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REVIEW ARTICLE

Recent advances in functionalization of plasmonic nanostructures for optical sensing

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Abstract

This review summarizes the progress that has been made in the use of nanostructured SPR-based chemical sensors and biosensors. Following an introduction into the field, a first large section covers principles of nanomaterial-based SPR sensing, mainly on methods using noble metal nanoparticles (spheres, cubes, triangular plates, etc.). The next section covers methods for functionalization of plasmonic nanostructures, with subsections on functionalization using (a) amino acids and proteins; (b) oligonucleotides, (c) organic polymers, and (d) organic compounds. Several tables are presented that give an overview on the wealth of methods and materials published. A concluding section summarizes the current status, addresses current challenges, and gives an outlook on potential future trends. This review is not intended to be a comprehensive compilation of the literature in the field but rather is a systematic overview of the state of the art in surface chemistry of plasmonic nanostructures. The ability of various ligands and receptors for functionalization of nanoparticles as well as their sensing capability is discussed.

Keywords Biosensors · Nanosensors · Surface chemistry · Oligonucleotides · Nanocubes · Triangular plates · Biosensor · Nanotetrahedron · Nanooctohedron

Introduction

Environmental monitoring for rapid and accurate determination of pollutant contamination in water, soil and air is one of the most pressing issues in our age. Every year, million tons of hazardous chemicals are entering the environment; hence finding a simple, rapid, and accurate determination technique is pivotal. The mentioned hazardous compounds include heavy metals, organic and inorganic pollutants, and chemical toxins [1–3]. Currently, conventional determination methods for such analytes are atomic absorption spectroscopy (AAS), atomic emission spectroscopy (AES), inductively coupled plasma/mass spectroscopy (ICP-MS), and ultraviolet-visible (UV-Vis) spectroscopy. Despite the high sensitivity and selectivity, these methods often need high operating costs, long sample preparation time, and well-trained operators [4, 5]. Therefore, there is a multidisciplinary effort for developing chemical sensors with specific abilities such as in situ multiplexed analyte determination, portability, sensitivity, and selectivity. Chemical sensors have the ability to transform the obtained chemical information upon binding of molecular guests to analytical information through different mechanisms such as electrochemistry, fluorescence, absorption, and scattering [6–9]. Chemical sensors based on nanostructured materials have received a close attention, and among them, colorimetric sensors based on plasmonic nanostructures have gained a great interest [10–21]. Localized surface plasmon resonance (LSPR)-based sensors for rapid and accurate determination of different analytes have been sufficiently covered in the previous literatures, and it is not the scope of this review. Here in this review, we are going to focus on the surface chemistry of plasmonic nanostructures for sensing applications, which was not adequately addressed in previous studies. First, a brief introduction on the theory and background of SPR phenomenon is presented which is followed by a comprehensive review on different surface functionalizing agents. To the best of our knowledge, this aspect was not comprehensively studied by previous literatures in the field, and this extensive review can be used as a guideline for the researchers.

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Surface plasmon resonance

Conversion of photons into collective oscillation of noble metals' conduction band electrons is known as localized surface plasmon resonance. This resonance coupling has opened new applications for plasmonic nanostructures due to their extraordinary absorption and scattering profile at these specific frequencies. The resonance frequency is highly depended on size, shape, composition, and environment medium of nanostructure [22–24]. The localized plasmonic fields in the immediate vicinity of nanostructures are highly sensitive to even minor changes in their near-fields, and this is the basis for their versatile applications as plasmonic sensors.

Plasmonic nanostructures have the ability to scatter or absorb light with several orders of magnitude larger than their physical sizes. In this regard, plasmonic nanostructures in different shapes and sizes were exploited largely due to their distinct optical properties even in the same composition and size [25, 26] (see Fig.1). Silver and gold nanostructures have been studied more in this field mainly due to their *d-d* electron transitions, which take place in visible range. The superior characteristics of silver compared with gold can be inferred from Mie's solution of Maxwell's equation for a simple spherical particle (Eq. 1):

$$C_{ext} = \frac{24N\pi^2R^3\varepsilon_m^{3/2}}{\lambda ln(10)}\left[\frac{\varepsilon_i}{(\varepsilon_r + 2\varepsilon_m)^2 + \varepsilon_i^2}\right] \quad (1)$$

where C_{ext} is the extinction (absorption + elastic scattering) cross section, R is the radius of particle, and N is the electron density. Refractive index of medium, imaginary, and real terms of particle's dielectric function were also written as ε_m, ε_i, and ε_r, respectively. According to this equation, the maximum extinction cross-section can be obtained if both $\varepsilon_r = 2\varepsilon_m$ and $\varepsilon_i \approx 0$ conditions are met. We have previously shown that imaginary part of silver's dielectric function is

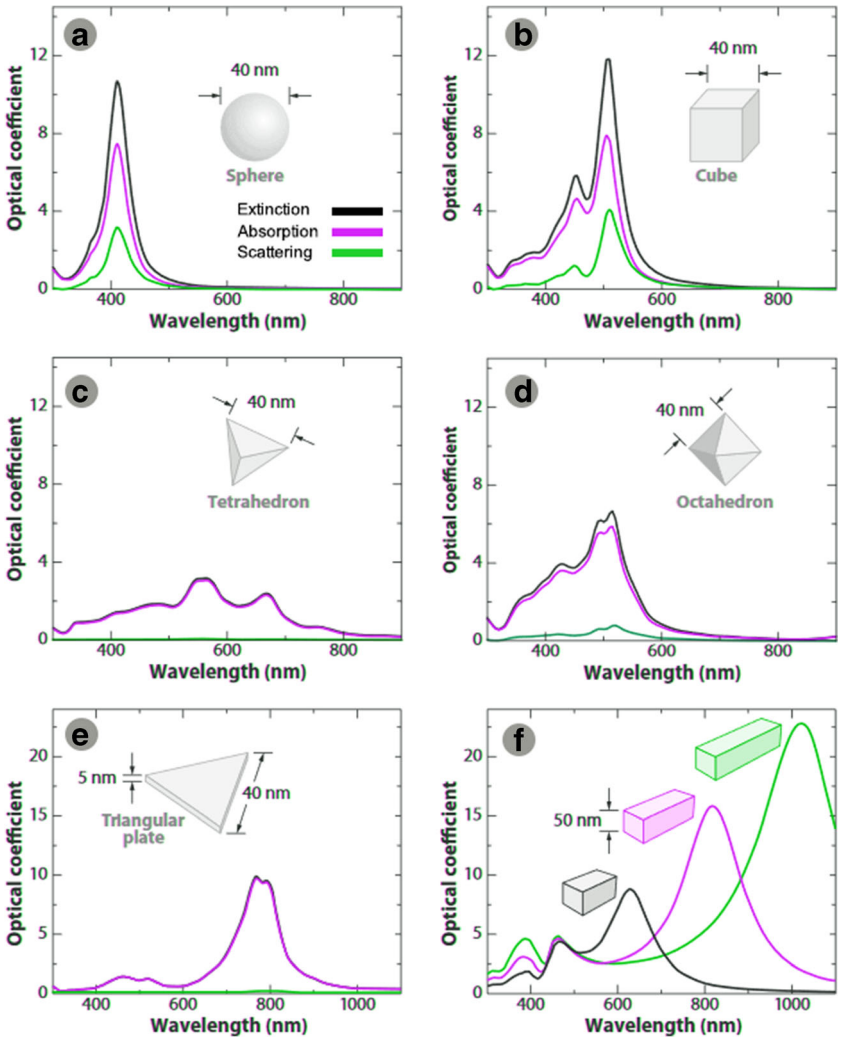

Fig. 1 Extinction (black), absorption (violet) and scattering (green) profiles of silver nanostructures with different shapes (a-f). Panel (f) shows the extinction profile of nano-bars with different aspect ratio of 2, 3, and 4 for black, violet, and green, respectively. Reproduced with permission from reference [25]

close to zero; also its imaginary part is more negative compared to gold [4]. In addition, the media in which the nanoparticles (NPs) are dispersed play a key role in their optical properties [27]. The optical extinction of the same nanoparticle in different media is not identical which can be attributed to the ε_m term in Eq. 1. These changes may occur in the intensity of SPR peak or spectral shift, but the main characteristic of the spectra (number of peaks and intensity ratio among different peaks) stays the same. However, from the sensing application point of view, the medium is not a parameter of choice, so the medium is not the main point of this review but these papers are suggested in this topic [28–30].

