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ORIGINAL PAPER

Ultrasensitive visual and colorimetric determination of dopamine based on the prevention of etching of silver nanoprisms by chloride

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Abstract The authors describe a selective and sensitive colorimetric method for determination of dopamine (DA) in serum. It is based on the protection of silver nanoprisms (AgNPRs) from being etched by chloride ion in the presence of DA. In the absence of DA, the shape of the AgNPRs is changed from triangle nanoplates to round nanodisk due to etching by chloride. This is accompanied by a stepwise color change from blue via purple and red to yellow. It is found that DA is strongly adsorbed on the surface of AgNPRs and thereby acts as a protective agent. As a result, etching by chloride is prevented and the color changes do not occur. This finding ease exploited to design a method for optical quantification of DA with either bare eyes or UV-vis spectrophotometry. The wavelength shift of the in-plane dipole surface plasmon band of the AgNPRs is linearly related to the DA concentration in the range from 0.5 to 100 nM. The detection limit is 0.16 nM (at an S/N ratio of 3) which is lower than that of most existing methods. Uric acid, ascorbic acid and other coexisting species do not interfere. The sensor is reproducible and stable and was applied to the determination of DA in spiked serum samples where it gave recoveries that ranged from 97.4 to 104.3%.

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Introduction

Dopamine (DA), chemically known as 4-(2-aminoethyl) benzene-1,2-diol, is one of the crucial catecholamine neurotransmitters [1]. It plays a central role in the mammalian central nervous system. Abnormal DA concentrations in brain are associated with neuropsychiatric disorders such as schizophrenia and Parkinson's disease [2, 3]. Hence, the sensitive and rapid determination of the concentration of DA in blood is of paramount importance.

Various detection methods have been employed to quantify DA including capillary electrophoresis techniques with laser-induced native fluorescence [4], high performance liquid chromatography techniques [5, 6] and chemiluminescence [7]. Most of these protocols display excellent performance but usually demand sophisticated operating, precise equipment and long analysis time. Electrochemical method has also attracted special attention since DA possesses good electroactivity [8–10]. However, coexisting species such as ascorbic acid (AA) and uric acid (UA), which are oxidized at potentials close to that of DA, can interfere with the detection of DA. Thus, it still remains urgent to fabricate a fast, sensitive and inexpensive method with good selectivity for determination of trace amount of DA in body fluid.

Rapid colorimetric detection method based on noble metal nanomaterials is gaining considerable attention due to its simplicity and practicality [11–13]. Spherical noble metal nanoparticles (NPs) are widely employed as colorimetric probe owing to their optical properties like the color of metal NPs

would transform as the change of their dispersion/aggregation states [14–17]. For example, Kong's group fabricated a colorimetric probe for sensitive DA detection based on AuNPs and double molecular recognition [18]. Feng's group introduced a colorimetric platform for the quantitative determination of DA, based on 4-amino-3-hydrazino-5-mercapto-1, 2, 4-triazol (AHMT) functionalized AuNPs as a model probe [19]. He's group reported a colorimetric detection of DA by using two specific ligands modified Ag (PBDA-DSP-Ag and MPBA-ABCE-Ag) nanoparticles [20]. Although high selectivity of these approaches, they are limited by the complicated pre-preparation and modification of nanomaterials.

Anisotropic noble metal nanomaterials of different shapes and sizes have attracted extensive interest in chemistry, physics and materials science [21–23]. Among them, plate-like triangular silver nanoprisms (AgNPRs) possess attractive optical, electronic and structural properties. Compared to regular noble metal nanoparticles, AgNPRs exhibit higher extinction coefficient, and the color and surface plasmon resonance (SPR) that sensitively depends upon their shape and size would change more elaborate [24–26]. Recently, it was found that AgNPRs can be easily etched into round nanodisk by heat [27, 28], H_2O_2 [29], UV light irradiation [30] and inorganic anions (e.g., Cl^-, Br^-, I^-, $H_2PO_4^-$, and SCN^-) [31], causing a concomitant blue shift of the SPR spectra depending on the particle morphology change, which instead of the modulation of aggregation/deaggregation of metal NPs. According to these unique properties, AgNPRs are promising to be utilized in the design of wavelength-variations sensing platforms. Recently, Chen's group proposed a colorimetric probe for Hg^{2+} based on 1-dodecanethiol-capped AgNPRs upon the presence of excess I^- [32]. Xia's group fabricated a homogeneous system consisting of AgNPRs and glucose oxidase for sensitive colorimetric sensing of glucose in serum [33]. To our knowledge, there is no work about AgNPRs-based colorimetric DA method reported up to now.

