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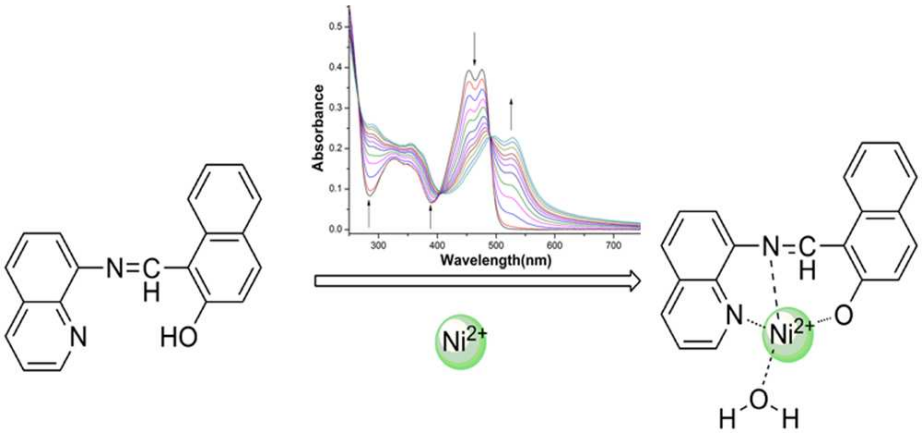
Graphical Abstracts

A highly selective colorimetric chemosensor **LX** was described, which could instantly detect Ni^{2+} without interference by other cations.



A Highly Selective Colorimetric Chemosensor for Detection of Nickel Ions in Aqueous Solution

Xin Liu, Qi Lin*, Tai-Bao Wei, You-Ming Zhang*

Key Laboratory of Eco-Environment-Related Polymer Materials, Ministry of Education of China; Key Laboratory of Polymer Materials of Gansu Province; College of Chemistry and Chemical Engineering, Northwest Normal University, Lanzhou, Gansu, 730070, P. R. China

* Corresponding author. E-mail: linqi2004@126.com (Dr. Q. Lin); zhangnwnu@126.com (Prof. Y. M. Zhang)

Tel:+86-931-7973120;

Abstract

A highly selective chemosensor **LX** based on quinoline was described, which could instantly detect Ni^{2+} in aqueous solution with specific selectivity and high sensitivity. The addition of Ni^{2+} to sensor **LX** induced a remarkable color change from yellow to red, these sense procedure could not be interfered by other coexistent competitive cations such as Fe^{3+}, Co^{2+}, and Cu^{2+}. Thus **LX** could be used as a potential Ni^{2+} colorimetric and naked-eye chemosensor. Moreover, test strips based on sensor **LX** were fabricated, which could act as a convenient and efficient Ni^{2+} test for "in-the-field" measurement of Ni^{2+}.

Keywords: Chemosensor; Nickel ions; Naked-eye detection; Ratiometric; Test strips

1. Introduction

Nickel is an essential trace element in biological systems such as respiration, biosynthesis, and metabolism [1-3]. Moreover, metallic nickel and its compounds are widely used in modern industry. Nickel compounds are used in electroplating and electroforming and for the production of nickel-cadmium batteries and electronic equipment. Nickel alloys, such as stainless steel, are used in the production of tools, machinery, armaments, and appliances [4-6]. However, the high usage of nickel in such industries inevitably leads to environmental pollution and directly impacts on people's physical health. The accumulation of nickel in the body can lead to lung fibrosis, and cardiovascular and kidney diseases [7-11]. Hence, the rational design and synthesis of efficient sensors to selectively detect Ni^{2+} ions at environmental and biological levels are necessary. Up to date, most of Ni^{2+}-selective sensors are based on potentiometric methods [12-13]. These sensors also display responses to other transition metal ions, such as Co^{2+}, Fe^{3+}, Cu^{2+} and so on [14-18]. According to literatures, only very few reports on detecting Ni^{2+} without interference had been published [19-20].