In our previous paper, we thoroughly reviewed the different mechanisms of plasmonic sensing, e. g., aggregation-based, oxidation-based and morphological-based detection mechanisms [4]. In this review, we have focused on functionalization of plasmonic nanostructures for sensing applications. Different categories of functional agents as well as their applications for plasmonic sensing were addressed and discussed in the following sections. The functionalization strategies of plasmonic nanostructures are different, but they mainly use thiol (-SH) and amine (-NH_2) groups for bonding to NP surface. Carboxylic (-COOH) and hydroxyl (-OH) groups are also used for this mean. The bonding of these groups on NPs surface is mainly described using hard and soft acid and base (HSAB) theory. Upon the interaction of functional agents with NP surface, the optical properties of NPs will change which can be observed as spectral shifts with possible decrease in the SPR peak intensity. In the following section, different functionalization strategies and the most recent works related to each category are discussed. The advantages and disadvantages of each strategy are also reviewed at the end of each section.

Functionalizing plasmonic nanostructures for sensing applications

Functionalizing ligands on the surface of plasmonic nanostructures can be classified on the basis of different criteria such as the ligand composition, target analyte, and its reaction with the nanoparticle. In this review, we have emphasized on the nature of the functional ligands and categorized them in four different groups namely as oligonucleotides, proteins and amino acids, organic polymers, and organic compounds (Fig. 2). The main advantage of such approach for classification is the direct comparison of different groups' ability for sensing applications. It has been demonstrated that plasmonic nanostructures (mainly gold and silver due to their LSPR in visible region) have strong binding affinity to many chemical and biochemical compounds which makes the functionalization process robust. In the following section, different common functional groups exploited for sensing purposes will be reviewed and discussed [31].

Functionalization using amino acids and proteins

Proteins are three-dimensional (3D) macromolecules consisting amino acids as building blocks. The 3D structure of protein highly depends on the physicochemical condition of the medium such as pH, salt concentration, and temperature [32, 33]. The size of proteins is in the range of 1–100 nm; hence they are classified as nanoparticles and are considered as ideal candidates for functionalization of nanostructures [34, 35]. Proteins have four different level structures, namely as primary, secondary, tertiary, and quaternary, which the former is the most important from the NPs interaction point of view. The primary structure of proteins which containing a set of 20 amino acids has different functional groups such as amino–NH_2 (lysine), carboxylic acid–COOH (aspartic, glutamic), hydroxyl–OH (serine, tyrosine), and –SH (cysteine) [36–38]. The functionalization of NPs with amino acids or proteins is usually endeavored using amine, carboxylic acid, hydroxyl, and thiol moieties available in their structures.

For instance, Jeevika et al. [39] have synthesized a colorimetric probe for determination of mercury ions by using gelatin functionalized silver nanoparticles (AgNPs) with a limit of detection of 25 nM. Upon the addition of Hg^{2+} to the gelatin functionalized AgNP colloids, a complete color change from yellow to colorless was observed due to redox reaction between silver and mercury and led to the aggregation and formation of silver and mercury (Ag/Hg) amalgam. Zhao and colleagues [40] have introduced a colorimetric sensor based on gold nanoclusters functionalized with glutathione for the determination of Cu^{2+} and Fe^{3+}. The detection limits of this sensor are 0.125 and 1.25 nM for Cu^{2+} and Fe^{3+}, respectively. In another study, a facile and selective optical sensor based on l-cysteine capped silver nanoparticles was developed for accurate determination of Hg^{2+} ions in aqueous solutions [26]. The synthesized AgNPs showed a high sensitivity in the range of 1×10^{-8} M. The selective response of l-cysteine-capped AgNPs towards Hg^{2+} ions is shown in Fig. 3(a), where other competing metal ions are not able to interact as much as mercury. The disappearance of the S-H vibrational band in the Fourier-transform infrared spectroscopy (FTIR) of AgNPs was attributed to the anchoring of l-cysteine to the AgNP surface via a thiolate linkage. Buduru et al. [43] have developed a simple, rapid, and sensitive colorimetric method with the determination limit of 0.90 μM for the determination of Hg^{2+} ion in water samples using glutamine (Gln)- and histidine (His)-functionalized silver nanoparticles (Gln-His-Ag NPs) as a probe. The stretching and vibrating bands of carboxylic and amino groups of Gln and His were shifted to lower and longer wave numbers, proving the interaction of Gln and His with the surfaces of AgNPs. Tyrosine-functionalized gold nanoparticles (AuNPs) were used to develop a colorimetric probe for the determination of Cr^{3+} and Pb^{2+} [28]. The LOD of Cr^{3+} and Pb^{2+} were found to be approximately 1 and 2 μM visually, respectively. Figure 3(b) illustrates the colorimetric sensing principle for the determination of Cr^{3+} and Pb^{2+} based on tyrosine-capped AuNPs. In the absence of Cr^{3+} or Pb^{2+}, AuNPs are well dispersed

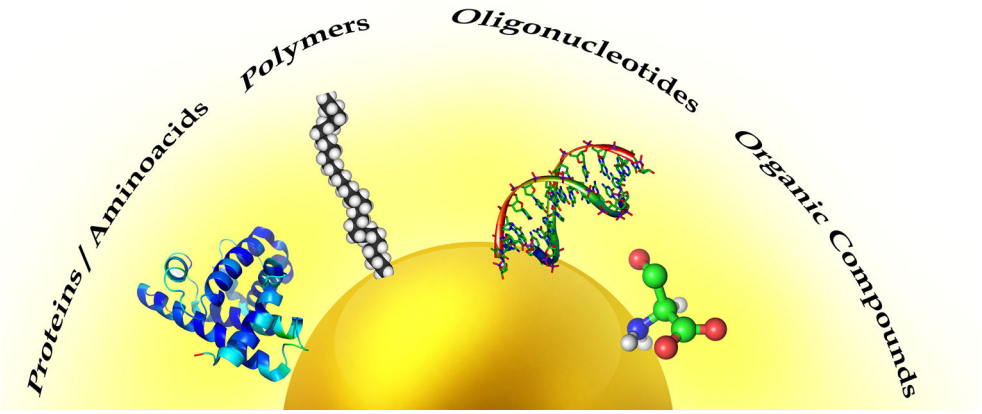

Fig. 2 Schematic representation of functional groups on plasmonic nanostructures for sensing application

in solution and the color of the solution remains red. Nevertheless, when Cr^{3+} or Pb^{2+} is added, their colors change from red to blue gray resulted from the aggregation of AuNPs induced by the binding between ions and tyrosine, accompanied by surface plasmon resonance (SPR) absorption peaks change in intensity and wavelength.

Ermini and co-workers [44] have synthesized peptide-functionalized gold nanoparticles as a biosensor for the determination of carcinoembryonic antigen (CEA) in blood plasma (Fig. 3(c)). It is shown that, for the same amount of target molecule, by tuning the surface properties of the peptide-functionalized NPs, it is possible to significantly enhance the sensor response for the

