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## Development a Simple and Sensitive Method for Determination Low Trace of Nickel by Localized Surface Plasmon Resonance of Citrate Capped Silver Nanoparticles

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**Abstract:** Nickel is a toxic transition metal which can create serious problems in health of humans and wildlife. Thus determination of nickel in environmental samples like waters is important and inevitable. In this research a simple and sensitive method for determination of nickel in water was described based on aggregation of citrate capped silver nanoparticles. Silver nanoparticles were prepared by reduction of silver nitrate with sodium borohydride. Cefixime was as chelating agent in the assay and when nickel was added to the mixture of citrate capped silver nanoparticles and cefixime, color of citrate capped silver nanoparticles changed from light yellow to red that depend on Ni<sup>2+</sup> concentration. As a result of aggregation, localized surface plasmon resonance (LSPR) band of silver nanoparticles around 395 nm decreased and a new peak appeared in 550 nm. Control experiments with 10 other ions carried out and did not a distinct change in color or spectrum. Under optimized conditions, linear relationship between Ni<sup>2+</sup> concentrations and the absorbance ratio of A<sub>550</sub>/A<sub>395</sub> and limit of detection were found in the range of 2.70 μmol L<sup>-1</sup> to 17.10 μmol L<sup>-1</sup> and 0.80 μmol L<sup>-1</sup>, respectively. The method was applied in tap and well waters that indicate the colorimetric method has acceptable accuracy and good stability.

**Keywords:** Silver Nanoparticle, Localized Surface Plasmon Resonance, Nickel, Colorimetric Detection.

## 1. INTRODUCTION

Heavy metals are a group of environmental chemicals that are ubiquitous and non-biodegradable. Harmful effects of heavy metals like mercury, cadmium and others are widely known in human nature. Many studies about effects of heavy metals on human nature and environment carried out all around the world. To protect health of humans in the world, many international organizations set standards in particular drinking water and etc. Due to pervious reasons, determination of heavy metals in human nature and environment is very important and necessary [1,2].

As the environmental concern, researchers have developed many of methods for determination nickel in water via various sensitive analytical methods like photochemical vapor generation-batch type ultrasonication assisted gas liquid separator-atomic absorption spectrometry (PVG-BT-UAGLS-AAS) [3], high-resolution continuum source atomic absorption spectrometry (HR-CS-AAS) [4], sequential injection-electrochemical analysis (SIECA) [5], inductively coupled plasma optical emission spectrometry (ICP-OES) [6]. Although these methods are accurate and sensitive, they are time-consuming and need to expensive instruments and complicated sample pretreatment procedures are necessary.

Localized surface plasmon resonance (LSPR) is a new method which attracted attention of many scientists in recent decade. LSPR refers to the collective oscillation of electrons on the metallic nanoparticles (NPs) excited by the incident photons at the resonant frequency [7,8]. The optical properties, especially the plasmonic properties, of noble metal nanostructures are used in the development of a new class of optical sensor in modern chemistry. Among these nanometals, gold and silver nanoparticles are the most extensively investigated due to their unique optical properties connected with this phenomenon [9,10]. Upon interaction of light and metal nanoparticles, free electrons of NPs undergo a collective coherent oscillation with respect to the positive metallic lattice [11]. As a result, the metal NPs display bright intense colors and corresponding specific extinction bands in their UV-Vis spectra [12,13]. When metal nanoparticles are dispersed in aqueous phase, solution shows a distinctive color attributed to their small size, and they exhibit a strong UV-Vis extinction band and display different bright colors. For example, in AuNPs or AgNPs, when surface resonance bands of noble metal nanoparticles are exposed to electromagnetic radiation, electrons on the surface can absorb light and color of the solution appears red and yellow, respectively [14,15]. The color changes of a solution in

presence of other ions, can be spectrometrically monitored and used as analytical signal for the detection of chemical species. For example, LSPR of silver nanoparticles have successfully investigated for the determination of some heavy metals like  $\text{Fe}^{3+}$ ,  $\text{Co}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Ba}^{2+}$ ,  $\text{Hg}^{2+}$  and  $\text{Al}^{3+}$  [16-24]. LSPR of silver and gold nanoparticles was also studied for the determination of serotonin, some toxins, n-hexane and several biologically important compounds [25-29].

In this research, we developed a sensitive and selective method for determination of nickel in water by localized surface plasmon resonance of silver nanoparticles. In the summary nickel added to the mixture of citrate capped silver nanoparticles and cefixime and after that, color of citrate capped silver nanoparticles changed from light yellow to red that depend on  $\text{Ni}^{2+}$  concentration. The absorbance spectra of the solution before and after adding  $\text{Ni}^{2+}$  are measured and compared for the quantitation of added amounts of nickel cation at trace levels.

