a visual approach for highly sensitive and selective detection of cysteine based on oligonucleotide-functionalized AuNPs that included placed thymidine–thymidine (T–T) mismatches which complexed with Hg^{2+} [113]. The mechanism relied on the formation of thymine–Hg^{2+}–thymine (T–Hg^{2+}–T) structure and strong coordination of Hg^{2+} with cysteine, leading to a purple-to-red color change. As a result, cysteine could be detected as low as 100 nM. Salt-induced aggregation of AuNPs has been reported all the time. In order to overcome this shortcoming, Wei et al. established a

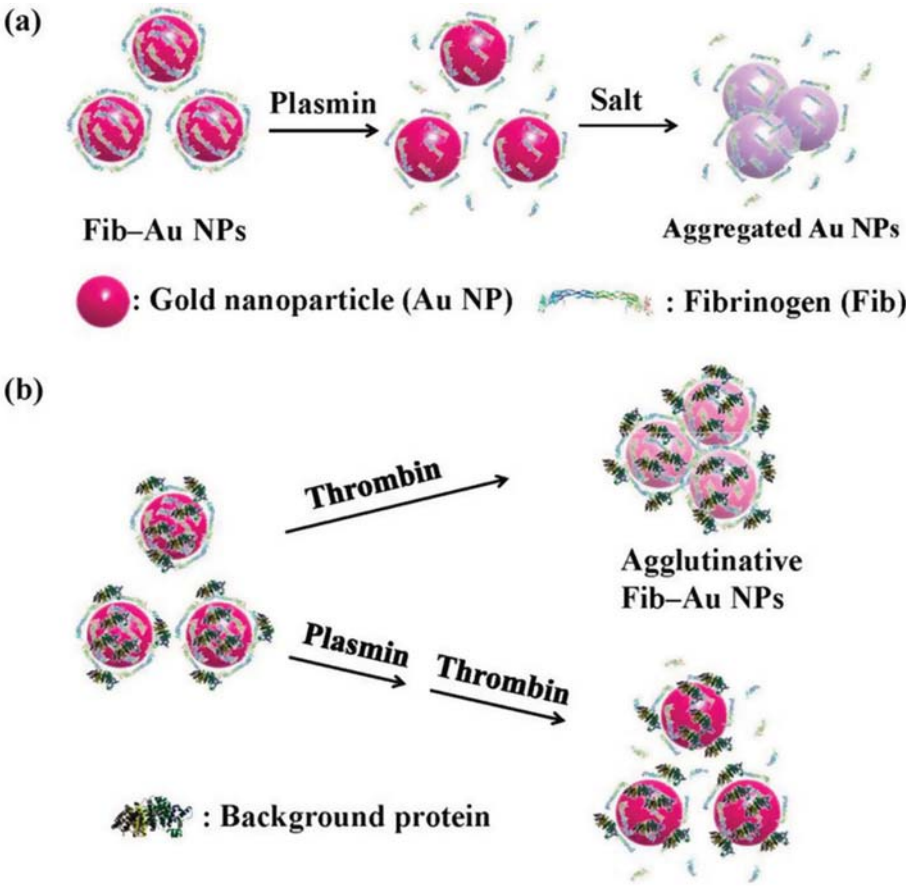

Fig. 3. Schematic illustration of plasmin (a) and plasminogen (b) sensors that design on the basis of fibrinolysis-mediated aggregation and thrombosis-mediated agglutination of Fib–AuNPs, respectively. Adopted from Ref. [133], Copyright (2013), with permission of Elsevier.

simple and sensitive colorimetric way to detect cysteine based on carboxymethyl cellulose-functionalized AuNPs (CMC-AuNPs) [114]. CMC can prevent the salt-induced aggregation of AuNPs and is specific for cysteine, leading to aggregation in the presence of cysteine with the good linear range of 10.0–100.0 μM. The application on real samples is crucial important for detecting methodology, such as serum, urine, or cerebral system [115]. Recently, Zhang et al. firstly reported a novel, specific, and sensitive sensor for cysteine using pectinase protected AuNPs, where AuNPs tightly combined with pectinase via the strong covalent Au—S bond and electrostatic binding [34]. This sensor provided wide linear range from 4.85 nM to 302 μM ($R^2 = 0.996$) and 3.25 to 10.3 mM and low LOD of 4.6 nM.

Monitoring of other amino acids such as arginine, histidine, methionine, and tryptophan has also been reported [116,117]. Importantly, parallel detection of them is difficult to obtain in detecting field, especially in colorimetric detection. However, Rawat and Kailasa have achieved this progress for parallel recognition of them by using 4-amino nicotinic acid-functionalized AuNPs [118]. The selectivity achieved mainly depends on the longer wavelengths at different wavelength of the surface plasmon resonance band which provided at 647, 676, 695, and 701 nm for tryptophan, arginine, histidine, and methionine, respectively. Furthermore, some new strategies not only use AuNPs but other materials such as graphene oxide (GO) and dots have been developed for colorimetric detection of amino acids, e.g., glutamate, aspartate, and cysteine [119–123]. The results suggested this kind of sensor had better stability and linearity in dose-dependent responses for amino acids than other particles.

3.1.2. Peptides and proteins

Peptides and proteins are very important for life, e.g., adjusting physiological functions of each system, composing cells and tissues, and taking charge of life [54]. Colorimetric detection of main peptides and proteins like glutathione (GSH) and enzymes based on AuNPs has been well developed [124]. The aggregation of AuNPs based on distance-dependent optical properties and interaction of a 2:1 GSH–Cu^{2+} complex has been utilized for detection of GSH [125]. It is interesting that the peptides–metal complex is always used to detect metal ions but this method uses this complex to hold a specific selective detection of GSH and reaches the LOD of 10 nM. On the contrary, the anti-aggregation of functionalized AuNPs for GSH detection has also been reported. For example, Uehara et al. described a GSH colorimetric assay based on GSH-triggered disassembly of AuNPs and exhibited a bluish purple color [126]. The degree of AuNPs anti-aggregation was proportional to the GSH concentration, and thus making sensitive detection of GSH (LOD of 29 nM). Similarly, the sodium piperazinebisdithiocarbamate [127] and 2-mercapto-1-methylimidazole (MMI) [128] -capped AuNPs based on anti-aggregation principle for determination of GSH have been reported, respectively. However, the principles are different. For the first one, the anti-aggregation is dependent on the stronger affinity between AuNPs and GSH. As for the other one reported in recent, the anti-aggregation relies upon the mixture between MMI and GSH, which leading to MMI lose the ability of attaching on the AuNPs surface rather than GSH had stronger binding ability with AuNPs.

The visual detection of proteins based on AuNPs over the past few years such as thrombin (Thr), lysozyme (Lysz), have been well

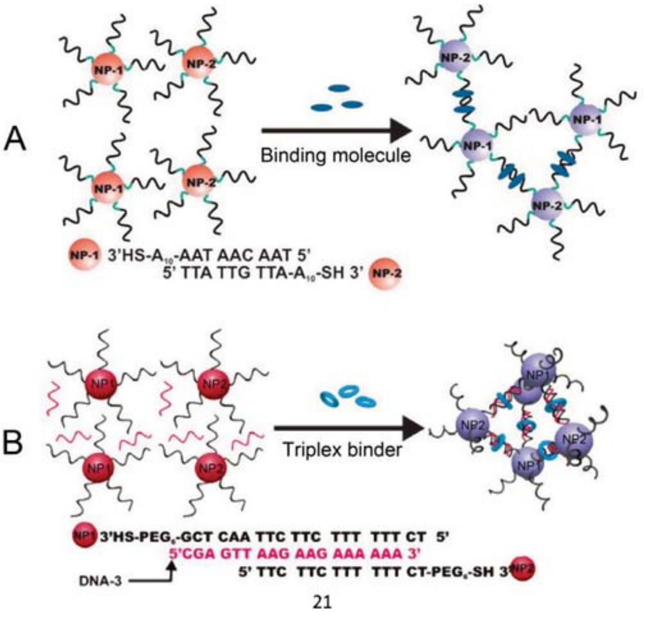

Fig. 4. Colorimetric detection of DNA concerning potential DNA-binding molecules based on the formation of duplex (A) or triplex DNA (B). (A) Adopted from Ref. [143], Copyright (2006), with permission of Wiley-VCH. (B) Adopted from Ref. [144], Copyright (2006), with permission of American Chemical Society.

reviewed by some reviews [129,130]. Hence, for this section, we just review some typical studies introduced by some groups and new studies reported recently. The groups of Huang and Chang are well known for studying on protein and DNA, especially the detection of proteins using various methodologies. In 2005, they have developed a highly selective sensor for the platelet-derived growth factors and receptors using aptamer modified AuNPs by monitoring the color changes and extinction of the UV–vis absorption peak [131]. In the aptamer-based strategy, unmodified AuNPs probes using a branched DNA amplification strategy have been employed by Chang et al. to detect vascular endothelial growth factor (VEGF) [132]. Additionally, by integrating the signal amplification capacity, this sensor allowed highly selective and sensitive VEGF detection with the LOD of 5 ng mL^{-1} (185 pM), which was much lower than that of other aptamer sensors. The modified-AuNPs for detection of fibrinolytic-related proteins was also reported by the group of Huang [133]. As shown in Fig. 3, the fibrinogen was self-assembled on the surface of AuNPs (Fib–AuNPs) for simultaneous detection of plasmin and plasminogen. In the first place, the salt-induced aggregation of Fib–AuNPs in the presence of plasmin leading to sensitive detection of plasmin. Next, the Thr- and Fib–AuNPs (Thr/Fib–AuNPs) were used to analyze the plasmin activity and plasminogen concentrations in serum by observing Thr-induced aggregation of Fib–AuNPs. On this basis, the concentration of plasmin as low as 0.4 nM has been determined. Recently, a facile AuNPs based colorimetric sensor array for various proteins detection was introduced using DNA as a nonspecific receptor [134]. 12 proteins in serum have been well discriminated with the naked eye, which provide novel insight into proteins recognition. DNA and carbon dots can also be employed to modify AuNPs for nucleic acid and protein detection and it were successfully applied to human serum samples [135].

3.1.3. Nucleic acids

In this part, we mainly introduce the studies demonstrated by the group of Mirkin and some papers reported recently. DNA-based rationally assembling AuNPs were firstly demonstrated by Mirkin in 1996 [136]. In fact, the hybridization of one oligonucleotide probe and target-induced aggregation of AuNPs contributes to the interparticle distance dependent effect and surface plasmon resonance of AuNPs [137]. Thiolated DNA strand-functionalized AuNPs make researchers study the properties of AuNPs probes according to this method [138]. The discovery has stimulated fabrication of oligonucleotide structured assemblies on AuNPs and extensive use of colorimetric detection of nucleic acids. The mechanism mainly lies in one or two base oligonucleotide or DNA sequences with thiol ligands or —SH attach to the surface of AuNPs and then the mismatches or complementary structure formed in the presence of target chains, thus leading to aggregation of AuNPs and following the color change from red-to-blue [139]. On this basis, they further investigated the factors controlling the melting properties of DNA-linked AuNPs assemblies and found that the melting temperature and linking DNA or oligonucleotides were the key controlling factors [140]. Therefore, some thermodynamic investigations on binding properties for DNA-functionalized AuNPs probes have been developed [141,142]. Furthermore, they also demonstrated new strategies for colorimetric detection of DNA concerning potential DNA-binding molecules which could form duplex or triplex DNA (Fig. 4A and B) [143,144]. Using these principle, Li et al. presented an ultrasensitive colorimetric method for microRNAs using the isothermal exponential amplification reaction-assisted AuNPs [145]. In the presence of DNA polymerase, the target RNA gradually detached by the sensing system and the bare AuNPs aggregated in high salt concentration without DNA protection. This method distinguished a single nucleotide among homologous microRNAs and provided a good linear range from 50 fM to 10 nM with the LOD of 46 fM.

In recent years, some new methods for nucleic acids detection based on AuNPs have been shown up. For example, Liu et al. reported a non-aggregated AuNPs based on colorimetric sensor that was dependent on oligonucleotide-functionalized AuNPs and magnetic beads, which played important role in removing the capped-AuNPs to obtain higher sensitivity [146]. Compared with other AuNPs sensors, the distinction was looking at the dispersed

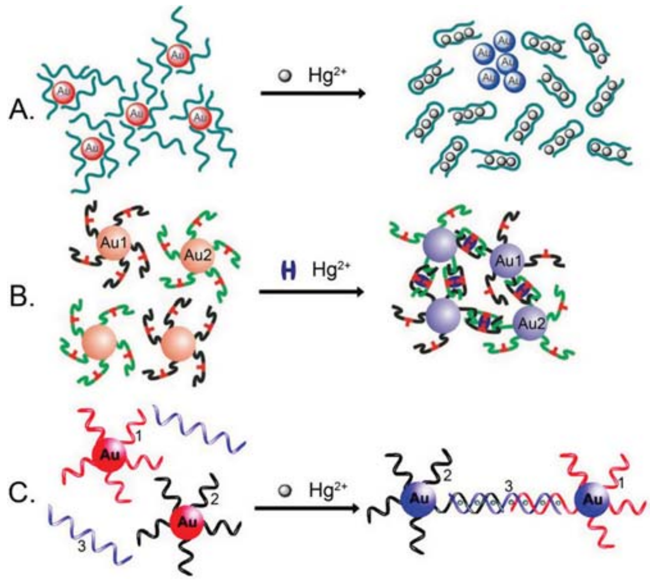

Fig. 5. Colorimetric detection of Hg^{2+} based on AuNPs aggregation by T–Hg^{2+}–T coordination chemistry on the basis of three formations, which lie in one (A), two (B), and three (C) DNA or oligonucleotide strand, respectively. (A) Adopted from Ref. [165], Copyright (2008), with permission of Wiley-VCH. (B) Adopted from Ref. [161], Copyright (2007), with permission of Wiley-VCH. (C) Adopted from Ref. [166], Copyright (2008), with permission of American Chemical Society.

AuNPs in the supernatant rather than the sedimentation of aggregated AuNPs. In addition, this strategy provided potential to establish a simple and simultaneous multi-color sensor based on the non-aggregated AuNPs. It is reported that because of the SPR phenomenon, namely the distance-dependent property of AuNPs, single stranded (ss) DNA can interact with AuNPs and well stabilize in high salt concentrations [147,148]. In this case, a direct colorimetric sensor for unamplified pathogen DNA has been developed based on dextrin-capped AuNPs, in which AuNPs are aggregated in the absence of target ssDNA and this strategy achieves very low LOD of 2.94 fM [149]. Strategies for nucleic acids detection based on AuNPs and other materials, such as grapheme [19] and GO [150], have also been developed. Yun et al. recently have introduced a novel strategy based on salt-induced aggregation of AuNPs for enzyme-free ultrasensitive DNA detection [151]. This platform relied on the three hairpin auxiliary probes are assembled on the surface of AuNPs in the absence of target DNA. AuNPs are well dispersed in high salt concentration. When target DNA existed, the hairpin auxiliary probes are activated and form additional branched junctions, which cannot prevent the salt-induced aggregation. This idea is simple and selective and is promising for colorimetric detection of other target molecules based on AuNPs.