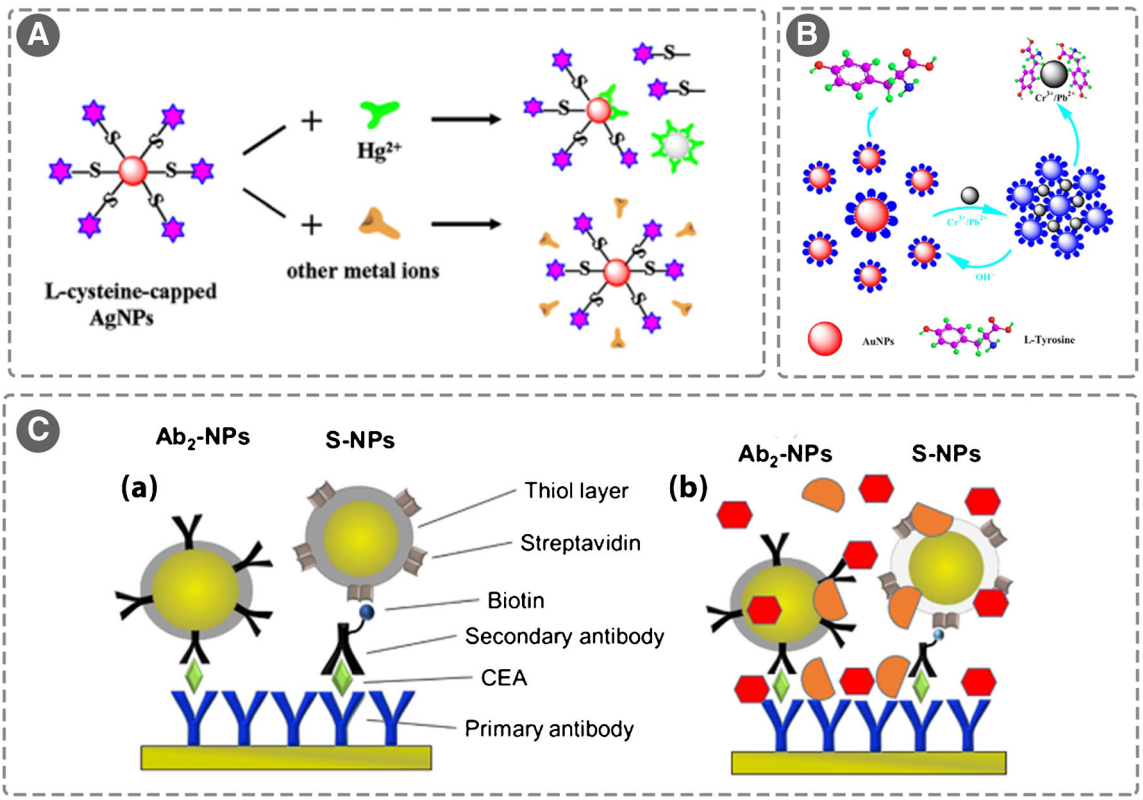

Fig. 3 (A) Schematic representation for the selective response of L-cysteine capped AgNPs towards mercury ions. Reproduced with permission from reference [41]. (B) Schematic illustration of Cr^{3+} and Pb^{2+} detection based on optical properties of AuNPs. Reproduced with permission from reference [42]. (C) Schematic of the sandwich assays using streptavidin functionalized NPs (S-NPs) and secondary antibody functionalized NPs (Ab_2-NPs). NPs for the determination of CEA in (a) phosphate buffer and (b) blood plasma. Reproduced with permission from reference [28]

target analyte. By using this SPR strategy, it is possible to distinguish the specific and non-specific interactions of analyte for in vivo applications. Satheeshkumar et al. [45] have proposed a label-free colorimetric assay for the determination of copper ions based on Tyrosine-functionalized silver nanoparticles with a linear range up to 10 μM. In this study, tyrosine has been used as both reducing and functionalizing agents. A photoactive species of tyrosine (Tyr) is used to reduce silver nanoparticle through a photochemical reaction, while the oxidized Tyr (Tyr^{Ox}) was exploited to functionalize the surface of the AgNPs at the same time. According to the FTIR measurements, the disappearance of the vibration band for phenolic C-O bending after functionalization may indicate the conversion of phenolic hydroxyl group in Tyr after photoreduction. In another study, L. D'souza and colleagues [46] have constructed a colorimetric sensor for determination of glutathione via ascorbic acid capped silver nanoparticles (AA-AgNPs) as the probe. The characteristic SPR peak of AA–AgNPs at 397 nm is redshifted to 468 nm only by the addition of a small amount of glutathione (GSH), resulting in a color change from yellow to orange-brown, which confirms the strong aggregation of AA–AgNPs by GSH due to the presence of multidonate anchoring groups (e.g., –SH, $–NH_2$ and $–COO^-$). The developed sensor had the ability to determine GSH in real samples. The interaction of AA molecules with AgNPs was proved by disappearance of –OH group stretching at 3212–3626 cm^{-1}. Tryptophan-functionalized gold nanoparticles were used for possible applications in detecting renal function deterioration by measuring Mg^{2+} concentrations in urine and artificial serum samples [32]. This assay has a rapid detection response of less than 1 min and a LOD of 0.2 μM. The visual detection was accompanied by the color change from purple to dark upon the addition of Mg^{2+}. Plasmonic nanostructures functionalized with antibodies are mostly endeavored due to their high sensitivity for detecting analytes in complex solutions. Therefore, this functionalization strategy was used for detecting several chemical and biochemical species such atrazine, C-reactive protein, protein biomarkers, tetracycline, human immunoglobulin G (hIgG), and diphtheria toxoid [47–55]. A comprehensive list of the recent works is summarized in Table 1.

The main advantage of functionalization using proteins and amino acids is the diversity of conjugation chemistry that can be implemented for a sensing mechanism. Several moieties are available in the structure of amino acids, peptides, and proteins, and other moieties can be also grafted by straightforward conjugation reactions. Highly sensitive sensors with the detection limits down to fM or pM could be designed using antibodies due to their specific interaction with the desired analyte.

Functionalization using oligonucleotides

Oligonucleotides are poly-nucleic acid chains made up from nitrogen-containing bases, five-carbon sugars, and phosphate groups. The monomers of oligos are adenosine (A), guanosine (G), cytidine (C), thymine (T), and uridine (U). The incorporation of specific ligands (such as thiol or amine modifications) at the 5′- or 3′-terminal of oligonucleotides enables the interaction of oligos with plasmonic nanoparticles. The easy synthesis process and the programmable assembly of oligonucleotides make them an ideal functionalization agent for NPs.

Zhu and co-workers [57] have proposed a facile Cr^{3+} and adenosine determination using the aptamer and 11-mercaptoundecanoic acid assembled gold nanoparticles. The detection limit of mentioned target is calculated to be 1.7×10^{-11} M and 1.8×10^{-8} M, respectively. The thiolated DNA and 11-mercaptoundecanoic acid (MUA) was simultaneously assembled to the surface of gold nanoparticles in one step by gold-sulfur interaction. The principle of detection was that Cr^{3+} bind preferentially with –COOH group in the structure of MUA through the chelation interaction. As a result, the interparticle distance of AuNPs was greatly decreased in the presence of Cr^{3+}, causing a red shift of the SPR peak and a visual color change from red to blue. Busayapongchai and Siri [58] developed a sensitive determination method for estradiol (E2) based on plasmonic properties of gold nanoparticles. This developed assay exhibited a wide dynamic range from 10^{-15} to 10^{-8} M for E2 determination. The mechanism of detection is based on the ligand binding domain of estrogen receptora (LBD-ERa) and gold nanoparticles using predesigned DNA aptamers. Jia et al. [59] have proposed a colorimetric sensing assay based on exonuclease I-triggered aggregation of DNA-functionalized gold nanoparticles for discriminations of different proteins. This sensor was able to discriminate 15 proteins with a detection limit of 10 nM in buffer solution and real serum samples. The oligonucleotides were immobilized on AuNP surfaces through the Au–S bond. Chu and colleagues [60] have introduced a facile method for the determination of mercury based on AuNPs and mercury-specific-oligonucleotide–conjugated resonators (MSOIRs) with a detection limit of 100 pM. The functionalization process of AuNPs is based on the binding of activated thiol groups at the end of DNAs and AuNPs surface (Fig. 4(a)). In another study, an optical biosensor was developed for the simultaneous detection of a variety of *Salmonella spp.* in environmental and food samples via oligonucleotide-functionalized gold nanoparticles [46]. This colorimetric sensor has a detection limit of < 10 CFU/mL for both pure culture and complex matrice setups. Highly specific oligonucleotides were designed and conjugated onto the surface of AuNPs via thiol linkage, $HS-(CH_2)_6$, which was initially introduced chemically to either 5′- or 3′- end of the oligonucleotide probes. Figure 4(b) shows the sensing strategy for positive and negative response for sensitive determination of *Salmonella spp.* using AuNPs.

