



Colorimetric detection of cadmium ions using modified silver nanoparticles

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Abstract

Metal ion sensors are a significant and challenging area in the analytical sciences. In this study, the use of grape juice for bio-synthesizing of silver nanoparticles (AgNPs) and using it for simple and rapid colorimetric detection of Cd²⁺ ions is described. The as-prepared AgNPs were characterized by standard analytical techniques including UV–visible and FTIR spectroscopic methods and transmission electron microscopy (TEM). The plasmon resonance band of the silver nanoparticles was observed at 410 nm and it was found that its intensity is related to the grape juice concentration and pH. The TEM image showed the average size of 5–10 nm for AgNPs. The detection of Cd²⁺ ions was based on the changes of absorbance due to complex formation of the metal ion. The colorimetric detection of Cd²⁺ was led to a linear dynamic range from 0 to 150 μmol/L ($r^2 = 0.9993$) and a low detection limit of 4.95 μmol/L for cadmium in aqueous solution. These results are close to or better than the previous reports. This bio-synthesized AgNPs can be used as simple alternative design for colorimetric sensing of Cd²⁺ in water samples.

1 Introduction

Heavy metal contamination is a great environmental concern worldwide. Cadmium is among the toxic and highly harmful metal ions. It is mostly used in industrial workplaces such as paint pigmentations, nickel–cadmium batteries, and electroplating. This metal ion can cause pollution of soil, water, and air [1, 2]. The metal can accumulate in human body in the form of cadmium salts and may affect the liver, kidneys, brain, lungs, heart, testes, and the central nervous system. The mechanisms for the toxicity of cadmium are not well understood. However, most often it is responsible for lipid peroxidation and free radical formation [3]. Accumulation in the kidney and the liver can result in long term toxicity because cadmium has a half-life of 4–19 years [4]. Therefore, according to the international standards for drinking

water such as WHO (world health organization) and ESEPA (United States Environmental Protection Agency), the permissible limit of Cd (II) ions in drinking water is 3.0 μg/L [5, 6]. Therefore, there is an increasing request for determination of low concentration of cadmium ions in water and wastewater samples.

To date, there are many classical analytical techniques used for the detection of cadmium ions in environmental- and biological samples. These include atomic absorption spectrometry [7], inductively coupled plasma mass spectrometry [8, 9], inductively coupled plasma-atomic emission spectrometry [10, 11], fluorescence spectroscopy [12], and graphite furnace atomic absorption spectrometry [13]. Many of these methods are expensive and time-consuming and need to be carried out by trained personnel due to the complex sample preparation and inability to use onsite operation. Therefore, it is highly desirable to use other types of methods and/or materials with lower costs and easier operation [14].

In the recent decades, nanoscience and nanotechnology have been developed and this has led to opening up new areas for the applications of nanomaterials [15]. In the analytical sciences, ultrasensitive detection and imaging methods have advanced [16]. Particularly, silver- and gold nanoparticles based on colorimetric sensors are now achieving increase attention. Because these nanoparticles have strong localized surface plasmon resonance (SPR)

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absorption and the optical properties related to inter-particles distances [17–19]. Speed, high sensitivity, cost-effectiveness, and ease of measurement are advantageous of colorimetric sensing methods [20–22]. The color changes associated with the surface plasmon absorption band is dependent on a number of parameters such as the size and shape of the particle, the adsorbed species, the dielectric properties of the medium, and the distance between particles [23]. Various analytes which have now been detected by SPR of gold- and silver nanoparticles which include metal ions [24], lectins [25] antibodies, and proteins [26, 27]. In general, metal nanoparticles surface modification is one of the best method for developing of metal nanoparticles as colorimetric detecting probes [28].

Chemical reduction of silver salts is the common synthetic route adopted for the preparation of silver nanoparticles (AgNPs). This involves the use of suitable solvents, reducing agents, and stabilizing agents [29, 30]. For preparation of silver or gold nanoparticles the use of naturally reducing and stabilizing agents in green chemistry are very important in comparison with the use of alternative highly toxic and reactive hazardous chemicals [31–36]. Fruit juices can act as a reducing agents as well as stabilizing agents in silver nanoparticles synthesis. This is due to the heteroaromatic rings, hydroxyl, carbonyl, and carboxyl groups existed in their structures [33]. In this work, grape juice was used for synthesis of silver nanoparticles. The reduction of Ag^+ ion and the stabilization of AgNPs were due to presence of polyphenols in the grape juice.