In view of this, and as a part of our research interest in molecular recognition [21-25], we have attempted to obtain an efficient colorimetric and/or fluorescent sensor that could sense Ni^{2+} with both high selectivity and sensitivity in aqueous. Our strategy for the design of such a sensor has been as follows. Firstly, colorimetric sensors are promising due to their simplicity, real-time and on-line analysis, especially a significantly lower capital cost than fluorescent sensors. Moreover, paramagnetic Ni^{2+} shows the fluorescence quenching nature. So we developed a colorimetric chemosensor that shows a significant color change when binding with Ni^{2+}. Secondly, it is well known Schiff base derivatives have been widely used in ion detection for a long time by virtue of their simple structures and good recognition performance, but the detection of Ni^{2+} ions based on Schiff base derivatives has rarely been reported. On the other hand, quinoline possess desirable photo-physical properties, they are ideal platforms for development of chemosensors for heavy and transition metal ions.

Therefore, we introduced C=N and quinoline groups into the sensor molecule. Finally, the sensor molecule was designed easy to synthesis. As a result, sensor **LX** could detect Ni^{2+} with specific selectivity and high sensitivity in DMSO-H_2O (v/v=1:1) HEPES buffer solutions at pH=7.4. Moreover, the sense procedure could not be interfered by other coexistent competitive cations (such as Cu^{2+}, Fe^{3+} and Co^{2+}) and anions.

2. Experimental

2.1. General information and materials

All reagents for synthesis were analytical grade, commercially and were used without further purification. All the cations were added in the form of perchlorate salts and anions were added in the form of tetrabutylammonium (TBA) or sodium salts, which were purchased from Sigma-Aldrich Chemical, stored in a vacuum desiccator. Melting points were measured on an X-4 digital melting point apparatus and were uncorrected. UV/Vis spectra were recorded on a Shimadzu UV-2550 spectrometer at room temperature. ^{1}H NMR spectra were recorded on a Varian Mercury Plus-400 MHz spectrometer with DMSO-d_6 as solvent and TMS as an internal reference. The infrared spectra were performed on a Digilab FTS-3000 FT-IR spectrophotometer. Elemental analyses were performed by Thermo Scientific Flash 2000 organic elemental analyzer.

2.2. General procedure for UV–vis experiments

Stock solutions of 4.0×10^{-3} mol·L^{-1} perchlorate salts of the respective cations (Fe^{3+}, Ag^{+}, Ca^{2+}, Cu^{2+}, Co^{2+}, Ni^{2+}, Cd^{2+}, Pb^{2+}, Zn^{2+}, Cr^{3+}, and Mg^{2+}) and 1.0×10^{-2} mol·L^{-1} tetrabutylammonium or sodium salts of the respective anions (F^-, Cl^-, Br^-, I^-, AcO^-, $H_2PO_4^-$, HSO_4^- ,ClO_4^- and CN^-) were prepared in water. A stock solution of sensor **LX** (2.0×10^{-4} mol·L^{-1}) was prepared in DMSO. The solution of sensor **LX** was then diluted to 2.0×10^{-5} mol·L^{-1} with DMSO-H_2O (v/v=1:1, pH=7.4) HEPES buffer solutions. In titration experiments, 2 mL solution of sensor **LX** (2.0×10^{-5} mol·L^{-1}) was filled in a quartz optical cell of 1 cm optical path length, and the ions stock solution were added into the quartz optical cell gradually by using a micropippet. Spectral data were recorded at 10 min after addition of the ions at room temperature.

2.3. General procedure for ^{1}H NMR experiments

For ^{1}H NMR titration, sensor **LX** was prepared in DMSO-d_6, $Ni(ClO_4)_2$ was prepared in D_2O. First of all, only sensor **LX** in DMSO-d_6 were added into NMR tube, and then added Ni^{2+} ions at 0.5, 1.0 and 1.5 equiv sequentially. All solutions were mixed directly in NMR tube.