According to previous literatures [34–36], the protonated DA molecule with catechol group in structure was found to easily adsorb on silver atoms surface through the robust Ag-catechol interaction. Inspired from the interesting mechanism, we fabricated a simple and highly sensitive colorimetric platform for DA detection based on the protection of Ag nanoprism from being etched by halide ion (Cl^-) in the presence of DA. The shape evolution of AgNPRs from nanoprisms to nanodisks can be prevented by DA. The wavelength shift of SPR before and after AgNPRs incubated with DA in the presence of Cl^- is recorded. It's worth mentioning that the strategy has three significant advantages: (i) the detecting procedure is quite convenient: AgNPRs can be directly employed as probe in detection without modification; (ii) the detecting phenomenon is obvious: the colors of AgNPRs at different etching degrees are rather abundant and bright (blue, purple, red and yellow), which is propitious to better observation by visually; (iii) the sensitivity of the method is excellent: the colorimetric assay has a detection limit as low as 0.16 nM of DA, exhibiting prominent superiority to other DA methods. The remarkable sensitivity is mainly benefit from the extremely high extinction coefficient of AgNRPs and steady combination between DA and AgNRPs. Therefore, such a rapid, facile colorimetric platform for DA detection with good sensitivity and selectivity is desirable for determination of trace amount of DA in human plasma samples.

Experimental

Reagents

All chemicals used were of analytical reagent grade without further purification and solutions were prepared with Milli-Q-purified distilled water. $AgNO_3$, $NaBH_4$, trisodium citrate and H_2O_2 were purchased from Sinopharm Chemical Reagent Beijing Co., Ltd. (Beijing, China, http://www.crc-bj.com/). Polyvinylpyrrolidone (PVP, MW ~ 29,000) was purchased from Sigma-Aldrich (Shanghai, China, http://www.sigmaaldrich.com). Dopamine (DA, 99%) and Ascorbic acid (AA, 99.7%) was purchased from Acros Organics (USA, http://www.acros.com/). Uric acid (UA, 99%) was bought from Alfa Aesar (Shanghai, China, http://www.alfachina.cn).

Apparatus

The UV–Vis absorbance spectra were recorded with a UV-2550 spectrophotometer at ambient temperature (20 ± 2 °C) with Quartz cuvette. The photographs were taken by a camera DSC-W150 from Sony Corporation, Ltd. Transmission electron microscopy (TEM) images were obtained by using a Philips EM-400. The Fourier transform infrared spectroscopy (FT-IR) characterization was carried out on a BRUKE Vertex 70 FT-IR spectrometer. The X-ray photoelectron spectrum (XPS) was measured by a PHI Quantera SXM™ Scanning X-ray Microprobe™.

Synthesis of AgNPRs

All glassware used in the following procedure was cleaned in a bath of fresh prepared 3:1 (*v*/v) HCl/HNO_3, rinsed thoroughly in water, and dried in air. Double-distilled water was used for all of the experiments. AgNPRs were prepared according to previous literature with little modification [26]. Briefly, aqueous solutions of $AgNO_3$ (0.05 M, 0.1 mL), trisodium citrate (0.1 M, 0.75 mL), PVP (3.5 mM, 1.0 mL) and H_2O_2 (30 wt%, 0.12 mL) were mixed with 49.75 mL of Milli-Q water in a beaker of 100 mL capacity and stirred vigorously at room temperature. After that, 0.5 mL aqueous solution of fresh $NaBH_4$ (0.1 M) was then rapidly added to the above

mixture with stirring to generate a pale yellow colloid. Almost 30 min later, the resulting colloid changed its color from pale yellow to blue, which was subsequently stored in the refrigerator.

