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RESEARCH ARTICLE

'Turn-on' fluorescent chemosensors based on naphthaldehyde-2-pyridinehydrazone compounds for the detection of zinc ion in water at neutral pH

Yuanyuan Liu[1] | Yuanyuan Li[2,3] | Qi Feng[1] | Na Li[4] | Kai Li[1] | Hongwei Hou[1] | Bing Zhang[2]

[1] College of Chemistry and Molecular Engineering, Zhengzhou University, Henan, P. R. China

[2] School of Chemical Engineering and Energy, Zhengzhou University, Henan, P. R. China

[3] College of Chemistry, Chemical and Environmental Engineering, Henan University of Technology, Henan, P. R. China

[4] Department of Pharmacy, Women & Infants Hospital of Zhengzhou, Henan, P. R. China

Correspondence
Yuanyuan Li, School of Chemical engineering and Energy, Zhengzhou University, Henan 450001, P. R. China.
Email: yuanyuanli@haut.edu.cn

Kai Li, College of Chemistry and Molecular Engineering, Zhengzhou University, Henan 450001, P. R. China.
Email: likai@zzu.edu.cn

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Abstract

A series of naphthaldehyde-2-pyridinehydrazone derivatives were discovered to display interesting 'turn-on' fluorescence response to Zn^{2+} in 99% water/DMSO (*v*/*v*) at pH 7.0. Mechanism study indicated that different substituent groups in the naphthaldehyde moiety exhibited significant influence on the detection of Zn^{2+}. The electron rich group resulted in longer fluorescence wavelengths but smaller fluorescence enhancement for Zn^{2+}. Among these compounds, **1** showed the highest fluorescence enhancement of 19-fold with the lowest detection limit of 0.17 μmol/L toward Zn^{2+}. The corresponding linear range was at least from 0.6 to 6.0 μmol/L. Significantly, **1** showed an excellent selectivity toward Zn^{2+} over other metal ions including Cd^{2+}.

KEYWORDS

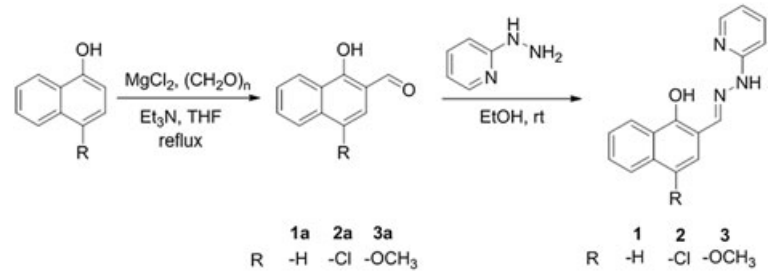
chemosensor, fluorescence, turn-on, zinc ion

1 | INTRODUCTION

Zinc is one of the necessary microelements for human beings, which is important and helpful for many biological activities such as gene code, DNA recombination or recognition, neural signal transmission and cellular metabolism [1–3]. Deficiency or surplus of zinc may cause physiological diseases [4–6]. Therefore, the measurement of zinc has significant values. Current analytical techniques such as flame atomic absorption spectrometry (FAAS), surface enhanced Raman scattering (SERS), colorimetry and ion selective electrode (ISE) have been applied for the detection of Zn^{2+} [7–10]. Among these methods, fluorescent chemosensors for Zn^{2+} have been developed as a widely used technique because of the merits such as convenience, emission signals being non-destructive and celerity [11–14]. Up to now, numerous fluorescent chemosensors based on different fluorogens have been utilized for the detection of Zn^{2+} [15–20]. Outstanding sensitivity and selectivity toward Zn^{2+} have been achieved. However, most of these reported fluorescent chemosensors worked in an environment containing high contents of organic co-solvents, which was not beneficial for the application in real samples [21,22]. Moreover, it is still a challenge to distinguish Zn^{2+} from Cd^{2+} due to their similar coordination modes and fluorescence responses [23,24]. Therefore, there is an urgent need to develop fluorescent chemosensors for Zn^{2+} detection to conquer the earlier mentioned challenges.