## 2. EXPERIMENTAL

### 2.1. Chemical and reagents

Silver nitrate( $\text{AgNO}_3$ ), nickel nitrate hexahydrate( $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ), trisodium citrate dihydrate( $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ ), sodium borohydride( $\text{NaBH}_4$ ), sodium chloride( $\text{NaCl}$ ), sodium hydroxide( $\text{NaOH}$ ), hydrochloric acid (HCl 37%) and methanol( $\text{CH}_4\text{O}$ ) were purchased from Merck(Germany). Cefixime ( $\text{C}_{16}\text{H}_{15}\text{N}_5\text{O}_7\text{S}_2$ ) purchased from Sigma-Aldrich (USA). Stock solution of  $\text{Ni}^{2+}$  was prepared in water and standard solutions of  $\text{Ni}^{2+}$  with different concentrations were prepared by dilution of the stock solution. Also, cefixime dissolved in methanol to prepare stock solution, and working solutions were prepared in different concentrations by successive dilution of the stock with methanol.

### 2.2. Apparatus and instruments

UV-Vis absorbance spectra were recorded on a UV-Vis spectrophotometer with a 1.0 cm quartz cell from 350 to 650 nm (PG T80<sup>+</sup>, PG instruments, United Kingdom). Transmission electron microscopy (TEM) was employed for morphology and size of silver nanoparticles (Philips CM30, Netherlands). Measurements of pH were made with a commercial pH meter with a combined glass electrode (Metrohm AG, Switzerland). Inductively coupled plasma-optical

emission spectrometer (730-ES, Varian, USA) was used for obtain atomic emission data.

### ***2.3. Preparation of citrate capped silver nanoparticles***

Citrate capped silver nanoparticles with average diameter of 15 nm were synthesized. In the summary, 0.012 g  $\text{NaBH}_4$  was added to 100 mL silver nitrate solution (0.0001 M) under vigorously stirring and after the moments, silver nanoparticles with light yellow color were achieved. Then 3 mL of tri sodium citrate solution (0.1 M) was added dropwise into the above-mentioned AgNPs solution under magnetic stirring. Citrate capped silver nanoparticles (cit-AgNPs) were prepared after 10 minutes and stored at 4°C in dark place for further utilization [24].

### ***2.4. Colorimetric detection of $\text{Ni}^{2+}$***

For colorimetric determination of  $\text{Ni}^{2+}$ , 210  $\mu\text{L}$  of cefixime solution (0.0001 M), 175  $\mu\text{L}$  of NaCl solution (0.1 M) , 3.00 mL of cit-AgNPs solution( suspension) and different known amounts of  $\text{Ni}^{2+}$  were successively added into a small volumetric flask and then the ultimate mixture was reached to a certain volume (3500  $\mu\text{L}$ ), with distilled water. Total volume of the mixture was Color of the solution was changed to red depending on  $\text{Ni}^{2+}$  concentration.

### ***2.5. Real sample analysis***

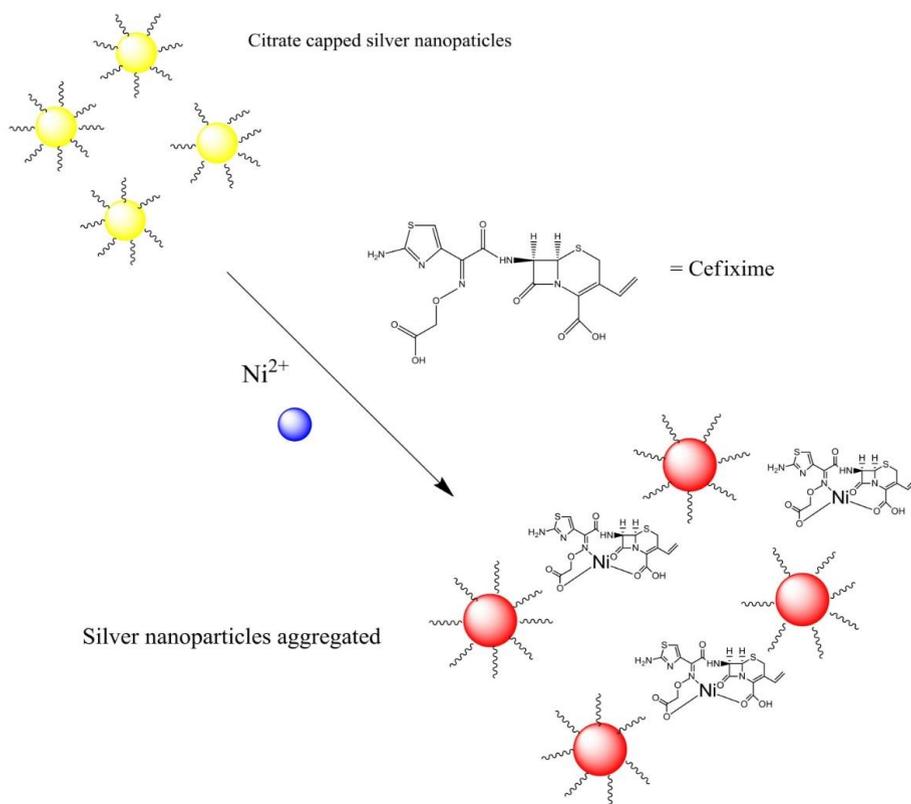
The performance of the cit-AgNPs in the determination of nickel was tested on different water samples. For studying performance of the method for determination of  $\text{Ni}^{2+}$  in real samples, 8  $\mu\text{mol}$  and 12  $\mu\text{mol}$  of nickel ion were spiked to the 1 mL well and tap waters. In summary, 28  $\mu\text{L}$  and/or 42  $\mu\text{L}$  of 0.001 M  $\text{Ni}^{2+}$  was spiked to 1 mL of water, then  $\text{Ni}^{2+}$  spiked water was added to the mixture of the silver nanoparticles, cefixime and NaCl solutions and total volume was reached at 3500  $\mu\text{L}$  with distilled water, after that the solution was transferred to 1cm quartz cell for colorimetric assay using spectrophotometer.