Procedures for dopamine detecting

Rational designed tests were carried out to optimize the detecting conditions. To a series of 5 mL calibrated test tubes, 50 μL aqueous solution of DA at different concentrations was mixed with 450 μL of unmodified Ag nanoprism and incubated for 30 min at room temperature, followed by 1.5 mL HAc/NaAc buffer solution (pH 6.5) diluted. Next, a 20 μL aqueous solution of 0.02 M KCl was added to the mixed solutions. After 10 min, the solutions were transferred separately into 1 cm quartz cuvette. A photograph was taken, and the in-plane dipole of SPR spectra of AgNPRs was directly measured by the UV-vis spectrophotometer.

Selective detection of dopamine

In the experiments of selectivity assay, various amino acids, tyramine (TIE), glutathione (GSH), glucose (Glc), UA, AA, 3,4 dihydroxyphenylacetic acid (DOPAC) and their mixture with DA were pre-mixed, which were then separately added to the Ag nanoprism solutions. Then the selectivity detection were accomplished by employing the protocol for the DA detection system, which is similar to the one described above.

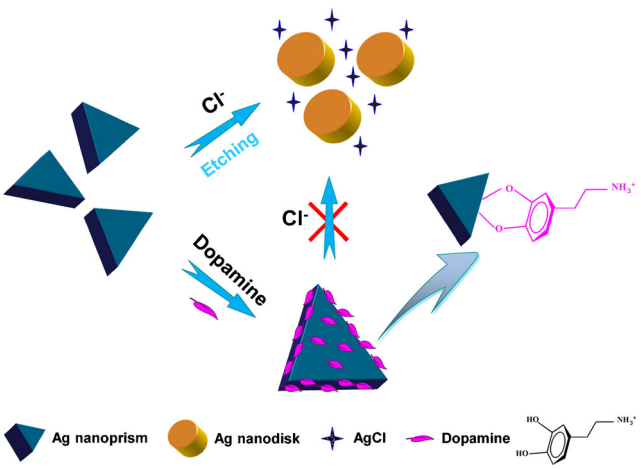
Results and discussion

Mechanism of the colorimetric platform

Scheme 1 outlines the detecting mechanism. In the absence of DA, Cl^- tends to attach to the corners and edges of the AgNPRs via Ag-Cl bond [37] and the triangle AgNPRs would be etched into smaller round Ag nanodisks. However, in the presence of DA, the catechol group of DA tends to readily adsorb onto the surface of AgNPRs via chemisorption-type interactions. Thus, DA acts as a protective agent for the AgNPRs to avoid etching by chloride and thereby increases the stability of silver atoms at corners and edges.

The mechanism for the detection of DA is verified by the SPR spectra. As presented in Fig. 1a, the initial Ag nanoprism colloid is blue and has three characteristic peaks in the extinction spectra at peak wavelengths of 716, 465 and 331 nm, corresponding to in-plane dipole, in-plane quadrupole, and out-of-plane quadrupole SPR modes of AgNPRs, respectively [24]. Figure 1b shows that the maximum absorption wavelength of AgNPRs changed from 716 to 503 nm with the addition of Cl^-, accompanying with subtle color variation process (blue → purple → red → yellow), indicating that triangle AgNPRs have been etched into smaller round Ag nanodisks. As shown in Fig. 1c, no obvious difference is observed for the absorption wavelength of AgNPRs in the presence of DA. When Cl^- is added into AgNPRs in the presence of DA, the maximum absorption wavelength of AgNPRs changed from 716 to 685 nm (Fig. 1d), the shift value of SPR peak prominently decreases compared with that of the Fig. 1b, which indicates DA validly prevents AgNPRs from Cl^- attack. Additionally, a set of contrast experiments utilizing AgNPs instead of AgNPRs is investigated in a manner similar to the one described above and the results are shown in Fig. S1. It's evident that in the DA-AgNPs detection system, the color and SPR spectrum are both proving no obvious change when AgNPs incubated with DA or Cl^-, which further indicating the unique property of the irreplaceable AgNPRs in this assay.