2 Experimental

2.1 Chemicals and materials

Silver nitrate, sodium hydroxide, and the standard solution (1000 ppm) of Ni^{2+} , Cr^{3+} , Pb^{2+} , Zn^{2+} , Cu^{2+} , Co^{2+} , Cd^{2+} , and Hg^{2+} ions were purchased from Merck chemical company (Germany). They were used without further purification. Other reagents and chemicals were of at least analytical reagent grade.

2.2 Grape juice preparation

The grape was bought from the local market and washed with double distilled deionized water. It was completely crushed using a blender and then, filtered using Whatman filter paper to get grape juice.

2.3 Synthesis of silver nanoparticles

Silver nanoparticles were synthesized with modification on previously reported procedure [37]. For this purpose,

the grape juice extract was diluted by deionized water. Different solutions ($V_{\text{juice}}/V_{\text{water}} = 20:0, 15:5, 10:10,$ and $5:15 \text{ mL:mL}$) were made and then cooled in ice cold water. The solution was made alkaline (pH 11) by adding NaOH and it was mixed for 10 min. Then, it was placed on a heating mantle. During heating 0.6 mL of 50 mmol/L aqueous silver nitrate solution was added drop wise with continuous stirring. It was heated for 5 min at 70 °C. The color of the solution gradually changed from light red to brownish-yellow. The brownish-yellow color is an indication of the formation of AgNPs [38].

2.4 Characterization

The absorption spectra of AgNPs were recorded using a DR-6000 Hach Lange spectrophotometer. The TEM images were taken using Philips CM-10 transmission electron microscope. The fourier transform infrared (FTIR) spectra of the synthesized AgNPs were obtained using Thermo FTIR (Avatar model).

2.5 Determination of Cd^{2+}

The colorimetric detection of aqueous Cd^{2+} ion was studied using the modified AgNPs solution at room temperature. To perform the determination of Cd^{2+} , 500 μL portions of several different concentrations of Cd^{2+} ion (0–150 $\mu\text{mol/L}$) were added one at a time to 3 mL of AgNPs. The resulting mixture was then allowed to stand for 10 min at room temperature, during which the brownish-yellow color changed to brownish-orange. The spectrophotometer was used for UV–vis absorption measurements.

2.6 Measurement of Cd^{2+} ions in real samples

The concentration of Cd^{2+} ion in different water samples were determined using our method. The water samples were taken from two different rivers (Shahr-e-Bijar and Sepidrood in Guilan province of Iran). The drinking water was also checked for Cd^{2+} . The different concentrations of Cd^{2+} ion (1, 50 and 100 $\mu\text{mol/L}$) were spiked into the collected water samples. For this purpose, the collected water samples were filtered through the 0.45 μm filter and then was spiked with the standard solutions of Cd^{2+} . Each water sample was analyzed three times by the above described procedure.

3 Results and discussion

3.1 Grape juice for formation of AgNPs

Chemical reductants such as NaBH_4 have been widely used to reduce Ag (I) to Ag (0) for production of AgNPs.

However, in this work, we propose a green route to synthesize AgNPs using grape juice as a bio-reductant. The freshly prepared grape juice consists of 70–80% water and many dissolved solids. These soluble solids include numerous organic compounds such as sugars, organic acids, phenolic compounds, nitrogenous compounds, etc. [39]. These compounds have functional groups that can react as reductant, modifier, and stabilizer. Scheme 1 shows possible reaction mechanism for AgNPs synthesis using the bio-reductant ascorbic acid (vitamin C) that is contained in grape juice. At the end of the reaction, the brownish-yellow color indicates formation of AgNPs.

3.2 Characterization of silver nanoparticles

FTIR spectra of the grape juice, AgNPs, and AgNPs complexed with Cd^{2+} are shown in Fig. 1. These spectra were recorded to identify the presence of biomolecules in grape juice such as ascorbic acid (vitamin C), riboflavin (vitamin B2), thiamine (Vitamin B1), etc. These are responsible for reduction of Ag(I) to metallic silver (Ag) and also for stabilizing AgNPs. The FTIR of the samples show broad peak between 3000 and 3600 cm^{-1} which may be due to hydroxyl (OH) stretching bands of alcohols and phenols [39]. Absorption bands at 2875 cm^{-1} are characteristic of C–H stretching vibration. In grape juice the peaks at 1720, 1020 cm^{-1} may result from the C=O stretching and C–OH bending, respectively [40, 41]. Comparison of the FTIR spectra of grape juice and AgNPs indicates that band at 1020 cm^{-1} decreases after bio-reduction. It seems that polyols are the