Zou and colleagues [62] have constructed a novel colorimetric sensing assay for biomolecule detection which integrates the signal amplification of hybridization chain reaction

Table 1 Summary of previous efforts for functionalization of plasmonic nanostructures using proteins and amino acids for sensing purposes

Analyte	Functional ligand/moiety	Limit of detection (LOD)	References
Hg^{2+}	L-Cysteine/NM*	10^{-8} M	[41]
Hg^{2+}	Glutamine and histidine/carboxylic and amino groups	0.90 μM	[43]
Cr^{3+}, Pb^{2+}	Tyrosine/NM	1, 2 μM	[42]
Copper ion	Tyrosine/phenolic and hydroxyl groups	150 nM	[45]
Glutathione	Ascorbic acid/multi-donate anchoring groups (SH, $-NH_2$, $-COO^-$)	2.4×10^{-7} M	[46]
Mg^{2+}	Tryptophan/NM	0.2 μM	[56]
Atrazine	N-methacryloyl L-aspartic acid (MAAsp)/NM	0.7134 ng/mL	[54]
Hg^{2+}	Gelatin/amalgam (Hg-Ag)	25 nM	[39]
Cu^{2+}, Fe^{3+}	Glutathione/NM	0.125, 1.25 nM	[40]
Carcinoembryonic antigen (CEA)	Peptide/NM	NM	[44]
Bacterial pathogens	Cysteine modified synthetic antimicrobial peptides (sAMPs)/peptide bonding (S-Au)	10^2 CFU/mL	[52]
hlgG	Human immunoglobulin G antibody (anti-hlgG)/NM	11 ng mL^{-1}	[47]
IFX and ATI	TNFα	2.5 μg/mL	[48]
Tetracycline (TC)	Mercaptoundecanoic acid (MUA) and antibody pair (anti (TC))/NM	10 aM	[50]
Alpha-1 antitrypsin (AAT) and Tau 381	Mixed antibody (anti-AAT and anti-Tau)/NM	μM and fM scale	[51]
C-reactive protein (CRP)	Aptamer antibody/3′-thiol-modified 6th-62-40	10 pM	[53]
Diphtheria toxin (DT)	Monoclonal anti-diphtheria IgG/NM	10 ng/mL	[55]

*NM = not mentioned

(HCR) with the assembly of gold nanoparticles through triplex formation with the detection limit of 5 pM, 10 pM, 5 nM, and 20 pM for DNAs, microRNAs, ATPs, and PDGF-BBs, respectively. DNA hairpin probes can form rigid triplex structures with triplex-forming oligonucleotide (TFO)-functionalized AuNPs in the absence of targets, which will aggregate in the presence of biomolecule targets. The reviewed literatures are summarized in Table 2.

When the sensitivity of the sensor is the ultimate goal, oligonucleotide functionalization is the best choice. Due to the specific interactions with the desired analyte, high selectivity could be obtained with the LOD usually lower than nM concentrations. Even though the oligonucleotides for specific analytes are well known, a time- and cost-consuming process should take over if the specific oligonucleotide is unknown for the desired analyte. On the other hand, the assembly of oligonucleotide on the NP surface is not a tough job and can be realized using amine or thiol modification in the oligos.

Functionalization using polymers

Polymer-functionalized nanostructures are known for their ability to create the desired surface functionalities. Both synthetic and organic polymer-functionalized NPs are widely used for biosensing because of their non-toxic and non-immunogenic properties [63–65]. Maruthupandy and colleagues [66] proposed a simple method for the rapid colorimetric and visual detection of glucose molecules in water medium with a linear range from 5 to 100 μM using chitosan capped-AgNPs (CS/AgNPs). Silver nanoparticles were interacted with the O_2 from hydroxyl group in chitosan as well confirmed with FTIR spectroscopy. The interaction of glucose molecules with CS/AgNPs decreased the interparticle distance significantly. The spectral relation as well as visual change upon the addition of glucose is shown in Fig. 5.

Also, a rapid and simple colorimetric method based on the surface plasmon resonance of polyvinylpyrrolidone (PVP)-stabilized AgNPs was developed for the detection of the Timolol (a cardiovascular drug) by Amirjani et al. [67] with the LOD of 1.2×10^{-6} M. Based on the proposed mechanism, the chemisorption of the Timolol drug on the AgNPs via Ag-S binding induces the aggregation of AgNPs.

In another study, a localized surface plasmon resonance sensor based on gold nanorods functionalized with polyethylene glycol was developed for the determination of activated leukocyte cell adhesion molecule (ALCAM) cancer

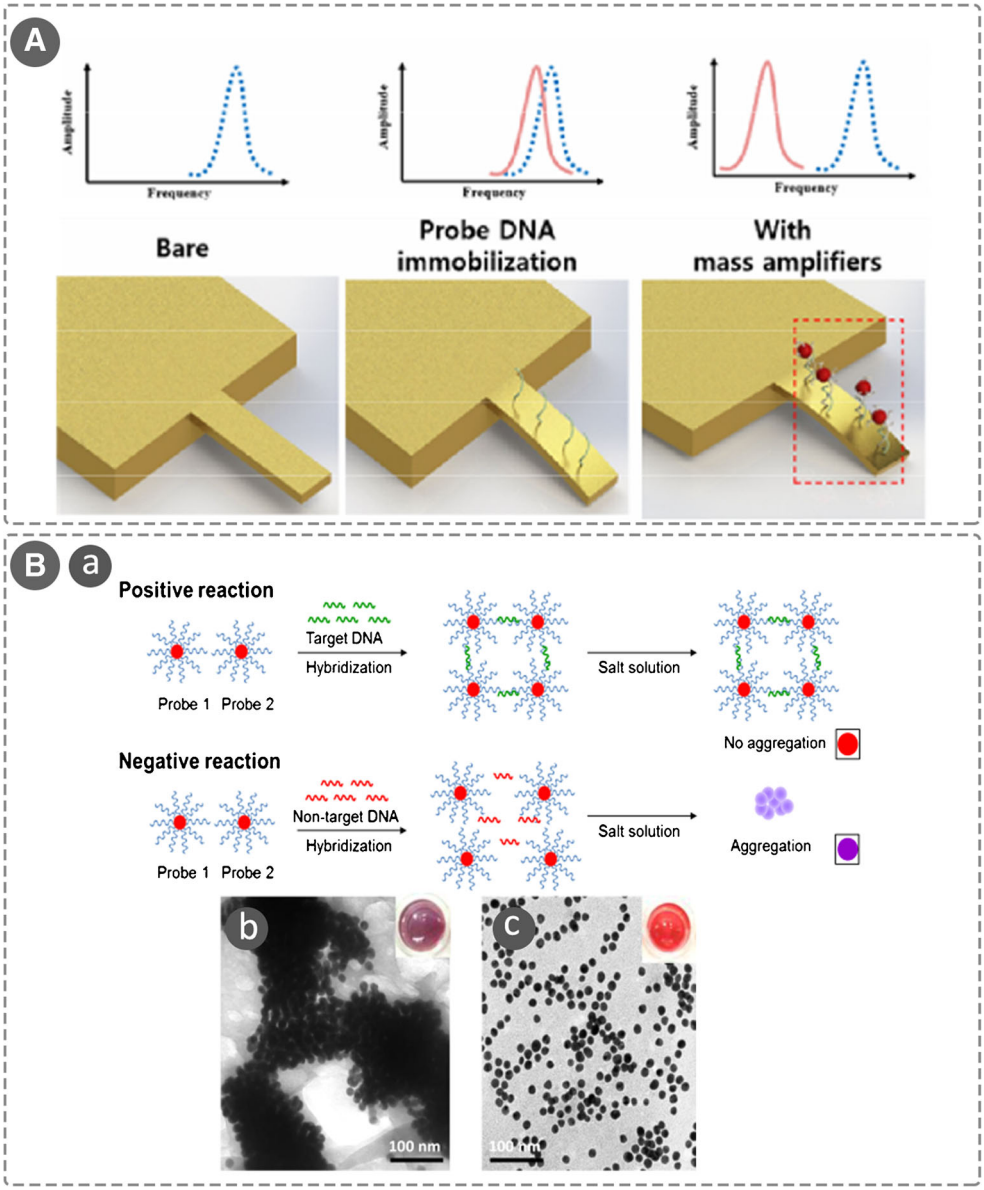

Fig. 4 (A) Schematic illustration of mercury ion detection using resonance frequency shift of a MSOIR on AuNPs surface. Reproduced with permission from reference [44]. (B) (a) Sensing strategy for colorimetric detection of *Salmonella spp*. using optical properties of aggregated versus non-aggregated AuNPs. The TEM images in (b) and (c) show the absence and presence of analyte in the solution, respectively. Reproduced with permission from reference [61]