## **3. RESULTS AND DISCUSSION**

### ***3.1. Mechanism of assay***

As shown in scheme 1, when  $\text{Ni}^{2+}$  was added to the solution contains cefixime and cit-AgNPs, stability of silver nanoparticles decreased due to the formation of of  $\text{Ni}^{2+}$ - cefixime complex and color of the solution

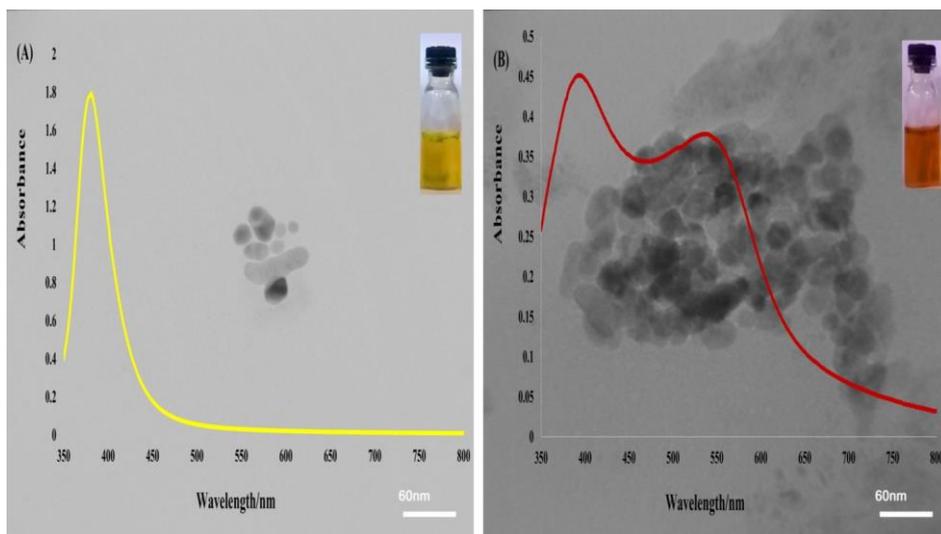
changed from light yellow to red consequently, that depends on  $\text{Ni}^{2+}$  concentration. As a result of aggregation, LSPR peak of silver nanoparticles around 395 nm decreases and a new peak appears around 550 nm. Cefixime is a thiol-containing drug, demonstrating a high affinity to the surface of silver nanoparticles through its S-H group, thus this drug having ability for interaction with citrate capped silver nanoparticles via hydrogen bonding or electrostatic interaction with amino and carboxyl groups of citrate.



**Scheme. 1.** The strategy of the assay

In the appropriate pH, citrate capped silver nanoparticles and cefixime were negatively and positively charged, respectively. Therefore, electrostatic attraction between silver nanoparticles and cefixime molecules and also hydrogen bonding between OH groups of citrate ions and  $\text{NH}_2$  of cefixime causes the aggregation of silver nanoparticles.