3.2. Detection of inorganic ions

3.2.1. Mercury ions

Different from other metal ions, Hg^{2+} can bond with many groups like —NH_2, —SH, and —COOH, which allows it form strong affinity with amino and amino acids. The basis of Hg^{2+}–thymine complex (T–Hg^{2+}–T) makes it is easily detected by oligonucleotide-functionalized AuNPs sensors. In particular, the affinity between nitrogen atom and Hg^{2+} is always used to fabricate Hg^{2+} sensors based on the blue shift of AuNPs process [152,153]. A simple approach for selective visual sensing of Hg^{2+} has been reported based on Hg^{2+} breaking the Au-S bonds between AuNPs thiol ligand modified AuNPs and inducing aggregation of AuNPs [154]. Furthermore, a highly sensitive and selective detection sensor for Hg^{2+} in drinking water through the sulfur–Hg^{2+}–sulfur interaction has been reported [155]. This strategy provided the widest detection range of 3×10^{-8}–1×10^{-2} M with a LOD of 30 nM.

Although the formation of Au–Hg alloy is unbeneficial to design Hg^{2+} sensor in electrochemical sensor using gold-based electrode (GE), recent researches provide new insight for designing Hg^{2+} colorimetric sensing probe [156]. Interestingly, a recent paper based on three color changes from red through sandy beige (Au–Hg alloy) to celandine green (larger Au–Hg alloy) has been proposed with a LOD of 8.76×10^{-9} M, which is much lower than 30 nM [157]. Another novel headspace colorimetric sensor for specific Hg^{2+} detection has been introduced based on the strong metallophilic Hg–Au interaction, and resulting in a LOD of 5 nM. In this strategy, AuNPs experience etching and amalgamation process by Hg^0 and a red-to-blue color change [158]. In addition, because of excellent peroxidase-mimicking activity, the Au–Hg amalgam strategy exhibits highly sensitive, rapid and low cost for Hg^{2+} and H_2O_2 detection at the same time [159]. Particularly, a gold nanozyme-based paper chip was further prepared on the basis of Au–Hg amalgam enhanced enzyme-like catalytic activity, which provided a simple, sensitive, rapid, and cost efficient sensor for trace of Hg^{2+} [160].

DNA or oligonucleotide-conjugated AuNPs have been used to colorimetric recognition of Hg^{2+} on the basis of T–Hg^{2+}–T coordination chemistry (Fig. 5). Sensors based on DNA or oligonucleotide were also well developed by the group of Mirkin. In 2007, they reported a colorimetric way by DNA-functionalized AuNPs [161]. This visual inspection sensor was highly sensitive and selective and simpler than the conventional methods, but the requirement of precise heating and careful monitoring of denaturation temperature limited the application. In order to overcome this drawback, Xue et al. developed this method, which the operating temperature could be adjusted in room temperature and simplify the step on detection of Hg^{2+} [162]. Owing to the properties of DNA or oligonucleotide conjugated AuNPs, Mirkin et al. designed a new class of G-rich oligonucleotides or guanosine monophosphate derivatives modified AuNPs to simple and efficient detection of metal ions [163]. On this basis, some recognition methods based on G-quadruplex-induced aggregation of AuNPs were developed. Li et al. have proposed the design on selective detection of Hg^{2+} using the T–T base pair in a DNA duplex, which Hg^{2+} can specifically bind to and form the folding G-quadruplex DNAs and T–Hg^{2+}–T complex [164]. Thus, the facial method to sense Hg^{2+} with colorimetry is proposed and the concentration was as low as 50 nM (10 ppb can be detected).

Amino acid-functionalized AuNPs to visual detection of Hg^{2+} have been proposed. A colorimetric sensing strategy for melamine (MA) and Hg^{2+} using cysteamine-modified AuNPs (Cys–AuNPs) has been introduced by Ma et al. [167]. This strategy achieved a LOD of 30 nM for Hg^{2+} detection with sensing mechanism of electrostatic attraction for N–Hg^{2+}–N structure in alkaline pH. Interestingly, this sensor could also detect MA by adjusting the pH to acidity since the surface of Cys–AuNPs was positively charged because of the protonated amine groups. The amine groups might electrostatically bind with MA and then induced aggregation of AuNPs. The proposed sensing strategy opened new opportunities for parallel detection of metal ions and organic environmental hormones. The idea of using N–Hg^{2+}–N structure to achieve efficient detection of Hg^{2+} is worth learning but many of them may only imitate this process to obtain a new method or use a new amino acid to modify AuNPs for Hg^{2+} detection. Different from these strategies, Sener et al. proposed novel thinking by using the same property. Citrate-stabilized AuNPs were well dispersed in aqueous solutions even Hg^{2+} coated on the surface [168]. Making use of the N–Hg^{2+}–N structure obtained from the lysine and Hg^{2+}, the Hg^{2+}-coated AuNPs aggregated and distinct color change from red to blue occurred. This proposal provided the LOD as low as 2.9 nM and was very fast and sensitive.

The groups of carboxylic and amino (—COOH and —NH_2) are easy to complex with metal ions, especially Hg^{2+} [169–171]. A carboxylated peptide-functionalized AuNPs (peptide–AuNPs) for detection of Hg^{2+} was introduced by Si et al. in 2007 [172]. The main driving force of this sensor was laid on the dipole–dipole interaction between carboxylated peptide and Hg^{2+}. In fact, the mechanistic pathway should owe to the interaction between Hg^{2+} and —NH_2 or —COOH. In addition, the dynamic light scattering (DLS) measurement was used for verifying successful assembly of the peptide–AuNPs. A paper developed by Xu et al. described a novel approach for simple and rapid detection of Hg^{2+} in high salt solutions based on Hg^{2+}-induced aggregation of mononucleotides-functionalized AuNPs (dTTPs–AuNPs) [173]. As we all known, the citrate-stabilized AuNPs are easy to aggregate in high salt solutions. However, the dTTPs-stabilized AuNPs were well dispersed in high salt solutions, and quickly reaggregated when Hg^{2+} presented.

Proteins are made up of one or more polypeptides, which are formed from many kinds of amino acids, and play various important roles in all aspects of biology. Hence, there is no doubt that protein-functionalized AuNPs can be used to detect metal ions. Guo et al. have developed a simple and rapid visual approach for Hg^{2+}, Cu^{2+}, and Pb^{2+} determination using papain, which is a protein with some cysteine residues, functionalized AuNPs [174]. To improve the sensitivity and selectivity, our group modified this sensor by adding the chelating agent, 2,6-pyridinedicarboxylic acid (PDCA), which could form strong complex with Hg^{2+}. The LOD was decreased from 200 to 9 nM and provided wide linear range of

0.01–14 μM [175]. To obtain the green colorimetric sensor based on AuNPs, Tripathi et al. have demonstrated a rapid and simple sensor of biogenic AuNPs by mixing the fungal biomass from *Trichoderma harzianum* with chloroauric acid solution [176]. The sensor based on proteins-functionalized AuNPs for colorimetric detection of metal ions provides a good idea, but the selectivity should be improved in some extent.

In addition to the principles mentioned above, the colorimetric sensing for Hg^{2+} based on blue shift of absorbance peak is popular since it is very sensitive and highly selective. The details of these sensors are showed in Table S2. Among these strategies, the most interesting one is the paper reported by Tripathi et al. [176]. In this work, the thiocyanuric acid (TCA) attached to the surface of AuNPs and induced aggregation of AuNPs with the color change of red to blue because the thiol group of TCA could easily bind with the AuNPs through Au—S bond. But the thiols were more apt to interact with Hg^{2+}, so when Hg^{2+} added, AuNPs were dispersed again and TCA separated from AuNPs forming complex with Hg^{2+}, which induced the color change from blue to red. Without the stabilization of citrate, AuNPs were easily aggregated in high concentration of Hg^{2+}, which showed reaggregation.

3.2.2. Lead ions

Table S3 exhibits the visual sensors for Pb^{2+} dealing with different capping agents based on AuNPs aggregation. The AuNPs aggregation approaches based on ions dependent chelation, including thiols and acids, were used to colorimetric recognition of Pb^{2+}. Also, the peptides, DNAzymes, and DNA can be the capping agents to functionalize AuNPs for Pb^{2+} detection. The group of Hupp pioneered the development of visual sensors for Pb^{2+} based on aggregated AuNPs using 11-mercaptoundecanoic acid (MUA) as the capping agent [177]. Later, Fan et al. improved this approach by performing the chelation mechanism on a polydimethylsiloxane microfluidic chip and the lower concentration with 10 μM of Pb^{2+} was determined [178]. Similarly, maleic acid-functionalized AuNPs tends to aggregate when Pb^{2+} exists. The free —COOH of maleic acid reacts specifically with Pb^{2+} because of the strong affinity between them [179]. A strong demonstration reported by Ratnarathorn et al. has introduced the mechanism clearly [180]. In a related study, Giannakopoulos et al. [181], Yoosaf et al. [182], and Huang et al. [183] have all demonstrated the determination of Pb^{2+} using the gallic acid (GA) stabilized AuNPs. These studies have all revealed that Pb^{2+} prefers to binding to the phenolic group that induced aggregation of GA–AuNPs in the presence of Pb^{2+}.

A series of colorimetric assays for detection of Pb^{2+} by using thiols-functionalized AuNPs have been reported by the group of Huang. Firstly, a label-free and nonaggregation-based AuNPs probe has been introduced in 2009 [184]. The mechanism based on the fact that AuNPs reacted with thiosulfate to form $Au(S_2O_3)_2^{3-}$ complexes, which lead to the decreasing of SPR absorption. When Pb^{2+} and 2-mercaptoethanol (2-ME) added, the Au^+–2-ME complexes formed immediately and induced the SPR absorption decreasing dramatically. Later, they reported an improved label-free AuNPs-based probe, operating on the mechanism that Pb^{2+} controlled the ligands shell of AuNPs using $S_2O_3^{2-}$ and 4-mercaptobutanol (4-MB). As a result, the SPR absorption of AuNPs started red-shift and became broadened. This probe showed high selectivity [185]. The group further developed a simple colorimetric approach for selective parallel determination of Hg^{2+}, Ag^+, and Pb^{2+} using the alkanethiols and label-free AuNPs [186]. For the detection of Pb^{2+}, they used the probe of 2-ME/AuNPs as mentioned above with a little modification, where Na_2S is the ligand instead of $S_2O_3^{2-}$. This method provided simultaneously selective detection of many different metal ions, but the sensitivity for detection of Pb^{2+} was not as good as the aforementioned approaches. In addition, Lin et al. reported a disaggregation-based method by using the crown ether (15-crown-5) and thioctic acid-bifunctionalized AuNPs [187]. In this procedure, the AuNPs aggregation formed because of the hydrogen bonds between carboxylic acid residues. However, the aggregation was disrupted because of the presence of Pb^{2+}, and leading to a red-shift, which exhibited with a color change from blue to red. On the contrary, Mehta et al. reported a dithiocarbamate (DTC) derivative of 4′-aminobenzo-18-crown-6 modified AuNPs colorimetric sensor based on the scheme of affinity between crown ether and Pb^{2+}, but it induced a red to blue color change [188].

Just like Hg^{2+}, peptides-functionalized AuNPs have been used for visual determination of Pb^{2+}. The group of Chai demonstrated the GSH-functionalized AuNPs (GSH–AuNPs) for cost-effective and sensitive colorimetric detection of Pb^{2+} in aqueous solution [189]. Later, Beqa et al. further developed this method to improve the sensitivity by using DLS probe at pH 8 [190]. Then, Zhu et al. reported a new colorimetric assay based on GSH and pentapeptide (CALNN) stabilized AuNPs in both aqueous solution and living cell [191]. In the real sample application, it detected Pb^{2+} in living cell downed to 2.9 fM with the wide linear relationship from 2.9 to 37.7 fM per cell. Recent year, an interesting ultrasensitive sensor using whispering gallery mode based on GSH–AuNPs complex came true on an optical microcavity [192]. The signal was found to be very proportional to the concentration of Pb^{2+} with the range of 2.4–48.26 nM.

Another kind of vital sensor for selective Pb^{2+} detection was based on the DNAzymes-functionalized AuNPs. For this work, the group of Liu and Lu have made outstanding contributions. Initially, they described a design of disaggregated sensors based on DNAzyme-directed assembly of AuNPs (“8–17” DNAzyme system), which provided high sensitivity and selectivity [193]. In this design, sequence of substrate strand (17DS) hybridized with AuNPs and enzyme carried out catalytic reactions, which led to the aggregation of AuNPs. When Pb^{2+} presented, “8–17” DNAzyme cleaved the substrate strand, thereby preventing the aggregation of AuNPs and retaining red color. This Pb^{2+} sensor was highly specific and provided a tunable dynamic range. However, the temperature dependent and requiring heating and cooling limited the simple application. Fortunately, they reported an improved version that allowed very fast and room temperature determination by using larger size of AuNPs (42 nm) with a tail-to-tail formation instead of the earlier head-to-tail formation [194]. Furthermore, they further developed a new design based on the controlled disassembly aggregated-AuNPs via a Pb^{2+}-dependent RNA-cleaving DNAzyme [195]. Later, they reported a sensor to tune the dynamic range based on unlabeled DNAzyme and AuNPs by changing the pH. They added NaCl to adjust ionic strength and EDTA to quench the chelating reaction [15]. As a variant of method introduced by Liu and Lu, Zhao et al. developed a simpler DNAzyme-based technique [196]. Through the Au—S chemistry, the sequence of S1-2 was modified on the surface of AuNPs (Au/S1-2) and then cleaved by the DNase I leading to well dispersed S1–AuNPs. On the other hand, the aforementioned 8–17 DNAzyme system added a significant negative charge, hence increasing the electrostatic stability in relatively high ionic strength. In the presence of Pb^{2+}, the 8–17 DNAzyme was cleaved. The electrostatic and steric stabilization are significantly reduced, which resulting in rapid AuNPs aggregation. Compared with previous reports mentioned above, this strategy is quite simple. In recent, the group of Guo [197], Yang [198] and Sang [199] have reported the colorimetric detection of Pb^{2+} based on DNAzyme–AuNPs amplification strategy and obtained a LOD of 2, 5, and 0.02 nM, respectively.