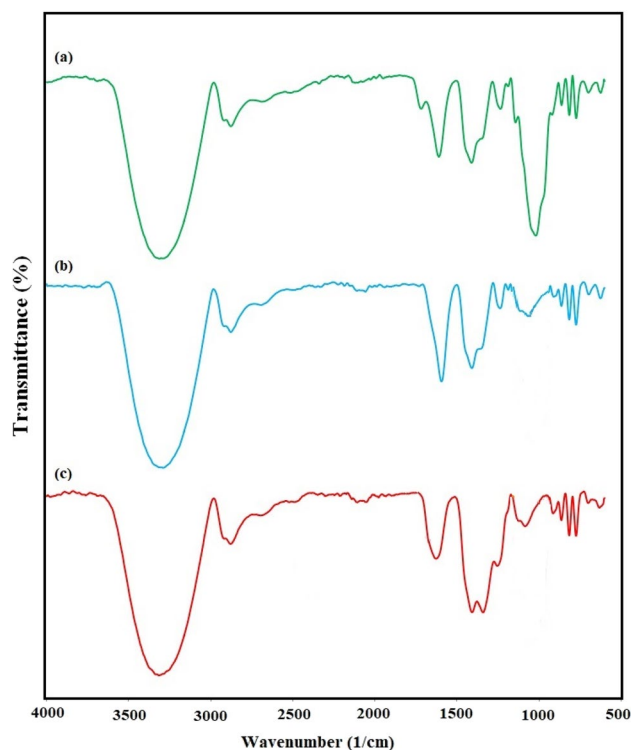
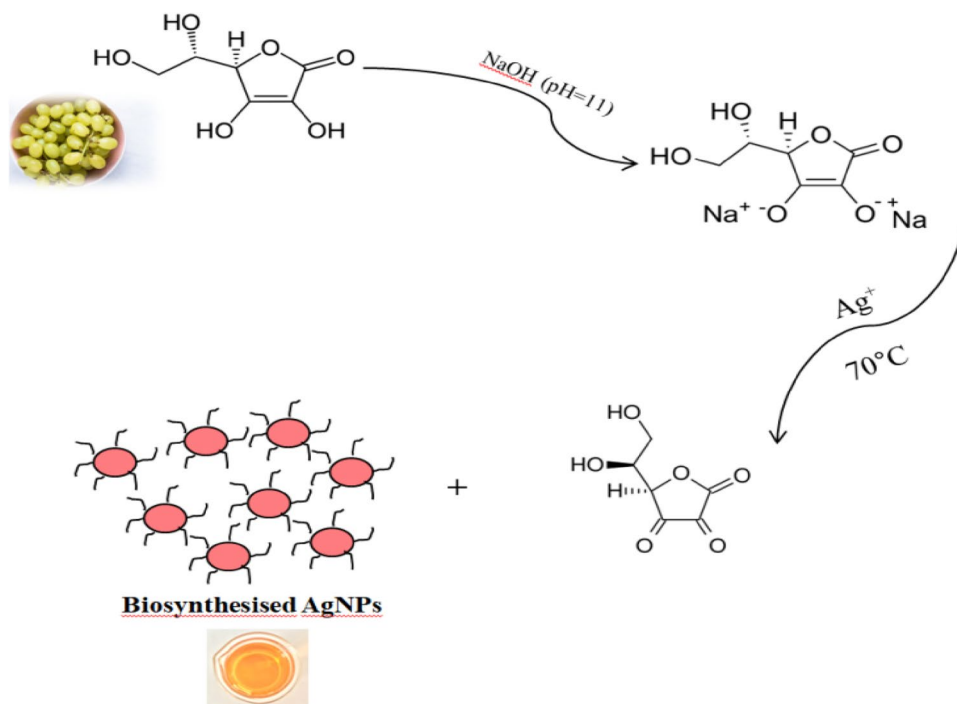


Fig. 1 FTIR spectra of **a** pure grape juice, **b** AgNPs, and **c** cadmium complex of AgNPs

main species in charge for reduction of Ag(I). The vibrational bands (O–H, C–H, C=C ring, C–OH and C–C ring) are derived from water soluble compound in juice such as

Scheme 1 Possible reaction mechanism for AgNPs synthesis. (●) Bio-synthesized AgNPs with capping agent