When nickel ions were added to citrate capped silver nanoparticles in absence of cefixime, did not affect the color or spectrum of the nanoparticles but in presence of cefixime as chelating agent, after addition of nickel ions to the above-mentioned mixture, color of the solution changed to red and a new peak appears in around 550 nm. As far as we know,  $\text{NH}_2$  and  $\text{COOH}$  groups of cefixime can interact with silver nanoparticles, therefore both of the groups can modified surface of the cit-AgNPs and also It has been reported that the both of the groups having high affinity to the metal ions [30,31], therefore the mixture of cit-AgNPs and cefixime aggregated in presence  $\text{Ni}^{2+}$  due to ion chelation. Fig. 1A shows absorption spectrum of silver nanoparticles that demonstrating silver nanoparticles have a peak around 395 nm (yellow). Fig. 1B also shows when aggregation of silver nanoparticles was happened, the peak around 395 nm decreased and new peak appears around 550 nm (red). Also Figure 1 shows typical TEM images, before and upon the addition of nickel ions to the mixture of citrate capped silver nanoparticles and cefixime. Therefore, aggregation of silver nanoparticles by adding  $\text{Ni}^{2+}$  was supported by TEM images (Fig. 1: A, B).



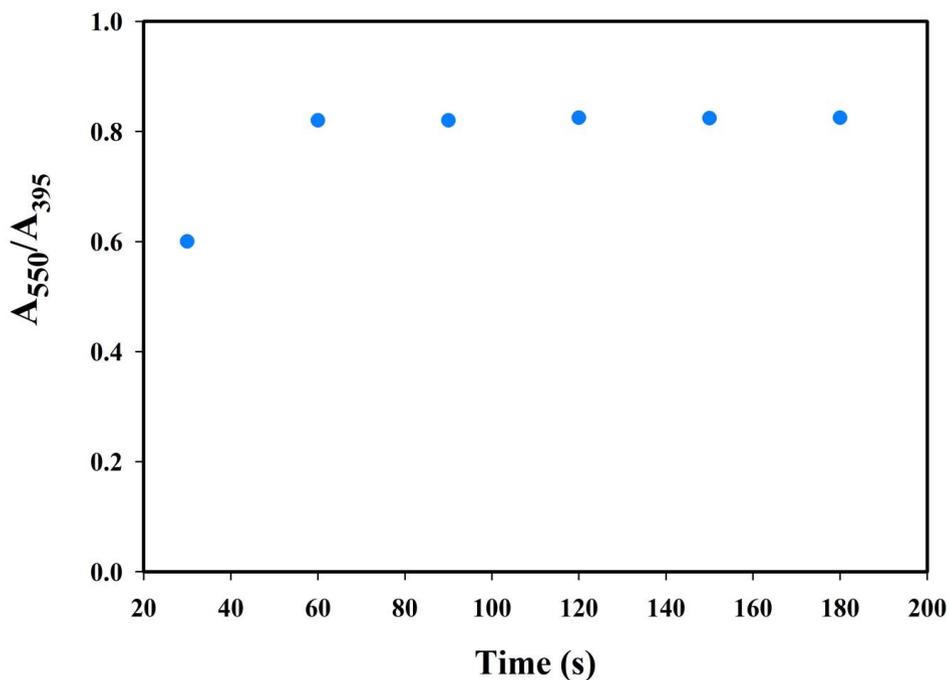
**Fig. 1.** Absorption spectra, TEM images and color of silver nanoparticles in the absence (A) and presence (B) of  $14.2 \mu\text{mol Ni}^{2+}$ .

### 3.2. Optimization of assay condition

Early experiments have been done to determine optimized conditions in the assay. We have explored the effect of NaCl and cefixime concentrations, pH and reaction time in the assay on the aggregation of citrate capped silver nanoparticles.