3.2.3. Cadmium and arsenic ions

Presence of cadmium (Cd^{2+}) and arsenic (As^{3+}) ions in rice and drinking water can lead to significant health hazards, especially Cd^{2+}, which easily accumulates in rice and then makes its way

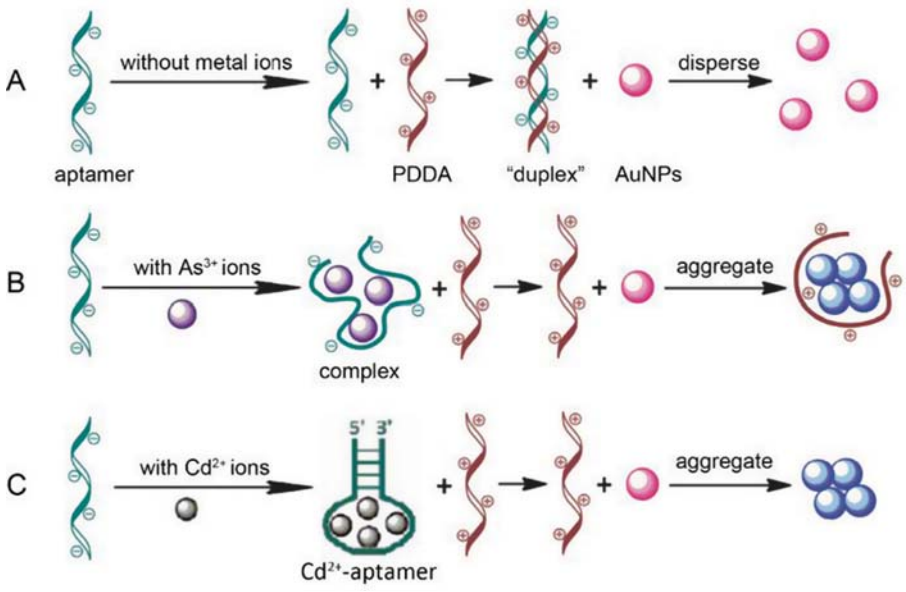

Fig. 6. Schematic description of (A) biosensor based on poly(diallyldimethylammonium chloride) (PDDA) and specific aptamer mediated AuNPs which are dispersed in the absence of metal ions; (B) biosensor for detection of As^{3+} based on PDDA and Ars-3 aptamer mediated AuNPs that is aggregated in the present of As^{3+}; adopted from Ref. [208], Copyright (2012), with permission of the Royal Society of Chemistry. (C) Colorimetric detection of Cd^{2+} on the basis of cationic polymer mediated aggregation of AuNPs using Cd-4 aptamer as a recognition element. Adopted from Ref. [209], Copyright (2014), with permission of the Royal Society of Chemistry.

via the food chain [200–203]. Colorimetric detection of Cd^{2+} ions mainly includes functionalized Ag nanoparticles and AuNPs [204,205]. The channels based on AuNPs can be divided three parts, the chemicals functionalized AuNPs, amino acid or peptides stabilized AuNPs, and thiols capped AuNPs. Kalluri et al. introduced a sensitive colorimetric sensor based on three kinds of chelating ligands functionalized-AuNPs through an As—O linkage or stable complex to sensitive detection of As^{3+} [206]. This method provided a LOD as low as 13 nM. In 2013, Wang et al. demonstrated a selective colorimetric detection method based on the 4-amino-3-hydrazino-5-mercapto-1,2,4-triazole (AHMT) functionalized AuNPs via chelation interaction between Cd^{2+} and chelating ligands of hydrazine and triazole groups [207]. This paper obtained a LOD of 30 nM with good linear range from 0 to 0.48 μM. As a confirmatory analysis, DLS was used to determine changes in particle radius. Furthermore, the aptamers-capped AuNPs for colorimetric detection of As^{3+} and Cd^{2+} were developed. The group of Zhou demonstrated an arsenic-binding aptamer sensor for As^{3+} via an As^{3+}–aptamer complex, which led to the aggregation of AuNPs and caused an obvious color change from red to blue [208]. It provided high selectivity and a low LOD of 70 nM (Fig. 6B). Afterward, they developed this method and used an aptamer to form Cd-4 aptamer for recognition of Cd^{2+} based on the aggregation of AuNPs (Fig. 6C) [209].

Peptides-functionalized AuNPs for determination of Cd^{2+} have been developed. A colorimetric assay using peptide-modified AuNPs (P-AuNPs) for parallel determination of Cd^{2+}, Co^{2+}, and Ni^{2+} has been developed by Zhang et al. [210]. The peptide ligand was self-assembled on the surface of AuNPs via S—Au bond. The P-AuNPs aggregated with the presence of Cd^{2+}, Co^{2+}, and Ni^{2+}, accompanying remarkable color change. Guo et al. introduced an interesting simple and label-free colorimetric approach to selective detection of Cd^{2+} utilizing unmodified AuNPs [211]. In this paper, GSH presented and could prevented AuNPs aggregating in high concentration of NaCl, which led to efficient detection in high-salt medium. In addition, the formation of spherical-shaped complex by coordinating Cd^{2+} with 4× GSH induced aggregation of AuNPs, which exhibited excellent selectivity to other metal ions. The details of other recent colorimetric sensors based on AuNPs for detection of Cd^{2+} are shown in Table S4.

3.2.4. Anions

Some anions, like I^-, F^-, cyanide (CN^- and SCN^-), sulfide (SO_4^{2-}, SO_3^{2-}, and S^{2-}), nitrite (NO_2^-), and nitrate (NO_3^-), etc., play important role in human activities [212]. They are widely present in environment and actively involved in food and biological systems [213]. However, excessive accumulation of these anions in living system with high quantities can result in a series of deleterious disorders [214]. Thus, the detection of anions has gained much attention. But the publications about detection of anions with AuNPs-based approaches are not so many compared with the detection of metal ions because of the high pH sensitivity, low charge to ratio, and solvent dependent binding affinity and selectivity [130]. Besides, some reviews have mentioned the detection of typical anions based on AuNPs-supported method [215–217]. Hence, this review just discusses some typical reports published in the last seven years. In this manner, the Yang group have made a tremendous contribution to highly sensitive and selective detection of anions. Besides, it is worth mentioning that the detection of anions based on AuNPs-supported electrochemical method usually relies on the catalytic or oxidation reaction of anions [218–220], while few of them have this ability. AuNPs on the electrochemical sensor for anions often serve as the medium connecting the ligands or assistant improving the performance, which is similar with the detection of metal ions [221–223]. On the other hand, metal ions such as Pb^{2+}, Hg^{2+}, and Cu^{2+} are capable of forming metal–ligands complexes with many groups or ligands, making them easier detection [158,224]. Hence, the detection of anions is only discussed in the colorimetric method.

The basic design principle of anions detection is mainly divided into two channels. One depends on exploiting a series of possible interactions, like hydrogen bonding interaction, electrostatic attraction, and metal coordination bonds. Another is the signaling subunit which is responsible for the signal transduction. It is reported that cyanide has the ability to etch metals to form a metal–cyanide complex, especially Au and Ag [225–227]. With this respect, many ligands, which served as the protecting and stabilizing agents, are removed from the surface of AuNPs by cyanide, thereby inducing the aggregation of AuNPs [228,229]. And cyanide can be detected selectively [230]. On the contrary, because the strong interaction between SCN^- and AuNPs, the aggregated AuNPs inducing by Au—N interaction are redispersed again. The color of AuNPs changes from blue to red [231]. In addition, in the "cyanide process", Au was leached out by CN^- to form an $[Au(CN)_2]^-$ soluble complex which resulted in on-site cyanide monitoring as low as 0.06 mg L^{-1} [232]. By combining the ligands removal and Au leached mechanism, Cheng et al. creatively proposed an AuNPs-based dual fluorescence–colorimetric sensor for highly sensitive and selective detection of cyanide with low and high concentration [233]. This strategy gives a new insight in pollutant detection. The visual detection of NO_2^- and NO_3^- based on AuNPs often relies on the Griess reaction, which combines two kinds of amides by NO_2^- and NO_3^- to form a N═N bond. The dispersed or aggregated AuNPs are aggregated or redispersed respectively, thus results in highly sensitive detection of NO_2^- and NO_3^- [234,235]. However, they sometimes interfere with each other.

Typical halogen ions including I^-, F^-, and ClO^- also have been detected by AuNPs-based colorimetric method. It is reported that ClO^- has strong oxidizing ability to some ligands, especially thiols. Zhang et al. introduced a visual method for selective detection of ClO^- by converting the alkanethiol to a sulfonate derivative, which induced a red to blue color change. However, the real sample detection were not so sensitive and selective [236]. Thus, Lu et al. proposed a different idea by taking advantage of the strong oxidizing ability of ClO^- to dithiothreitol (DTT). In the present of ClO^-, the aggregated DTT–AuNPs were dispersed again because DTT was oxidized and could not be chemisorbed on AuNPs surface, hence achieving highly sensitive and selective detection of ClO^- [237]. Recently, on the basis of oxidizing Ag^0 to Ag^+ ions by ClO^- in the Au/Ag alloy NPs, an effective colorimetric method for ClO^- detection was reported with the LOD of 300 nM in the linear range from 2400 to 24,000 nM [238]. I^- and F^- can combine with metal ions and ligands to form metal–ions complex or ligand–ions complex, making AuNPs aggregate as a result of lose the protector or changes of SPR [239–241]. Interestingly, a novel sensing strategy for F^- detection was introduced based on F-thiols, providing specific fluorous–fluorous interactions, which resulted in the generation of precipitation, thus inducing a color change of red to colorless [242]. Besides, simultaneous detection of metal ions and anions has gained much more attention. With this respect, Kumar et al. proposed a sunlight induced reduction of AuNPs method, using polyacrylate functionalized AuNPs, to detect Al^{3+} and F^- simultaneously [100].

Sulfide is another kind of important anion, which exists in environment widely. Hydrogen bonding interaction plays important role in sulfide detection since sulfide has strong binding ability with some molecular receptors, like Cys and DTC, which induced aggregation of protected AuNPs [105,243]. Conversely, Yang group proposed a different report [244]. Owing to the directional hydrogen bonding of —NH···O between sulfite and probe, the electrostatic repulsion increases dramatically, hence contributing to the enhancement in the stability of AuNPs. It is proved that AuNPs have an intrinsic peroxidase-like activity that can catalyze oxidation of 3,30,5,50-tetramethylbenzidine (TMB) by H_2O_2, thus generating blue colored products [245]. However, S^{2-} has confirmed that it is capable of reducing Au^{3+} to form AuNPs, Au_2S, or their mixture [246–248]. Therefore, S^{2-} shields the peroxidase-like activity and triggers the aggregation of ligands-capped AuNPs through hydrogen bond and electrostatic interaction, which dramatically decreases the catalytic activity of AuNPs [249]. Inevitably, the

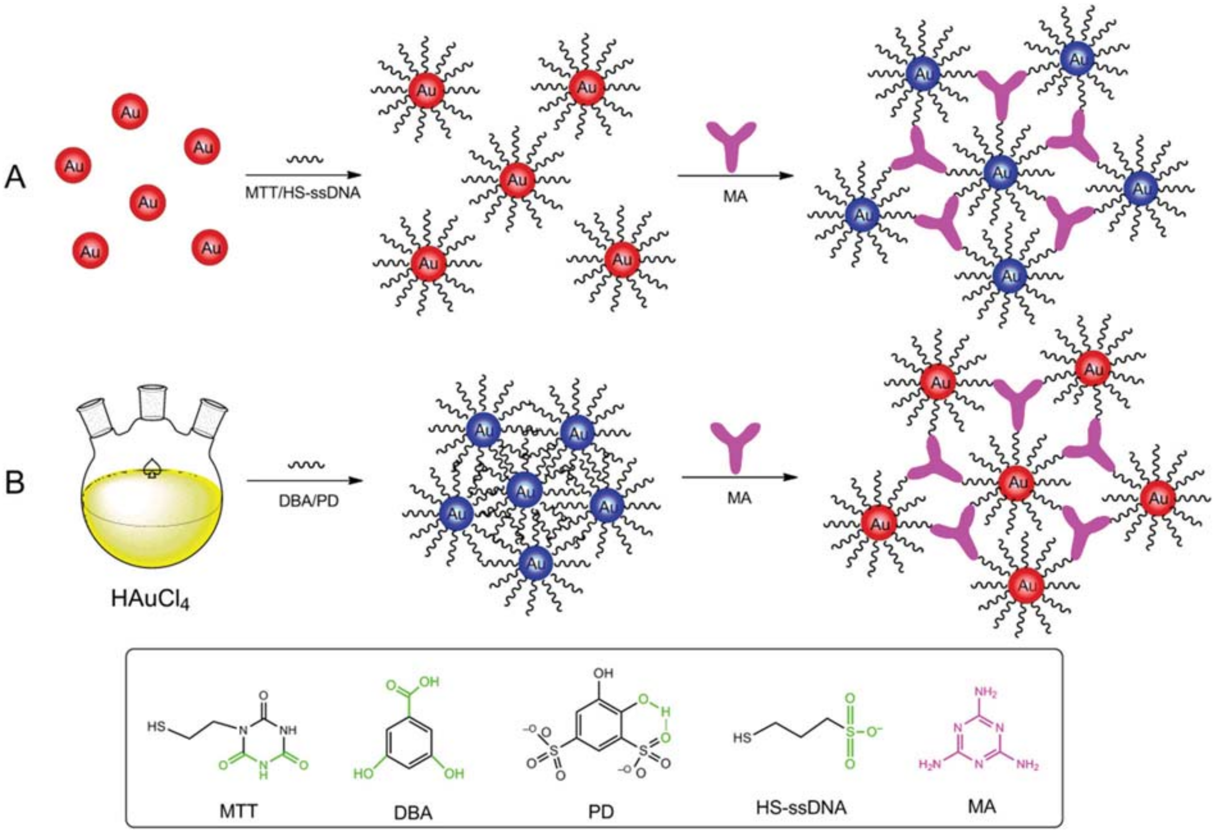

Fig. 7. Hydrogen bonding interaction induced aggregation (A) and anti-aggregation (B) of AuNPs between MTT, HS-ssDNA, DBA and PD and MA, respectively. The color-labeled chemical construction is the potential hydrogen bonding sites, which can form the hydrogen bonding interaction.

chromogenic reaction of TMB is inhibited by S^{2-}, which contributes to the sensitive and selective detection of S^{2-} [250].

3.3. Detection of organic environmental hormones

Recent years, the organic environmental hormones including some persistent organic pollutants, food additives, and medicines have been widely used in agriculture, industry, food, and medical field [251–255]. Some of them are released into the environment, resulting in serious environmental problems [256,257]. On the other hand, these molecules accumulate through biologic chain and eventually bring about serious side effects of health for all living species [258,259]. In the above section, we discussed AuNPs-based colorimetric assays for biomolecules and inorganic ions. Similar detection systems also proposed for organic environmental hormones such as MA, pesticides, drugs, and others (Table S5).

3.3.1. Melamine

As reported by many papers, MA contains a three nitrogen hybrid ring and three exocyclic amino groups which makes it easily bind onto the surface of AuNPs and be recognized via hydrogen bonding. Ai et al. reported the use of hydrogen bonding recognition for MA based on 1-(2-mercaptoethyl)-1,3,5-triazinane-2,4,6-trione (MTT) stabilized AuNPs, with the LOD of 0.2 nM and linear range from 4 to 200 nM and 280 to 440 nM [260]. Given the mechanism, it is the first time to demonstrate the triple hydrogen-bonding recognition between MTT and MA based on AuNPs can be used to detect MA. Later, as shown in Fig. 7, some papers reported that many materials such as DBA [261], PD [262], and HS-ssDNA [263] could form hydrogen-bonding interaction with MA due to it had the ability to provide nine hydrogen-bonding sites [264]. Besides the aggregation of AuNPs based on hydrogen-bonding recognition, colorimetric detection of MA based on non-aggregation of AuNPs has reported by Cao et al. [261] and Wu et al. [262] with the LOD of 0.8 and 0.64 nM respectively. Interestingly, the principle of this two assays was similar that AuNPs were reduced by the stabilizers and aggregated at the same time. However, in the presence of MA, AuNPs were well dispersed in solution attribute to hydrogen-bonding interaction between the stabilizing agents and MA (Fig. 7B).

