

# Synthesis of a Novel Fluorescent Schiff Base as a Possible Cu(II) Ion Selective Sensor

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**Abstract** In this study a new fluorescent Schiff base; 1,1'-(4,4'-oxybis(4,1-phenylene)bis(azan-1-yl-1-ylidene))bis(methan-1-yl-1-ylidene)dinaphthalen-2-ol (2-HNA) was synthesized and characterized by FT-IR, UV-vis, and  $^1\text{H}$  and  $^{13}\text{C}$ -NMR techniques. Photoluminescent properties of 2-HNA were investigated in different solvents including methanol, THF, DMF, DMSO, acetone, acetonitrile, and dichloromethane. 2-HNA was found to have higher emission intensity and Stoke's shift value ( $\Delta\lambda_{\text{ST}}$ ) in methanol solution. Relative emission intensity changes ( $I_0-I/I_0$ ) of 2-HNA in methanol/water mixtures depending on different  $\text{Cu}^{+2}$  ion concentrations were determined and a linearized plot was obtained. Possible interference of some other transition metal ions was also determined. Sensitivity limit of the new sensor was found to be higher than  $5 \times 10^{-7}$  mol/L. 2-HNA has quite high selectivity against  $\text{Cu}^{+2}$  ion and, thus, can be used as a new fluorescence  $\text{Cu}^{+2}$  ion sensor in practice.

**Keywords** Fluorescence sensor · Cu(II)sensor · Schiff base · Chemosensor

## Introduction

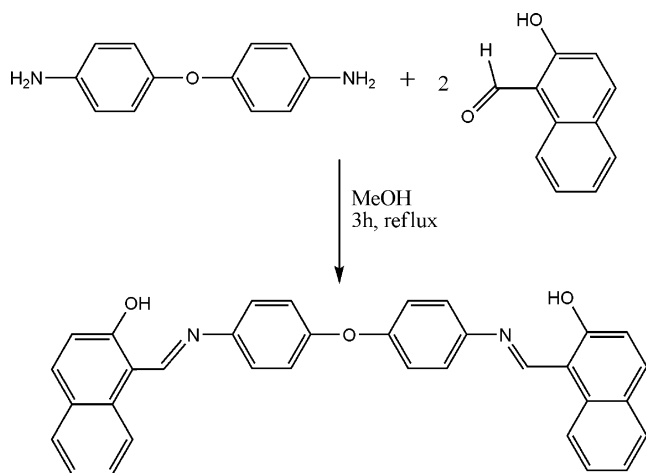
Detection of heavy metals in any aqueous sample has been a significant matter in Analytical Chemistry. Various analytical methods including atomic absorption spectroscopy (AAS), inductively coupled plasma atomic emission or

mass spectroscopy (ICP-AES, ICP-MS) have been widely used for detection of heavy metals even at very low concentrations. However, these methods are relatively expensive and difficult to be applied. To solve this problem a series of fluorescence sensors have been developed so far [1–5]. Fluorescence signaling supplies high sensitivity and can be directly used for chemosensors with fiber optic systems [6, 7]. Also, a significantly advantage of this sensing method is easy handling as well as cheaper to use than the other methods mentioned above. So, developing of new generation fluorescent agents containing chelating groups can help to detect heavy metals more efficiently. Polymeric compounds were also used in this field to obtain more selective-sensitive sensors [8, 9].

Copper is one of the important trace elements existing in human body, plants, and animals. However, high amount of this element can cause serious health problems including irritation of nose and throat, nausea, vomiting, and diarrhea as well as damage to liver and kidneys [10]. In the view of the biological and environmental importance a considerable attention has been focused on detection of Cu(II) ion [11, 12]. The studies developed in this area have been usually depended on quenching of fluorescence intensity of the employed sensing material [13–16]. This is because of the paramagnetic nature of Cu(II) ion. A series of spectrofluorometric Cu(II) selective sensors have been investigated with their selectivity, sensitivity, and also interference of other possible heavy metal kinds [17, 18].