#### 3.2.1. Effect of reaction time

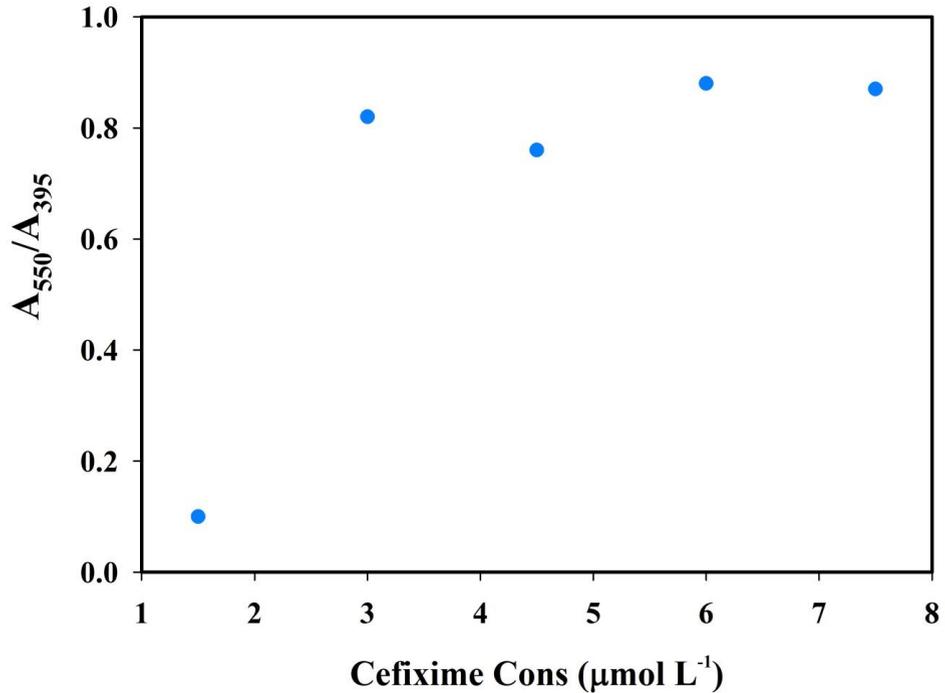
Reaction time or in other words time interval between addition of cefixime reagent to solution and absorbance measurement is a critical parameter in the assay that must be optimized to enhance the sensitivity of the method. Fig. 2 shows the changes of the absorbance ratio of Cit-AgNPs colloid after different times over the range of 30-180 seconds. The results showed that signal of plasmon intensity reaches a maximum in 60 seconds then remains constant. Thus, a 60 sec time interval was selected for subsequent studies.



**Fig. 2.** Effect of time on aggregation of silver nanoparticles in the presence  $14.2 \mu\text{mol L}^{-1} \text{Ni}^{2+}$ ,  $3 \mu\text{mol L}^{-1}$  Cefixime,  $3 \text{mmol L}^{-1}$  NaCl and  $\text{pH} = 8$ .

### 3.2.2. The effect of cefixime concentration

Effect of cefixime concentration on the plasmon absorbance intensity of the citrate –capped silver nanoparticles was studied. It was observed that the concentration of added cefixime within the range of  $1.5 \mu\text{mol L}^{-1}$  to  $7.5 \mu\text{mol L}^{-1}$  had significant effect on the plasmon absorbance and aggregation of silver nanoparticles. A concentration of  $6 \mu\text{mol L}^{-1}$  of cefixime was chosen as optimum (Fig. 3).

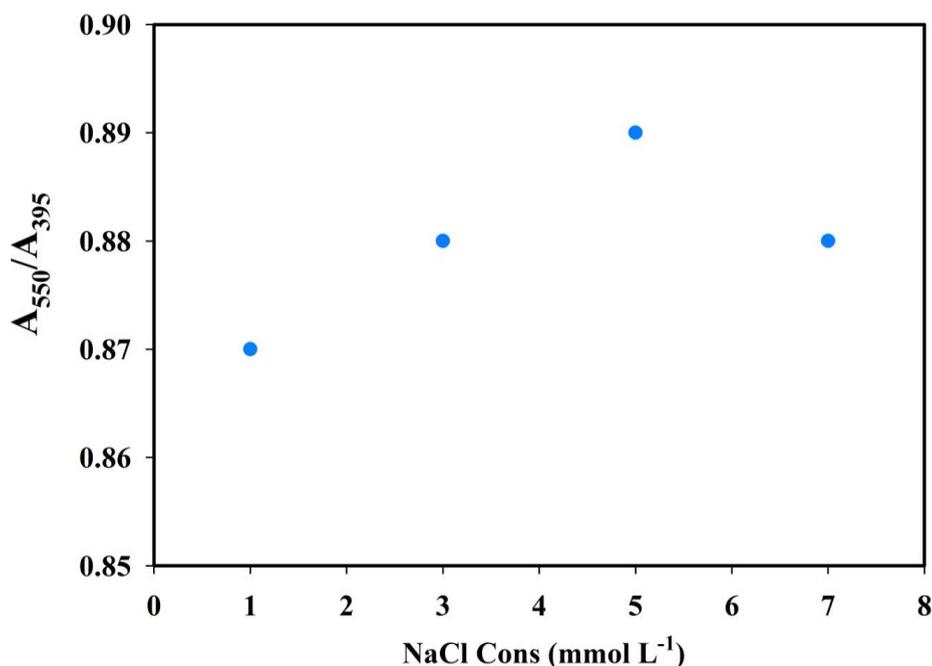


**Fig. 3.** Effect of cefixime concentration on aggregation of silver nanoparticles in the presence  $14.2 \mu\text{mol L}^{-1} \text{Ni}^{2+}$ ,  $3 \text{mmol L}^{-1} \text{NaCl}$  and  $\text{pH} = 8$ .