Except for the hydrogen-bonding recognition, other colorimetric strategies based on aggregation of AuNPs have been reported. Chen et al. [265] and Ni et al. [266] demonstrated strategies based on MA inducing aggregation of bare AuNPs and provided high sensitivity. The difference was that the paper reported by Ni et al. utilized the proxidase-like activity of AuNPs, which produced a blue color change. In addition, Guan et al. designed a simple label-free approach for sensitive detection of MA based on chitosan-stabilized AuNPs. The presence of MA induced a red-shift in surface plasmon bond and a red-to-dark blue color change [267]. Recently, methanobactin (Mb) reduced Au^{3+} to Au^{0} and mediated the fabrication of AuNPs for colorimetric detection of MA has been reported [268]. In the presence of MA, the oxazolone ring of Mb interacts with MA, the formation of AuNPs is interrupted and aggregation of AuNPs shows up simultaneously. This strategy provides the synthesis of AuNPs and analysis of MA in one-step.

3.3.2. Pesticides

Visual detection of pesticides based on AuNPs has been well developed in recent years since the pesticide contamination has become a serious problem all over the world [269]. Organophosphorus pesticides (OPs), a kind of important pesticide, are widely used in agriculture. Acetylcholinesterase (AChE) is a very important cholinesterase enzyme. It sustains the level of neurotransmitter acetylcholine for living organisms via catalyzing the hydrolysis reaction from acetylcholine to thiocholine. However, the catalytic activity of AChE can be thoroughly inhibited by trace amount of OPs. This property makes AChE inhibition biosensor become the excellent way for colorimetric detection of OPs based on non-aggregation of AuNPs (as shown in Table S5) [270,271]. The mechanism of this approach can be classified into two steps. The first step is that the catalytic hydrolysis of AchE promotes the growth of AuNPs seeds and then leads to a red-to-blue color change. The second step is that OPs inhibit the enzymatic catalytic hydrolysis product and thus preventing AuNPs aggregation with well dispersion. This approach is very sensitive for recognition of OPs. However, the sensitivity and real sample applications for some are still needed to improve. For example, the paper reported by Sun et al. demonstrated a simple label-free visual sensor for OPs detection based on the catalytic reaction of AchE and aggregation of lipoic acid-capped AuNPs, but the sensitivity was not so satisfied [272]. In order to overcome these weaknesses, the group of Wu developed a highly sensitive colorimetric method for parathion detection based on the enzymatic hydrolysis reaction of AchE and dissolution of AuNPs in Au^{3+}–CTAB solution [273]. As a result, parathion was sensitively and selectively detected in tap water, sea water, and apple washing solution with the LOD of 2.4 nM under optimal condition.

Bai et al. reported a quick colorimetric detection approach for recognition of six OPs based on aptamer-stabilized AuNPs in high-salt concentration solutions [274]. Aptamer prevented the aggregation of AuNPs in high-salt concentration solutions. Upon the addition of targets, aptamer bond to the targets and detached from AuNPs, leading to eventual aggregation of AuNPs. The results showed that only isocarbophos had the highest sensitivity. Although the specific of this method is still needed to improve, it provides potential application of aptamer-functionalized AuNPs to detect OPs. Similarly, Bala et al. used an aptamer and a positively charged, water-soluble polyelectrolyte, PDDA to stabilize AuNPs and detect Malathion selectively [275]. PDDA and aptamer could form the PDDA–apt duplex and prevented the AuNPs aggregation. When Malathion presented, the aptamer bond to Malathion and further formed apt–mal complex, the aggregation of AuNPs was induced with a color change from red to blue. In addition, this approach provided high sensitivity and selectivity with the linear range of 0.5–1000 pM and LOD of 0.06 pM.

3.3.3. Drugs and other organic environmental hormones

Colorimetric detection of drugs based on AuNPs has been developed because of the good biocompatibility and easy functionalization [276,277]. As shown in Table S5, the principle of this sensor almost lies in the hydrogen-bonding interaction mentioned above. Particularly, Zhang et al. [278] proposed a novel one step preparation of AuNPs using pyrocatechol violet (PCV) as the reducer agent to parallel detect four kinds of antibiotics, including kanamycin, neomycin, streptomycin, and bleomycin. This sensor was simple and practical, but the selectivity should be further improved. On the basis of hydrogen-bonding interaction, our team demonstrated the colorimetric detection of kanamycin using AHMT- [279] and chitosan [280] -functionalized AuNPs, respectively. The hydrogen-bonding interaction between kanamycin and functionalized agents allows the AuNPs aggregate rapidly with the color change from red to blue. In addition, these sensors can be used for various real samples detection such as environmental water, milk, and urine with good sensitivity and satisfied selectivity. However, more specific sensors should be further developed for visual detection of antibiotics. The colorimetric aptasensor has been designed for selective and fast detection of target either [281]. Ramezani et al. developed a triple-helix molecular switch and AuNPs assembled biosensor for colorimetric detection of tetracycline [282]. In the present of tetracycline, the salt-induced aggregated AuNPs dispersed again due to aptamer bond to its target and induced obvious blue-to-red color change.

4. Electrochemical applications of AuNPs in sensing

Electrochemical sensor has gained much attention because of its distinct properties such as rapidness, convenience, sensitivity, good handling, in situ monitoring, low cost, and miniaturization [283,284]. Until now, indirect electrochemical methods for various samples detection have been well developed [285,286]. Specifically, the electrochemical sensor is usually performed by a three-electron system including a working electrode (WE), counter electrode (CE), and reference electrode (RE). A platinum wire and Ag/AgCl (saturated KCl) usually act as the CE and RE, respectively. Different electrodes, such as GE, GCE, are used as the WE broadly [287]. Because of the high price and easy abrasion, GE is used less. To improve the specificity, WE is often covered with different materials. The presence of substances causes the change of current, potential, capacitance, or electrochemical impedance, which indicates the change of different concentration of substances and provides specific detection approach [22]. Based on the detection signals, the common electroanalytical techniques employed for sensor system mainly include electrochemiluminescence (ECL), electrochemical impedance spectroscopy (EIS) and voltammetry which can be briefly divided into these parts: differential pulse voltammetry (DPV), cyclic voltammetry (CV), square wave voltammetry (SWV), and anodic stripping voltammetry (ASV). Generally, electrochemical sensor provides rapid response and well portability and offers high sensitivity and selectivity, which makes the chemical recognition of species being determined [288,289]. Moreover, the surface area of WE is the most important factor that affects the sensitivity because more functional groups or chemical moieties can be exposed to the target in the case of enhanced surface area [290]. Owing to the properties of good biocompatibility, huge surface area, effective amplification of electric signal, and easy to modify, electrochemical sensor employing AuNPs has many advantages [291]. Firstly, an electrode employing AuNPs provides much larger surface area, which results in higher current signals. Secondly, the biologic chain can contact with the AuNPs more "intimately", which can facilitate electron transport. Thirdly, the sensitivity, selectivity, and robustness can be further enhanced.

4.1. Detection of biomolecules

4.1.1. Amino acids and proteins

AuNPs have been used for electrochemical detection of amino acids and proteins with very low LOD, even picomole. The techniques include aptasensor, immunosensor, molecularly imprinted polymers (MIPs), and ECL, etc. For example, Miao et al. developed a simple ultrasensitive electrochemical sensor for picomole determination of GSH using two GE and two complementary thiolated oligonucleotides [288]. One GE was immobilized with one oligonucleotide at first and then replaced by GSH. Subsequently another GE immersed in the solution and captured the released oligonu-

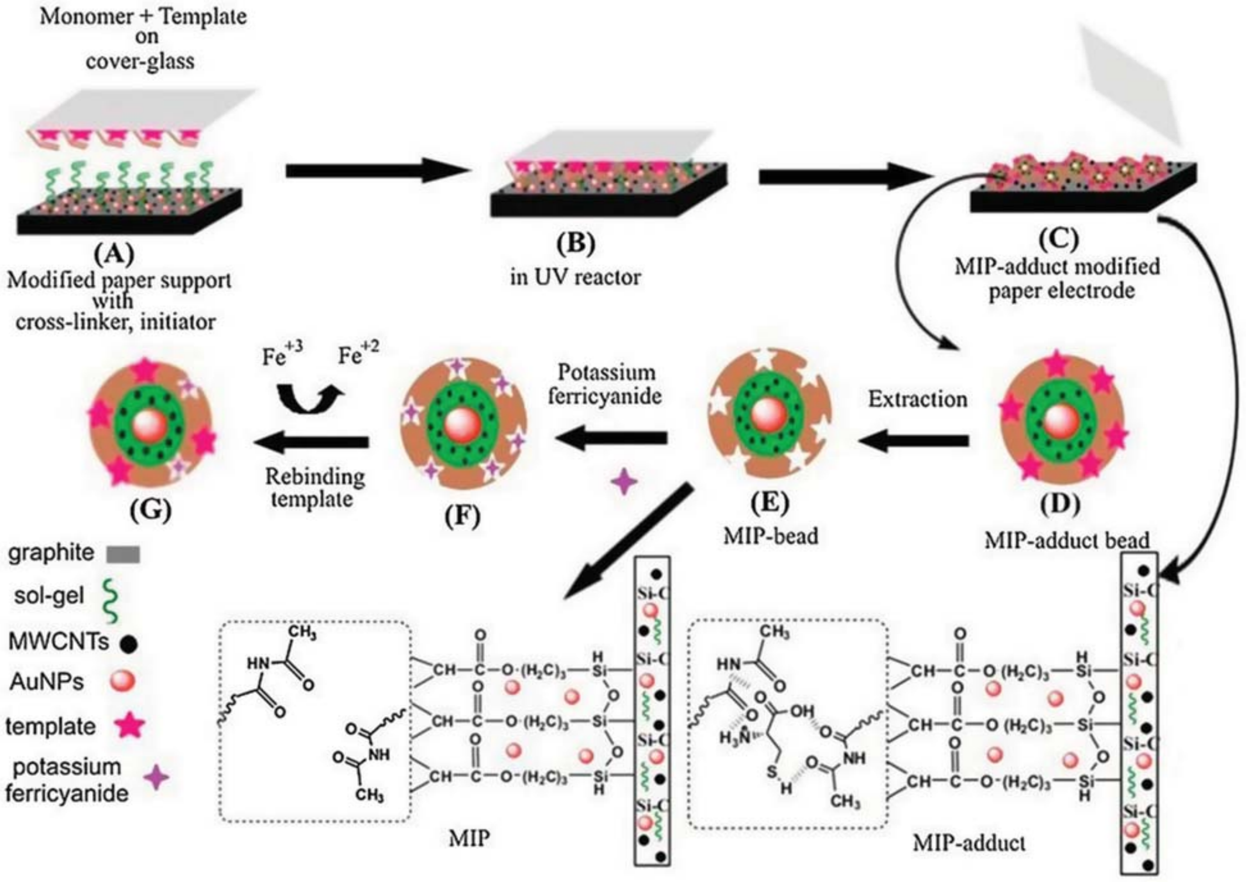

Fig. 8. Outline of L-cysteine micro-contact imprinting: (A) graphite/MWCNTs/AuNPs/sol–gel layered Si—C abraded composite paper support modified with cross-linker and initiator, and the cover-glass is coated with template and functional monomer; (B) the cover-glass was brought into contact with the modified paper support and then placed in UV reactor; (C) the cover-glass is removed; (D) a single bead presentation of MIP-adduct; (E) template is extracted by washing; (F) potassium ferricyanide is introduced into MIP cavities; and (G) rebinding of template by replacing potassium ferricyanide from MIP-cavities. Adopted from Ref. [298], Copyright (2015), with permission of Elsevier.

cleotide. The goal reached by immobilizing the complementary oligonucleotide functionalized AuNPs, which located large numbers of $[Ru(NH_3)_6]^{3+}$, and measured the chronocoulometry (CC) response. The stronger response meant more $[Ru(NH_3)_6]^{3+}$ were adsorbed onto the surface, which indicated a higher concentration of GSH existed. On the contrary, Chen et al. developed a cationic polyelectrolyte-functionalized graphene/AuNPs composites modified electrode for angiogenin sensing [292]. Upon the aptamer combined with AuNPs and $[Fe(CN)_6]^{3-}/^{4-}$ redox couple presented, the angiogenin pushed the structure conversion of loose random coil DNA sequence into the secondary stem–loop structure. Then $[Fe(CN)_6]^{3-}/^{4-}$ redox couple released in the solution and led to an increase in the electron transfer resistance, which induced a significant decrease in SWV current and the sensing process came true. Moreover, the electrochemical immunoassay supported by AuNPs composited with carbon nanotubes (CNTs) and GO has also been used for highly sensitive detection of alpha-fetoprotein [293], antibodies [294], and biotin [295]. AuNPs are used to improve the reaction activity and enhance the electrical conductivity.

AuNPs are used to provide a large surface area for effective immobilization of ligands, ascertain the bioactivity and stability for immobilization of target, as well as trigger the catalysis reaction in some case [296,297]. This design provides excellent selectivity. Similar strategy based on MIPs technique has been further proposed. For instance, Prasad and Singh reported the concerted effect of MIP@graphite/multi-walled carbon nanotubes (MWCNTs)/AuNPs/sol–gel composite for L-cysteine detection by measuring the signal intensity of differential pulse anodic stripping voltammetric (DPASV) (Fig. 8) [298]. On one hand, the incorporation of MWCNTs and AuNPs was significant in terms of the possible detrimental effect on ohmic contact due to the sol–gel × graphite interface. On the other hand, the electro active surface area was obviously enhanced owing to its abundant porous structure via incorporation of MWCNTs and AuNPs, which resulted in a sensitivity augmentation. Other electrochemical biosensors based on AuNPs using different techniques such as ECL and bi-enzyme biosensor were used for detection of glycosylated hemoglobin [299], inosine monophosphate [300], etc. In ECL, luminol and Ru $(bpy)_3^{2+}$ are significant luminophores and always used as the ECL indicators [301–304]. Owing to the excellent electrical conductivity of AuNPs, ECL of indicators can be promoted [305]. There are many reports demonstrated that AuNPs could promote the electron transfer between electrode and indicator and increase the sensitivity of proposed ECL sensor [306,307]. When composed with other materials like MWCNTs and polyamidoamine dendrimer, the energy transfer can be further improved [308]. Particularly, the group of Huang and co-workers developed the pulsed-laser desorption/ionization mass spectrometry (LDI–MS) for detection