Table 2 Summary of previous efforts for functionalization of plasmonic nanostructures using oligonucleotides for sensing purposes

Analyte	Functional ligand/moiety	Limit of detection (LOD)	Reference
Cr^{3+}, Adenosine	Aptamer and 11-mercaptoundecanoic acid / -COOH	1.7×10^{-11} M and 1.8×10^{-8} M	[57]
Estradiol (E2)	Estrogen receptora (LBD-Era)/specific interaction	2.62×10^{-14} M	[58]
15 different proteins	DNA sequence/NM*	10 nM	[59]
Mercury	Mercury-specific-oligonucleotide-conjugated resonators (MSOIRs)/NM	100 pM	[60]
Variety of *Salmonella* spp.	Specific oligonucleotides/NM	<10 CFU/mL	[61]
DNA, microRNAs, ATPs, PDGF-BBs	Specific oligonucleotides/NM	5 pM, 10 pM, 5 nM, 20 pM	[62]

*NM = not mentioned

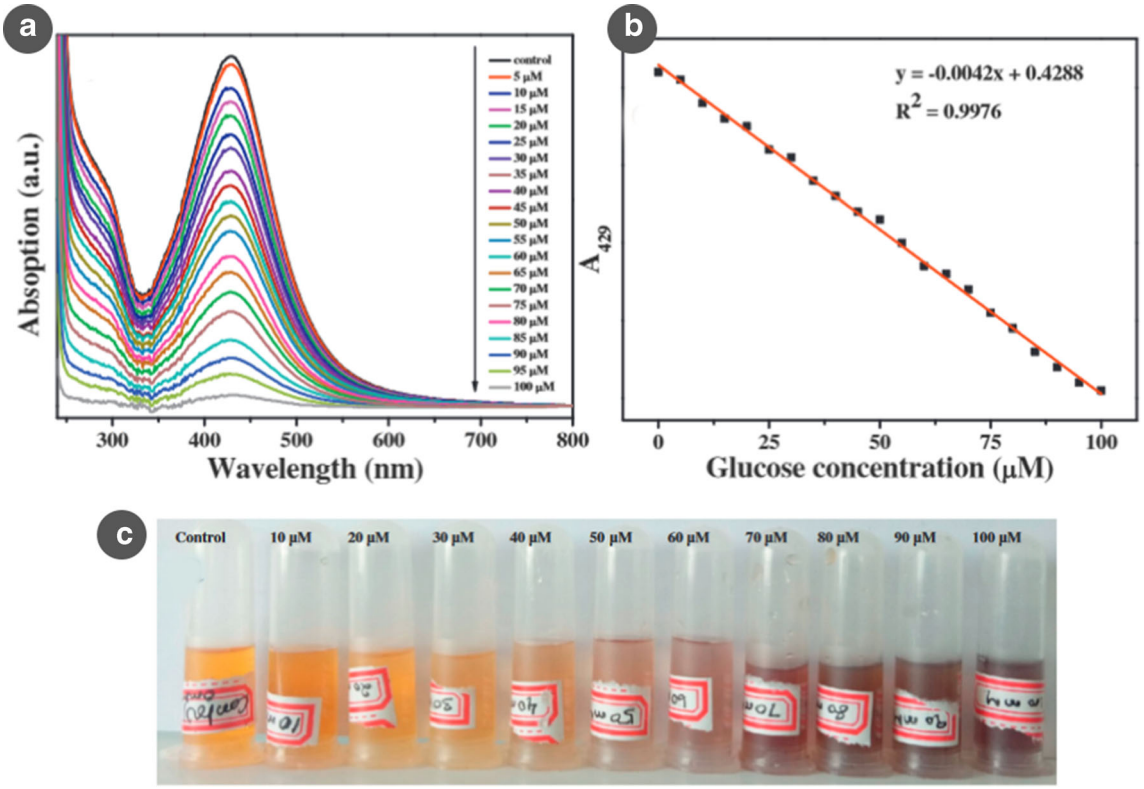

Fig. 5 (**a**) and (**b**) show the relationship between absorbance at 429 nm and the concentration of glucose in the solution in the range of 5–100 μM. (**c**) shows the visual color change in the presence of different glucose concentrations. Reproduced with permission from reference [66]

biomarker [53]. Both high- and low-molecular weight thiolated PEG molecules were used to provide effective steric hindrance as well as ample reactive groups for conjugation with the biomolecular probes. This strategy leads to increased sensitivity of the developed sensor and allowed the detection of the ALCAM antigen concentration down to 15 pM. A sensitive and selective Hg^{2+} optical sensor has been developed based on the redox interaction of Hg^{2+} with starch-coated silver nanoparticles in the presence of 0.005 $molL^{-1}$ HNO_3 by Vasileva and coworkers [68] with the detection limit of 0.9 μgL^{-1}. The formation of Ag-Hg amalgam due to the sorption and reduction of positively charged Hg^{2+} on the surface of negatively charged AgNPs was known as the responsible mechanism of this sensor (Fig. 6).

Buccolieri et al. [69] have developed a colorimetric sensor for ammonia sensing in aqueous solutions based on bio-synthesized AgNPs using sucralose and glucose. The mentioned sensor could detect ammonia in the range of 10^{-2} to 10^{3} ppm in aqueous solutions. In another study, Ban and colleagues [70] have developed a spectroscopy based method for sensing Hg^{2+} and cellular-free oxygen radical via starch-functionalized silver nanoparticles. Starch was used as the reducing agent as well as capping agent, and NaOH played the role of a catalyst for converting $AgNO_3$ to AgO_2 and Ag, respectively. It was observed that starch-functionalized AgNPs were highly sensitive to Hg^{2+} ions as reflected from the blue shift in the absorption spectra. In the presence of Hg^{2+}, the interaction between silver and mercury forms amalgam. Li et al. [71] have presented a paper-based colorimetric sensor using $–NH_2$ and –SH decorated AuNPs for rapid determination of Fe^{3+} ions. The leaching of gold nanoparticles in the presence of thiourea or hydrogen peroxide can speed up by using catalytic Fe^{3+} ions, and this method is capable to detect ferric ions as low as 0.85 μM. In another study, poly(allylamine hydrochloride) (PAH) and poly(sodium 4-styrenesulfonate) (PSS) were used to functionalize gold nanoparticles for colorimetric determination of Hg^{2+} and Cd^{2+} in water samples [58]. Two bi-layers of polyelectrolytes were deposited on the AuNP-functionalized sensor probes for giving a better RI sensitivity (down to 0.5 ppb) compared with single bi-layer. In another study, a rapid and straightforward method was developed for colorimetric determination of ammonia using smartphones based on PVP-stabilized AgNPs [59]. In order to evaluate the effect of ammonia on the UV–vis spectrum of the synthesized silver nanoparticles, different levels of ammonia (in the range of 10–1000 mg L^{-1}) were added to the colloidal solution of AgNPs containing a constant level of the nanoparticles. The mechanism of the detection is based on the formation of a complex ($Ag(NH_3)^{2+}$) which is accompanied by the decrease in the number of individual AgNPs and their related characteristics surface plasmon band. The main point of this study was the use of a smartphone for