### 3.2.3. Effect of ionic strength

Effect of sodium chloride on aggregation of silver nanoparticles was explored because ionic strength of reaction media has a critical role on aggregation of AgNPs. Fig. 4 shows the changes of the absorbance ratio

of the colloidal silver nanoparticles by various concentrations of NaCl over the range of 1-7 mmol L<sup>-1</sup>. It has been found with increasing NaCl concentration, the absorbance ratio ( $A_{550}/A_{395}$ ) was increased, because sodium chloride can constrict the electrical double layer of capping citrate ions [32]. According to the results, 5 mmol L<sup>-1</sup> was selected as optimum NaCl concentration for subsequent investigations.

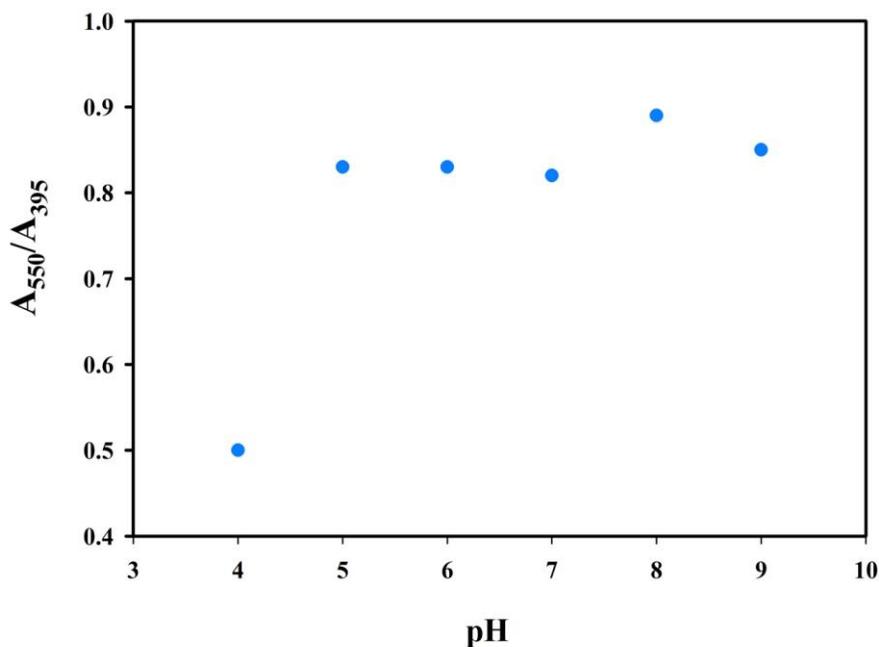


**Fig. 4.** Effect of NaCl concentration on aggregation of silver nanoparticles in the presence 14.2  $\mu\text{mol L}^{-1}$  Ni<sup>2+</sup>, 6  $\mu\text{mol L}^{-1}$  Cefixime and pH = 8.

#### 3.2.4. Effect of pH

pH has a crucial effect on aggregation of silver nanoparticles, thus effect of pH was studied in the range of 4.0-9.0 and as shown in Fig. 5, pH=8.0 was selected as optimized. In pH<4.0 due to protonation of citrate molecules, surface charge of the silver nanoparticles was neutralized. In 5.0< pH< 8.0, cefixime and citrate molecules were protonated and for this reason, aggregation of the silver nanoparticles was increased. In

pH > 8.0, citrate becomes negative and cefixime is neutralized and electrostatic attraction between silver nanoparticles decreases and aggregation of silver nanoparticles is lowered [33].