Apart from the SPR investigation, a series of characterization is carried out to confirm the feasibility of the strategy discussed above as well. The morphological transition process of AgNPRs before and after incubation with DA and Cl^- is also investigated by TEM. As shown in Fig. 2a, triangle



Scheme 1 The schematic diagram of the colorimetric detection of DA based on prevention of etching of silver nanoprisms by chloride

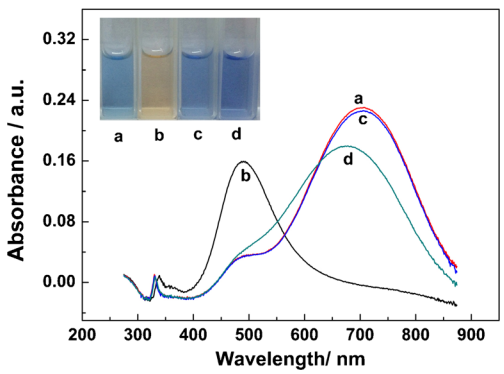


Fig. 1 The SPR spectrum of AgNPRs (insert photograph corresponds to the colorimetric response) under different conditions (**a**) AgNPRs; **b** AgNPRs incubated with 0.2 mM Cl^-; **c** AgNPRs in the presence of 80 nM DA; **d** AgNPRs incubated with 0.2 mM Cl^- in the presence of 80 nM DA

nanosheet-like AgNPRs are in uniform shapes. Their sizes are observed evidently. All AgNPRs have an edge length of 40 ± 6 nm. Figure 2b shows a TEM image of AgNPRs incubated with 0.2 mM Cl^-. It is obvious that the morphology of AgNPRs is etched to 25 ± 5 nm round nanodisk after incubation with Cl^-, which is consistent with the above analysis of SPR shifts. The TEM image of AgNPRs incubated with Cl^- in the presence of 80 nM DA is shown in Fig. 2c. It's worth mentioning that in the presence of DA, the morphology of AgNPRs almost remains triangle shape even after incubation with Cl^-. This result reconfirms the mechanism that the shape evolution of AgNPRs can be prevented by DA. Moreover, FT-IR and XPS spectra are employed to further reveal the interaction between DA and AgNPRs (Fig. S2). These results further demonstrate that DA protects the AgNPRs.

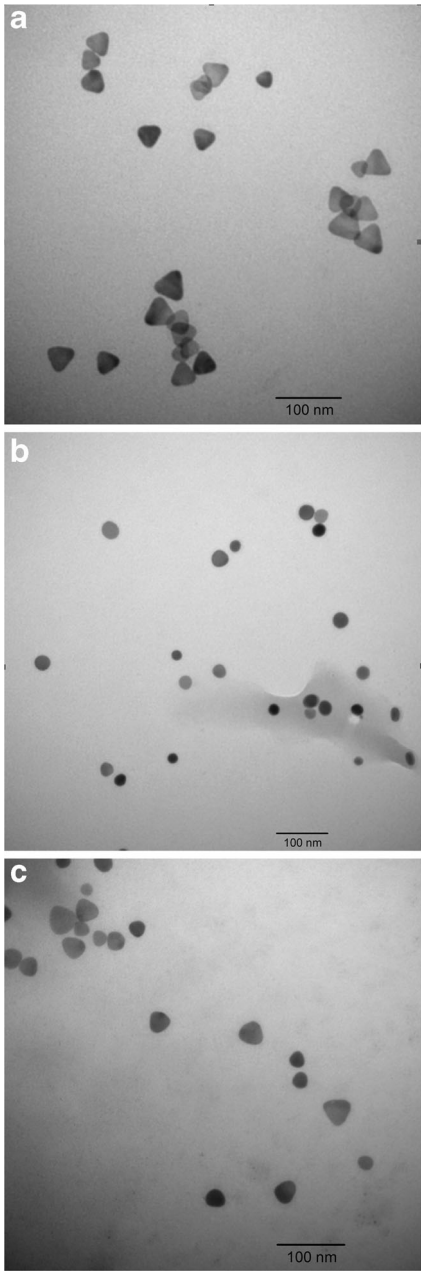


Fig. 2 TEM images of (**a**) fresh AgNPRs; **b** AgNPRs incubated with 0.2 mM Cl^-; **c** AgNPRs incubated with 0.2 mM Cl^- in the presence of 80 nM DA