In this work, naphthaldehyde-2-pyridinehydrazone based 'turn-on' fluorescent chemosensor of 1-hydroxy-2-naphthaldehyde-2-pyridinehydrazone (**1**) and its control compounds 1-hydroxy-4-chloro-2-naphthaldehyde-2-pyridinehydrazone (**2**) and 1-hydroxy-4-methoxy-2-naphthaldehyde-2-pyridinehydrazone (**3**) for detecting

Abbreviations used: CHEF, chelation-enhanced fluorescence; DMSO, dimethyl sulfoxide; ESI-MS, electrospray ionization mass spectrometry.



SCHEME 1 Synthesis of **1**–**3**

Zn^{2+} in 99% water/dimethyl sulfoxide (DMSO) (*v*/*v*) at neutral pH were reported (Scheme 1). Enhancement of cyan fluorescence emission was detected with a UV lamp after mixing Zn^{2+} with an aqueous solution of **1** at neutral pH, enabling fluorescence 'turn-on' detection of Zn^{2+}. Compound **1** showed good sensitivity toward Zn^{2+} with a detection limit of 0.17 μmol/L. In fact, **1** was a Zn^{2+}-selective fluorescent chemosensor, which could distinguish other metal ions including Cd^{2+} from Zn^{2+}.

2 | EXPERIMENTAL

2.1 | Reagents

All the materials utilized in this experiment were of analytical grade unless otherwise noted. 1-Naphthol, 4-chloro-1-naphthol, hexamethylenetetramine, trifluoroacetic acid were purchased from Energy Chemical Co., Shanghai, China. 4-Methoxy-1-naphthol, 2-hydrazinopyridine were ordered from J&K Chemical Co., Beijing, China. All the other materials were purchased from Sinopharm Chemical Reagent Beijing Co., Beijing, China. Deionized water (distilled) and DMSO were applied throughout the experiments. All the metal ions solutions used in the experiment were prepared from their nitrate salts or perchlorate salts. Tris–HCl buffer solutions which possessed a wide pH range were prepared by utilizing 10 mmol/L Tris and suitable amount of hydrogen chloride (HCl) or sodium hydroxide (NaOH) modulated by a pH meter.

2.2 | Apparatus

Absorption spectra were determined on a JASCO V-550 UV–vis spectrophotometer. Fluorescence spectra were measured on a JASCO FP-8300 spectrofluorimeter equipped with an ETC-815 peltier thermostatted single cell holder. All pH tests were measured with a MettlerToledo FE20/EL20 pH meter. NMR spectra were recorded using a BRUKER400 spectrometer operated at 400 MHz. Mass spectra (electrospray ionization mass spectrometry [ESI-MS]) were measured on a Bruker Esquire 3000 pius ion trap mass spectrometer.

2.3 | Synthesis of 1-hydroxy-2-naphthaldehyde derivatives (1a–3a)

1-Hydroxy-2-naphthaldehyde derivatives were prepared as previous reported [25,26]. Then anhydrous magnesium chloride ($MgCl_2$) (15 mmol) and triethylamine (Et_3N) (37.5 mmol) were added to the mixture of 1-hydroxy-2-naphthaldehyde derivatives (14 mmol) and paraformaldehyde (84 mmol) in acetonitrile solution (50 mL). After being stirred at 90°C for 24 h, water (50 ml) was used to dilute the mixture and then extracted with ethyl acetate (300 ml). The organic layer was washed with 1% aqueous HCl solution and dried over anhydrous sodium sulfate. All volatiles were took away under reduced pressure and the products **1a–3a** were isolated by flash chromatography (EA/PE) on silica gel (yield of 41%, 36% and 31%, respectively).

ESI-MS spectrometry: *m*/*z* calc. For **1a**: 171.1 ([*M* - H]$^-$), found: 170.8. ^{1}H–NMR ($CDCl_3$) *δ* (ppm): 7.28 (d, 1H, *J* = 8.0 Hz), 7.36 (d, 1H, *J* = 8.0 Hz), 7.51 (t, 1H, *J* = 8.0 Hz), 7.62 (t, 1H, *J* = 8.0 Hz), 7.73 (d, 1H, *J* = 8.0 Hz), 8.41 (d, 1H, *J* = 8.0 Hz), 9.88 (s, 1H), 12.68 (s, 1H). ^{13}C–NMR ($CDCl_3$) *δ* (ppm): 114.23, 119.41, 124.27, 124.44, 126.09, 126.42, 127.62, 130.55, 137.46, 161.77, 196.26.