2.4. Synthesis of the sensor **LX**

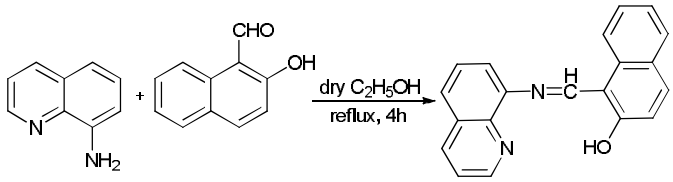
The structure and synthesis of sensor **LX** is shown in Scheme 1. 8-aminoquinoline (0.29 g, 2 mmol), 2-hydroxy-1-naphthaldehyde (0.35 g, 2 mmol) in dry ethanol (30 mL) was stirred under reflux condition for 4 h, and get yellow product (0.51 g, 85% yield) after recrystallization from C_2H_5OH-DMF.

m.p. 227-229℃. IR (KBr, cm^{-1}): ν= 3447(OH), 1624(C=N), 1589(C=N), 1535(C=C). ^{1}H NMR (400 MHz, DMSO) δ 15.91 (d, J = 10.8 Hz, 1H, OH), 9.66-9.51 (m, 1H, =CH), 9.05 (dd, J = 4.1, 1.7 Hz, 1H), 8.47 (dd, J = 14.9, 4.8 Hz, 3H), 7.91-7.16 (m, 7H, Ar-H), 6.72 (dd, J = 9.5, 5.8 Hz, 1H). ^{13}C NMR (151 MHz, DMSO) δ 181.42 (s), 150.17 (s), 147.25 (s), 139.38 (s), 138.74 (s), 136.45 (s), 136.26 (s), 134.24 (s), 129.12 (s), 128.39 (d, J = 6.4 Hz), 126.96 (s), 126.29 (s), 125.92 (s), 124.44 (s), 123.46 (s), 122.59 (s), 119.93 (s), 114.63 (s), 108.24 (s). Anal. calcd for $C_{20}H_{14}N_2O$: C 80.52, H 4.73, N 9.39; found: C 80.60, H 4.58, N 9.37. ESI-MS: calcd for $[C_{20}H_{14}N_2O+H]^+$ 299.1, found 299.3.

2.5. Synthesis of the **LX**-Ni

The DMF solution of **LX** (0.030 g, 0.1mmol) and the water solution of $Ni(ClO_4)_2{\cdot}6H_2O$ (0.055 g, 0.15mmol) were mixed and stirred at room temperature for 2 h. The red solid formed was filtered, washed with water and dried under vacuum.

m.p. 262-264℃. IR (KBr, cm^{-1}): ν= 3441(OH), 1614(C=N), 1534(C=C), 1492(C=C). ESI-MS: calcd for $[\mathbf{LX}+Ni^{2+}+H_2O]^+$ 373.1, found 373.3.



Scheme 1. Structure and synthesis of the sensor **LX.**

3. Results and discussion

The sensing abilities of **LX** toward various cations (Fe^{3+}, Ag^{+}, Ca^{2+}, Cu^{2+}, Co^{2+}, Ni^{2+}, Cd^{2+}, Pb^{2+}, Zn^{2+}, Cr^{3+}, and Mg^{2+}) were investigated by UV-vis spectroscopy. When 10 equivalents of these cations ($c=2\times10^{-4}$ M) was added to the DMSO-H_2O (v/v=1:1, pH=7.4) HEPES buffer solutions of sensor **LX** ($c=2\times10^{-5}$ M) respectively at room temperature, a dramatic color change from yellow to red was observed by the naked-eye only upon the addition of Ni^{2+} to sensor **LX** (Figure 1). In the corresponding UV-vis spectra (Figure 2), the formation of a new absorption band at about 525 nm is in good agreement with this color change. All examined cations such as Fe^{3+}, Co^{2+} and Cu^{2+} didn't cause any obvious color and spectra changes. The plot of changes in the absorbance at 525 nm upon addition of various cations clearly showed excellent selectivity of sensor **LX** towards the Ni^{2+} (Figure 3). Moreover, we also explored the optical response of sensor **LX** by monitoring the changes in absorption spectra upon addition of various anions (F^-, Cl^-, Br^-, I^-, AcO^-, $H_2PO_4^-$, HSO_4^-, ClO_4^- and CN^-) under the same conditions, no obvious color and spectra changes were observed (Supporting Information, Figure S1).

