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Label-Free and Turn-on Fluorescent Cyanide Sensor Based on CdTe Quantum Dots Using Silver Nanoparticles

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Abstract

Silver nanoparticles were used to develop a simple turn-on fluorescent assay based on glutathione-capped CdTe quantum dot for the determination of trace amounts of the lethal poison, cyanide. It was found that the fluorescence intensity of glutathione-capped CdTe quantum dot increased with increasing cyanide concentration. Several experimental variables such as pH, the amounts of quantum dots and silver nanoparticles were affect the analytical signals were optimized. Using this optical sensor under optimum conditions, cyanide was measured in the range of 0.01 – 2.5 μg mL^{-1} with a detection limit as low as 0.004 μg mL^{-1}. Relative standard deviations of 2.0% (for 0.5 μg mL^{-1}, n=10) and 1.8% (for 2.0 μg mL^{-1}, n=10) were obtained. Investigation of the effects of potential interfering anions on the response of the sensor revealed the high selectivity of the sensor for the detection of cyanide in real samples. High sensitivity, superior selectivity, low detection limit (0.004 μg mL^{-1}) and ease of production are the most important advantages of the present sensor. Finally, the sensor was lucratively applied for the determination of cyanide in real samples.

Keywords: Cyanide, CdTe quantum dot, Silver nanoparticles, Turn-on fluorescent.

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1. Introduction

Quantum dots (QDs) are brightly luminescent semiconductor nanoparticles that have found wide applications in bioanalysis and bioimaging in recent decades. This is due to their unique photoproperties such as wide UV–Vis absorption spectrum and narrow photoluminescence spectrum.[1] Compared to organic dyes, QDs have a brighter fluorescence by about 10–20 times and a better photostability by 100–200 times.[2] A lot of studies in a variety of fields have been devoted over decades for the interesting optical properties of nanoparticles such as silver and gold nanoparticles (SNPs and GNPs, respectively).[3]

Plasmon resonance absorptions of SNPs and GNPs have molar extinction coefficients (~ 3×10^{11} M^{-1} cm^{-1}) that make them good energy acceptors so that they often serve the role of quenchers in fluorimetric methods.[4–8] The fluorescence of CdTe-QDs is significantly reduced by SNPs to the extent that their self-quenching effect could be eliminated.

Cyanide is one of the most lethal toxins; 300 µg mL^{-1} of this poison would be enough to kill a man quickly. Cyanide's toxicity to humans lies in its ability to suppress oxygen transfer via its binding to the active sites of cytochrome C oxidase that results in hypoxic.[9] Long-term exposure to low amounts of cyanide also affects the central nervous system.[10] Cyanide poisoning may also occur through inhaling emissions from residential applications, metal plating, metal mining, plastic manufacturing, and metal processing industries.[11]

Several analytical methods have been developed for the detection of cyanide including liquid choromatography-mass spectrometry,[12] fluorimetric[13–15] chemiluminescence,[9,16] colorimetric[17,18] and electrochemical methods.[19] A few methods have also been reported for the fluorimetric determination of cyanide using QDs.[20–24]

Among these, fluorescent sensors enjoy many advantages. They are highly sensitive, inexpensive, easy to use, and especially suitable as a diagnostic device for analytical purposes. Moreover, they can be employ hands-free via remote controlling.

Quantum dots based on organic dyes as fluorophore have attractive properties and several advantages.[25] Cyanide could enhance the fluorescence of CdTe QDs, however the linear range was narrow and the selectivity was poor. For resolving this problem, Shang and et al. used copper ion-modified CdTe quantum dots.[20] The fluorescence of CdTe QDs was quenched by the copper ions. However, in the presence of CN^-, copper ions could be desorbed from the surface of the QDs and therefore the fluorescence of CdTe QDs increased. In similar report for CN^- detection, copper ion was used as a carbon dot fluorescence quencher.[24] Cyanide is well-known to be capable of dissolving nanometals such as Ag in the presence of oxygen and in basic solution.[26] In addition the fluorescence of CdTe QDs could be quenched by silver nanoparticles.

Here, a novel and simple fluorimetric sensor was reported for cyanide detection based on glutathione-capped cadmium telluride quantum dot (GSH-capped CdTe QDs). One limitation in the fluorescence detection at high fluorescence intensity is self-quenching. In this study, SNPs were used as a quencher to overcome the problem. The method was observed to be highly selective and sensitive for detection of cyanide contents as low as 0.004 μg mL^{-1}. Moreover, it was found that the detection limit and dynamic range of the proposed sensor was comparable with other fluorescence methods reported in the literature for measuring cyanide (Table 1).

2. Experimental

2.1. Chemicals

Glutathione, sodium tellurite, $CdCl_2$ and $NaBH_4$ were purchased from Aldrich. $AgNO_3$ and trisodium citrate were purchased from Merck. All other analytical reagent grade chemicals (with the highest degree of purity available), 0.01 mol L^{-1} carbonate buffer solution and deionized water were used throughout.

A 50.0 µg mL^{-1} stock solution of cyanide was prepared by dissolving an appropriate amount of NaCN into a 100–mL standard flask.

A 0.040 mol L^{-1} $CdCl_2$ and Na_2TeO_3 (0.010 mol L^{-1}) as precursors were prepared with highest purity available chemicals using deionized water.

2.2. Apparatus

UV–VIS absorption spectra were obtained using a Jasco V–570 UV/Vis/NIR spectrophotometer.

The luminescence spectra were measured on a Jasco FP–750 spectrofluorometer. The slit widths of the excitation and emission were fixed at 10.0 nm.

Transmission electron microscopy (TEM) experiments were carried out with a Philips CM30 300 kV TEM.

2.3. Preparation of GSH-capped CdTe QDs

The GSH-capped CdTe QDs were prepared according to the protocol reported in the literature.[27] Briefly, 2.0 mL of 0.04 mol L^{-1} $CdCl_2$ was diluted to 50 mL. Then, 0.050 g trisodium citrate dihydrate, 0.025 g glutathione, 2.0 mL, 0.010 mol L^{-1} Na_2TeO_3, and 0.025 g $NaBH_4$ were added to the $CdCl_2$ solution, while stirring at room temperature. After 2 h, the mixture was refluxed for 12 h at 90 °C. The resulting solution was transferred to a dark container where it was kept at 4 °C.