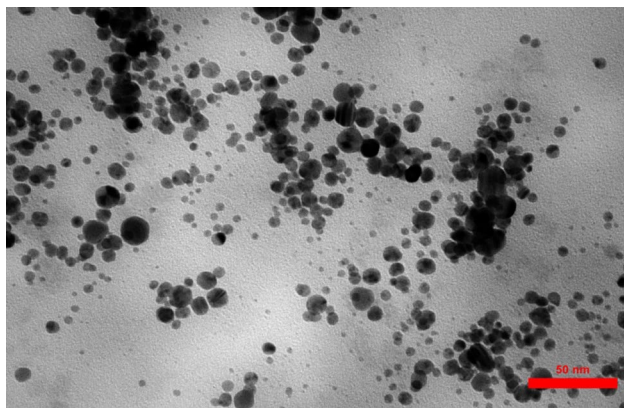


Fig. 2 Transmission electron micrographs of AgNPs

ascorbic acid, as well as flavoids and polyphenols. They are responsible for the efficient stabilization or capping of AgNPs [42]. When Cd^{2+} ions form complexes with AgNPs, the peak at 1593 cm^{-1} shifts to 1627 cm^{-1} .

The TEM image of AgNPs are shown in Fig. 2. It was observed that the particles were mostly spherical and their sizes are varied within 5–10 nm.

3.3 Formation and stability of AgNPs

For preparation of AgNPs, four different volume/volume compositions of juice: water ratio at pH 11 was examined. For the 20:0 ratio, a broad absorption band was observed at about 410 nm which is sharp but not smooth. But with 15:5 ratios, the plasmon band appeared. It is sharp and smooth with a maximum at 410 nm. For the composition 10:10 and 5:15 ratios, containing less amount of grape juice, the intensity of the absorption band was decreased (Fig. 3). So, the sample with 15:5 ratio (15 mL juice: 5 mL water) was selected as the best. Also UV–visible spectra of the pure juice (extracted grape juice without any treatment for synthesizing AgNPs) did not show any absorption band in 300–800 nm area wavelength.

We found that formation of AgNPs in addition to the concentration of the grape juice is depended on pH too. Thus, the dependency of AgNPs formation on pH was investigated. The pH of the selected juice/ water = 15:5 sample varied from 5 to 12 by addition of NaOH and then AgNPs were synthesized. The relationship between pH and the relative amount of AgNPs formation is shown in Fig. 4a. The relative amount of AgNPs is defined as the absorbance at 410 nm divided by the maximum absorbance at the same wavelength (which is gained at pH 11). The mathematical relation in percent is described as below:

$$\text{Relative amount(\%)} = \frac{\text{Absorbance in pH:5}}{\text{Absorbance in pH:11}} \times 100.$$