ESI-MS spectrometry: *m*/*z* calc. For **2a**: 205.0 ([*M* - H]$^-$), found: 204.9. ^{1}H–NMR ($CDCl_3$) *δ* (ppm): 7.54 (s, 1H), 7.63 (t, 1H, *J* = 4.0 Hz), 7.79 (t, 1H, *J* = 8.0 Hz), 8.18 (d, 1H, *J* = 12.0 Hz), 8.46 (d, 1H, *J* = 12.0 Hz), 9.88 (s, 1H), 12.56 (s, 1H). ^{13}C–NMR ($CDCl_3$) *δ* (ppm): 114.07, 122.44, 124.53, 124.74, 125.59, 125.65, 126.93, 131.61, 134.41, 160.73, 195.24.

ESI-MS spectrometry: *m*/*z* calc. For **3a**: 201.1 ([*M* - H]$^-$), found: 200.9. ^{1}H–NMR ($CDCl_3$) *δ* (ppm): 4.02 (s, 3H), 6.75 (s, 1H), 7.63 (t, 1H, *J* = 8.0 Hz), 7.72 (t, 1H, *J* = 8.0 Hz), 8.24 (d, 1H, *J* = 8.0 Hz), 8.45 (d, 1H, *J* = 8.0 Hz), 9.94 (s, 1H), 12.40 (s, 1H). ^{13}C–NMR ($CDCl_3$) *δ* (ppm): 55.74, 101.85, 113.07, 122.09, 124.27, 125.37, 126.80, 130.27, 130.30, 148.54, 156.72, 195.95.

The original files for the NMR spectrometry are provided in the Supporting Information.

2.4 | Synthesis of 1-hydroxy-2-naphthaldehyde-2-pyridinehydrazone derivatives (1–3)

Componds **1a–3a** (1 mmol) and 2-hydrazinopyridine (1 mmol) were dissolved in 20 ml pure ethanol. The mixture were stirred for 3 h at room temperature to form a precipitate. After being filtered, the precipitate was washed with 30 ml absolute ethanol three times. The precipitate was then dried under reduced pressure for a suitable time to gain the products **1–3** (yield of 82%, 77% and 87%, respectively).

ESI-MS spectrometry: *m*/*z* calc. For **1**: 264.1 ([*M* + H]$^+$), found: 264.0. ^{1}H–NMR (DMSO-d_6) *δ* (ppm): 6.82 (t, 1H, *J* = 8.0 Hz), 7.00 (d, 1H, *J* = 8.0 Hz), 7.43 (d, 1H, *J* = 8.0 Hz), 7.52 (m, 3H), 7.70 (t, 1H, *J* = 8.0 Hz), 7.84 (t, 1H, *J* = 8.0 Hz), 8.21 (d, 1H, *J* = 4.0 Hz), 8.31 (t, 1H, *J* = 4.0 Hz), 8.42 (s, 1H), 11.15 (s, 1H), 12.13 (s, 1H). ^{13}C–NMR (DMSO-d_6) *δ* (ppm): 106.63, 113.44, 115.80, 119.44, 122.71, 124.94, 126.00, 126.38, 127.58, 128.02, 134.27, 138.64, 142.38, 148.66, 153.30, 156.09.

ESI-MS spectrometry: *m/z* calc. For **2**: 298.1 ([*M* + H]$^+$), found: 297.9. ^{1}H–NMR (DMSO-d_6) δ (ppm): 6.84 (dd, 1H, *J* = 8.0 Hz), 7.01 (d, 1H, *J* = 8.0 Hz), 7.65 (m, 1H), 7.71 (m, 2H), 7.81 (s, 1H), 8.09 (d, 1H, *J* = 8.0 Hz), 8.22 (t, 1H, *J* = 4.0 Hz), 8.35 (d, 1H, *J* = 8.0 Hz), 8.38 (s, 1H), 11.24 (s, 1H), 12.14 (s, 1H). ^{13}C–NMR (DMSO-d_6) δ (ppm): 106.97, 114.54, 116.00,121.22, 123.41, 124.17, 125.68, 126.31, 126.97, 128.90, 130.56, 138.64, 140.28, 148.64, 152.31, 155.94.