Optimization of experiment conditions of the detecting assay

In this assay, in order to promote the analytical performance of the colorimetric platform, several experiment conditions including pH value of AgNPRs solution, incubation time of DA and etching time of Cl^- are investigated by measuring the blue shifts of SPR band of AgNPRs in the absence and presence of DA under the etching of Cl^-.

Figure 3a shows Δλ of AgNPRs solution at different pH from 5.5 to 8.0 ($\Delta\lambda = \lambda - \lambda_1$, λ and λ_1 denotes the maximum absorption wavelength of AgNPRs incubated with Cl^- in the absence and presence of DA, respectively). Results indicate that the colorimetric probe exhibits better sensitivity when pH is 6.5, which may due to the protonated DA molecule is more readily attached to AgNPRs. Moreover, when pH is lower than 6, the nanostructure transformation of AgNPRs from nanoprism into nanodisc would be induced [38]. Hence, pH 6.5 is chosen for the entire detection. Figure 3b illustrates that Δλ of AgNPRs in the presence of DA changed with incubation time from 10 to 50 min. It is noteworthy that Δλ increases as the incubation time increases from 10 to 30 min and almost keeps a constant over 30 min, indicating the amount of DA reached saturation on the surface of AgNPRs from then on. As shown in Fig. S2, the SPR spectrum of AgNPRs shifts apparently as the etching time of Cl^- increases. However, the SPR spectrum of AgNPRs shifts slowly in the presence of DA. The increase of Δλ is fast from 0 to 10 min and keeps a constant over 10 min (Fig. 3c). Thus, 10 min is chosen as the optimal etching time of Cl^-.

Sensitive colorimetric detection of dopamine

To evaluate the analytical performance of the method for DA detection, under the optimized conditions, different concentrations of DA are added to AgNPRs in the presence of Cl^-. As illustrated in Fig. 4a, the change of the maximum absorption wavelength of AgNPRs (Δλ) decreases and the color shows less change with the increase of DA concentration, which is ascribed to the better protection effect of DA. Thus, the concentration of the DA was quantified based on the relationship between the concentration of DA and Δλ. As shown in Fig. 4b, a good linear relationship between Δλ and DA concentration ranging from 0.5 to 100 nM is observed. The linear regression equation is $\Delta\lambda = 9.7125 + 2.2998\ C$ (unit of C is nM) with a linear coefficient of 0.9920. The detection limit is as low as 0.16 nM ($S/N = 3$). Compared to other existing