*2.4. Preparation of citrate-stabilized SNP*s

SNPs were prepared via a previously reported method.[26] In a brief, 0.125 mL of 0.10 mol L^{-1} $AgNO_3$, and 0.125 mL of 0.10 mol L^{-1} sodium citrate were added to 50 mL of water while stirring continued. Then, 3 mL of a newly prepared 5.0 mmol L^{-1} $NaBH_4$ was added into the above aqueous solution and stirred for 30 min. The resulting yellow colloidal SNPs solution was stored at 4 °C overnight before use.

2.5. Measurement procedure

For the determination of cyanide, a freshly prepared mixture containing 0.45 nmol L^{-1} of GSH-capped CdTe QDs and 8.0 nmol L^{-1} of SNPs, that buffered with the carbonate buffer (pH 10.0), plus an appropriate volume of sample solution were mixed. Then, the fluorescence spectrum of the solution was recorded at 550 – 700 nm upon excitation at 400 nm. The slit widths of both the excitation and the emission were set 10.0 nm. The response function (F–F0) values of the sensor were obtained with different concentration of cyanide, where F and F0 are the fluorescence intensities at 615 nm in the presence and absence of cyanide, respectively.

2.6. Sample preparation

Real samples of human serum and of wastewater were filtered to remove any particles, before analysis. Standard addition method was used for the determination of cyanide. 1.0 mL of each real sample (with and without standard solution) was transferred into a vial containing 0.45 nmol L^{-1} of GSH-capped CdTe QDs and 8.0 nmol L^{-1} of SNPs at pH 10.0. Then, the fluorescence spectrum was recorded at 550 – 700 nm upon excitation at 400 nm.

3. Results and discussion

3.1. TEM and UV–Vis absorption spectrum

TEM was used to characterize the GSH-capped CdTe QDs and SNPs. The image of the colloidal GSH-capped CdTe QDs solution (Fig. 1A) shows that the nanoparticles are mostly round in shape with an average particle size of ~8 nm. The UV–Vis absorption spectrum of the GSH-capped CdTe QDs (Fig. 2A) shows the wide UV–Vis absorption spectrum as expected of the quantum dots. The spectrum was employed to determine the GSH-capped CdTe QDs content.[28] The amount of GSH-capped CdTe QDs was calculated to be 0.34 μmol L^{-1}.

TEM analysis was also used to verify the size of the SNPs (Fig. 1B). Based on the TEM image of the colloidal SNPs solution, the nanoparticles are mostly round in shape with an average particle size of ~11 nm. The UV–Vis absorption spectrum of the synthesized SNPs is shown in Fig. 2B, as can be seen the wavelength of maximum is ~400 nm.

3.2. Operating principles

The fluorescence intensity of GSH-capped CdTe QDs was raised in the presence of cyanide. Given the fact that the photoluminescence of QDs arises from electron-hole recombination it is logical to expect that changes in the surface charges or QD components, caused by chemical or physical interactions between ions or small molecules and the QDs, might affect the efficiency of the electron–hole recombination and, thereby, the luminescent emission.[29]

At high cyanide concentrations (above 0.5 μg mL^{-1}), the fluorescence intensity of GSH-capped CdTe QDs (in the absence of SNPs) reduces because of the self-absorption effect of GSH-capped CdTe QDs (Fig. 3). In this work, this technical problem (self-absorption) was resolved by adding SNPs.

The energy transfer or inner filter effect (IFE) of fluorescence refers to the absorption of the excitation and/or emission of light by absorbers in the detection system. As can be seen in Fig. 2B (from the absorption spectrum of CdTe-QDs and that of SNPs), there is no obvious overlap between the spectrum of CdTe-QDs and that of SNPs. Therefore, it does not reflect the evidence of IFE. But, as can be seen in this figure, the maximum wavelength of SNPs is about 400 nm that is equal to the excitation wavelength of CdTe-QDs. Therefore, the photoluminescence emission intensity of GSH-capped CdTe QDs is reduce in the presence of SNPs. After addition of cyanide ions, the luminescent emission of GSH-capped CdTe QDs was increased. This is due to the fact that in the presence of cyanide, the SNPs is dissolved in the form of $Ag(CN)_n^{(n-1)-}$, so the plasmon absorption band of the SNPs was decreased[26]. As evidenced by the absorption spectroscopy (Fig. 4), the absorption band of SNPs decreased gradually.

Scheme 1 shows trend of the fluorescence signal of the sensor by addition of cyanide ions. The zeta potential of CdTe-QDs was found to be negative at pH 10. Also, citrate-stabilized SNPs were negatively charged; thus, no obvious interactions (e.g., electrostatic binding) were expected to occur with GSH-capped CdTe QDs. Fig. 5 shows the fluorescence emission spectra of a mixture solution containing different amounts of cyanide (under the optimum conditions). As can be seen in Fig. 5, the proposed sensor works in a 'turn-on' mode, which is generally more sensitive than the turn-off assay mode because the enhanced fluorescence of the signal transduction gives a much better signal-to-noise ratio for the sensing scheme.[30]

3.3. Effect of sample solution pH

One method most commonly used for dispersing QDs in aqueous solution is to modify their outer surface with anionic carboxylate groups. In this experiment, the

fluorescence of the CdTe solution was depended strongly on the solution pH. This means that a suitable buffer should be used to maintain the pH of the aqueous solution at a constant level. For this purpose, the response of the sensor was measured at different pH (6.0–11.0) using solutions containing 0.28 nmol L^{-1} of GSH-capped CdTe QDs, 8.0 nmol L^{-1} of SNPs and 1.0 μg mL^{-1} cyanide (Figs. 6A and 6B). As can be seen, the best pH value for the determination of cyanide by the sensor is 10.0. Since the pK_a of the –COOH group in glutathione is 3.6, the zeta potential of the GSH-capped CdTe QDs is negative at pH 10.0. At an adequately basic pH, the electrostatic repulsion between GSH-capped CdTe QDs affords a stable colloidal suspension, whereas an acidic pH yields insoluble aggregates of GSH-capped CdTe QDs.[1] At higher pH levels, however, GSH-capped CdTe QDs may dissolve and the response may also decrease simultaneously at higher pH medium solutions (pH > 10). This is due to the fact that production of too much –OH groups on the surface of QDs hinders cyanide and GSH-capped CdTe QDs interactions. Therefore, pH 10.0 was selected as the optimum pH for further experiments.