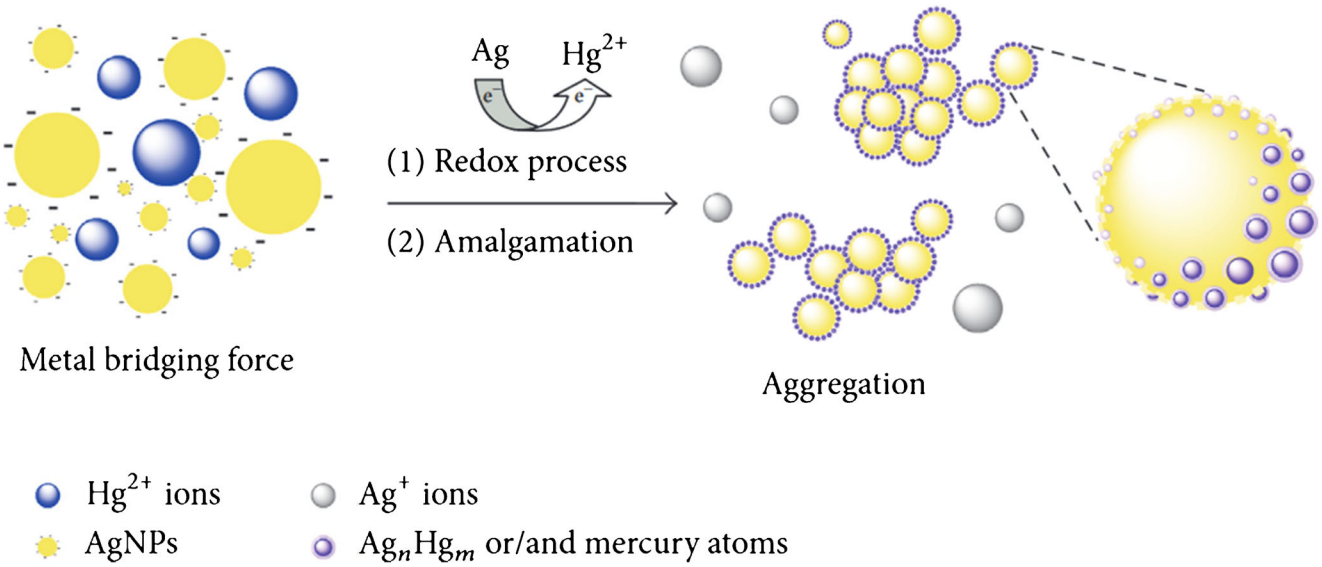

Fig. 6 The amalgamation process by interaction of Hg^{2+} ions with negatively charged silver nanoparticles. Reproduced with permission from reference [68]

colorimetric measurements instead of a UV-Vis spectrophotometer. The smartphone-based detection has the capability to detect ammonia with the LOD as low as 200 ppm. Acrylamide, cellulose, sodium alginate, and sodium cholate were also used in previous literatures for functionalization of nanoparticles [72–76]. Table 3 presents a complete list of polymers for functionalization with responsible moiety in their structure.

The main advantage of polymers as functional agents is their ability to host different moieties with a simple polymerization reaction. The bonding to NPs can be realized using amine or thiol modification as well as the desired moiety for analytical process, which can be grafted in the structure of polymers. However, most of the works in this field are based on non-specific interaction of polymers with NPs and the analytes, which will not yield extremely low detection limits.

Functionalization using organic compounds

Functionalization of plasmonic nanoparticles using organic compounds is the mostly endeavored mechanism for designing colorimetric sensors. The main reason is the diversity of chemical compounds and the ability to design the required surface chemistry for selective determination of specific analyte. The main strategy for linking to metal NPs is via S-H and NH_3 linkage and several chemical moieties (e.g., hydroxyl, carboxyl, carbonyl, etc.) can be available as an anchor to the analytes.

For instance, a simple colorimetric citrate-capped silver nanoparticle-based sensor have been proposed by Zheng et al. [80] for the determination of thiophanate-methyl (TM) in the range of 2–100 μM with a detection limit of 0.12 μM. Their approach is based on the color change of cit-AgNPs from yellow to cherry red with the addition of TM to Cit-AgNPs that caused a redshift on the SPR band from 394 to 525 nm due to the hydrogen-bonding and substitution. The absorbed citrate ions on the surface of AgNPs are capable of forming the hydrogen bonding with thiophanate-methyl through –COOH group of citrate and –NH, –C=S, –C=O, –CH_3 groups of thiophanate-methyl. In another study, a colorimetric sensor based on sulfoanthranilic acid dithiocarbamate (SAA-DTC)-functionalized AgNPs was developed by Mehta and colleagues [81] for the detection of Mn^{2+} and Cd^{2+}, with the detection limit of 1.7 and 5.7 μM, respectively. Disappearing of stretching and bending modes of S-H group indicates the successful attachment of SAA-DTC on the surface of AgNP via thiolate linkage. Sensing mechanism of the above-mentioned sensor is based on the aggregation of SAA-DTC AgNPs in the presence of Mn^{2+} and Cd^{2+}. In another study, a sensitive and low-cost colorimetric probe was developed by introducing a linkage between 1-amino-2-naphthol-4-sulfonate (ANS) and triangular silver nanoplates by electrostatic interaction of the sulfo groups, for Cd^{2+} sensing in narrow linear range of 30–70 μM with a limit of detection of 30 nM [67]. ANS can bind to Cd^{2+} ions through NH_2, SO_3, and OH groups, which leads to the aggregation of triangular AgNPs. Song and colleagues [82] have developed a low-cost, rapid, simple, and sensitive assay using sulfanilic acid-functionalized silver nanoparticles (SAA-AgNPs) for melamine detection in pretreated milk samples, with a LOD of 10.6 nM. In the presence of melamine, the SAA-AgNPs aggregated rapidly through hydrogen bonding between –NH_2 groups on the outer surface of the SAA and the melamine molecule. Surface modification of AgNPs was done by Neem Gum (NG), containing complex polysaccharides, proteins, and other organic compounds, as the reducing and stabilizing agent [69]. FTIR analysis has demonstrated the

Table 3 Summary of previous efforts for functionalization of plasmonic nanostructures using polymers for sensing purposes

Analyte	Functional ligand/moiety	Limit of detection (LOD)	Reference
Glucose	Chitosan/NM*	5 μM	[66]
Timolol	Polyvinylpyrrolidone (PVP)/-SH	1.2×10^{-6} M	[67]
Activated leukocyte cell adhesion molecule (ALCAM) cancer biomarker	Polyethylene glycol/NM	15 pM	[77]
Hg^{2+}	Starch/NM	0.9 μgL^{-1} (in range of 0–12.5 μgL^{-1}) 2.7 μgL^{-1} (in range of 25–500 μgL^{-1})	[68]
Ammonia	Surcralose and Glucose/NM	10^{-2}–10^{-3} ppm	[69]
Hg^{2+} & cellular free oxygen radical	Starch/NM	NM	[70]
Fe^{3+}	$-NH_2$, -SH	0.85 μM	[71]
Hg^{2+}, Cd^{2+}	Poly(allylamine hydrochloride) (PAH) and Poly(sodium 4-styrenesulfonate) (PSS)/NM	0.5 ppb	[78]
Ammonia	Polyvinylpyrrolidone (PVP)/NM	200 ppm	[79]
Ag^{+}	Hyperbranched polyethylenimine (HPEI)/-SH	8.76×10^{-8} M (naked eye) 8.76×10^{-9} M (UV-Vis)	[73]
Acrylamide	Acrylamide/C=C	0.2 nM	[72]
H_2O_2	Polysaccharide (cellulose nanowhiskers)/NM	0.014 μM (in range of 0.1–30 μM) 112 μM (in range of 60–600 μM)	[74]
Hg^{2+}	Sodium alginate/NM	5.29 nM	[75]
Hg^{2+}, Pb^{2+}	Sodium Cholate/COO^{-} and -OH	12 nM, 60 nM	[76]

*NM = not mentioned

binding of hydroxyl, carbonyl, carboxyl, and amine groups of amino acid and NG proteins to the surface of AgNPs. In another study, 2-Mercaptoethanesulfonate (MES) was used to develop a selective and sensitive sensor for alkaline and alkaline earth metal cation determination and monitoring in biological samples [70]. Their SERS-based sensor exhibits limits of detection of 10 nM for Ca^{2+} and 1 μM for Co^{2+}, Fe^{2+}, and Mn^{2+} with a mechanism based on attractive interaction between negative charges of MES attached to the surface of AgNPs and cations present in the solution. Patel and colleagues [83] have developed a simple, rapid, and sensitive colorimetric method for the determination of carbendazim using 4-aminobenzenethiol-functionalized silver nanoparticles (ABT-AgNPs) as a colorimetric sensor. Under optimum conditions, the absorbance ratio at A_{510}/A_{397} is linearly related to the concentration of carbendazim in the range of 10–100 μM, with a detection limit of 1.04 μM. This colorimetric method has been successfully utilized to detect carbendazim in environmental water and food samples. Since ABT-AgNP surface exhibits positive charges (pKa of ABT is 5.70) whereas carbendazim bears negative charges (pKa is 4.48), the conjugation of carbendazim with ABT-AgNPs results to aggregation of NPs via strong ion-pair interactions. The π–π interactions between the neighboring carbendazim-conjugated ABT-AgNPs are responsible for the aggregation that result a color change from yellow to orange and a red-shift in SPR band of ABT-AgNPs from 397 to 510 nm. The formation of a new bond between ABT and AgNPs was approved by disappearance of –SH group located at 2543–2550 and 935–945 cm^{-1}.