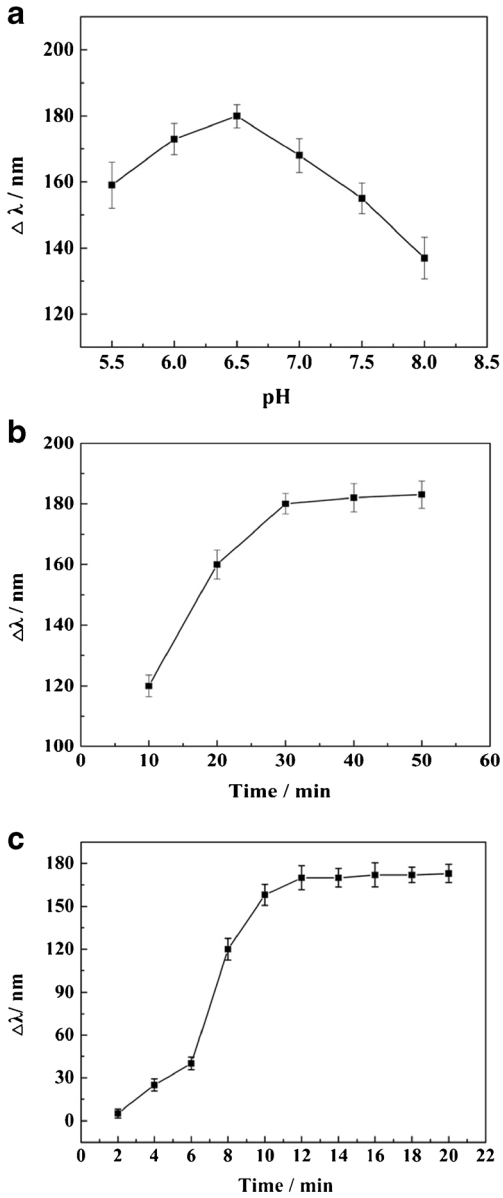


Fig. 3 **a** The plot of Δλ vs. pH of AgNPRs in the presence of 80 nM DA and 0.2 mM Cl^-; **b** The plot of Δλ vs. incubation time of 80 nM DA in the presence of 0.2 mM Cl^-; **c** the plot of Δλ vs. etching time of 0.2 mM Cl^- in the presence of 80 nM DA (Δλ = λ - $λ_1$, λ and $λ_1$ denotes the maximum absorption wavelength of AgNPRs incubated with Cl^- in the absence and presence of DA, respectively)

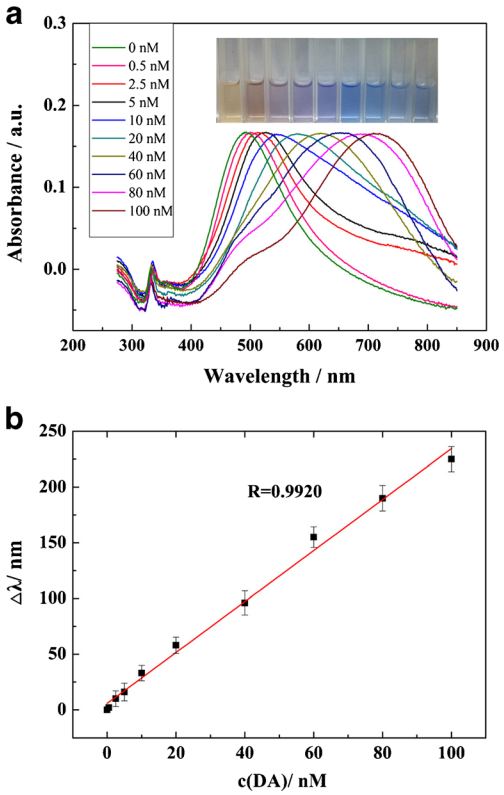


Fig. 4 **a** SPR spectrum of the AgNPRs incubated with 0.2 mM Cl^- in the presence of DA at various concentrations (insert photograph corresponds to the colorimetric response of DA in the range of 0.5 to 100 nM from left to right); **b** the linear relationship between the blue shift value of SPR wavelength with the concentration of DA ranging from 0.5 to 100 nM (Δλ = λ - $λ_1$, λ and $λ_1$ denotes the maximum absorption wavelength of AgNPRs incubated with Cl^- in the absence and presence of DA, respectively)

methods of DA detection, our assays exhibits prominent superiority both in sensitivity and easily operate (Table 1).

Selectivity of the detection system

Under the etching of Cl^-, the selectivity of the AgNPRs-based approach for the detection of DA is evaluated by monitoring the change of the maximum absorption wavelength of AgNPRs (Δλ) in the absence and presence of the coexisting species in cerebral systems such as tyrosine (Tyr), tryptophan (Try), glutamic acid (Glu), Glc, GSH, TIE, UA, AA and DOPAC. As shown in Fig. 5, compared with that of DA, low values of Δλ are observed in the presence of amino acid, Glc, GSH, UA and AA. The DOPAC, which is the metabolity of DA and has a similar catechol structure with DA causes a certain amount of interference within the acceptable scope. These results further proved that the catechol structure plays an important role in the detection of DA. It is found that the value of Δλ in the presence of the mixture of DA and other interferences is almost similar to that in the presence of DA only. The satisfied selectivity is mainly attributed to the high affinity binding between DA and AgNPRs via Ag-catechol bonds In addition, the coexisting species such as AA and UA are negatively charged under pH 6.5, so the electrostatic repel interaction with negatively charged citrate-capped AgNPRs prevents their access to AgNPRs [34]. Therefore, the selectivity is further enhanced.