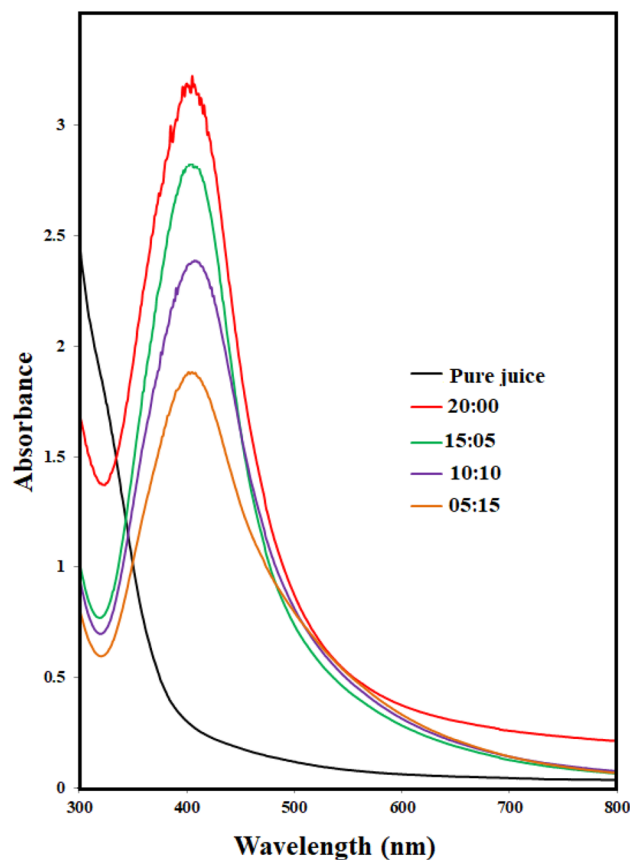


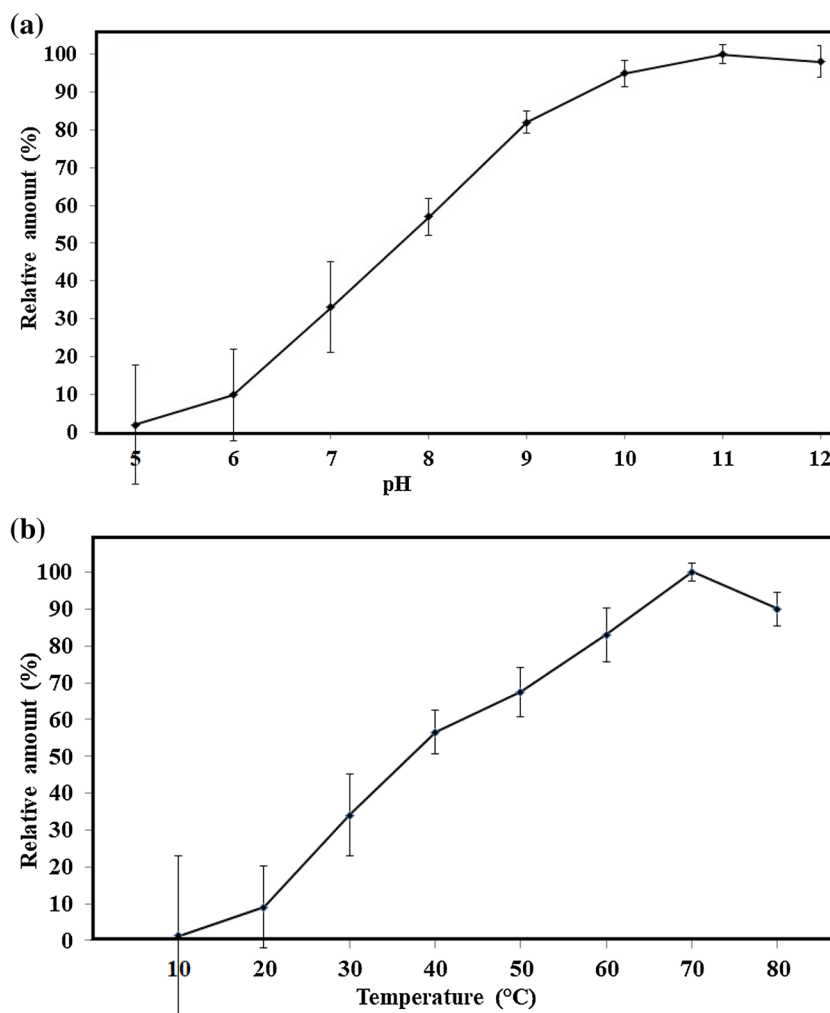
Fig. 3 UV–visible spectra of AgNPs prepared with different $V_{\text{juice}}/V_{\text{water}}$ ratio. The numbers in figure is for designation of the spectra related to juice: water ratios. The spectrum for pure grape juice is also shown

It was found that at pH 11, the narrow and sharp peak with high absorbance is obtained. With the decrease of pH, the plasmon band gradually broadened and the absorbance is lowered. At below pH 5, the AgNPs were not formed at all. The lack of the formation of silver nanoparticles at low pH have been observed by other researchers as well [37].

The relation between the temperature and relative amount of the colorimetric object is shown in Fig. 4b. By increasing the temperature from 10 to 70 °C, the relative amount of AgNPs increased from 1.2 to 100%. In contrast, by further increasing the temperature the relative amount of the silver nanoparticles declined to 90%. Thus, temperature of 70 °C was found to be the optimal temperature.

UV–visible spectroscopy is one of the simplest techniques to determine particle formation and its stability. It is known that the surface plasmon resonance spectra of nanoparticles are influenced by the size, shape, inter-particle interactions, free electron density, and surrounding medium [43]. Therefore, UV–vis is an efficient tool for monitoring the stability of AgNPs. To investigate the stability of our plasmonic silver nanoparticle, its absorbance intensity was

Fig. 4 Formation of AgNPs related to **a** pH and **b** temperature variables. (The error bars represent the standard deviation of three measurements)



measured for 30 days every 2 days (Fig. 5). There were no considerable changes in the shape, position, and the symmetry of the absorption peak during the tested 30 days period. A very short decrease in the absorbances is the only difference among them. This result proves that the plasmonic AgNPs are stable.