In another research by Devadiga and colleagues [84], aqueous extract of an agrowaste: *Terminalia catappa* leaves was used to reduce and functionalize AgNPs with possible application for Hg^{2+} sensing. Authors believed that the multifunctional groups (e.g., hydroxyl, carboxyl, and heteroaromatic rings) present in the extract are responsible for interaction with mercury ion and enhanced stability of the nanoparticles. There are also other reports on using biosynthesized nanoparticles as optical sensors for Hg^{2+}, Cr^{6+}, Zn^{2+}, and hydrogen peroxide determination [85–87]. Silver triangular nanoplates conjugated with gallic acid were designed as a probe for colorimetric detection of reduced GSH with a limit of detection of 0.12 nM [76]. The functionalization of nanoplates was easily done through the phenolic hydroxyl groups (–OH) of gallic acid. The authors believed that the interaction of deprotonated carboxylate (COO^{-}) of gallic acid with protonated amine (NH_3^{+}) is responsible for aggregation of nanostructures. Muthivhi et al. [88] have developed a green method for sensing Hg^{2+} in aqueous media via gelatin-noble metal polymer nanocomposites with a detection limit of

10^{-3} nM. The interaction of gelatin with AgNPs was done via coordination with nitrogen from the amide group.

Silver nanoparticles capped with 3-mercapto-1propanesulfonic acid (AgNPs-3MPS) were used to develop a colorimetric sensor for Ni^{2+} or Co^{2+} ions in water based on the change of the intensity and shape of SPR peak [78]. The reported sensor has a good sensitivity for the detection of Ni^{2+} or Co^{2+} ions in aqueous solutions up to 500 ppb. The interaction of MPS with AgNPs was done via thiol-linkage. Pinyorospathum and co-workers [89] have presented a sensitive colorimetric sensor for the determination of phosphate ions (P_i) performed on paper-based analytical devices (PADs) based on the anti-aggregation of 2-mercaptoethanesulfonate (MS)-modified silver nanoplates. This sensor has a detection limit of 0.33 mg L^{-1} and a limit of quantification equal to 1.01 mg L^{-1} for determination of phosphate ions in the range of 1–30 mg L^{-1}. The presence of -C-H, -COO-, or -COOH stretching modes in FTIR spectra proved the assembly of 2-mercaptoethanesulfonate on AgNPs surface. In addition, the absence of thiol band (-S-H) at 2550 cm^{-1} can be attributed to the formation of Ag-S bonds. Chen and colleagues [90] have introduced an adrenaline sensor based on 4-amino-3-hydrazino-5-mercapto-1,2,4-triazol (AHMT)-functionalized gold nanoparticles with a wide linear range of 7 nM–0.1 mM and the detection limit of 1 nM. Sensing mechanism is based on aggregation of AuNPs which was specially induced by the binding of AHMT to adrenaline as a result of hydrogen bonding between the two molecules, leading to a color change from wine-red to purple-blue. Adrenaline molecule has one amine group and three hydroxyl groups. Each adrenaline molecule has four sites to form hydrogen bonds of NH-O and NH-N. Thus, the aggregation of AHMT-AuNPs was induced by hydrogen bonding between the AHMT and adrenaline. Organically functionalized gold nanoparticles were developed as a prototype gas sensor for formaldehyde detection with possible applications in non-invasive diagnosis through exhaled breath analysis [81]. In this study, 2-mercaptobenzoxazole (C_7H_5NOS) was used to functionalize the AuNPs. A colorimetric method for the detection of Fe^{3+} in water and biological samples is introduced via oxamic acid (OA) and p-aminobenzoic acid (PABA) functionalized gold nanoparticles (OA-PABA-Au NPs) as a probe [82]. This sensor exhibits a detection limit of 5.83 μM. According to the FTIR measurements, OA and PABA molecules were successfully assembled on the surfaces of AuNPs via Au–N linkage. The addition of Fe^{3+} ion leads to a decrease in the SPR band intensity of OA-PABA-AuNPs at 523 nm and to generate a new SPR peak at 685 nm, confirming that the aggregation of OA-PABA-AuNPs induced by Fe^{3+} ion, which results a color change from red to blue. Khodaveisi and colleagues [91] have proposed a colorimetric sensor for the determination of naproxen (NAP) based on the aggregation of the thiolated β-cyclodextrin (Tβ-CD)-functionalized gold nanoparticles (Tβ-CD-AuNPs) in the presence of NAP and Zn^{2+} with a detection limit of 0.6 μg L^{-1} in the range of 4–180 μg L^{-1}. It is known that NAP can act as a unidentate ligand through its carboxylate group and form complexes with several transition metal ions while the other end of NAP is hydrophobic and has high affinity to interact with molecules such as CD. Furthermore, due to the hard and soft acid-base interaction, the thiolated molecules have the ability to interact with the surface of AuNPs and displace the shell of citrate groups. The β-CD was thiolated and immobilized on the surface of synthesized AuNPs. Then, Zn^{2+} which forms a colorless complex with NAP was selected as transition metal ions and along with NAP was added to the Tβ-CD-AuNP solution. This resulted in the formation of $(T\beta\text{-CD:NAP})_2Zn$ complex through aggregation of AuNPs, and because of the near-field coupling in the resonant wavelength peak of the interacting particles, the original LSPR peak of AuNPs decreases, and a new red-shifted band at 650 nm appears. Qin et al. [92] have employed AHMT-AuNP for sensitive determination of kanamycin (KA) in the range of 0.005–0.1 μM and 0.1–20 μM with a limit of detection of 0.004 μM. AHMT contains one mercapto group, which can strongly coordinate to the surface of AuNPs. In addition, AHMT has two exocyclic amino groups and a three nitrogen hybrid ring. On the other hand, as an aminoglycoside antibiotic, KA has four amino groups ($-NH_2$) and seven hydroxyl groups (-OH) which may combine with the AHMT through hydrogen-bonding interaction. The aggregation of AHMT-AuNPs in the presence of KA was studied by monitoring the shift of SPR band.

Khodaveisi and co-workers [93] have reported a colorimetric sensor based on the aggregation of the Tβ-CD functionalized gold nanoparticles for the determination of nabumetone (NAB) in the presence of PVP with a LOD of 0.2 μgL^{-1}. In this study, PVP has the key role to increase the affinity of β-CD for NAB. Formation of the ternary complex of NAB:$(\beta\text{-CD})_2$-PVP resulted in the aggregation of NPs.

Chen and colleagues [94] have synthesized Rhodanine-stabilized gold nanoparticles in order to construct a colorimetric sensor for selective determination of Hg^{2+} in the range of 0.02–0.5 μM. The detection limit of this sensor was measured to be 6.0 nM. The assembly of Rhodanine on AuNPs was done via thiol sub-unit molecules through gold-thiol (Au-S) affinity interactions. Upon the addition of Hg^{2+} to AuNPs@R, a new absorption band at 650 nm appeared and dispersed AuNPs@R are induced to aggregate via the formation of the R-Hg^{2+}-R structure. A sensitive biosensor based on functionalized nanoporous gold (NPG) has been constructed for the determination of human serum albumin (HSA) [95]. In order to study the Raman signal produced by modified NPG substrates, four different compounds (i.e., cysteamine, 3-mercaptopropionic acid, 4-aminothiophenol, 4-mercaptobenzoic acid), all provided with a sulfidrilic group to be bound to the gold surface, were tested after their immobilization on nanostructured gold surface of given porosity. The structural differences among the selected molecules concern the functional group (i.e., amino or carboxyl) used to link covalently to the antibody and the aliphatic or aromatic nature of the structure themselves. All these molecules are bifunctional with a thiol

Table 4 Summary of previous efforts for functionalization of plasmonic nanostructures using organic compounds for sensing purposes