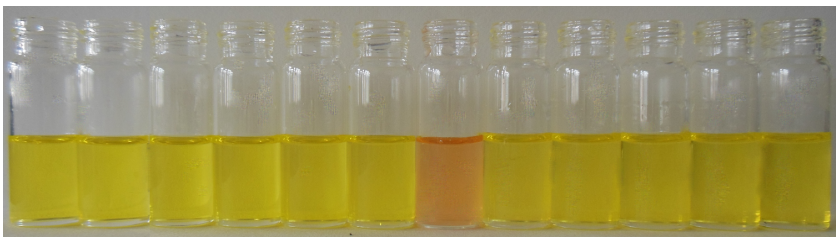


Figure 1. Color changes of **LX** ($c=2\times10^{-5}$ M) after addition of 10 equivalents various cations in DMSO-H_2O (v/v=1:1) HEPES buffer solutions at pH=7.4. From left to right: only **LX**, Fe^{3+}, Ag^{+}, Ca^{2+}, Cu^{2+}, Co^{2+}, Ni^{2+}, Cd^{2+}, Pb^{2+}, Zn^{2+}, Cr^{3+}, Mg^{2+}.

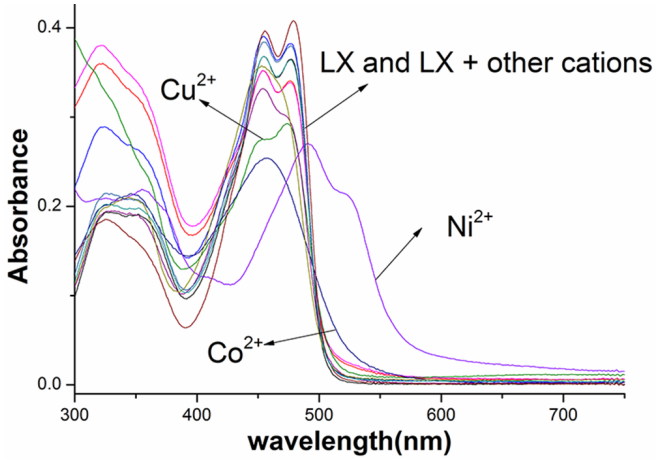


Figure 2. Changes in the UV/Vis spectra of **LX** ($c=2\times10^{-5}$ M) after addition of 10 equivalents various cations in DMSO-H_2O (v/v=1:1) HEPES buffer solutions at pH=7.4.

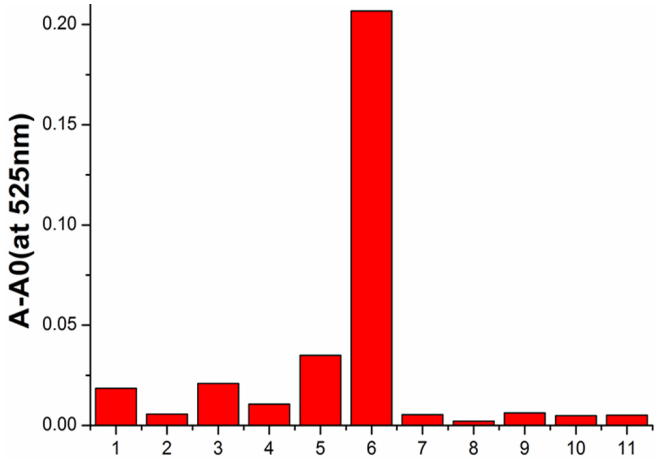


Figure 3. Normalized changes in the absorbance at 525 nm of **LX** ($c=2\times10^{-5}$ M) after addition of 10 equivalents various cations, from 1 to 11: Fe^{3+}, Ag^{+}, Ca^{2+}, Cu^{2+}, Co^{2+}, Ni^{2+}, Cd^{2+}, Pb^{2+}, Zn^{2+}, Cr^{3+}, Mg^{2+}.