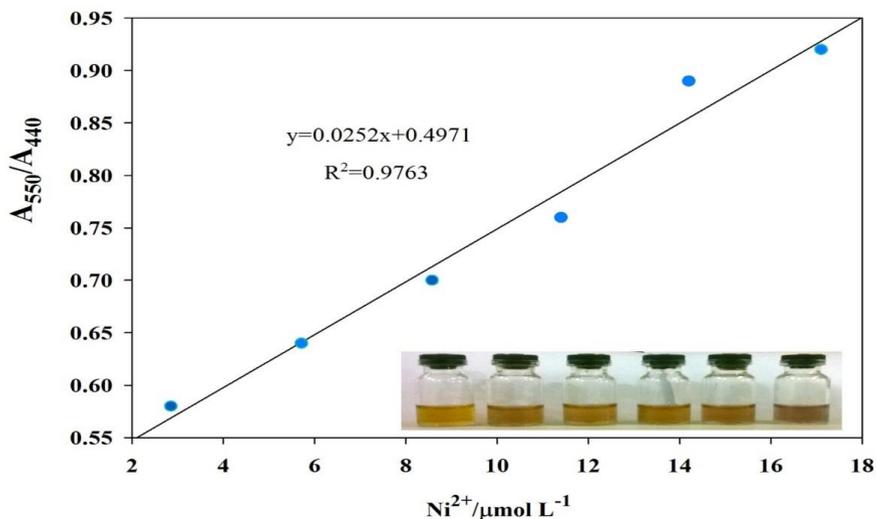


**Fig. 5.** Effect of pH on aggregation of silver nanoparticles in the presence  $14.2 \mu\text{mol L}^{-1}$   $\text{Ni}^{2+}$ ,  $6 \mu\text{mol L}^{-1}$  Cefixime and  $5 \text{mmol L}^{-1}$  NaCl.

### 3.3. Analytical Figures of Merit

The linear range, detection limit, reproducibility, sensitivity and selectivity of the method were evaluated under the optimum. Absorbance spectra of Ag-NPs in the presence of different concentrations of  $\text{Ni}^{2+}$  were measured. Fig. 6 shows a linear correlation of the absorbance ratio ( $A_{550}/A_{395}$ ) and color image of the glass bottles containing Ag-NPs formed in the presence of different concentrations of  $\text{Ni}^{2+}$ . As the concentration of the  $\text{Ni}^{2+}$  increases, the absorbance ratio ( $A_{550}/A_{395}$ ), corresponding to the change in plasmon of the Ag-NPs is increased. Under the specified experimental conditions, the calibration curve for  $\text{Ni}^{2+}$  was linear from  $2.70$ - $17.10 \mu\text{mol L}^{-1}$  with a correlation coefficient

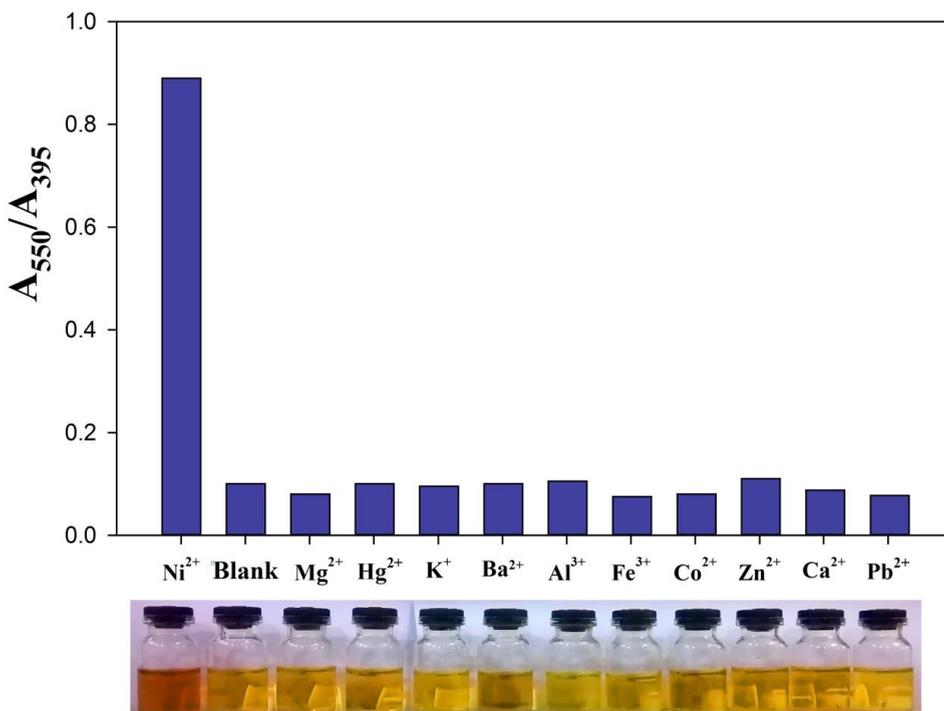
(R) of 0.9881 and equation of  $(A_{550}/A_{395}) = 0.0252[Ni^{2+}] + 0.4971$  (Figure 6). Also, the detection limit ( $3S_b/m$ ) [34], obtained for the determination of  $Ni^{2+}$ , was  $0.81 \mu\text{mol L}^{-1}$ .