3.4. Effect of the amount of GSH-capped CdTe QDs

In order to optimize the amount of GSH-capped CdTe QDs in solution, six solutions were prepared with different volumes of GSH-capped CdTe QDs (15 to 50 μL of 34.0 nmol L^{-1}) in the presence of 8.0 nmol L^{-1} of SNPs and 1.0 μg mL^{-1} cyanide at pH 10.0. The results are shown in Fig. 7A, clearly confirm that a solution containing 0.45 nmol L^{-1} of GSH-capped CdTe QDs yields the best response to cyanide. Moreover, the sensitivity of the sensor obviously declines with decreasing the amounts of GSH-capped CdTe QDs. A high GSH-capped CdTe QDs content may lead to the self-absorption of the GSH-capped CdTe QDs fluorescence.

3.5. Effect of the amount of SNPs

SNPs were used to inhibit the self-absorption during fluorescence measurement. The influence of the amount of SNPs on the sensor response was investigated by preparing several solutions with different volumes (25 to 120 µL) of 0.24 µmol L^{-1} SNPs in the presence of 0.45 nmol L^{-1} of GSH-capped CdTe QDs and 1.0 µg mL^{-1} cyanide at pH 10.0. Based on the results (Fig. 7B), a solution containing 4.0 nmol L^{-1} of SNPs yielded the best response to cyanide. Higher amounts of SNPs were found to lead to reduce the sensor sensitivity.

4. Analytical figures of merit

Under the optimized conditions, the response function (F–F0) values of the sensor were obtained for different cyanide concentrations. Fig. 8 shows the calibration curve under the optimum conditions. The detection limit ($3S_b/m$, where S is the blank standard deviation (n = 10), and m is the slope of the calibration curve) was obtained to be 0.004 µg mL^{-1}.

To consider the repeatability of the sensor, 0.5 and 2.0 µg mL^{-1} of cyanide solution were measured ten times. The results showed RSD% values of 2.0% and 1.8% for cyanide solutions of 0.5 and 2.0 µg mL^{-1}, respectively.

5. Selectivity

The potential interference of common anions on the selectivity of the sensor was investigated under the optimum conditions. For this purpose, the optical sensor responses to several anions were examined. The tested anions were including 20.0 µg mL^{-1} Br^-, Cl^-, ClO_3^-, $C_2O_4^{2-}$, F^-, I^-, NO_2^-, SO_3^{2-}, SO_4^{2-}, S^{2-}, NO_3^- and SCN^-, and 0.20 µg mL^{-1} CN^-. As

can be seen in Fig. 9, the proposed sensor exhibits a better selectivity for cyanide than for the other ions examined.

6. Application

The applicability of the sensor for real sample analysis was investigated by using the sensor to analyze spiked human serum samples. In addition, to check the accuracy of the sensor, a potentiometric standard method (using CN^- selective electrode) was used to measure the cyanide content in wastewater sample. The results are given in Table 2, confirm the acceptable recovery and accuracy of the sensor.

7. Conclusion

A new optical sensor based on GSH-capped CdTe QDs was developed for the detection of cyanide at ultra-trace levels. The fluorescence intensity of the sensor was considerably enhanced in the presence of cyanide. The sensor was capable of determining cyanide content in the range of 0.01 – 2.5 μg mL^{-1}. The sensor was also found to work in a 'turn-on' mode, which is usually more sensitive than a "turn-off" assay. Higher sensitivity, superior selectivity, low detection limit (0.004 μg mL^{-1}) and ease of production are the most important advantages of the proposed sensor. Finally, the sensor was successfully employed for the determination of cyanide in real samples.

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Table 1. Comparison of analytical data of fluorescence methods for the determination of cyanide.

Fluorophore	Linear dynamic range ($\mu g\ mL^{-1}$)	Detection limit ($\mu g\ mL^{-1}$)	Mode of assay	Ref.
Poly[2-methoxy-5-(3,7-dimethyloctyloxy)-1,4 phenylenevinylene]	0.026 – 15.6	0.0156	Enhancement	13
Rhodamine B	0.01 – 3.12	0.001	Enhancement	14
Copper ion-modified CdTe-QD	0.0078 – 0.312	0.0039	Enhancement	20
2-Mercaptoethane sulfonate-modified CdSe-QD	Up to 6.5	0.029	Quenching	21
2-Mercaptoethanol-capped ZnS-QD	0.063 – 0.67	0.0044	Quenching	23
Glutathione-capped CdTe-QD	0.01 – 2.50	0.004	Enhancement	This work

Table 2. Determination of cyanide in real samples.

Samples	CN^- added, ($\mu g\ mL^{-1}$)	CN^- found*, proposed sensor ($\mu g\ mL^{-1}$)	Recovery (%)	CN^- found*, potentiometric method ($\mu g\ mL^{-1}$)
Human serum sample	0.50	0.49±0.01	98.0	–
	1.50	1.52±0.03	101.3	–
	2.00	1.97±0.02	98.5	–
Wastwater	–	2.38 ± 0.02	–	2.30 ± 0.13

* Average values of six determinations ± standard deviations.