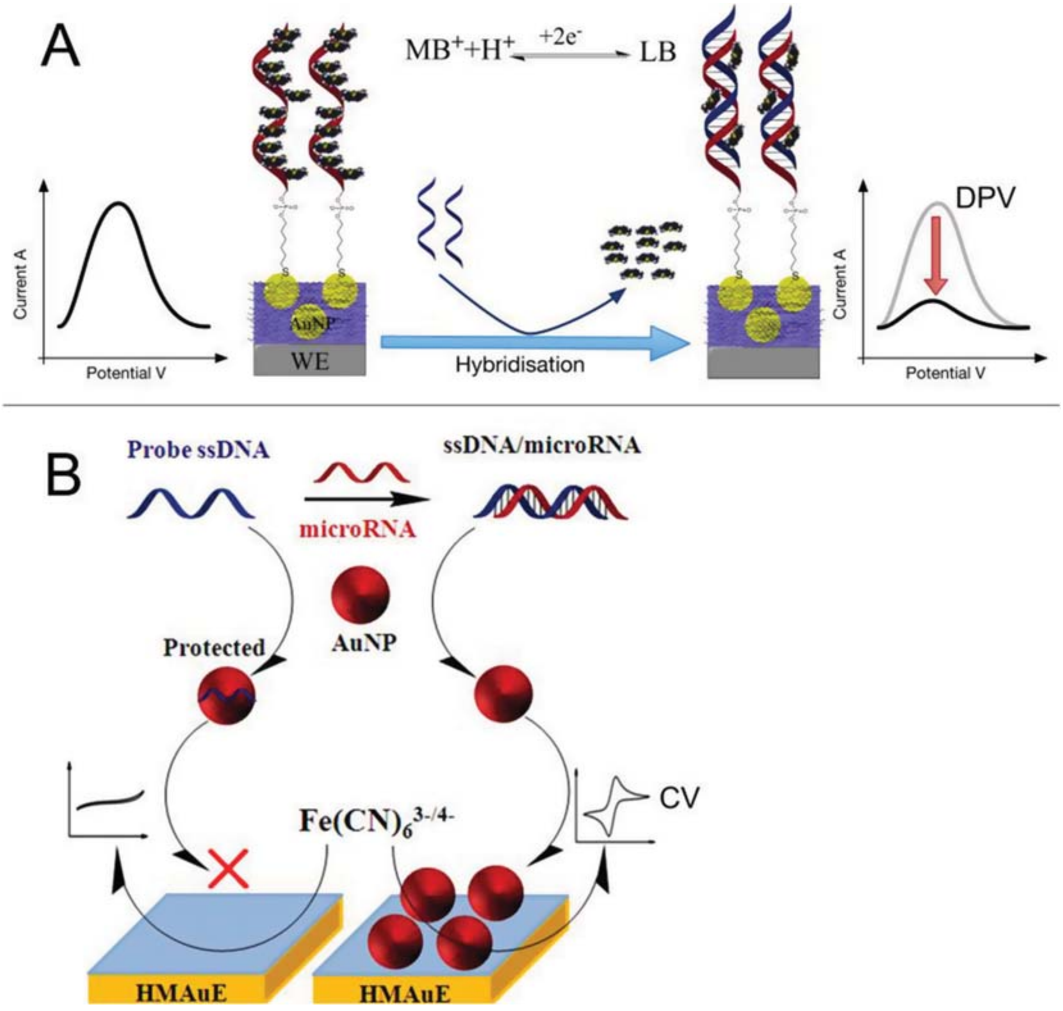

Fig. 9. Schematic representation of electrochemical biosensor (A) for DNA recognition based on polylactide-stabilized AuNPs modified electrode; adopted from Ref. [317], Copyright (2016), with permission of Elsevier. (B) Cartoon drawing for electrochemical detection of target nucleic acid sequence. Adopted from Ref. [322], Copyright (2016), with permission of Elsevier.

of Thr [309] and plasmin [310] by analyzing gold clusters desorbed from aptamer or Fib-modified AuNPs on a nitrocellulose membrane or mixed cellulose ester membrane. This kind of technique provided ultrahigh sensitivity and excellent application on human serum. Importantly, it showed high potential for aptamers, proteins, even other biological signals recognition. QDs are well-known for their ability to generate photocurrent. However, a recent paper demonstrated that an AuNPs/CdAgTe QDs hybrid photoelectrochemical (PEC) immunosensing system was successfully synthesized to sensitively detect cardiac troponin I based on energy transfer between QDs and AuNPs [311]. The electrons produced by QDs were quickly transferred to the surface of AuNPs and finally moved onto GCE to generate photocurrent.

4.1.2. Nucleic acids

There is no doubt that most of the electrochemical sensors for nucleic acids detection are on the basis of nucleic acid hybridization since it prefers to form complementary, non-complementary base-pairing, and mismatched oligonucleotides. The signal response is enormously increased in the presence of AuNPs [312]. The attachment of AuNPs to sensor can dramatically decreased the electron transfer resistance [313]. AuNPs can serve as the signal transduction probes [314], which trapping and storing the electrons to further increase the signal for nucleic acids detection [315]. A label-free ECL sensor using a CdTe quantum dots (QDs) co-immobilized with AuNPs on a chitosan-modified electrode has been designed for DNA detection [316]. The target DNA hybridizes with a molecular beacon to form the hemin/G-quadruplex architecture for signal inhibition. The results reviewed that the ECL signal could be decreased linearly following the logarithmic value of DNA concentration in the range of 5 fM–0.1 nM, with a LOD of 0.9 fM. As shown in Fig. 9A, an original sensitively electrochemical-based DNA biosensor on the basis of direct relation between redox complex concentration and hybridization has been reported by using methylene blue (MB) as electroactive indicator [317]. The peak current of DPV decreased with the increasing concentration of target DNA, which attributed to the complementary base-pairing interaction leading to MB turn to leucomethylene blue. In addition, it was found that when the redox complex bond more strongly with the unhybridized biorecognition molecule, higher selectivity was achievable. Recently, a highly efficient PEC sensor was reported for microRNA-21 detection based on the energy transfer between CdS:Mn doped structure and AuNPs [318]. AuNPs were employed to adjust the exciton states in CdS:Mn doped structure through energy transfer. This method was realized for microRNA-21 detection in a linear range of 1.0 fM–10.0 pM with LOD of 0.5 fM.

Increasing electrical signal, namely signal amplification, is a typical way for target recognition. AuNPs used here can amplify the signal and be the attachment between targets and ligands [319]. The dual-amplification strategy for analyte recognition

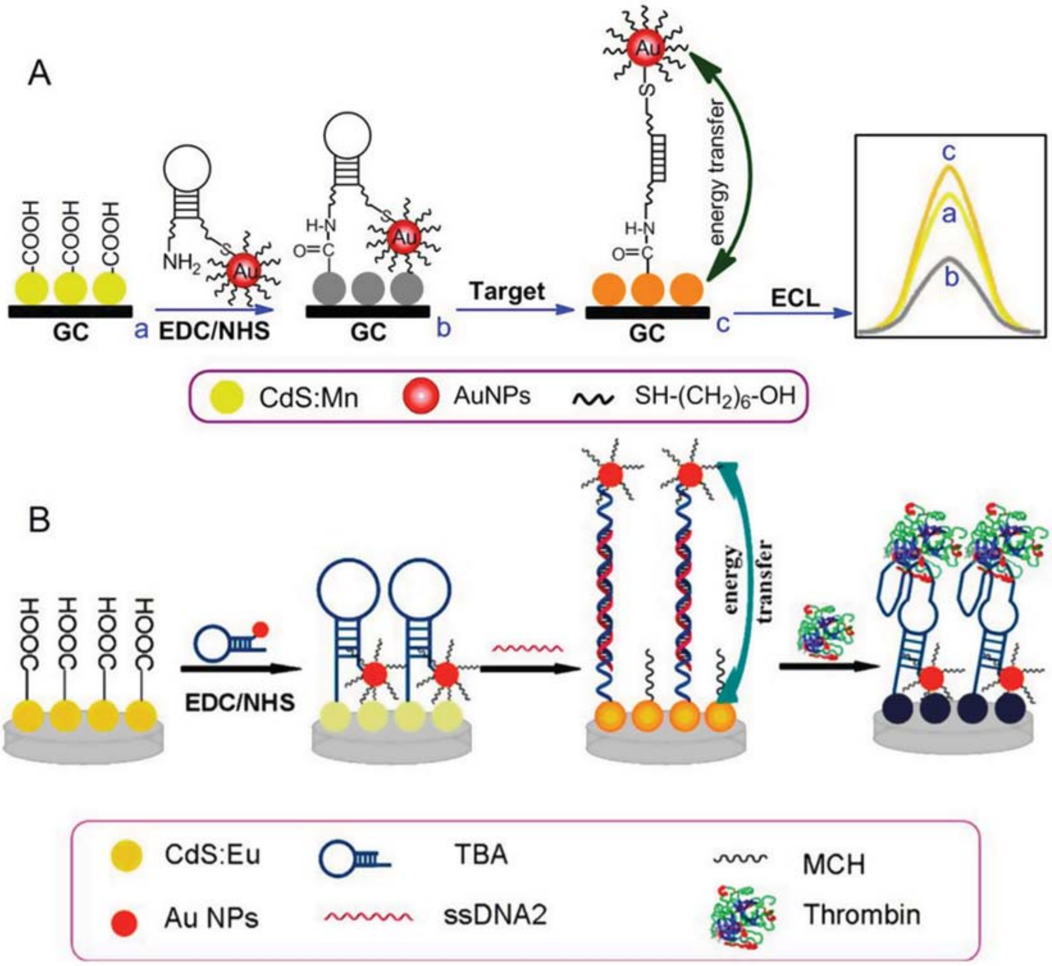

Fig. 10. (A) DNA ECL sensing platform based on energy transfer between CdS:Mn NCs and AuNPs; adopted from Ref. [327], Copyright (2009), with permission of the Royal Society of Chemistry. (B) TBA-related ECL "off–on–off" sensing platform based on energy transfer between CdS:Eu NCs film and AuNPs. Adopted from Ref. [328], Copyright (2014), with permission of Elsevier.

highly improves the sensitivity and has well application on real samples [320]. Besides, a highly sensitive and selective aptasensor for adenosine on the basis of target-induced strand release from the electrode and AuNPs amplification labels was developed [321]. The results showed excellent selectivity toward adenosine recognition in serum samples. The optical approach for nucleic acid detection supported by unmodified AuNPs using differential affinity of single- or double-stranded nucleic acids is always limited by relatively low sensitivity. To improve the sensitivity, the group of Li demonstrated an amplified electrochemical strategy based on the selective preconcentration of AuNPs [322]. As exhibited in Fig. 9B, the protected AuNPs formed by capturing the probe ssDNA on the surface, which produced significant negligible amperometric signal in the presence of $Fe(CN)_6^{3-}$. However, experiencing a hybridization reaction, the ssDNA/microRNA heteroduplex could not bind with AuNPs anymore. And lately, the free-AuNPs were preconcentrated on the surface of electrode, inducing significant improvement of amperometric signal. In another one-pot assay concept, the target microRNA served as a shear, which opened the biotin–MBs, and captured AuNPs, resulting efficient detection of microRNA-21. Furthermore, the LOD was further improved from 100 fM to 4 fM by depositing Ag onto AuNPs [323].

4.1.3. Enzymes

Thr plays central role in large number of cardiovascular diseases and regulates many processes like inflammation and tissue repair at the blood vessel wall [324]. It is necessary for assessing its concentration at trace level and many efforts have been tried. Zhang et al. [325] and Bai et al. [326] both introduced the detection method of Thr based on the hybridization chain reaction depending on DPV response. The difference is that Zhang et al. used two kinds of DNA as aptasensor and Pb^{2+} was necessary here to indicate the concentration of targets. AuNPs served as the connection between aptamerII and barcode binding DNA to capture more targets. However, Bai et al. used HRP and DNA as the aptasensor and obtained the DPV signal through exonuclease-catalyzed target recycling. AuNPs were deposited on the surface of GCE to attach the capture probe. These aptasensors are sensitive and selective but the achievement of target detection is dependent on other materials, which limits the further development. ECL, a supersensitive electrochemical approach for tracing detection of target, has been proved very useful as simple strategy in Thr recognition. As displayed in Fig. 10A, a ECL DNA sensing platform on the basis of energy transfer between CdS:Mn nanocrystals (NCs) and AuNPs has been introduced [327], where CdS:Mn NCs served as ECL luminophores and AuNPs functionalized as both the ECL quencher and enhancer. The quenching of fluorescence by AuNPs is because of the Förster resonance energy transfer (FRET) when luminophores and AuNPs are at close proximity. When the target DNA hybridizes with hairpin-DNA probe, an ECL enhancement achieved due to the interactions of the excited CdS:Mn NCs with ECL-induced SPR in AuNPs at large separation. Inspired by this sensor, Deng et al. developed a Thr-binding aptamer-related ECL "off–on–off" platform as well based on the energy transfer between AuNPs and CdS:Eu NCs film (Fig. 10B) [328]. AuNPs played a critical role in triggering on or turning off the energy transfer. Results showed that this "off–on–off" ECL sensor exhibited a maximum 7.4-fold changes of ECL intensity and offered obvious sensitivity improving for Thr detection in a widespread detection range from 50 aM to 1 pM with the low LOD of 1 aM.

Other aptamer-based ECL protocols have been reported for highly sensitive detection of Thr [329,330]. Other material such as graphene was used to composite with AuNPs for further amplifying the signal [331]. In addition, the label-free bifunctional ECL aptasensor to simultaneously detect many kinds of targets trough switching structures of aptamers was introduced [332]. But the study was not intended to eliminate the interference. Especially, Chen et al. further simplified the design by using TCA/AuNPs modified with ssDNA and improved the sensitivity ranging from 5 pM to 1 nM and LOD as lower as 0.1 pM [333]. AuNPs were used as the carriers to load more ssDNA and further enhance the sensitivity owing to their huge surface area. Recently, an interesting work for Lysz detection has been proposed by Shamsipur et al. [334], who also used AuNPs to capture large amount of aptamer. The most interesting thing is that they used two kinds of electrochemical assay with two different redox probes to indicate the electrochemical signal and induced responses of "signal-on" and "signal-off" respectively. Generally, it is very difficult to express opposite signal in the same system but this strategy indicates a new trend for application of sensor. Not only for Thr and Lysz, other enzyme sensors have been developed either [335,336], as well as other electrochemical techniques such as PEC [337]. They also provide excellent selectivity and high sensitivity and are worthy of consideration for wider application.

4.2. Detection of heavy metal ions

4.2.1. Mercury ions

Similar with the principle reviewed about the colorimetric sensor for Hg^{2+} above, the main mechanism of electrochemical sensor can be divided into four parts: (1) the interaction between Au and Hg^{2+}; (2) the formation of T–Hg^{2+}–T coordination chemistry; (3) the formation of cooperative metal–ligand complex; and (4) the strong affinity of S–Hg^{2+} bond. Table S6 reviews the developments in the design of electrochemical sensor for Hg^{2+} with different strategies in recent years. The details of these strategies will be elaborated as follows.

CNTs-supported AuNPs composites have been synthesized by Xu et al. and first applied for the detection of trace Hg^{2+} via ASV based on the strong interaction between Au and Hg^{2+} [338]. It provides remarkable decrease of reaction time and improves the performance comparing with solid GE. However, the performance on real samples and interferences are not clear. CNTs serve as the bridge links and transmit the electron between GCE and AuNPs. Except for CNTs, other materials such as GO and carbon fiber can also be the bridge between electrode and AuNPs [339,340]. Recent research using 3D graphene/gold film has been proven for highly efficient detection of Hg^{2+} [341]. This newly-designed strategy opens the door toward fabricating 3D graphene-based AuNPs hybrid films simply, and such structures possesses excellent properties of high structure stability, substrate binding strength, significantly large specific area, and good electrical conductivity due to the synergistic effect of AuNPs and graphene. Most importantly, it offers an ultralow LOD of 50 aM, which is the lowest one among we have ever mentioned in this review. On the other hand, the bare GCE deposited with AuNPs for detection of Hg^{2+} has been developed by using a Square Wave Anodic Stripping Voltammetry (SWASV) procedure [342].