ESI-MS spectrometry: *m/z* calc. For **3**: 294.1 ([*M* + H]$^+$), found: 294.0. ^{1}H–NMR (DMSO-d_6) δ (ppm): 3.96 (s, 3H), 6.83 (dd, 1H, *J* = 8.0 Hz), 7.02 (s, 1H), 7.04 (d, 1H, *J* = 8.0 Hz), 7.56 (m, 2H), 7.70 (t, 1H, *J* = 8.0 Hz), 8.10 (d, 1H, *J* = 8.0 Hz), 8.20 (d, 1H, *J* = 8.0 Hz), 8.25 (d, 1H, *J* = 8.0 Hz), 8.41 (s, 1H), 11.14 (s, 1H), 11.33 (s, 1H). ^{13}C–NMR (DMSO-d_6) δ (ppm): 56.14, 103.37, 105.61, 113.35, 115.70, 121.96, 122.75, 126.02, 126.12, 126.63, 127.00, 138.63, 141,71, 146,97, 148.34, 148.59, 156.32.

The original files for the NMR spectrometry are provided in the Supporting Information.

2.5 | Absorption and fluorescence measurements

The absorption and fluorescence spectra were recorded at 25°C. A stock solution of **1** (2 mmol/L) was prepared in absolute DMSO. In 4 ml glass tubes 0.03 ml of the stock solution was added to 2.97 ml buffer (10 mmol/L Tris–HCl with desired pH). The final concentration of 20 μmol/L was obtained for further experiments. Metal ions dissolved in water were added to the solution of **1** under the same conditions. After being blended well, the solutions were allowed to stay at 25°C for 1 min. Then absorption or fluorescence spectra were recorded.

3 | RESULTS AND DISCUSSION

3.1 | Binding of 1 with Zn^{2+}

The binding properties of **1** with Zn^{2+} were investigated by UV–vis absorption and fluorescence spectroscopy in an aqueous solution buffered by 10 mmol/L Tris–HCl at pH 7.0. As shown in Figure 1a, the absorption bands of **1** originally appeared at 376 nm and 396 nm in the absence of Zn^{2+}. With the addition of Zn^{2+}, these two absorption bands decreased gradually and a new absorption band at 416 nm appeared. The isosbestic point at 406 nm suggested the formation of **1**–Zn complex. Under the same condition, the fluorescence spectra showed obvious enhancement at 484 nm with the increase of Zn^{2+}. When more than 1 equiv. Zn^{2+} was added, the fluorescence intensity reached to the maximum and varied very slightly (Figure 1b). A 19-fold 'turn-on' fluorescence enhancement of **1** toward Zn^{2+} could be observed.