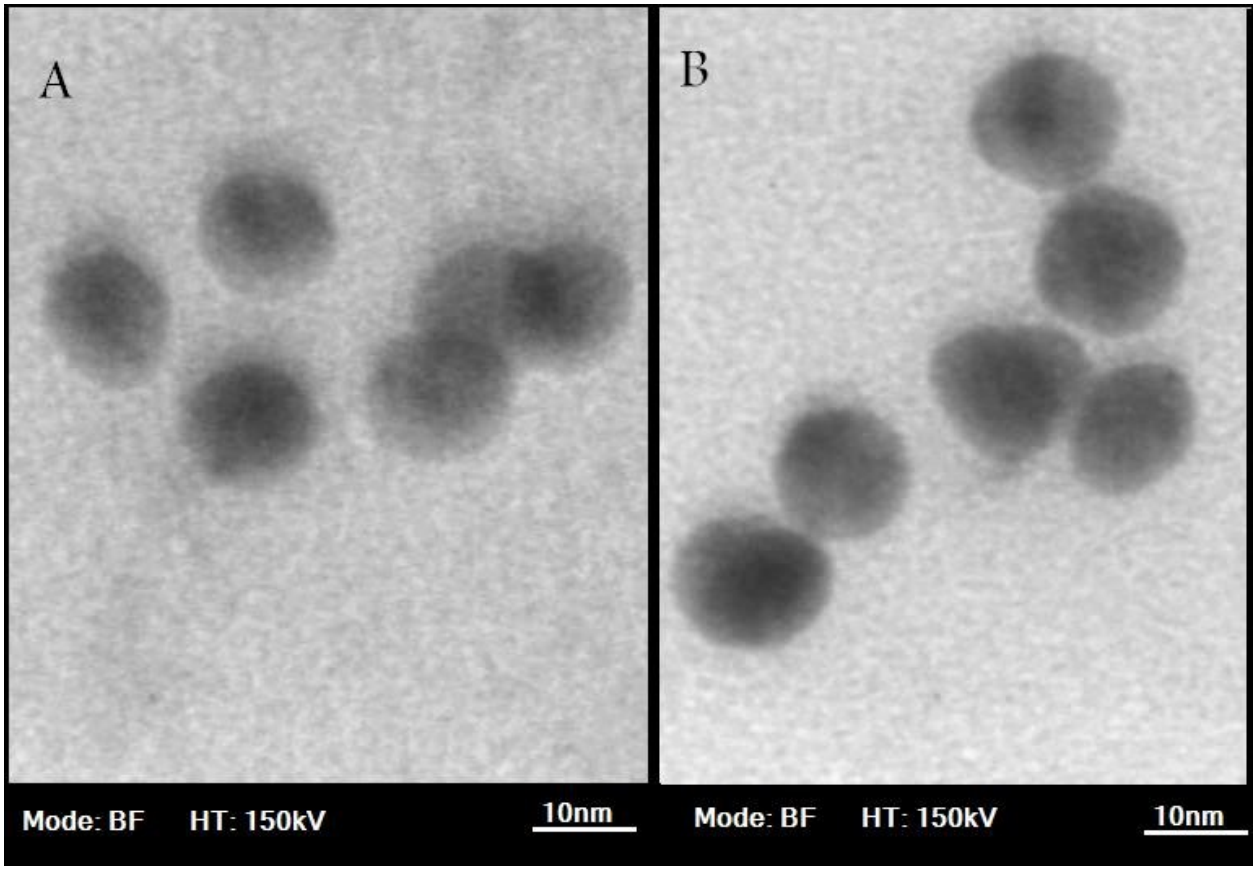


Fig. 1. TEM images of applied nanoparticles A): GSH-capped CdTe QDs; and B): SNPs.

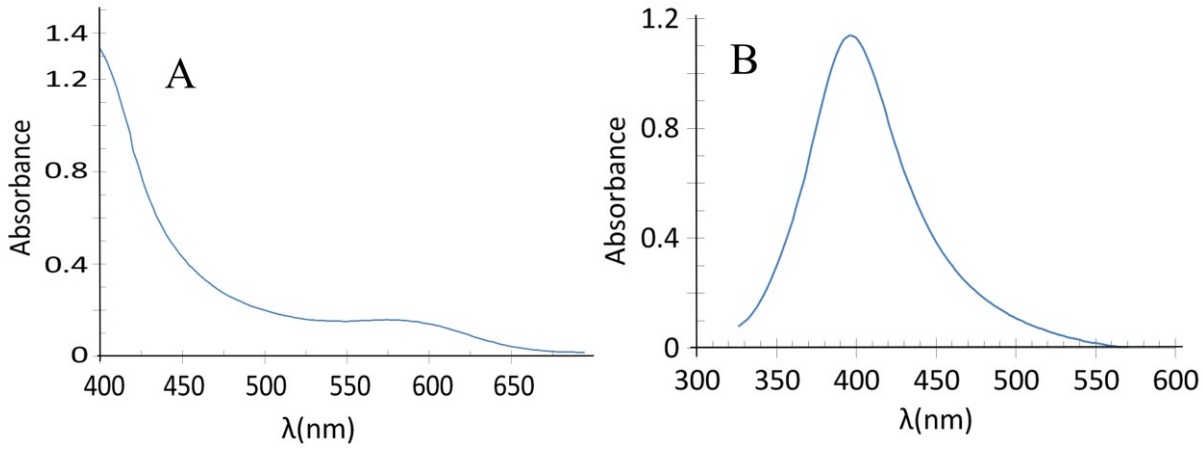


Fig. 2. UV–Vis absorption spectrum of prepared nanoparticles A): GSH-capped CdTe QDs; and B): SNPs.

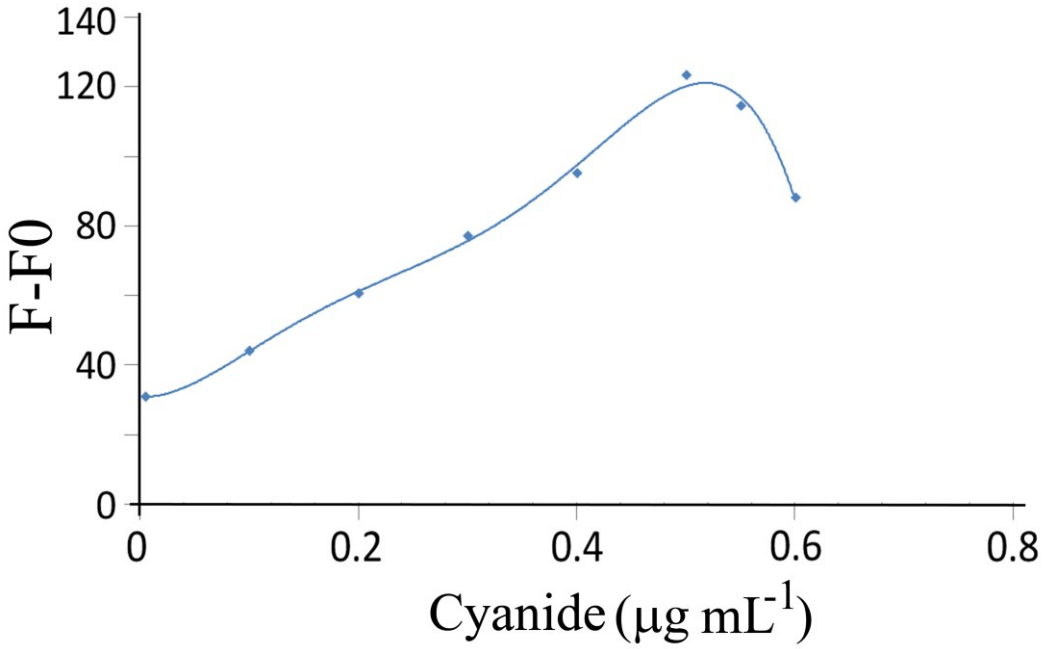


Fig. 3. Effect of CN^- on the fluorescence intensity of 0.45 nmol L^{-1} of GSH-capped CdTe QDs at pH 10.0 in the absence of SNPs.