Reproducibility and stability of the detection system

The reproducibility of the colorimetric probe is determined at the DA concentration of 20 and 80 nM, and the relative

Table 1 Comparison between the colorimrtric platform with other reported methods for the detection of dopamine

Materials	Detection method	Linear range(μM)	Detection limit(nM)	References
F-$CuInS_2$ QDs	fluorescence	0.5–40	200	[39]
Tungsten trioxide modified electrode	electrochemical	0.1–600	24	[40]
Graphene/SnO_2 modified electrode	electrochemical	0.1–10	80	[8]
AgNPs	colorimetry	0–0.6	60	[34]
AHMT-AuNPs	colorimetry	0.2–1.1	70	[19]
MBA-DSP-AuNPs	colorimetry	0.005–0.18	0.5	[18]
AgNPRs-Cl^-	colorimetry	0.0005–0.1	0.16	This work

standard deviation (RSD) for five times were 5.52% and 4.40%, respectively, implying that this platform has a high degree of reproducibility. The stability of AgNPRs is evaluated by recording SPR spectrum of AgNPRs during a month. As shown in Fig. S4, the SPR spectrum exhibits no obvious change even after a month, indicating that the method has excellent stability.

Real serum sample analysis

In order to demonstrate whether the colorimetric method can used for the detection of DA in real samples, different concentrations of DA are doped into serum samples by standard addition method. The experimental results are shown in Table S1. The recoveries are in the range of 97.40% to 105.2%, indicating that the colorimetric platform might be applied for the detection of DA in human serum. Therefore, the colorimetric probe may have potential applications for DA analysis in biological assays.

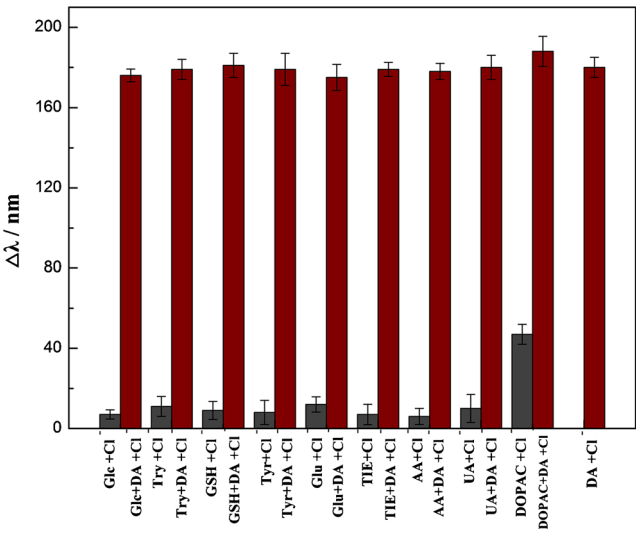


Fig. 5 Under the etching of 0.2 mM Cl^-, the change of the maximum absorption wavelength of AgNPRs (Δλ) in the presence of 80 nM DA or 800 nM other interferences or their mixtures

Conclusion

In this study, a rapid, simple, sensitive, and selective colorimetric platform is developed for detection of DA in human serum, based on prevention of etching of silver nanoprisms by chloride without any modification of AgNPRs. No sophisticated instrumentation is required. The prepared DA detection platform takes advantage of the formation of robust Ag-catechol interaction between AgNPRs and protonated DA, which provides a potential application for monitoring DA related to physiological and pathological events in brain chemistry. But up to now, the research about AgNPRs based colorimetric probe is still lacking of particularly highly selectivity when the catechol compounds is coexisted with dopamine, and further improvement is demanded in the future study. To summarize, based on these results, we consider AgNPRs-based colorimetric method can offer new trends in determination of dopamine in human serum samples.

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Compliance with ethical standards The authors declare that they have no competing interests.

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