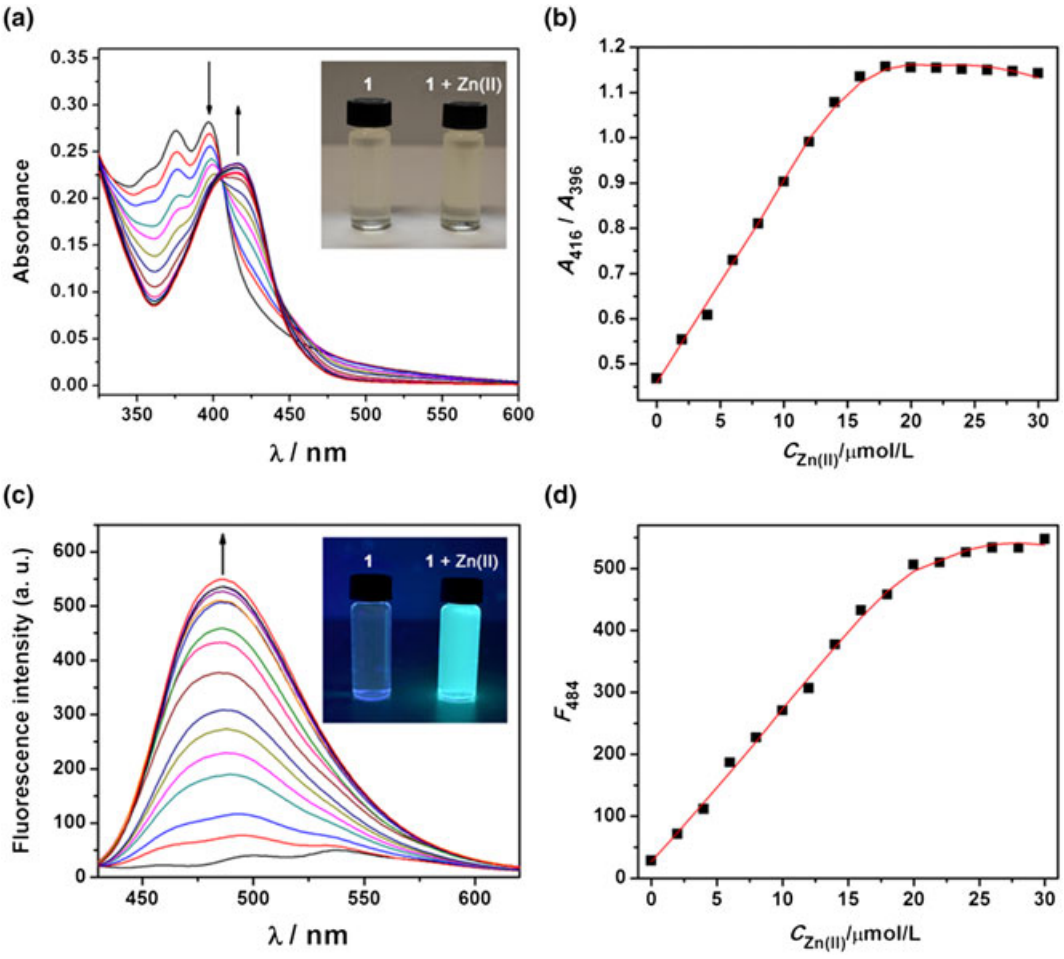


FIGURE 1 (a) absorption spectra and (c) fluorescence spectra of **1** (20 μmol/L) upon the addition of Zn^{2+} (0–30 μmol/L). (b) the absorbance ratio (A_{416}/A_{396}) and (d) the fluorescence intensity (F_{484}) as a function of Zn^{2+} concentration. Inset: The photographss are **1** in the absence and presence of 1 equiv. Zn^{2+}in a glass cuvette excited by (a) sun-light and (c) UV light (365 nm). Conditions: 99% water/DMSO (v/v) at pH 7.0 buffered by 10 mmol/L Tris–HCl

The non-linear fitting of the titration curve indicated that the combination of **1** and Zn^{2+} was probably a 1:1 stoichiometry. The association constant K_a value was about 4.94×10^6 L/mol. Fluorescence emission enhancement at 484 nm of **1** upon the addition of Zn^{2+} is considered to be on account of the generation of the **1**–Zn complex, which is a typical chelation-enhanced fluorescence (CHEF) process [27]. To further confirm the metal-to-ligand ratio, Job's plot of **1** and Zn^{2+} was tested from the fluorescence spectra with a whole concentration of 20 μmol/L (Figure 2), which also suggested the formation of a 1:1 complex.

According to the reports, the introduction of electron rich groups will bathochromic the fluorescence wavelength of the CHEF process [28]. Therefore, control compounds **2** and **3** with electron rich groups of chloric and methoxy were synthesized. Both of them exhibited fluorescence 'turn-on' response to Zn^{2+} (Supporting Information Figure S1), which was similar to that of **1**. Meanwhile, the metal-to-ligand ratios of Zn^{2+} to **2** and **3** were also 1:1 from Job's plots in Figures S2 and S3, which suggested that they exhibited analogous response mechanism as **1**. As expected, both of the fluorescence wavelengths of **2**–Zn (490 nm) and **3**–Zn (510 nm) were longer than that of **1**–Zn. Nevertheless, the fluorescence enhancements of the two conpounds were only seven- and five-fold, respectively, which were due to their stronger fluorescence backgrounds than that of **1**.

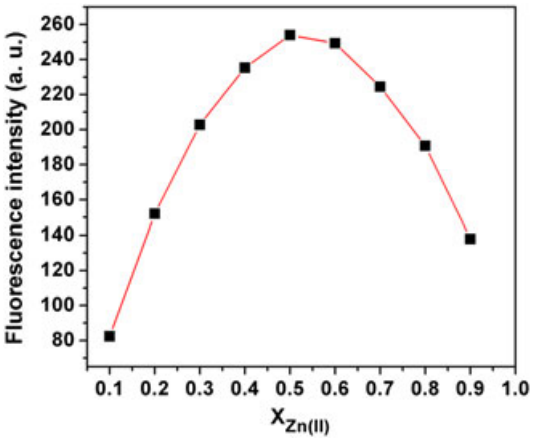


FIGURE 2 Job's plot method for evaluating the stoichiometry of **1**–Zn complex. [**1**] + [Zn^{2+}] = 20 μmol/L. conditions: 99% water/DMSO (*v*/*v*) at pH 7.0 buffered by 10 mmol/L Tris–HCl. Excitation and emission was at 395 nm and 484 nm, respectively