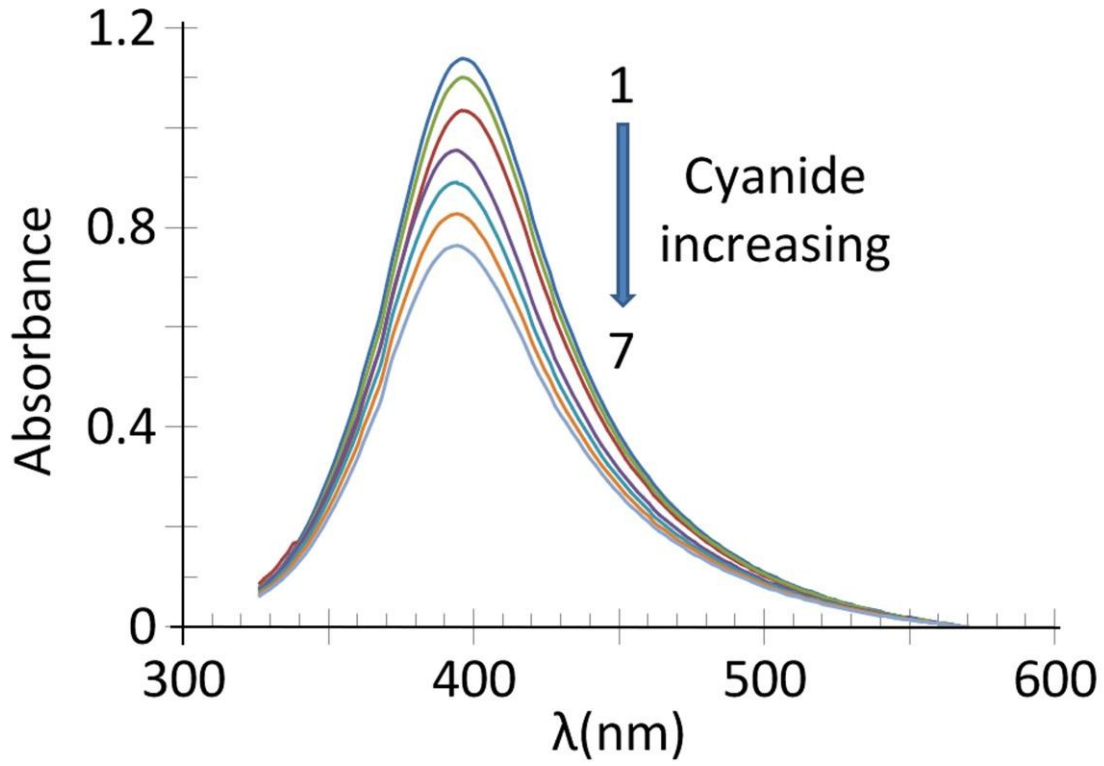


Fig. 4. UV–Vis absorption spectra of SNPs at pH 10.0 containing different concentration of cyanide as: 1) 0.00; 2) 0.015; 3) 0.5; 4) 0.15; 5) 1.00; 6) 1.50; 7) 2.00; and 8) 2.50 µg mL^{-1}.