Figure 4 shows the family of absorption spectra obtained over the course of the titration of sensor **LX** with Ni^{2+} in DMSO-H_2O (v/v=1:1) HEPES buffer solutions at pH=7.4. With the gradual addition of pure water solution of Ni^{2+} to sensor **LX**, the intensity of absorption bands at 525 nm, 389 nm and 284 nm increased, while an absorption bands at 464 nm began to decrease until it reached a limiting value. Moreover the presence of three isosbestic points at 490, 265 and 405 nm indicated that sensor **LX** reacts with Ni^{2+} to form a stable complex. Simultaneously, the ratio of A525/A464 rises along with the increase in Ni^{2+} concentrations, which allows the Ni^{2+} concentration to be determined ratiometrically (Supporting Information, Figure S2). From titration plots in UV-visible spectroscopy, the 1:1 stoichiometry between Ni^{2+}

and sensor **LX** has been proved.

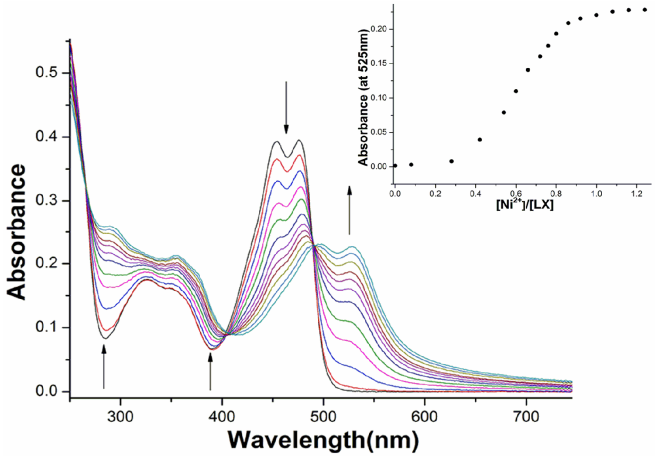


Figure 4. UV-vis spectra of **LX** (c=20 uM) upon the addition of Ni^{2+}. [Ni^{2+}]= 0, 5.6, 8.4, 10.8, 12.0, 13.2, 14.4, 15.2, 16.4, 18.4, 20.0, 21.6 uM. Inset: plot of absorbance at 525 nm vs. number of equivalents of Ni^{2+}.

To know stoichiometry between the guest (Ni^{2+}) and host **LX** molecule in DMSO-H_2O (v/v=1:1) HEPES buffer solutions at pH=7.4. Job's plot has been drawn (Figure 5). When molar fraction of Ni^{2+} was 0.5, the absorbance at 525 nm got to extreme value, indicating that forming a 1:1 complex between **LX** and Ni^{2+}. The association constant (K_a) of **LX** with Ni^{2+} was determined using the Benesi-Hildebrand equation [26-28]. The measured absorbance [1/(A-A_0)] varied as a function of 1/[Ni^{2+}] in a linear relationship (R=0.9887), indicating formation of 1:1 sotchiometry between Ni^{2+} and **LX**. This conclusion is consistent with corresponding Job's plot. The association constant of **LX** with Ni^{2+} in DMSO-H_2O (v/v=1:1) HEPES buffer solutions at pH=7.4 was calculated to be 1.33×10^5 M^{-1} (Supporting Information, Figure S3).

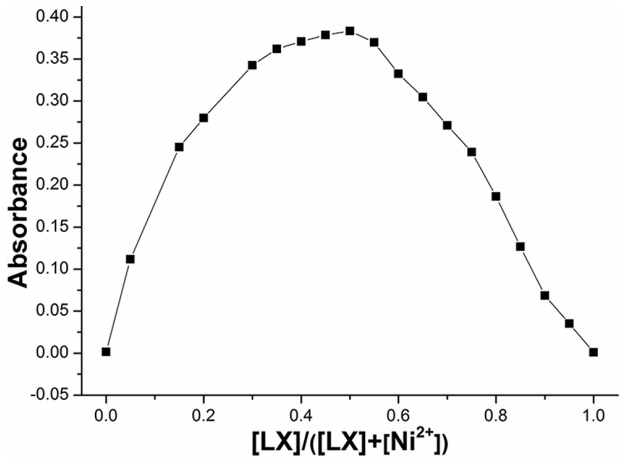


Figure 5. Jobs plot for complexion of sensor **LX** with Ni^{2+} in DMSO-H_2O (v/v=1:1) HEPES buffer solutions at pH=7.4.