Analyte	Functional ligand/moiety	Limit of detection (LOD)	Reference
Hg^{2+}	Citrate/amalgam (Hg-Ag)	4 nM	[5]
Thiophanate-methyl (TM)	Citrate/hydrogen bonding	0.12 μM	[80]
Mn^{2+}, Cd^{2+}	Sulfonathranilic acid dithio carbamate (SAA-DTC)/NM	1.7, 5.7 μM	[81]
Cd^{2+}	1-amino-2-naphthol-4-sulfonate (ANS)/NH_2, SO_3, OH	30 nM	[97]
Melamine	Sulfonic acid (SAA)/-NH_2	10.6 nM	[82]
Ca^{2+}, Co^{2+}, Fe^{2+}, Mn^{2+}	2-Mercaptoethanesulfonate (MES)/NM*	10 nM, 1 μM, 1 μM, 1 μM	[98]
Carbendazim	4- aminobenzenethiol/NM	1.04 μM	[83]
Hg^{2+}	Extract of *Terminalia catappa* leaves/NM	NM	[84]
Hydrogen peroxide	Kiwifruit extract/NM	5.0×10^{-7} M	[85]
Cr^{6+}, Ammonia	Durenta erecta (*D. erecta*)/metal-oxygen bonding	Up to 0.1 ppm	[86]
Hg^{2+}	*Matricaria recutita* (Babunah) plant extract / NM	100 ppm	[87]
Reduced glutathione (GSH)	Gallic Acid/NM	0.12 nM	[99]
Hg^{2+}	Gelatin/NM	10^{-3} nM	[88]
Ni^{2+}, Co^{2+}	3-mercapto-1 propanesulfonic acid (3MPS)/NM	500 ppb	[100]
Phosphate ion	2-mercaptoethanesulfonate (MS)/NM	NM	[89]
Adrenaline	4-amino-3-hydrazino-5-mercapto-1,2,4-triazol (AHMT)/NM	1 nM	[90]
formaldehyde	2-mercaptobenzoxazole (C_7H_5NOS)/NM	NM	[101]
Fe^{3+}	Paminobenzoic acid (PABA), Oxamic acid (OA) / NM	5.83 μM	[102]
Naproxen (NAP)	Thiolated β-cyclodextrin (Tβ-CD) and Zn^{2+}/carboxylate group	0.6 μgL^{-1}	[91]
Kanamycin (KA)	4-amino-3-hydrazino-5-mercapto-1,2,4-triazole (AHMT)/amine and hydroxyl groups and hydrogen bonding		[92]
Nabumetone (NAB)	Thiolated β-cyclodextrin (Tβ-CD) / NM	0.2 μgL^{-1}	[93]
Hg^{2+}	Rhodanine/NM	6.0 nM	[94]
Human serum albumin (HSA)	Cysteamine, 3-mercaptopropionic acid, 4-aminothiophenol, 4-mercaptobenzoic acid/amine or carboxyl (amide bond with antibody)	NM	[95]
Pencycuron fungicide	6-aza-2-thiothymine (ATT)/NM	0.42 μM	[96]
Dopamine (DA)	S-doped carbon dots (S-CD)/carboxylic group of CDs and amine groups of DA	0.23 μM	[103]
HER2-positive breast cancer cell (SKBR3)	Liposome/anti-HER2	5 single cells	[104]
Cu^{2+}	1,3-alternate calix[4]arene/NM	2.5×10^{-6} M	[105]
Hg^{2+}	Mercaptobenzoheterocyclic compounds (MBO, MBI, MBT)/Ag-Hg interaction	9.2 pM (MBO) 46 pM (MBI) 92 pM (MBT)	[106]
As^{3+}	Polyethylene glycol (PEG) / - OH	1 ppb	[107]
Hg, Hg^{+}	Calixarene / Ag-Hg interaction	0.5 nM (UV-Vis) 10 nM (Amperometry)	[108]

*NM = not mentioned

group able to form the Au-S bond to NPG and an amino or carboxylic terminal group that gives an amide bond with the antibody. Aromatic moieties have been preferred to aliphatic ones due to a more oriented interaction of the molecule with the NPG surface, while the choice between the two aromatic moieties (i.e., 4ATP and 4MBA) has been affected by the strategy used to generate the amide bond with the antibody.

Kailasa and colleagues [96] have proposed a facile method for developing a colorimetric sensor based on the pencycuron-induced aggregation of 6-aza-2-thiothymine (ATT)-functionalized gold nanoparticles for the determination of pencycuron fungicide in rice, potato, cabbage, and water samples with the detection limit of 0.42 μM in the range of 2.5–100 μM. ATT can easily displace citrate molecules on the surfaces of AuNPs

and tune the visual readout ability of AuNPs towards a specific analyte. ATT contains a mercapto group that can easily form a covalent bond (via Au-S bond) with the surface of AuNP. In another study, a selective colorimetric sensor has been proposed based on LSPR of S-doped carbon dots-functionalized gold nanoparticles for detection of dopamine (DA) with a detection limit of 0.23 μM [89]. In this study, phenylamine-4-sulfonic acid with abundant thiol functional groups interacted with Au NPs through soft-soft acid-base interaction. It was observed that addition of DA molecules followed by Fe^{3+} causes aggregation of DA-S-CDs@Au NPs resulting in a decrease in the LSPR band of modified AuNPs around 520 nm and the appearance of a new band at 670 nm. This observation is due to the assembly of DA molecules on the surface of S-CDs@Au NPs through the bonding between its primary amine with the carboxylic group of CDs and aggregation of DA-S-CDs@Au NPs by coordination of Fe^{3+} with DA molecules. In another study, Amirjani et al. [5] have proposed a rapid and sensitive colorimetric detection method for the determination of Hg^{2+} based on citrate-functionalized silver nanotriangles with a limit of detection of 4 nmol L^{-1} which was below the safety level of Hg^{2+} ions (10 nmol L^{-1}) in drinking water. The ability of Hg^{2+} ion to interact with Ag and form the Hg-Ag alloy (amalgam) over the surface of nanotriangles resulted in an obvious color change from blue to violet. A comprehensive list of different organic compounds as functional ligand can be found in Table 4 [104–108].

Organic compounds include a large library of chemicals with the ability to link to plasmonic nanoparticles thorough S-H and NH_3 linkage. One can simply choose the desired compound based on the required moiety for a specific analyte. Even though low detection limits can be obtained by this functionalization strategy, because the ligand is not specifically designed for the analyte the selectivity of the sensor is debatable.

Conclusion and future prospects

During the last decade, many plasmonic sensors were designed and developed for a wide range of analytes from neurotransmitters to explosive chemicals [109–116]. The basis for their versatile applications is the sensitivity of localized plasmonic fields in the immediate vicinity of nanostructures. In this paper, recent advances in functionalization of plasmonic nanostructures for optical sensing were reviewed. With the emphasis on the nature of the functional ligands, they were categorized in four different groups namely as oligonucleotides, organic polymers, proteins and amino acids and organic compounds. Different scenarios for attachment of functional agents to NPs as well as different approaches for analyte chelation were reviewed and discussed in each category. Proper functionalization of nanostructured probe is essential for selective determination of desired analyte. Engineered oligonucleotides as functional groups can be designed for selective determination of specific analytes with detection limit as low as pM. The efficiency and performance of plasmonic sensors are totally comparable or even superior to conventional detection methods. The ability of colorimetric detection in solution phase using plasmonic nanostructures makes the process rapid and straightforward. Nowadays, there is growing interest in immobilization of nanostructures on substrates (glass, paper, indium tin oxide (ITO), Polyethylene terephthalate (PET), etc.) for realization of lab-on-a-chip concept [117–119].

By immobilizing the nanostructured probe, sensors can be used for several detection cycles. In addition, there is a huge demand for using portable and easy accessible signal readers for such sensors such as smart phones instead of conventional spectrophotometers. The future prospect of plasmonic sensors is mainly dominated by immobilized NPs arrays on substrates, which are able to detect analytes on-site by the aid of a portable image analyzer unit (such as smartphones). These sensor arrays also make the multi-analyte determination possible by using different ligands for every specific analytes. In this decade, plasmonic sensors can become the gold standard for determination of chemical and biochemical species.

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Compliance with ethical standards

Conflict of interest The author(s) declare that they have no competing interests.

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