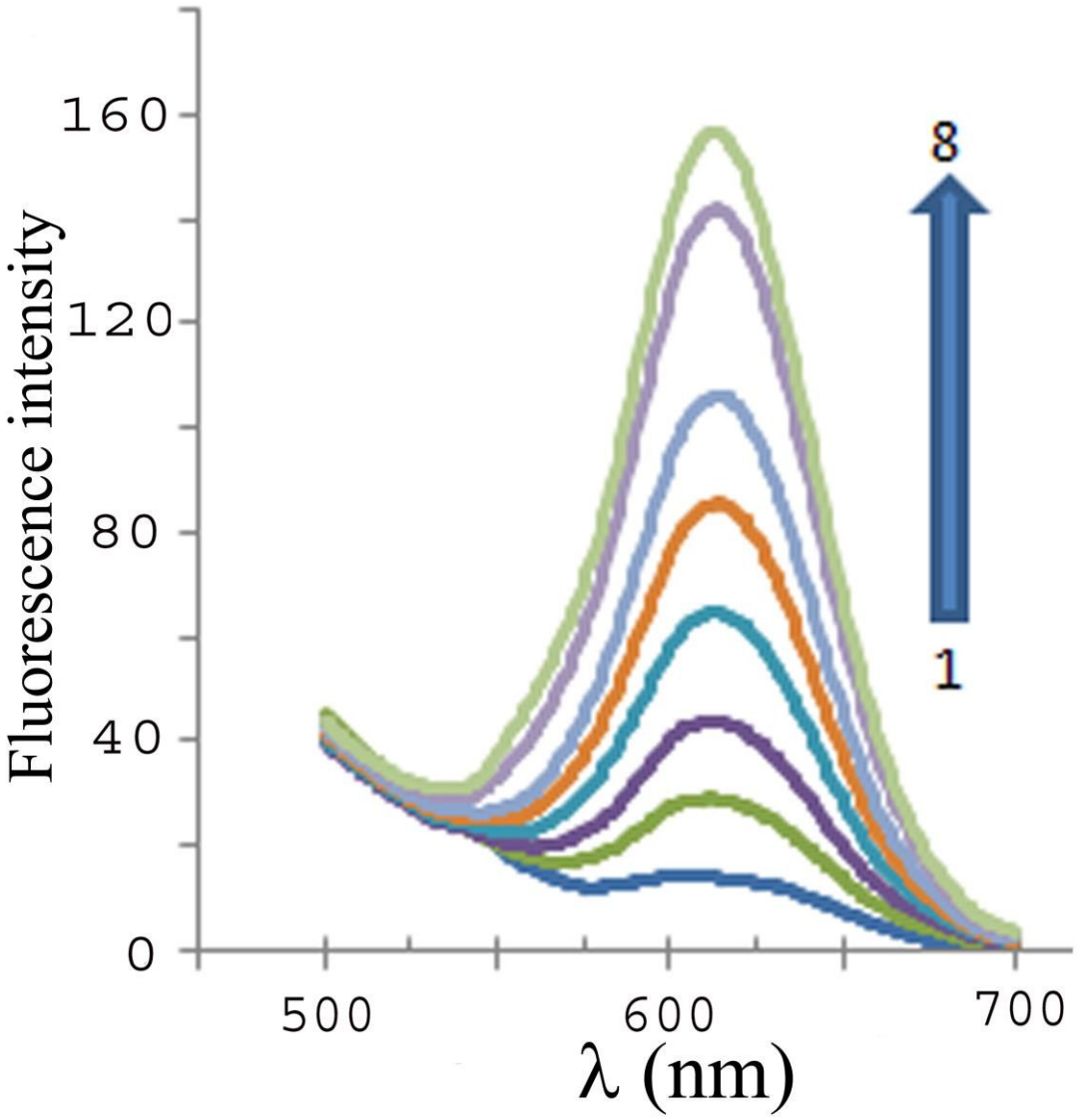


Fig. 5. Fluorescence spectra of the optical sensor. Conditions: 0.45 nmol L^{-1} of GSH-capped CdTe QDs, 4.0 nmol L^{-1} of SNPs at pH 10.0 containing different concentration of cyanide as: 1) 0.00; 2) 0.01; 3) 0.25; 4) 0.50; 5) 1.00; 6) 1.50; 7) 2.00; and 8) 2.50 μg mL^{-1}.

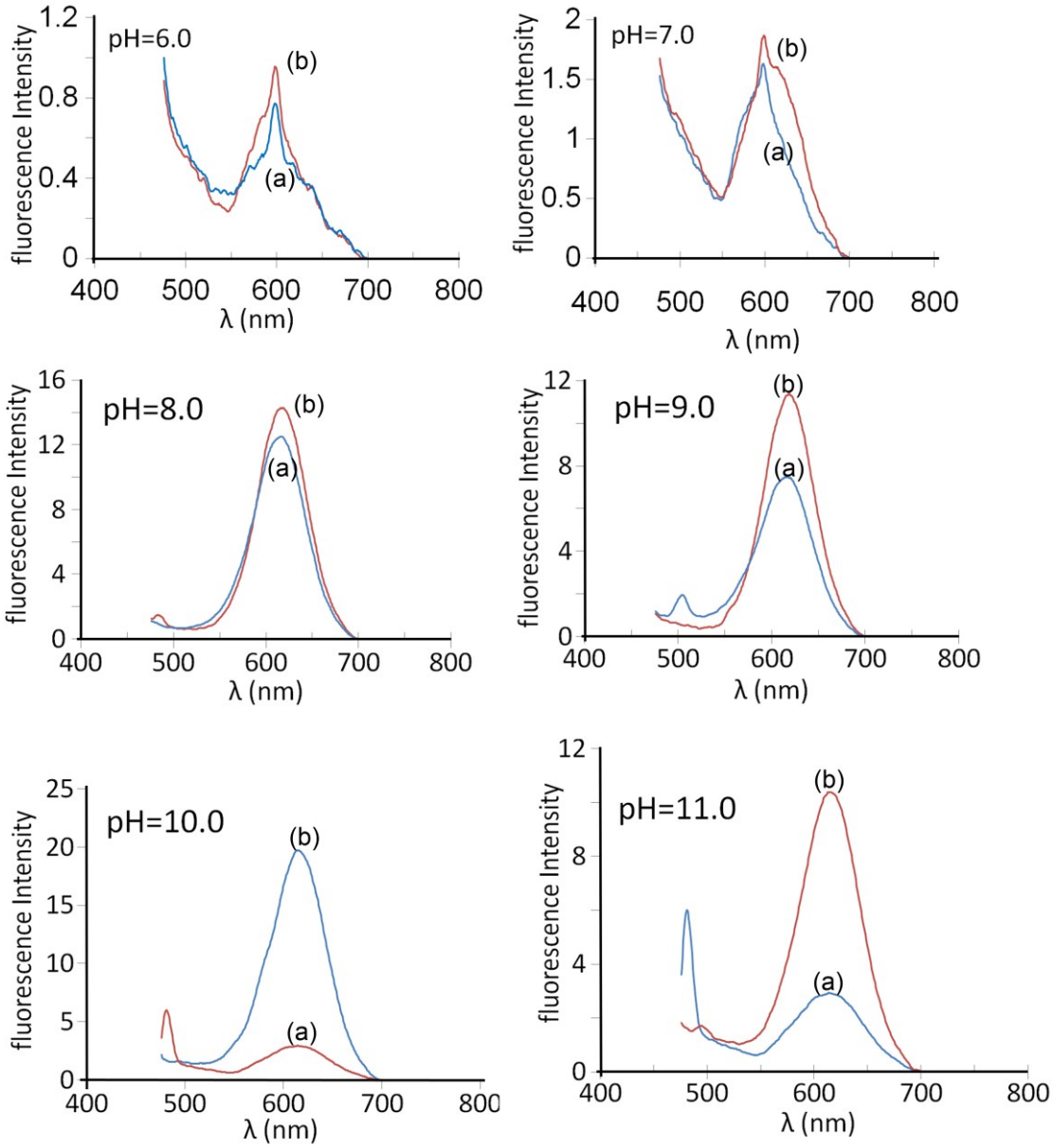


Fig. 6. A): Fluorescence emission spectra of the blank (a) and sample solutions (b), in the presence of 1.0 µg mL^{-1} of cyanide at different pH;