3.2 | Selectivity of 1 to Zn^{2+} over other metal ions

To explore the selectivity of **1** to Zn^{2+}, competition experiments in the presence of Zn^{2+} mingled with other metal ions were carried out. As shown in Figure 3, most of the metal cations, especially Cd^{2+}, showed scarce interference when detecting Zn^{2+}, suggesting that **1** possessed excellent selectivity for Zn^{2+} detection. It should be noted that **1** exhibited relatively poor detection selectivity in the presence of Cu^{2+}, which is due to the paramagnetic metal ions leading to fluorescence quenching [29]. Both **2** and **3** showed similar selectivity with **1** toward Zn^{2+} as well as toward other metal ions under the same conditions. However, the fluorescence enhancements of **2** and **3** with Zn^{2+} were not as good as **1** (Figures S4 and S5).

3.3 | Optimizing the pH for fluorescent determination of Zn^{2+}

The effect of pH on Zn^{2+} detection was also investigated. As shown in Figure 4, the fluorescence intensity of **1** was independent on the pH of the solution, which was due to the protonation of **1** in its pyridine unit. In contrast, for **1**–Zn complex, the fluorescence intensity reached a maximum at neutral pH. The reason could be explained as follows: in

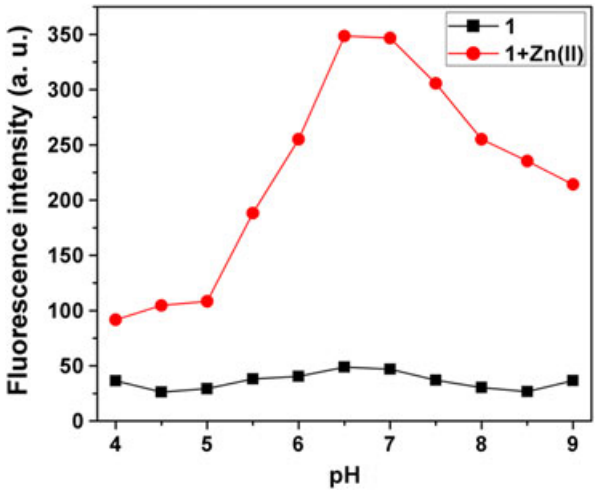


FIGURE 4 Fluorescence intensity of **1** in the absence and presence of Zn^{2+} at different pH. Conditions: [**1**] = 20 μmol/L, [Zn^{2+}] = 20 μmol/L, 99% water/DMSO (*v*/*v*) at different pH buffered by 10 mmol/L Tris–HCl. Excitation and emission was at 395 nm and 484 nm, respectively