As known, chemosensors always have a problem of long response time. In our case, the binding process of Ni^{2+} to **LX** was found to be very fast (Figure 6). After adding Ni^{2+}, the absorbance of **LX** was increased at 525 nm and reached the plateau region less than 12 s, and remains quite stable, suggesting that the binding process might be completed instantly and the chemosensor has rapid detection ability for nickel cation.

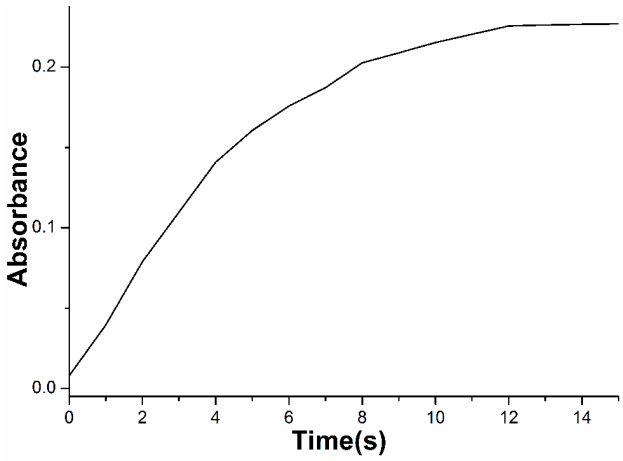


Figure 6. The time-dependent absorbance at 525 nm for **LX** (2.0×10^{-5} M) in DMSO-H_2O (v/v=1:1) HEPES buffer solutions at pH=7.4 after addition of 10 equivalents Ni^{2+}.

An important feature of a sensor is its selectivity toward analyze relative to other competitive species. Therefore, competition experiments were carried out by adding Ni^{2+} ions ($c=2\times10^{-4}$ M) to solution of **LX** ($c=2\times10^{-5}$ M) in the presence of miscellaneous ions including anions ($c=1\times10^{-3}$ M) and cations ($c=2\times10^{-4}$ M), respectively. These miscellaneous competitive ions did not induce significant absorption changes of **LX** in the absence of Ni^{2+}. However, upon addition of Ni^{2+} to the solution, the unique spectral and color changes were still displayed under the above conditions (Figure 7&8). These results revealed that **LX** had a remarkable selectivity toward Ni^{2+} over other competitive ions, and more, the detection of Ni^{2+} by **LX** was hardly affected by these common coexistent cations and anions.

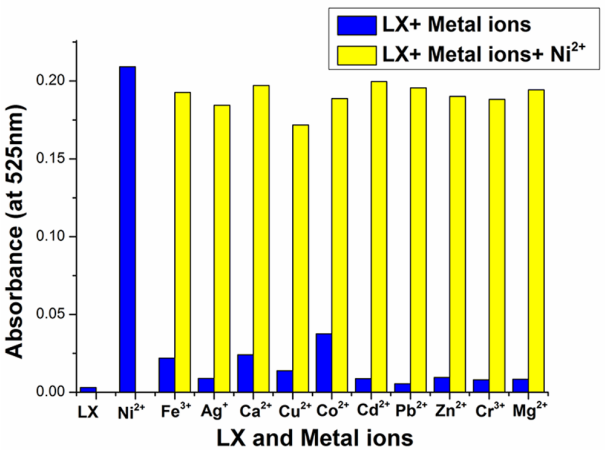


Figure 7. Absorbance responses of **LX** (c=2×10^{-5} M) in the presence of 10 equivalents Ni^{2+} with 10 equivalents various metal ions in DMSO-H_2O (v/v=1:1) HEPES buffer solutions at pH=7.4. Bars represent absorbance at 525 nm. The blue bars represent the addition of the competing metal ions to solution of **LX**. The yellow bars represent the addition of competing metal ions and Ni^{2+} to the solution of **LX**.