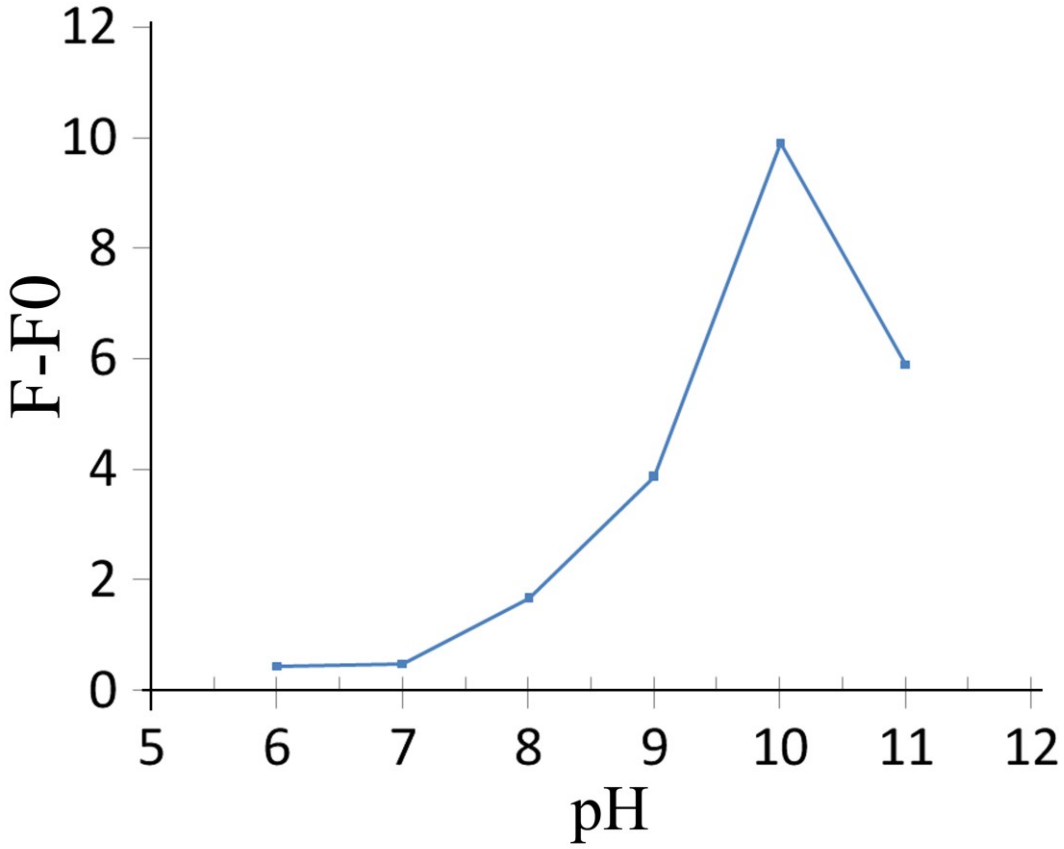


Fig. 6B): Diagram of F–F0 vs. the solution pH. Conditions: 0.28 nmol L^{-1} GSH-capped CdTe QDs in the presence of 8.0 nmol L^{-1} of SNPs.

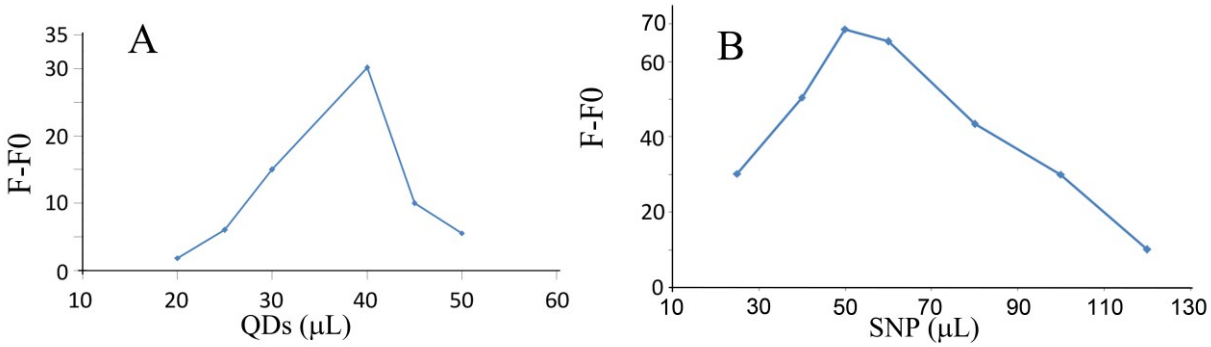


Fig. 7. A): Influence of the amount of GSH-capped CdTe QDs on the sensor response to cyanide ions. Conditions: 1.0 µg mL^{-1} of cyanide, 8.0 nmol L^{-1} of SNPs, pH 10.0 and different volume of GSH-capped CdTe QDs (34.0 nmol L^{-1}). B): Influence of the amount of SNPs on the response of the sensor to cyanide ions. Conditions: pH, 10.0; 1.0 µg mL^{-1} of cyanide, 0.45 nmol L^{-1} of GSH-capped CdTe QDs and different volume of SNPs (0.24 µmol L^{-1}).