3.4 Sensing ability

The UV–visible absorption responses were used to evaluate the sensitivity of our AgNPs' system. Therefore, a series of different concentration of Cd^{2+} ions (0–150 $\mu\text{mol/L}$) were added into AgNPs solutions. Figure 6 shows the UV–visible spectra of AgNPs following the addition of Cd^{2+} ions. The SPR peak at 410 nm gradually decreases upon the increase of Cd^{2+} ions concentration. This is accompanied with the increase of absorbance in the range of 500–600 nm. Also, the color of AgNPs solutions changed from yellow to orange upon increasing concentrations of Cd^{2+} ions.

The color changes could be due to the presence of functional groups responsible for the stabilization of

AgNPs. The structure of riboflavin, thiamine and phenolic compounds have $-\text{NH}_2$, $-\text{OH}$ and $\text{O}=\text{C}-\text{NH}-\text{C}=\text{O}$ functional groups (Fig. S1). Based on the reported works, free $-\text{NH}_2$ and hydroxyl group were believed to take part in cross-linking and formation of the complexes with Cd^{2+} ions. The cadmium ions are known to bind well with these groups which contain lone pair electrons. Based on the hypothesis, this complexation leads to the decrease in the inter-particle distances among the nearby nanoparticles. This eventually resulted as a red-shift in the SPR peak from 410 to 580 nm [42, 44]. Upon increasing the concentration of Cd^{2+} ions, the color varies to orange. This is correlated with the reduction of the absorbance at 410 nm and increasing the absorbance at about 580 nm.

The linear range for cadmium ions is from 0 to 150 $\mu\text{mol/L}$ as shown in Fig. 6b. The $\text{Ex}580/410$ was used instead of absorbance for the calibration plot. The extinction coefficients at 410 and 580 nm are related to the quantities of the dispersed and aggregated AgNPs, respectively. Therefore, the parameter $\text{Ex}580/410$, which is the ratio of the extinction coefficients at these two

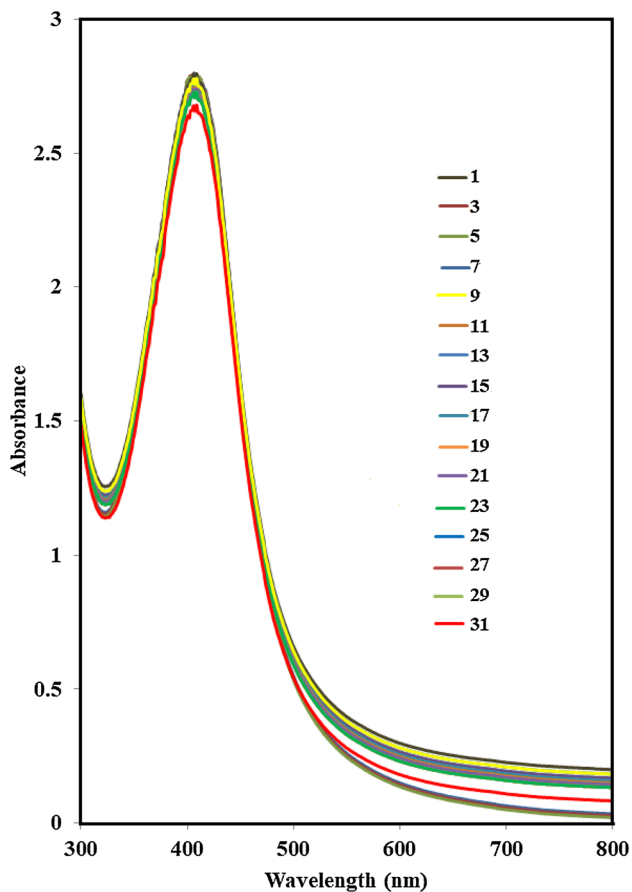


Fig. 5 Effect of time on stability of AgNPs. The numbers inserted into the figure are the day of reading absorbance

wavelengths is more appropriate for the quantities AgNPs related to cadmium concentration. The detection limit was found to be 4.95 $\mu\text{mol/L}$. The resultant linear range and the detection limit of our proposed colorimetric procedure is comparable with those reported previously. Table 1 compares the linear range and limit of detection (LOD) for the procedures using different plasmonic nanoparticles/capping agents for determination of cadmium ions reported by other workers.