Most of the current researches related to electrochemical methods for detection of Hg^{2+} using AuNPs are based on T–Hg^{2+}–T coordination chemistry. For the strong affinity between gold and some groups, GE is always used relatively early based on T–Hg^{2+}–T coordination chemistry [343,344]. It is simple and convenient, but GE is expensive, easy to abrade and structurally change because of amalgam formation, the methods using GCE have been developed. The groups of Zhang [345] and Tang [346] with their co-workers have been developed an electrochemical sensor using GCE for Hg^{2+} detection based on target-induced formation of AuNPs, coupling with the cycling signal amplification strategy using DPV. The main mechanism of the two strategies is shown in Fig. 11. They are both sensitive and selective and provide a low LOD of 0.02 and 0.06 nM with the linear range of 0.5–120 nM and 0.02–1000 nM, respec-

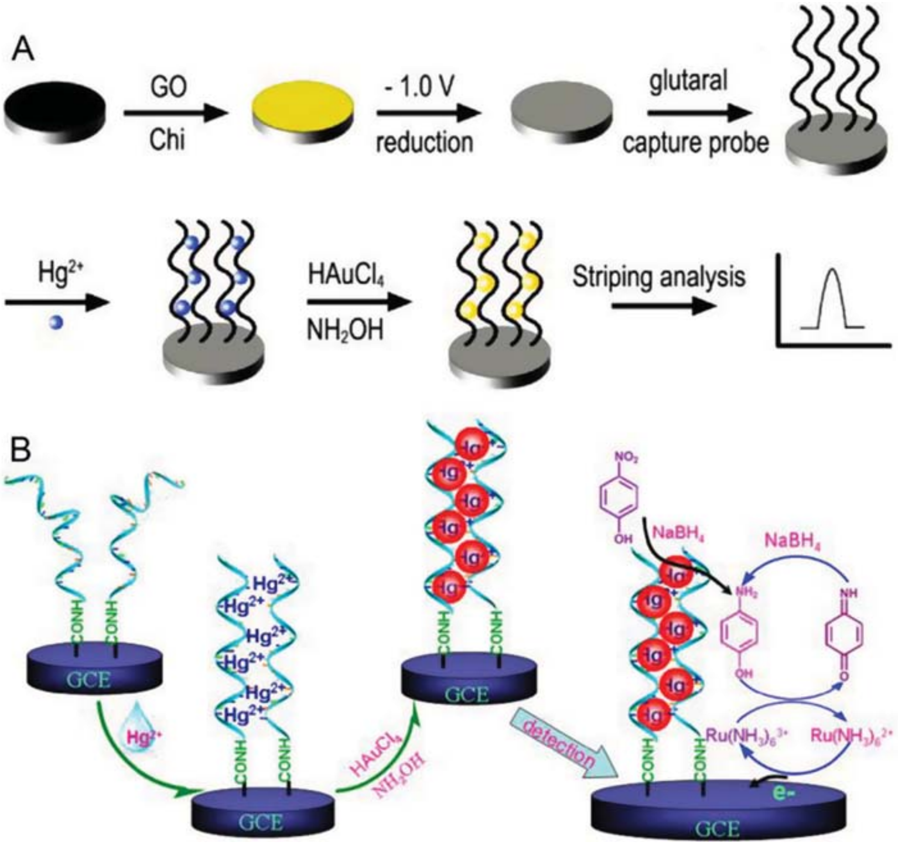

Fig. 11. Schematic diagram of electrochemical sensor for Hg^{2+} detection based on (A) catalytic formation of AuNPs; adopted from Ref. [346], Copyright (2014), with permission of Elsevier. (B) Target-induced AuNPs aggregation formed on DNA-based polyion complex membrane by coupling with cycling signal amplification strategy. Adopted from Ref. [345], Copyright (2014), with permission of Elsevier.

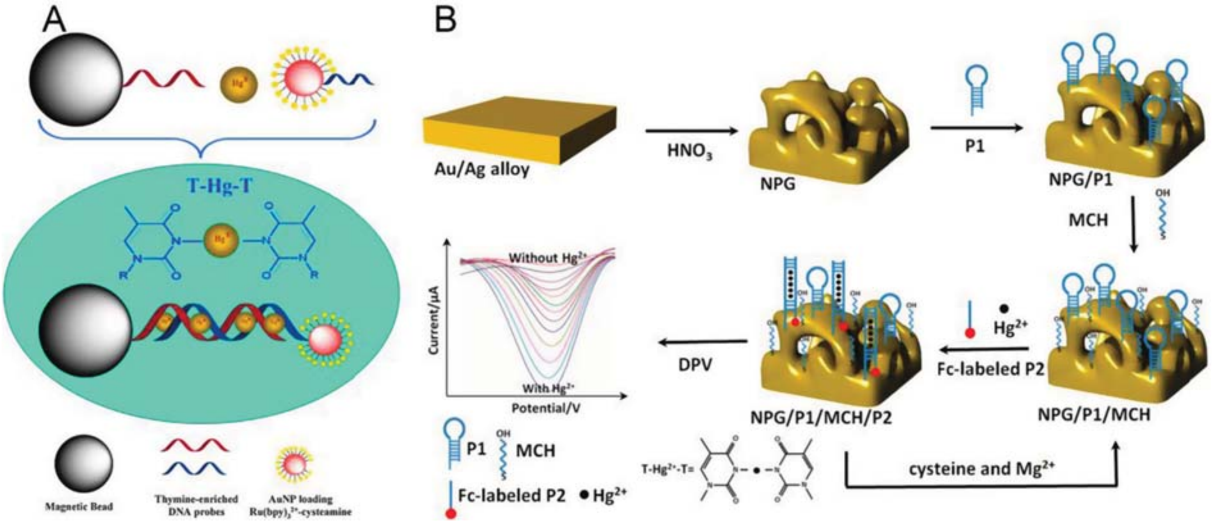

Fig. 12. Schematic illustration of electrochemical detection of Hg^{2+} based on T–Hg^{2+}–T mismatches using the cooperative binding between Hg^{2+} and N atoms. (A) Adopted from Ref. [354], Copyright (2012), with permission of Elsevier. (B) Adopted from Ref. [355], Copyright (2017), with permission of Elsevier.

tively. Recently, a thymine (T)/CA/AuNPs/rGO modified electrode has been fabricated and obtained high sensitivity in the range of 0.05–5 nM and LOD of 0.008 nM [347]. In this case, AuNPs always serve as the connection to link rGO and T. Moreover, they can be the capture probe to attach more T. It is reported that DPV is one of the most sensitive approaches for heavy metal ions detection because of the low LOD [348]. Interestingly, an enzyme-triggered formation of enzyme–tyramine concatamers on AuNPs-functionalized dendrimer using EIS with enhanced sensitivity was reported [349]. A wide dynamic working range from 0.001 to 100 nM and LOD of 0.0004 nM were obtained, and the method exhibited high selectivity and specificity toward Hg^{2+} against other metal ions. ECL is also one of the important approaches for electrochemical detection of Hg^{2+} [350]. For example, a “signal-on” ECL

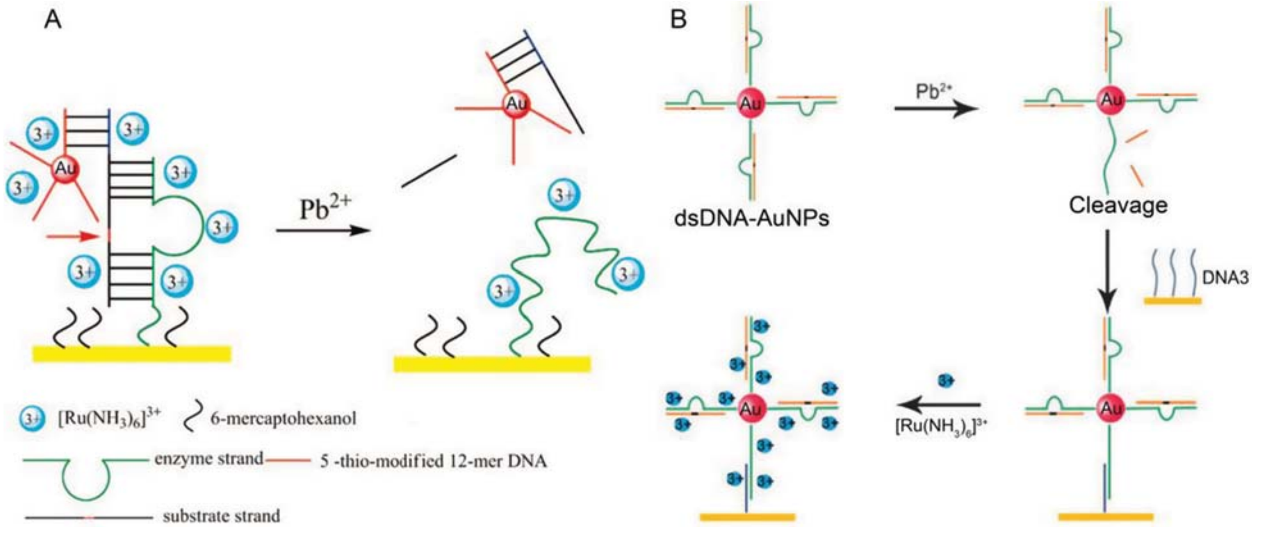

Fig. 13. Principle of electrochemical sensor for Pb^{2+} on the basis of (A) DNAzyme catalyzed Pb^{2+}-induced hydrolytic cleavage of the substrate; adopted from Ref. [358], Copyright (2008), with permission of American Chemical Society. (B) Pb^{2+}-induced cleavage of DNA strand. Adopted from Ref. [359], Copyright (2010), with permission of the Royal Society of Chemistry.

biosensor based on DNA hybridization was designed on the AuNPs nanopatterned electrode surface [351]. In the presence of Hg^{2+}, the loop sequence of hairpin-DNA opened and the ECL "signal-on" was triggered at the same time. Cai et al. designed a multiple signal amplification protocol based on graphene conjugated with CdSe QDs and horseradish peroxidase (HRP) labeled with AuNPs [352]. The presence of AuNPs greatly accelerated the electron transfer and enhanced the electrochemical reaction efficiency owing to their excellent electrical conductivity and large effective surface areas. The ECL intensity was linearly dependent on wide Hg^{2+} concentration from 1 to 4985 nM with the LOD of 0.3 nM. Lately, a label-free ECL assay was developed through oligonucleotide mediated assembly of AuNPs, in which AuNPs well preserved electronical conductivity and significantly enhanced the ECL signal [353]. This method provided specific Hg^{2+} detection over the range of 8 pM–2 nM with a LOD of 2 pM.

Cooperative metal T-rich DNA sequences ligand interaction is very attractive for metal ions detection. The groups, including —COOH and —NH_2, are typically and can form metal–ligand complex with Hg^{2+}. In fact, as shown in Fig. 12, the T–Hg^{2+}–T mismatches as described above use the cooperative binding between Hg^{2+} and N atoms [354,355]. On these basis, Safavi and Farjami [356] reported a new strategy based on thiolated amino acids-functionalized AuNPs through cooperative Hg^{2+}–ligand interactions. The carbon ionic liquid electrode served as the WE to avoid amalgam. The proposed electrode showed high sensitivity and selectivity and good applicability in tap and waste water. Moreover, attempts have been done by using new method such as imprinting films to enhance the performance. For example, Fu et al. [357] demonstrated a surface imprinted electrode utilizing the strong coordination between Hg^{2+} and S, which produced excellent selectivity in natural water samples. AuNPs served as a reinforcing substrate material to synthesis core–shell structural ion imprinting material, due to the high surface area, excellent catalytic and sensing properties.

4.2.2. Lead ions

Electrochemical sensor for specific recognition of Pb^{2+} consisting of AuNPs-supported electrode has been successfully applied for the enhanced sensitivity and selectivity. On the basis of G-quadruplex formation between Pb^{2+} and aptamer, DNAzyme cleavage-based sensors are the most popular techniques (Fig. 13). As an example, a DNAzyme-immobilized GE through thiol–gold interaction has been designed [358]. On the basis of binding Pb^{2+} to DNAzyme, the hydrolytic cleavage of substrate is catalyzed by DNAzyme, which leads to the removal of substrate strand and bound $Ru(NH_3)_6^{3+}$ from the GE surface (Fig. 13A). Different with this procedure, Yang et al. have demonstrated a DNAzyme functionalized AuNPs amplification strategy to enhance the sensitivity [359]. As shown in Fig. 13B, the DNA2 of ds-DNA coated on AuNPs is cleaved by Pb^{2+}, remaining DNA1 which could be matched with the complementary strand (DNA3) and resulting in the CC signals when $Ru(NH_3)_6^{3+}$ existed. Similar to this strategy, our team provided an amplified detection strategy employing AuNPs and nanoporous Au for Pb^{2+} detection in various samples [287]. As the result of Pb^{2+}-specific DNAzyme, the aptasensor showed high selectivity and sensitivity with wide linear range of 0.05–100 nM, following a LOD as low as 0.012 nM.

In terms of G-quadruplex-based sensor, Hai et al. [360] reported a GE coated Fe_3O_4–AuNPs–Aptamer ECL sensor, which formed a G-quadruplex owing to the opening of "stem-loop" structured hairpin aptamer by Pb^{2+}. This "turn-on" ECL sensor could be separated easily and enhanced the selectivity and sensitivity, resulting in a linear relationship in the range from 0.2 to 10 nM and LOD of 0.00108 nM. Based on the distance-dependent quenching of ECL from CdSe QDs by nanocomposites of graphene and AuNPs, Lu et al. developed a highly sensitive ECL method and provided the linear range of 0.1–10 nM and LOD of 0.1 nM for Pb^{2+} detection [361]. RET, providing indeed energy transfer between the donors and acceptors at a certain distance, is a novel and powerful strategy for highly sensitive recognition on DNA binding events. Based on this strategy, a "signal-on" PEC detection system supported by the energy transfer between CdS QDs and AuNPs was used to verify the presence of Pb^{2+} [362]. Compared with other methods reported previously, this sensing device provided acceptable linear range and LOD. On the development of RET technique, a novel ECL–RET system from $O_2/S_2O_8^{2-}$ to the amino-terminated perylene derivative (PTC–NH_2) was demonstrated, in which AuNPs and fullerene nanocomposites coated on GCE and then the thiol-modified assistant probes attached on [363]. $O_2/S_2O_8^{2-}$ served as an ECL donor and PTC–NH_2 acted as the acceptor. The developed aptasensor offers an alternative analytical method with outstanding properties of high sensitivity, selectivity, and accuracy (Table S7).