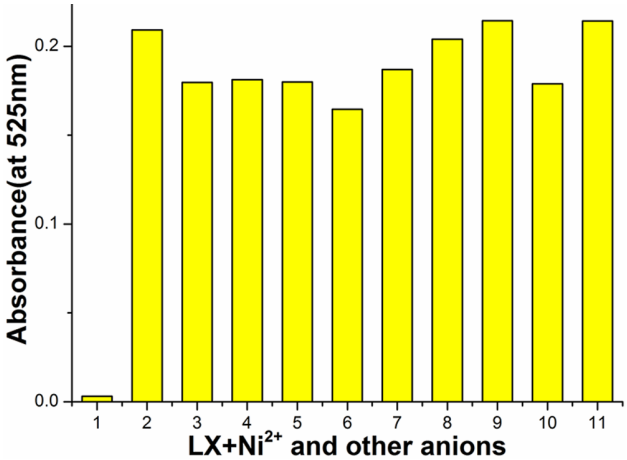


Figure 8. Absorbance at 525 nm of **LX** (c=2×10^{-5} M) in the presence 10 equivalents Ni^{2+} with 50 equivalents various anions in DMSO-H_2O (v/v=1:1) HEPES buffer solutions at pH=7.4. From 1 to 11: **LX**, **LX+Ni^{2+}**, **LX+Ni^{2+}**+F^-, **LX+Ni^{2+}**+Cl^-, **LX+Ni^{2+}**+Br^-, **LX+Ni^{2+}**+I^-, **LX+Ni^{2+}**+AcO^-, **LX+Ni^{2+}**+$H_2PO_4^-$, **LX+Ni^{2+}**+HSO_4^-, **LX+Ni^{2+}**+ClO_4^-, **LX+Ni^{2+}**+CN^-.

The colorimetric detection limits of sensor **LX** for Ni^{2+} were also tested. As is shown in Figure 9, the minimum concentration of Ni^{2+} for color change observed by the naked-eye was 5.0×10^{-6} M and the detection limit of the absorption spectra changes calculated on the basis of $3s_B/S$ [29] was 2.2 ×10^{-7} M for Ni^{2+} cation (Supporting Information, Figure S4).

Figure 9. Naked-eye detection limit.

Motivated by the favorable features of sensor **LX** in solution, we prepared test strips by immersing filter papers (3×1 cm^2) into the DMSO solution of sensor **LX** (1×10^{-3} M) and then dried them in air to determine the suitability of a "dip-stick" method for the detection of Ni^{2+}, similar to that commonly used for the pH measurement. When the test strips coated with **LX** (1×10^{-3} M) were immersed into the pure water solutions of Ni^{2+} with different concentrations, the obvious color change from yellow to red was observed (Figure 10). The development of such a "dip-sticks" approach was extremely attractive for "in-the-field" measurements that did not require any additional equipment. Therefore, the test strips of **LX** have excellent application value in detection Ni^{2+}.

Figure 10. Color change of the test strips of **LX** (1×10^{-3} M) to various concentrations of Ni^{2+} in water, from left to right: 0, 1×10^{-3} M, 1×10^{-4} M, and 1×10^{-5} M.

To explore the sensing mechanism of sensor **LX** to Ni^{2+}, the IR, ^{1}H NMR titration and ESI-MS were investigated, which illustrated the characteristic structural changes occurring upon interaction with Ni^{2+}. In the IR spectra of **LX**, the stretching

vibration absorption peaks of HC=N, quinoline C=N appeared at 1624 cm^{-1}, 1591 cm^{-1} and in plane bending vibration absorption peak of O-H at 1301 cm^{-1}, respectively. However, when **LX** coordinated with Ni^{2+}, the stretching vibration absorption peaks of HC=N and in plane bending vibration absorption peak of O-H disappeared, meanwhile the stretching vibration absorption peak of quinoline C=N shifted to higher wavenumbers at 1614 cm^{-1} (Figure 11). These changes indicated that nickel ions had coordination with imine nitrogen atoms, oxygen atoms of phenolic hydroxyl groups and nitrogen atoms of quinoline of **LX**, respectively (Scheme 2).