3.5 Interferences in cadmium detection

The selectivity of a diagnostic experiment is very important in real sample applications. To test the selectivity of developed procedure in detection of Cd^{2+} , some control experiments were performed using Ni^{2+} , Cr^{3+} , Pb^{2+} , Zn^{2+} , Cu^{2+} , Co^{2+} , and Hg^{2+} . The selectivity experiments were carried out using 200 $\mu\text{mol/L}$ of different ions (Fig. 7). The parameter $\left[\left| \frac{A-A_0}{A_0} \right| \times 100 \right]$ is used for the selectivity measurement. In this equation, A_0 and A are the absorbance at 410 nm before and after addition of the interfering ion. It is clear that the synthesized AgNPs were highly selective to Cd^{2+} ions, resulting in the largest absorbance difference, and thus providing a positive indicator for qualitative detection and quantities application.

3.6 Determination of Cd ions in real samples

Cadmium ions in different water sources were determined using our method (Table 2). The water samples were drawn from two different rivers (Shahr-e-Bijar and Sepidrood) and from two different drinking water suppliers. Each water sample was measured three times. In our colorimetric method, samples were spiked with known concentration of Cd^{2+} to check the recovery and precision of this method. The unspiked water had no detectable Cd^{2+} while the recovery percentages of the spiked samples were between 95 and 102% with a precision better than 5%.

4 Conclusion

A fast and effective spectrophotometric detection method for cadmium ions based on AgNPs was developed. The bio-synthesized AgNPs were prepared via a simple method using grape juice as the reductant and stabilizer. The AgNPs tend to aggregate upon addition of Cd^{2+} due to strong coordination bond between Cd^{2+} and functional groups such as peptide linkage or hydroxyl of riboflavin, ascorbic acid, and other phenolic compounds in grape juice. The detection limit for this method was found to be 4.95 $\mu\text{mol/L}$ with precision better than 5% for replicate samples analysis. This method can be used for precise and accurate measurement of cadmium in various environmental water samples.