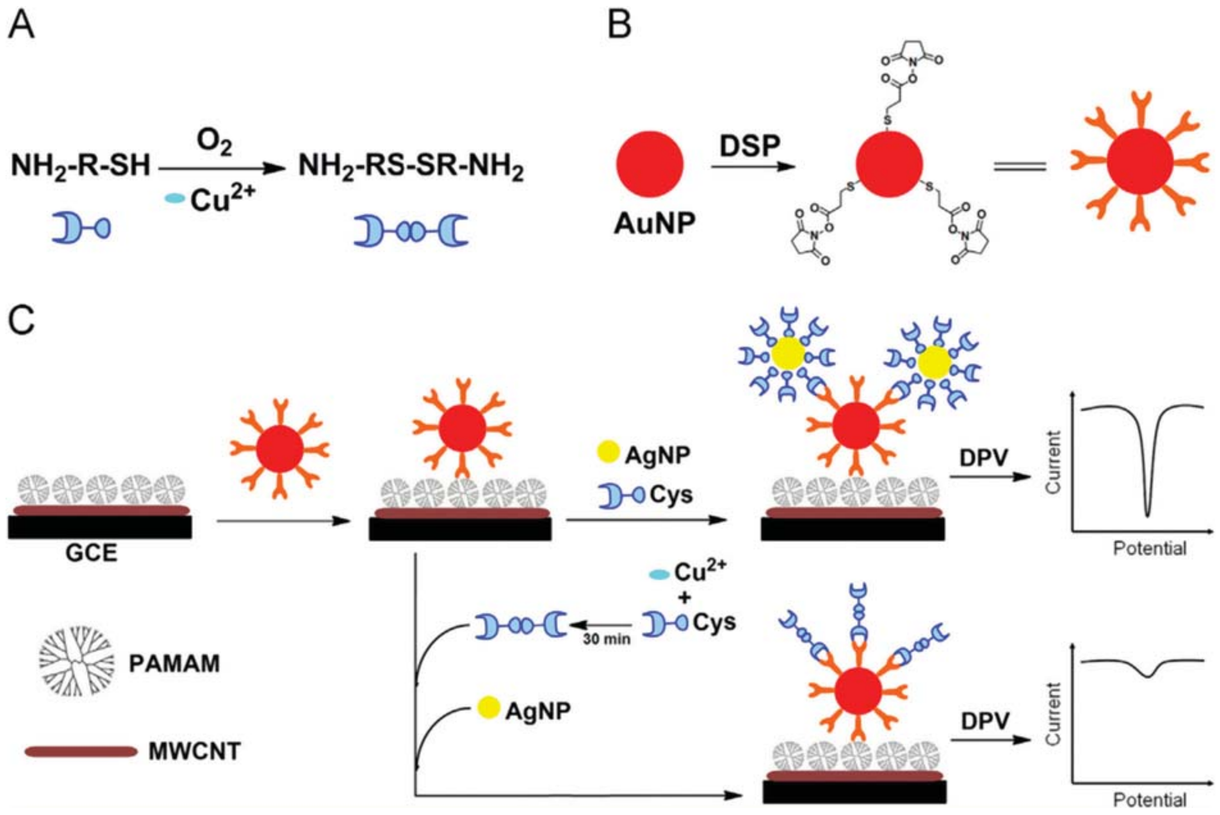

Fig. 14. Schematic drawing of (A) Cu^{2+}-catalyzed oxidation of Cys to disulfide cystamine by O_2, (B) preparation of dithiobis[succinimidyl-propionate] encapsulated AuNPs and (C) electrochemical detection process of Cu^{2+}. Adopted from Ref. [367], Copyright (2014), with permission of Elsevier.

4.2.3. Copper ions

Avenues have emerged making use of electrochemical approach supporting with AuNPs for Cu^{2+} detection due to the similar properties with Pb^{2+}. Cu^{2+} ions prefer to bind with ligands containing N donor atoms like cysteine, Cys, and aptamer, etc. Based on this theory, aminoundecylthiol base layer modified GE supporting with cysteine-functionalized AuNPs was fabricated via CV analysis [364]. Another example is the protocol that two partial complementary scaffold DNA strands are linked to AuNPs, which attach on the surface of GE through —SH of 4-aminothiophenol [365]. Upon Cu^{2+} binding, self-cleaving occurs at two different locations of the recognition strand into three fragments, driving $[Fe(CN)_6]^{3-/4-}$ to approach the surface of electrode and produce redox signal. AuNPs are used to increase the surface area of the electrode and anchor more aptamers, which helps obtaining the LOD of 0.1 pM for Cu^{2+} via SWV. On the basis of the same mechanism that the distance between signal tag and electrode surface determines the electrochemical signal producing or not, an ultrasensitive DNA biosensor through electroactive MA–Cu^{2+} complex attached on the end of hairpin-like probe using AuNPs as the signal amplification platform has been introduced [366]. When complementary chain presented, the distance of signal tag and electrode surface was so far that the response of CV signal was broken off, which leading the intensity of signal was proportional to the concentration of Cu^{2+}. Besides N atom-based coordination, some electrochemical sensors with the catalytic reaction are prepared. As shown in Fig. 14, Jumade group use the interaction between Cu^{2+} and Cys to selectively detect Cu^{2+} via copper-catalyzed oxidation of thiol compounds (R—SH) to disulfide compounds (R—S—S—R) [367]. For all of the proposals mentioned above using AuNPs, few of them have paid their attention to the size effects of AuNPs. Interestingly, taking the advantage of 13 and 45 nm AuNPs, Foroushani et al. [368] prepared a novel ultrasensitive Cu^{2+} sensor based on L-cysteine-functionalized core-satellite AuNPs networks to obtain tunneling the recognition current.

4.2.4. Bimetallic ions

Simultaneously detecting two kinds of metal ions (bimetallic ions) supported by AuNPs is the advantage of electrochemical techniques. Most of them use stripping voltammograms technique, as the quantitative range of metal ions is calculated by the changes of current peak value in accordance with concentration of metal ions. That is to say, as long as the changes of current peak value under a certain potential are regular, many kinds of ions can be detected simultaneously. But it is worth noting that these ions should have similar properties. For example, Ting et al. [369] demonstrated a sensitive electrochemical detection method for Hg^{2+} and Cu^{2+} using Graphene quantum dots (GQDs)-functionalized AuNPs modified GCE with ASV. In this sensor, the —COOH of Cys, which capped AuNPs, interacted with Hg^{2+} in the form of R—COO—(Hg^{2+})—OOC—R and Cu^{2+} competed for the limited binding sites on the surface of electrode. Importantly, Cu^{2+} has higher binding affinity. Thus, a peak of stripping potential at 0.42 V and 0 V was observed for Hg^{2+} and Cu^{2+} respectively and the peak intensity of Cu^{2+} was higher. Another typical kind of bimetallic detection is for Cu^{2+} and Pb^{2+}. As mentioned above, Cu^{2+} and Pb^{2+} have some similar properties, which provide excellent strategy for determining them at the meantime. Wang et al. [370] developed a L-aspartic acid/L-cysteine/AuNPs-modified microelectrode for parallel detection of Cu^{2+} and Pb^{2+} using SWV with the peak potential locating at 0.27 V and −0.09 V, respectively. The complex of —COOH and AuNPs leads to satisfied sensitivity and selectivity. Other supporting materials equipped AuNPs including carbon-based materials like GO, single-walled carbon nanotubes (SWCNTs), and carbon foams have been used for bimetallic detection as well. The sensitivity and selectivity are acceptable. More information of these researches is exhibited in Table S8. Recently, development for simultaneous recognition of metal ions and other inorganic salt has been proposed. Bui et al. [371] reported the first dual functional electrochemical sensor based on selenium particles (SePs) and AuNPs-functionalized disposable carbon paper electrodes for Hg^{2+} and NO_3^- detection in water. The main mechanism for Hg^{2+} detection was the high binding affinity between SePs and Hg^{2+}, and AuNPs here served as a nucleation sites for Hg^{2+}.

4.3. Detection of organic environmental hormones

4.3.1. Pesticides

The electrochemical detection of pesticides based on AuNPs as the signal tags-supported electrodes is one of the most popular techniques since it provides high sensitivity, super selectivity, low LOD, and fast responses [372,373]. Colorimetric method mentioned above mostly uses AChE due to OPs can inhibit the catalytic activity of AChE. On this basis, AChE inhibition-based electrochemical biosensor has also been developed. Using AuNPs offers faster signal transduction and larger surface area, which improves the performance of sensor [374]. Du group have made a tremendous contribution to detection of pesticides based on electrochemical method. In 2011, they designed a versatile processing approach for biosensing of organophosphorus compounds with in situ AChE–AuNPs immobilization [375]. When OPs presented, the electrochemical signal became weakened, resulting in the intensity decreasing proportional to the concentration of OPs. Further, they carried out a nanohybrid strategy using AuNPs, polypyrrole, and rGO as the hybrids (Au–PPy–rGO) based on the same property to enhance the sensitivity. The Au–PPy–rGO nanocomposite exhibited excellent conductivity and high electrocatalytic activity for AChE, and thus obtaining a wide detection range of 1.0–5.0 μM [376]. An AChE/AuNPs/rGO-based sensor for detecting paraoxon by using 'all in one' hybrid synthesis strategy in the presence of PDDA was developed [377]. Compared with many steps synthesis, this method is simpler and faster for assessing low-dose exposure to paraoxon. Wei et al. [378] reported a highly sensitive DPV biosensor for determination of chlorpyrifos and methyl parathion using an AChE enzyme immobilized boron-doped diamond electrode modified with AuNPs and carbon spheres (AuNPs–CS). As many OPs have the ability to inhibit the catalysis of AChE, the selectivity of this method should be improved in the future.

The electrochemical immunosensors have attracted significant interest for pesticides detection because of their high sensitivity, inherent miniaturization, and low cost [379]. In this field, carbon and noble nanomaterials especially AuNPs have been used extensively [380]. In addition, inhibition biosensors based on AChE are not selective to OPs because AChE itself is the target of carbamate pesticide. Thus, Du and the coworkers also turned to seek effective method to solve this problem. An amperometric biosensor for methyl parathion detection was developed by using a dual-signal amplification, which included a large amount of enzyme attached on the electrode surface because of the CdTe QDs covalent attachment and enzymatic catalysis improvement in the present of AuNPs and MWCNTs [381]. Unlike the AChE-based sensor, this sensor can be potentially reused and selectively detect methyl parathion with LOD of 1.0 ng mL^{-1} in garlic samples. Compared with the enzyme inhibition biosensor, the sensitivity should be enhanced further. Hence, for sensitive and specific detection of methyl parathion, they demonstrated a nanocomposite biosensor based on formation of AuNPs on silica particles and mixing with MWCNTs. It obtained linear concentration of methyl parathion in the range of 0.001–5.0 μg mL^{-1} with the LOD of 0.3 ng mL^{-1}, which was lower than the amperometric biosensor [382]. Additionally, they presented the first report on $Fe_3O_4@TiO_2$ magnetic nanoparticles-based immunosensor combining with multi-enzyme, including HRP and anti-BChE antibody (Ab), labeled AuNPs amplification strategy for selective and sensitive detection of OPs [383]. The magnetic nanoparticles not only provided easy separation of samples, but also overcame the difficult availability of commercial OP-specific antibody. The immunosensor for sensitive and selective detection of herbicide, another typical pesticide, has also been well developed. A rapid disposable immunomembrane-based electrochemical sensor for sensitive and selective detection of picloram in the agricultural field samples was developed by our team [258]. This sensor possessed good reproducibility for fabrication in batch and provided a congenial microenvironment for picloram. AuNPs shuttled the electron

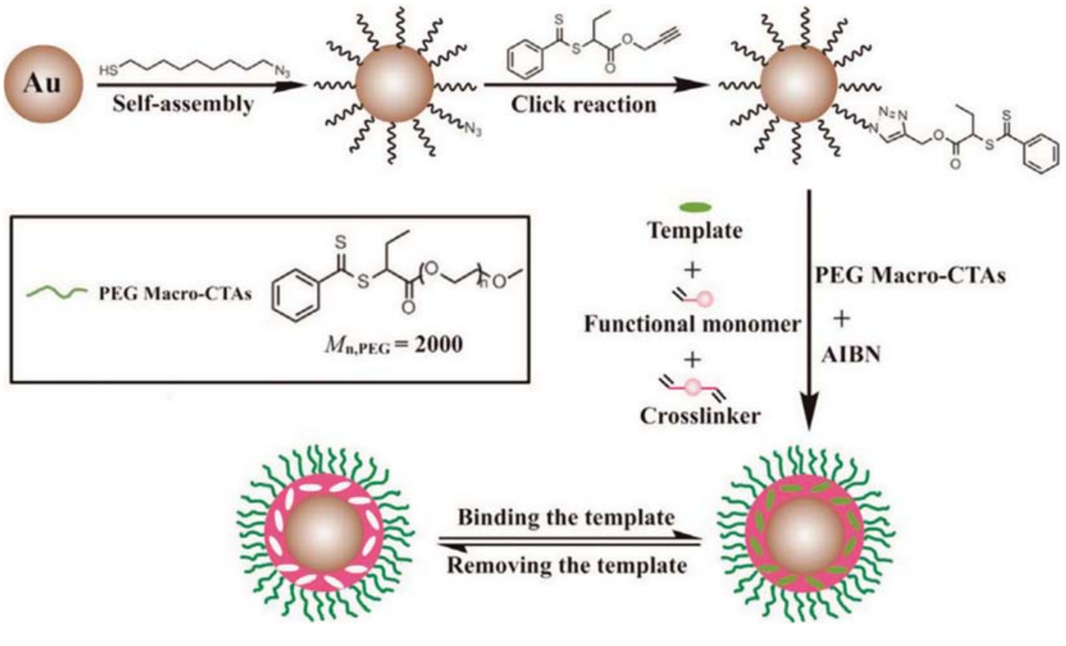

Fig. 15. Preparation of surface-imprinted polymer coated AuNPs grafted with water-compatible external layer via reversible addition–fragmentation chain transfer precipitation polymerization. Adopted from Ref. [397], Copyright (2014), with permission of Elsevier.

transfer between the electrode surface and immobilized enzyme to improve the sensitivity. The immunosensor based on antigen–antibody reaction has been developed for estimation of atrazine and diuron, etc. AuNPs serve as the electronic transmitters or antibody attachment and promote high sensitivity and excellent selectivity [384–386]. These methods all successfully detect herbicide in plant samples that provides tremendous future for other pesticides analysis in practice. Recently, an effective ECL aptasensor switch for aldicarb detection has been introduced on the basis of energy transfer, which occurs between AuNPs and the ECL signal indicator (Ru(bpy)$_3^{2+}$). The signal is enhanced because of effective energy transfer [387].

Electrochemical designers based on AuNPs and other materials such as DNA [388], Nafion film [389,390], graphene nanosheets [391,392], and Ca^{2+} cross linked pectin [35] modified electrode have been developed for highly sensitive recognition of pesticides. However, the selectivity of these sensors is still needed to be improved. Another electrochemical strategy like MIPs-based sensor has been reported to overcome this shortcoming due to its excellent selectivity [393,394]. The MIP techniques usually include preassembled template molecule, functional monomer, and final copolymerization with the cross-linking monomers [395]. The recognition sites for analysis of target are generated by removing template molecules from the polymer matrix. In other words, the recognition sites directly decide the sensitivity. Hence, how to provide more recognition sites is crucial. The simplest way is to introduce a larger surface area electrode, via the immobilization of AuNPs on the surface. In this respect, Sun et al. [396] introduced a visible light PEC sensing platform for selective pentachlorophenol detection by combining the MIP technique and microfluidic paper-based analytical device (μ-PAD). As shown in Fig. 15, a water-compatible surface-MIPs based sensor prepared on AuNPs was synthesized via click chemistry and reversible addition–fragmentation chain transfer precipitation polymerization (RAFTPP) for highly selective and sensitive detection of fenitrothion [397]. When this system immobilized on the ionic liquid-functionalized graphene coated GCE, the resulting electrode provided the linear range of 0.01–5 μM, with the sensitivity and LOD of 6.1 μA μM^{-1} mm^2 and 8 nM, respectively.