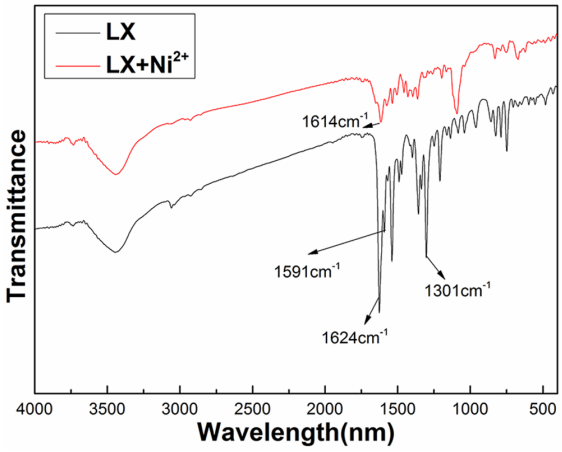
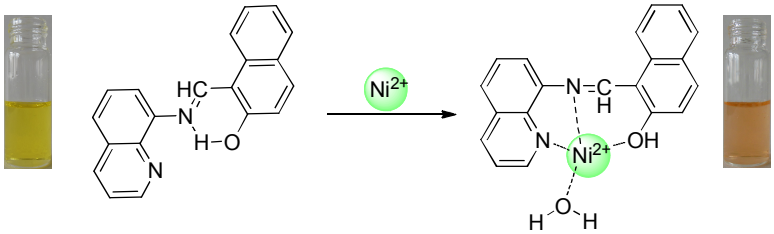


Figure 11. IR spectra of compound **LX** and **LX**-Ni^{2+} in KBr disks.



Scheme 2. A possible sense mechanism of the sensor **LX** to Ni^{2+}.

The results of ^{1}H NMR experiments also support this proposed mechanism (Supporting Information, Figure S5). There is one intramolecular hydrogen bond in the molecular structure of **LX**: OH···N=C. The formation of this strong hydrogen bond leads to the ^{1}H NMR chemical shift the OH group appearing at very low-field, 15.9 ppm. With gradual addition of Ni^{2+}, the OH peak at 15.9 ppm shifted to 11.4 ppm and became broad, which can be attributed to the breaking of intramolecular hydrogen bond by Ni^{2+}···O-H interactions. Meanwhile, the signal of the hydrogen atoms in

aromatic rings and CH=N showed a significant downfield shift, indicating a charge transfer from aromatic groups to the Ni^{2+} ions. These results also suggested that Ni^{2+}-**LX** complex was formed via the coordination of Ni^{2+} with OH, C=N and nitrogen atoms of quinoline on **LX**.

Further evidence was obtained by ESI-MS experiments also support this proposed mechanism. In the ESI-MS spectra of sensor **LX** (Figure S6), the $[\mathbf{LX}+H]^+$ peak appeared at 299.3 (m/z_{calcd}=299.1). However, when 1 equivalent Ni^{2+} was added to the solution of **LX**, a new peak appeared at 373.3, coinciding well with that for the species $[\mathbf{LX}+H_2O+Ni^{2+}]^+$ (m/z_{calcd}=373.1) and indicating the formation of the stabilized cationic species **LX**-Ni^{2+} (Figure S7).

4. Conclusion

In summary, we have developed a Ni^{2+} sensor, which could detect Ni^{2+} in aqueous solution with specific selectivity and high sensitivity in a very short time. A unique colorimetric response to Ni^{2+} is realized through the coordination with sensor **LX**. In particular, competitive cations such as Fe^{3+}, Co^{2+} and Cu^{2+} did not afford any obvious interference response. The detection limits were 5.0×10^{-6} M and 2.2×10^{-7} M of Ni^{2+} using the naked-eye color changes and absorption spectra changes respectively. Moreover, test strips based on sensor **LX** was fabricated, which could serve as practical colorimetric sensor for "in-the-field" measurement of Ni^{2+} and did not require any additional equipment but just by virtue of "dip-sticks" approach. We believe the test strips could act as a convenient and efficient Ni^{2+} test kit.

Acknowledgment

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