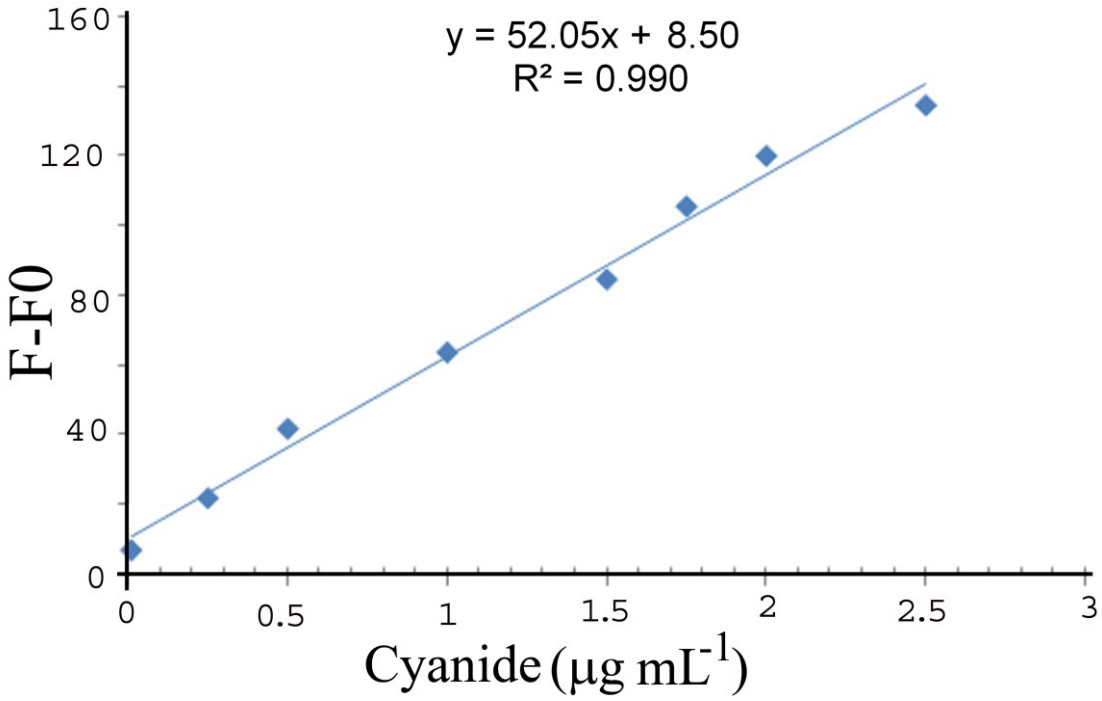


Fig. 8. Calibration graph for the cyanide determination at the optimum conditions. Conditions: pH, 10.0; 0.45 nmol L^{-1} of GSH-capped CdTe QDs and 4.0 nmol L^{-1} SNPs.

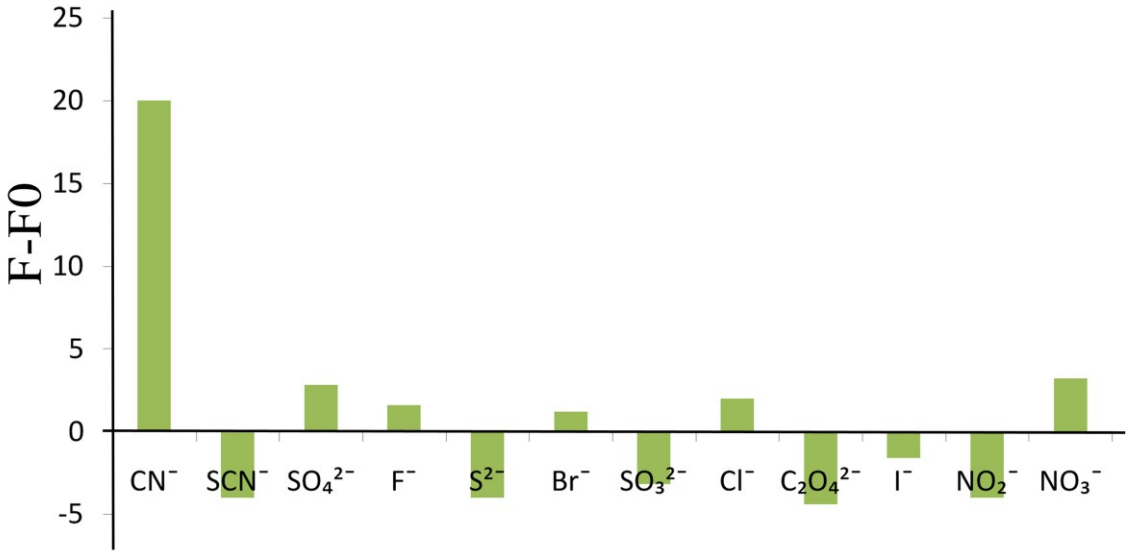
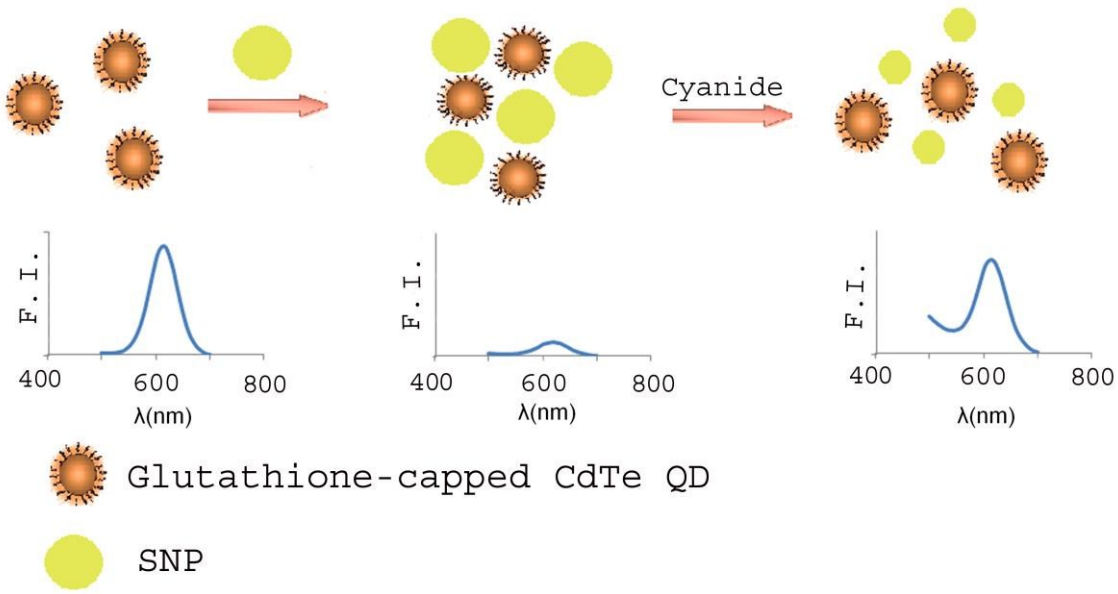


Fig. 9. The fluorescence response of a mixture containing 0.45 nmol L^{-1} of GSH-capped CdTe QDs, 4.0 nmol L^{-1} of SNPs at pH 10.0 in the presence 0.20 µg mL^{-1} cyanide and 20.0 µg mL^{-1} of the other anions.

145



146

147 Scheme 1. A turn-on fluorescent cyanide sensor based on GSH-capped CdTe QDs using

148 silver nanoparticles.