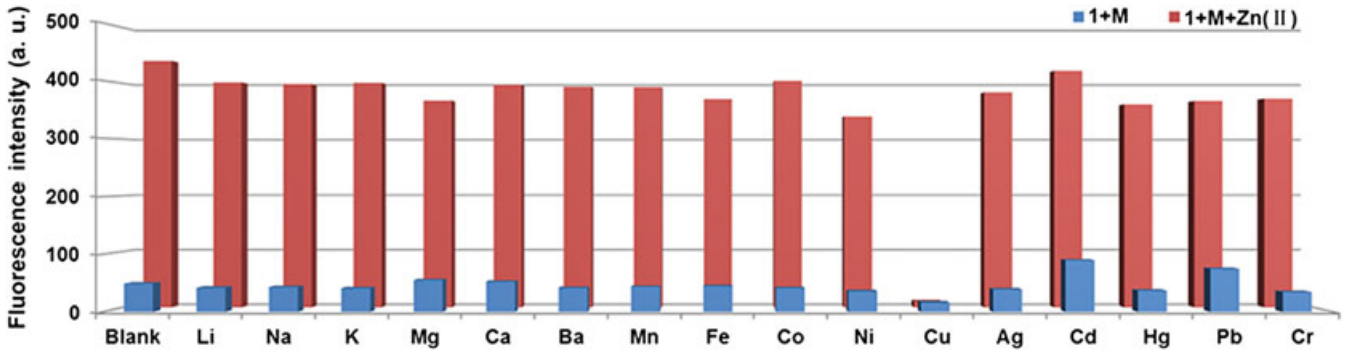


FIGURE 3 Fluorescence intensity of **1** in the presence of different metal ions with or without Zn^{2+}. Ions: Blank, Li^+, Na^+, K^+, Mg^{2+}, Ca^{2+}, Ba^{2+}, Mn^{2+}, Fe^{3+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Ag^+, Cd^{2+}, Hg^{2+}, Pb^{2+}, Cr^{3+}. Conditions: [**1**] = 20 μmol/L, [M] = 20 μmol/L, [Zn^{2+}] = 20 μmol/L, 99% water/DMSO (*v*/*v*) at pH 7.0 buffered by 10 mmol/L Tris–HCl. Excitation and emission was at 395 nm and 484 nm, respectively

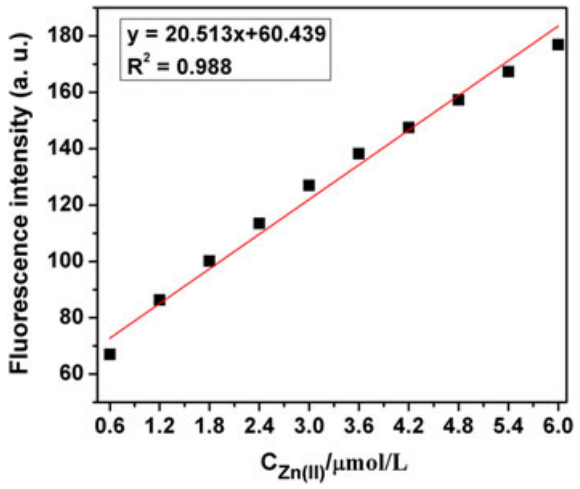


FIGURE 5 Fluorescence intensity of **1** as a function of Zn^{2+} concentration (0.6–6.0 μmol/L). Conditions: [**1**] = 20 μmol/L, 99% water/DMSO (*v*/*v*) at pH 7.0 buffered by 10 mmol/L Tris–HCl. Excitation and emission was at 395 nm and 484 nm, respectively

acidic solutions, **1** protonated its pyridine unit which weakened the binding ability with Zn^{2+}. In alkaline solution, the combination of OH^- with Zn^{2+} decreased the fluorescence of **1**–Zn. For the sake of achieving the highest signal-to-noise ratio, pH 7.0 was used for Zn^{2+} detection in the experiment. Similar optimal conditions of pH 7.0 could also be found in compounds **2** and **3** (Figures S6 and S7).

3.4 | Analytical figures of merit

The calibration curves for the determination of Zn^{2+} by **1** were established in the optimum condition of 99% water/DMSO (*v*/*v*) at pH 7.0 buffered by 10 mmol/L Tris–HCl (Figure 5). Compound **1** displayed a linear range of 0.6 to 6.0 μmol/L for Zn^{2+} detection with correlation coefficient of R^2 = 0.988 (n = 3). 0.17 μmol/L was computed as the detection limit based on the definition by IUPAC ($C_{DL} = 3S_b/m$) from 10 blank solutions. The relative standard deviation (n = 3) was 0.2% at 3 μmol/L Zn^{2+}. The corresponding calibration curves of control compounds **2** and **3** were dispiayed in Figures S8 and S9. The detection limits were 0.16 μmol/L and 0.24 μmol/L while the linear ranges were 0.4–4.0 μmol/L and 0.2–2.0 μmol/L, respectively.

4 | CONCLUSION

In conclusion, we have prepared a series of naphthaldehyde-2-pyridinehydrazone derivatives fluorescent chemosensors for Zn^{2+} detection in aqueous solution at neutral pH. As a sensitive and selective fluorescent chemosensor for Zn^{2+}, **1** displayed the detection limit of 0.17 μmol/L toward Zn^{2+} with a linear range of 0.6 to 6.0 μmol/L. Especially, when other physiological relevant metal ions including Cd^{2+} existed, **1** also showed excellent selectivity to Zn^{2+}.

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SUPPORTING INFORMATION

Additional Supporting Information may be found online in the supporting information tab for this article.

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