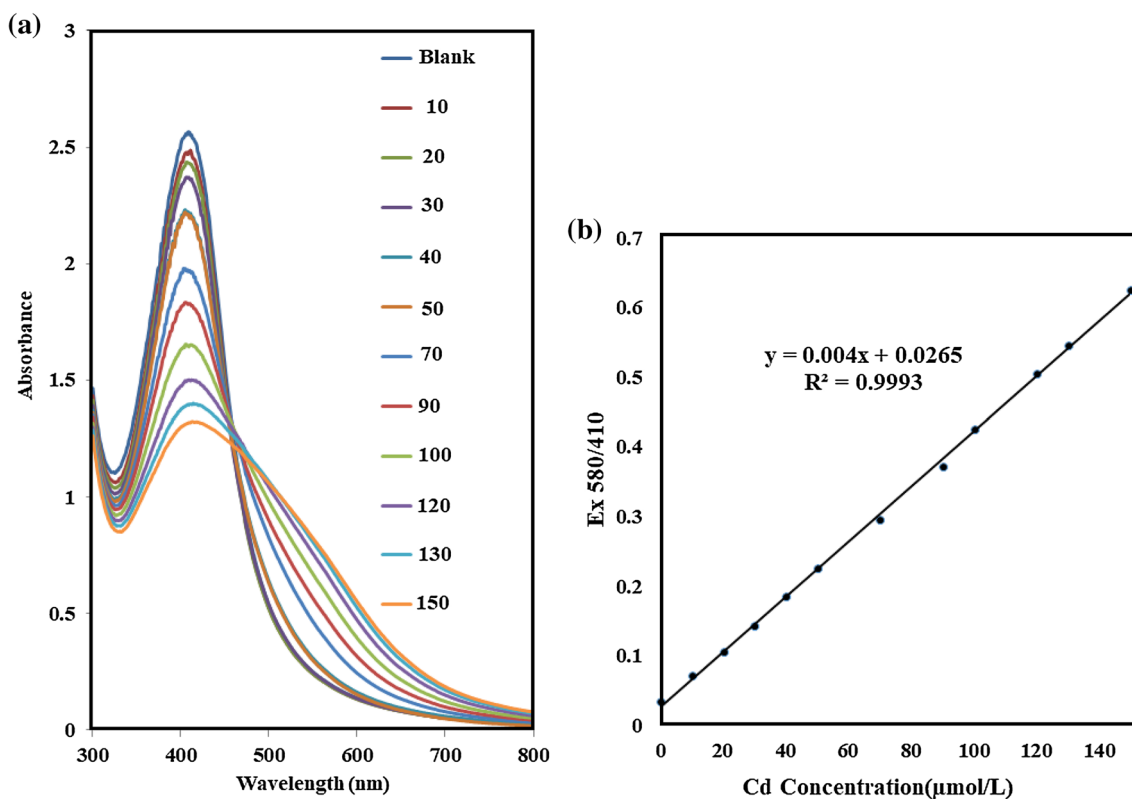


Fig. 6 UV-vis spectra **a** and calibration curve **b** for AgNPs containing different concentration of Cd²⁺ ions. The numbers inserted in part a are cadmium concentration in µmol/L)

Table 1 Comparison of limit of detection (LOD) for Cd(II) determination using different plasmonic nanoparticles

Plasmonic NP/capping agent	LOD (µmol/L)	References
AgNPs/5-sulfoanthranilic acid dithiocarbamate	5.8	[44]
AgNPs/perylene dye	10	[45]
AuNPs/glutathione	5	[46]
AgNPs / <i>N</i> -(2-hydroxybenzyl)-valine, <i>N</i> -(2-hydroxybenzyl)-isoleucine	1	[47]
AgNPs / 1,13-bis(8-quinolyl)-1,4,7,10,13-pentaoxatridecane	2	[48]
AuNPs/peptide ligand	0.05	[49]
AgNPs/5-sulfosalicylic acid	0.003	[50]
AgNPs/grape juice	4.95	This work

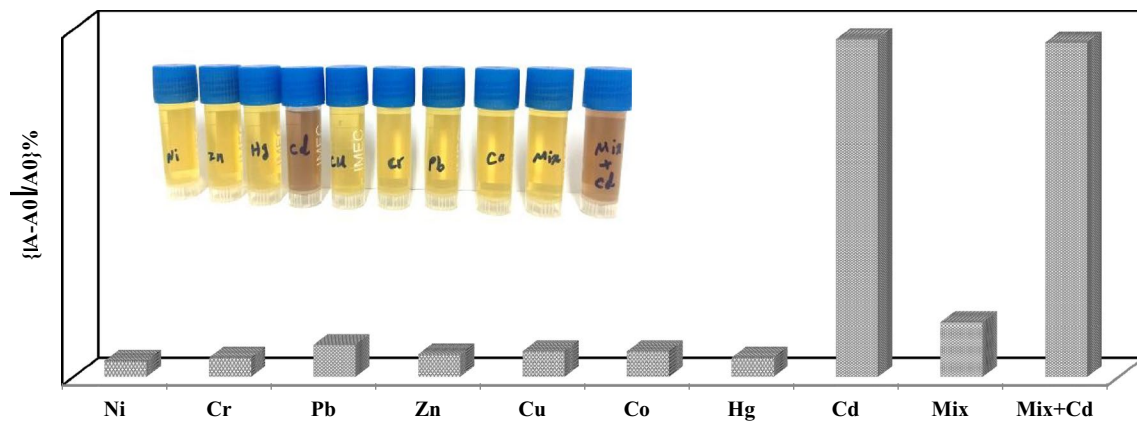


Fig. 7 The selectivity of our method for Cd^{2+} determination in presence of 100 $\mu\text{mol/L}$ Cd^{2+} and 300 $\mu\text{mol/L}$ other metal ions

Table 2 Determination of cadmium ions in different selected water samples

Sample	Added ($\mu\text{mol/L}$)	Found ($\mu\text{mol/L}$)	%Recovery	%RSD
Drinking water 1 (Sepidrood)	10	9.85	98.55	1.40
	20	19.92	99.62	1.38
	50	50.30	100.61	1.30
	100	100.45	100.45	1.02
Drinking water 2 (shahre Bijar)	10	9.88	98.75	2.07
	20	19.69	98.42	1.13
	50	50.55	101.09	1.64
	100	100.31	100.3	1.46
River 1 (Sepidrood)	10	9.62	96.10	1.99
	20	19.82	99.05	1.79
	50	50.61	101.2	1.47
	100	100.45	100.44	1.05
River 2 (shahre Bijar)	10	9.79	97.78	1.23
	20	19.79	98.89	1.42
	50	50.54	101.06	1.29
	100	100.53	100.52	0.87

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