**Fig. 6.** Silver nanoparticles linear calibration curve under optimized conditions in the presence of different concentrations of  $Ni^{2+}$  ( $2.85 \mu\text{mol L}^{-1}$ ,  $5.7 \mu\text{mol L}^{-1}$ ,  $8.5 \mu\text{mol L}^{-1}$ ,  $11.4 \mu\text{mol L}^{-1}$ ,  $14.2 \mu\text{mol L}^{-1}$ ,  $17.1 \mu\text{mol L}^{-1}$ )

### 3.4. Interferences

Selectivity of the method with other ions was investigated with ten other ions. Ten ions including  $Mg^{2+}$ ,  $Hg^{2+}$ ,  $K^+$ ,  $Ba^{2+}$ ,  $Al^{3+}$ ,  $Fe^{3+}$ ,  $Co^{2+}$ ,  $Zn^{2+}$ ,  $Ca^{2+}$ ,  $Pb^{2+}$  were added with high concentration to the solution and did not change in color or spectrum (Fig. 7). As shown in Fig. 7 the proposed method is free from interferences of many ions. It is evident from the data that with adding  $Ni^{2+}$  to the mixture of citrate capped silver nanoparticles and cefixime, absorbance ratio ( $A_{550}/A_{395}$ ) was significantly increased.



**Fig. 7.** The absorption ratio value ( $A_{550}/A_{395}$ ) and photographic images in the presence interferences including  $Mg^{2+}$ ,  $Hg^{2+}$ ,  $K^+$ ,  $Ba^{2+}$ ,  $Al^{3+}$ ,  $Fe^{3+}$ ,  $Co^{2+}$ ,  $Zn^{2+}$ ,  $Ca^{2+}$ ,  $Pb^{2+}$ . Concentration of  $Ni^{2+}$   $14.2 \mu\text{mol L}^{-1}$  and other ions considered 50 times higher than  $Ni^{2+}$ .

### 3.5. Analysis of spiked samples

In order to evaluate of colorimetric assay for the determination of nickel in real water samples, the proposed method was used for determination  $Ni^{2+}$  in real samples like well and tap water. The results show that the colorimetric method has good stability in different real water samples (summarized in Table 1). The results indicate good recoveries in the range of 97 to 97.5% in well water and 96 to 104% in tap water. Also the relative standards deviations (RSD) of the method were found to be <4% in tap and well water. The results were in good agreement with those obtained by ICP-OES, confirming the practical analytical utility of proposed method. Repeatability and reproducibility of the proposed were evaluated as precision indicators. Three similar experiments were performed for the analysis of samples on the same day and on five

consecutive days. The obtained RSDs for the intra-day and inter-day spiked samples were to be <3% and <4%, respectively.

Table 1  
Determination Ni<sup>2+</sup> in well and tap water (n=3)

Samples	Spiked ( $\mu\text{mol L}^{-1}$ )	Found ( $\mu\text{mol L}^{-1}$ )	Recovery (%)	RSD (%)	ICP-OES ( $\mu\text{mol L}^{-1}$ )
Well water	0	ND	-	-	ND
	8	7.79	97	2.5	8.1
	12	11.7	97.5	3.1	11.8
Tap Water	0	ND	-	-	ND
	8	7.74	96	3	7.8
	12	12.5	104	3	12.2

### 3.6. Comparison to other reported methods for determination Ni<sup>2+</sup>

The performance of the developed method was compared with other methods for determination Ni<sup>2+</sup> in real water samples. As shown in Table 2, the proposed method provided good detection limit and linear range compared with similar reported methods for determination Ni<sup>2+</sup> as presented in Table 2.

Table 2  
Comparison of figures of merit of the present method with other reported colorimetric methods for determination of Ni<sup>2+</sup>

Method	LOD <sup>a</sup> ( $\mu\text{g L}^{-1}$ )	LDR <sup>b</sup> ( $\mu\text{g L}^{-1}$ )	Matrix	Reference
Colorimetry (LSPR <sup>c</sup> )	17	NR	River Water	35
Colorimetry	5	NR	Water	36
Colorimetry	0.3	NR	Water	37
Colorimetry	290	324-1252	Water	38
Colorimetry	34.8	232-3480	Water	39
Colorimetry	214	NR	Water	40
Colorimetry (LSPR <sup>c</sup> )	51	165-991	Water	This work

NR= Not reported

<sup>a</sup> Limit of detection. <sup>b</sup> Linear dynamic range. <sup>c</sup> Local surface plasmon resonance. <sup>d</sup> Fluorescence quenching method.

#### 4. CONCLUSION

In this study citrate capped silver nanoparticles were employed for determination  $\text{Ni}^{2+}$  in real water samples including well and tap water. The presence of  $\text{Ni}^{2+}$  induced the aggregation of silver nanoparticles through metal-ligand interaction. Eventually, the proposed method applied successfully in tap and well water. The proposed method also demonstrated an excellent interference-free ability against heavy metal ions. The results show that the proposed method is suitable for determination trace amounts of  $\text{Ni}^{2+}$  in real water samples.

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