4.3.2. Drugs

The detection of drugs that used in animals and humans based on AuNPs supported electrochemical sensor can be divided into three parts: ligands combined with AuNPs, carbon-based material decorated AuNPs, and aptamer sensors. It is well known that thiol ligands can capture AuNPs via Au—S bond and this is widely used for sensors [398]. Gevaerd et al. [399] prepared a modified biphasic method for diltiazem detection based on dodecanethiol-captured different size of AuNPs and investigated the electrochemical activity through changing the molar ratio of Au: thiol. AuNPs decorated poly-MA modified GCE has been used for CV detection of domperidone in pharmaceuticals and biological fluids as urine and serum [400]. The response of this sensor shows stable, selective, and sensitive with the linear range of 0.05–100 μM and a LOD of 6 nM. Recently, AuNPs-supported porous covalent organic polymers (COPs) applied to sensors have received attention due to the high surface area, porosity, high stability, and nitrogen content of COPs, which are very favorable for the decoration of nanoparticles distribution without aggregation [401]. Typical examples are the studies reported by Vilian et al. Initially, they reported a paper for vanillin detection based on palladium nanoparticles supporting porous aromatic frameworks (Pd/PAF-6) and received good reproducibility, high selectivity, as well as good stability [402]. In light of the role of PAF-6, they further demonstrated an AuNPs decorated PAF-6 modified electrode for determination of quercetin (QC) via electrocatalytic oxidation of QC at reduced potential and obtained remarkable analytical performance [403]. The Au–PAF-6 modified electrode showed a good DPV response in the QC concentration of 1–600 pM with the LOD of 0.2 pM.

Carbon-based materials such as GO, graphene sheets, and WCNTs equipped with AuNPs have gained much attention in electrochemical sensor because of their properties of direct electron transfer and large availability of active sites [404]. Pruneanu et al. [405] has developed a novel carbamazepine sensor with the help of graphene–AuNPs/MgO composite-deposited electrode, which shows excellent oxidation toward carbamazepine. However, the selectivity should be further investigated. In terms of the selectivity, a new generation electrochemical sensor using electrochemical oxidation of graphene nanosheets/AuNPs/Nafion nanocomposite

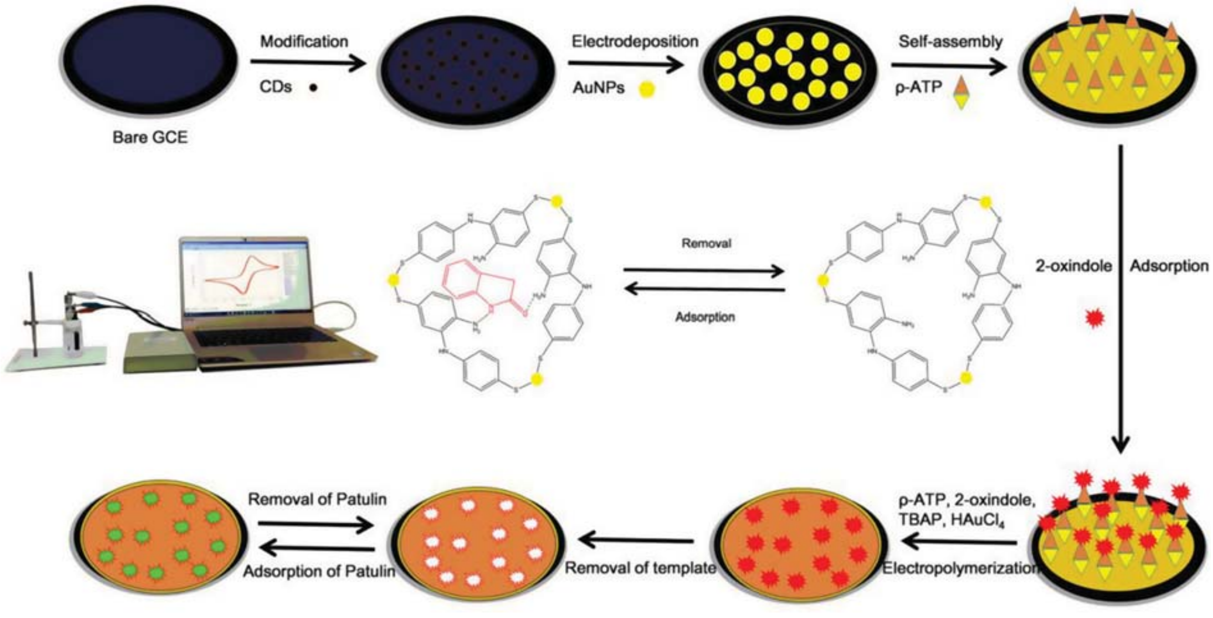

Fig. 16. Schematic illustrations of the fabrication procedure of chitosan and carbon dots combined with AuNPs to modify MIP sensor for patulin recognition. Adopted from Ref. [408], Copyright 2017, with permission of Elsevier.

has been developed for selective determination of silodosin for the first time [406]. Compared with the voltammetric response of 400-fold concentration of other molecules, the nanocomposite-modified electrode has satisfied selectivity. As a graphite-like structure, graphite-like carbon nitride (g-C_3N_4) is good candidate for potential application on sensors. AuNPs functionalized g-C_3N_4 nanosheets was introduced to amplify the ECL signal for penicillamine enantiomers detection [407]. This strategy exhibited high selectivity, stability and reproducibility and opened a new door based on ECL to discriminate drugs. MIP technique is also applied to detect patulin using carbon dots, chitosan, combined with AuNPs (Fig. 16) [408]. This strategy provides a wide detection range of 1–1000 pM with a low LOD of 7.57 pM.

Aptamer-based biosensors for picomolar detection of drugs have been proposed [36,409]. In addition to the promising sensors for one drug, a novel "dual potential" ECL aptasensor using CdS QD and luminol–AuNPs as labels for simultaneous determination of chloramphenicol and malachite green has been reported by Feng et al. [410]. Different with other dual potential sensors, this designed sensor is consisted of two screen-printed carbon WE for simultaneous detection in one single assay. AuNPs are deposited on electrode to immobilize luminol, which hybridizes with the complementary DNA sequences. This procedure provides high sensitivity with the linear ranges of 0.2–150 nM and 0.1–100 nM for chloramphenicol and malachite green respectively. Meanwhile, it has successfully applied to real fish samples, which holds great potential in the food analysis. Besides the "dual potential" ECL, a triple-amplification ECL for detection of chloramphenicol was developed [411]. Because of the SPR of AuNPs, the ECL intensity was effectively enhanced. Interestingly, incorporating the merits of ECL and MIP, a new MIP–ECL sensor was creatively introduced for sensitive detection of fumonisin B_1, where AuNPs greatly amplified the ECL signal of MIP on the basis of LSPR and electrochemical effect [412]. New methods not only cooperate different techniques, but also combine bimetallic NPs have been developed. For instance, a recent paper reported by Hu et al. has introduced an ECL brombuterol immunoassay using Ag@Au core shell NPs as the substrate [413]. AuNPs are used to facilitate electron transfer, as well as capture more biomolecules. This is a novel tendency for target analysis.

4.3.3. Other organic environmental hormones

A considerable effort of studies are devoted to explore effective methods which can be used to detect trace organic environmental hormones like polycyclic aromatic hydrocarbons (PAHs), phenolic, and nitroaromatic compounds due to their inherent property of poisonousness, persistence, bioaccumulation, and non-biodegradable [414–419]. Electrochemical strategy supported by AuNPs provides a novel effective approach, where AuNPs have high effective surface area and excellent catalysis properties, enhancing electrode conductivity and facilitating the electron transfer [420,421]. Kang et al. [415] carried out a PEC immunosensor for ultrasensitive detection of PAH amplified by bifunctional AuNPs modified with PAH antigen and HRP. The electron transfer prompted by HRP-catalyzed reaction caused an excellent photocatalytic performance, which achieved linear range of 0.315 pM to 3.15 nM on the logarithm of Benzo(a)anthracene (BaP) concentration and LOD of 0.315 pM. Another interesting electrochemical immunosensor for bisphenol-A (BPA) determination via competition and displacement assays has been developed [422]. Other immunosensors are rarely focused on the comparison of different assay formats, limiting the application of immunosensor. Through comparison, the results demonstrated that the displacement assay took longer time to detect target but LOD was much lower than the competition assay. The electrochemical oxidation and photocatalytic technique of AuNPs-based electrochemistry for pollutant treatment have been used recently owing to their effective surface area of resulting electrode and high-efficiency [416]. The most interesting is Hu et al. performed a photorefreshable and photoenhanced electrochemical sensing platform for BPA recognition on the basis of AuNPs decorated carbon doped TiO_2 nanotube assays [423]. Under UV irradiation, the performance of resulting electrode was significantly improved because it provided fresh reaction surface continuously. The photogenerated electron-hole pairs, which derived from the consumption of holes by BPA, improved the separation efficiency and further increased photocurrent. It is a promising sensor for long-time BPA monitoring.

MIPs and ECL-based electrochemical sensor has been reported. The details of these sensors are reviewed in Table S9. However, it is worth noting that the papers reported by Yuan et al. [424] and Lu et al. [425], which both use the L-Cys assembled AuNPs-modified electrode for detection of various kinds of phenolic compounds at the same time. This is a new trend for electrochemical sensor, but it also can be regarded that the selectivity should be further improved in the future. The material employed as an electrochemical sensor consists of AuNPs supporting by polymer, which has been used for organic contaminant detection successfully. In 2011, Liu et al. [426] demonstrated that on polyacrylonitrile electrospun nanofibers, AuNPs could be synthesized simply and effectively and exhibited excellent catalytic effect on the ECL of Ru $(bpy)_3^{2+}$. Recently, Li et al. [427] have provided a similar strategy supporting by poly(caffeic acid)/AuNPs composite modified GCE to detect acetaminophen effectively by using electrocatalytic activity of modified electrode toward the oxidation of acetaminophen. The response of catalytic current versus target concentration exhibits two linear segments in the ranges of 0.2–20 μM and 50–1000 μM. It has been successfully applied and validated by analyzing acetaminophen in urine, blood and pharmaceutical samples. Making use of polydopamine, MWCNTs and AuNPs composites modified electrode, a selective sensor for hydroquinone and catechol monitoring has been developed by Wang and his group [428], in which MWCNTs provide large specific area and AuNPs offer superexcellent electroconductibility. It is found that under optimal conditions, the CV and DPV can be used to perform the response of hydroquinone and catechol separately. Compared with other approaches, this strategy shows good stability and reproducibility.

5. Summary and outlook

Detection of chemical and biological molecules plays important role in medical, industrial, and environmental researches. Unique properties of nanoparticle materials make them have tremendous potential in creating new sensors and improving the sensitivity and selectivity of sensors. This review has provided a brief overview of the history of AuNPs from discovery to application in sensing fields, the fabrication of AuNPs and sensors supported by AuNPs for colorimetric and electrochemical detection in the past few years. There are many wonderful researches on the synthesis of AuNPs, as well as the control of size, shape, and application in various fields. They have made a significant contribution to the application of AuNPs. Turkevich–Frens and Brust–Schiffrin methods are still the most popular approaches because the synthetic AuNPs are uniform, stable, and regular, as well as easy to operate. The seed-mediated growth method provides a good idea for fabricating core–shell coated nanoparticles. And green strategies make excellent contribution to biological and medical researches. However, it is still necessary to develop synthesis approaches for regular, uniform, stable, and non-toxic AuNPs because many factors including pH, ligand, solvent, temperature, ionic strength, and reaction time may have a side effect on AuNPs. It is difficult to obtain large size of AuNPs, which can well disperse in aqueous solution. Non-toxic strategies are more and more popular and more suitable approaches synthesizing AuNPs for clinical research are well needed.

Research on AuNPs is obviously evolving rapidly. It takes a long time for AuNPs to be applied in chemical and biological sensing and analysis since it was discovered. The advantages of well stability, biocompatibility to aqueous medium, easy to surface functionalization, huge surface area, and fast electron transport have allowed them preferred candidates in colorimetric and electrochemical sensing compared to the conventionally used techniques. It provides an effective platform for cost-effective and feasible analysis by overcoming the traditional techniques that contain lengthy protocols and complicated instrumentation. Particularly, many of the sensing systems offer LOD at low level even aM level for analytes testing. Recently, many of the developed sensors were successfully applied to determine the analytes in real-life samples not only in environmental waters, but also in biological samples. Owing to the unique optical properties (i.e., resonance light scattering and SPR absorption) of AuNPs, colorimetric sensor based on analytes-induced AuNPs aggregation and anti-aggregation has well developed for sensitive detection. It starts turning to detect organics and small molecules, not only inorganic ions. Interestingly, Au–Hg alloy, considered as unpopular reaction before, has been used in specific detection of Hg^{2+} due to the excellent catalytic activity. More and more new colorimetric strategies with excellent selectivity will be developed in the future. Colorimetric method is intuitive, sensitive, and simple, but requires high experimental conditions due to the easy aggregation of AuNPs. The strategies based on aggregation of AuNPs are easier to obtain but the selectivity is still needed to improve. The anti-aggregation approach has good selectivity, but the process is difficult to realize. In addition, the simultaneous detection of different kinds of analytes is a new trend, but the colorimetric sensor based on AuNPs are limited in this field. Hence, more sensitive, selective and simper colorimetric sensor is still necessary.

Electroanalytical method well overcomes some drawbacks of colorimetric sensor. It provides satisfied sensing platform for simultaneous monitoring of different kinds of analytes, but is also expected to have further development with higher sensitivity. In generally, AuNPs are widely used in electrochemical sensor as the assisting portion. Their larger specific surface areas, small dimensions, and effective increase in electron transfer rate between the base electrode and analytes can bring more recognition sites, leading to more rapid and sensitive current response. Functionalized AuNPs may act as both signal transducer and molecular receptor in one single sensing platform, thereby simplifying the sensor design and enhancing the sensitivity. In addition, some electrochemical sensors prefer to search the electro-catalytic performance of AuNPs and make it in the sensing system, especially the redox property. Such effort results in robust development of AuNPs-based sensor. The electrochemical sensor are ultrasensitive and highly selective, it can be used for various analytes simultaneously. However, it is more complex and tedious for procedure when compared with the colorimetric sensor.

Currently, the single detection of one contaminant has been well developed, but the combined pollution detection is just at the start line. The portable devices have a great interest in the determination of both biological and environmental analytes. Recognition of more biological fluids not only blood, such as sweat, saliva, tears, is being considered for developing portable sensors. On the other hand, it will make a big difference if the biomolecules such as DNA can be monitored accurately with the wearable sensors, which can be applied to criminal investigation. In the near future, it is very expected that AuNPs-supported materials with lower detection limits, better sensitivity, and selectivity with high robustness will be developed as sensing materials. In terms of commercialization, the development of effective sensor with reusability and system integration and mass production is very necessary. Strategies simultaneously detecting vary kinds of analytes such as heavy metal ions, anions, organic contaminants, small molecules, and nucleic acids etc., supported by AuNPs and other nanomaterials based nanocomposites are worth trying. Incorporation of different analysis methods including colorimetric, electrochemical or fluorescent approach is also a new trend and needed to be further investigated. Except for sensing, the investigation of catalysis and other properties of AuNPs can be combined. Furthermore, the long-term possible impacts of AuNPs supported materials on environment and human health should be pay attention.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at https://doi.org/10.1016/j.ccr.2018.01.006.

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