In this study a newly fluorescence Schiff base, 1,1'-(4,4'-oxybis(4,1-phenylene)bis(azan-1-yl-1-ylidene))bis(methan-1-yl-1-ylidene)dinaphthalen-2-ol (2-HNA), was synthesized and developed as a possible Cu(II)-selective fluorescence sensor. An important advantage of the proposed Cu(II) sensor is easy production (only one step reaction) with fine sensing properties. It was observed that 2-HNA is capable

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**Scheme 1** Synthesis of 2-HNA

of selective complexation with Cu(II) ion which causes great quenching in the emission intensity. Selectivity study of the new Cu(II) sensor was investigated and a considerable selectivity was obtained according to relative intensity change values. Also, sensitivity of the present Cu(II) sensor was found quite high above  $10^{-6}$  mol/L.

## Experimental

### Materials

2-Hydroxy-1-naphthaldehyde, bis(4-aminophenyl)ether, methanol, acetonitrile, acetone, dichloromethane, dimethylformamide (DMF), dimethylsulfoxide (DMSO), tetrahydrofuran (THF),  $\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$ , and  $\text{Mn}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$  were supplied from Merck Chemical Co. (Germany),

$\text{Cu}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$ ,  $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ ,  $\text{Co}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ ,  $\text{Cd}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ , and  $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$  were from Fluka, and  $\text{ZrCl}_4$  and  $\text{CrCl}_3$  were from Riedel Dehaen and they were used as received.

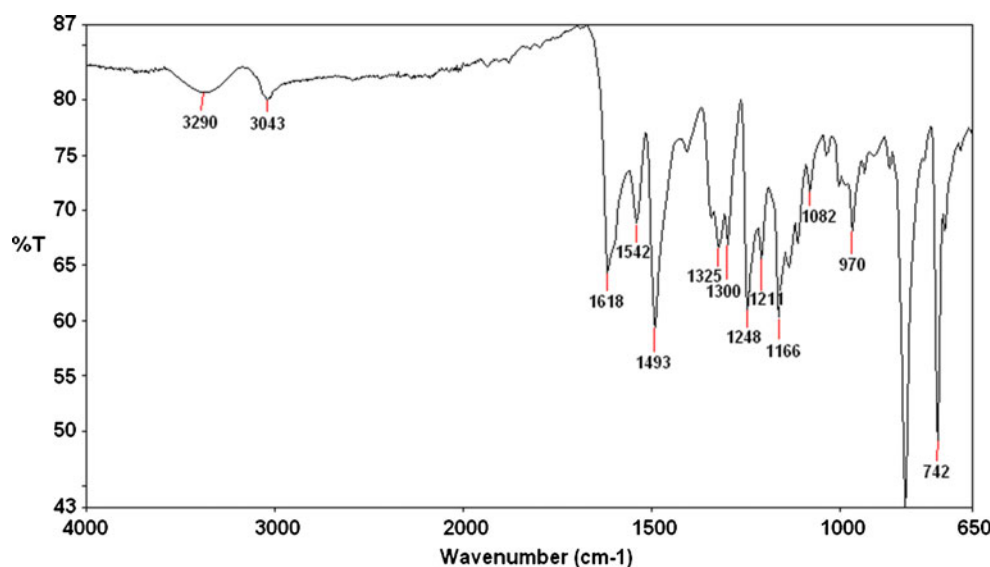
### Synthesis of 1,1'-(4,4'-oxybis(4,1-phenylene))bis(methan-1-yl-1-ylidene)dinaphthalen-2-ol (2-HNA)

2-HNA was synthesized by the condensation reaction of bis(4-aminophenyl)ether with 2-hydroxy-1-naphthaldehyde. Schiff bases with similar structures have been previously synthesized and reported in the literature [19, 20]. Reaction was made as follow: Bis(4-aminophenyl)ether (0.2 g, 0.001 mol) was placed into a 250 ml three-necked round-bottom flask which was fitted with condenser, thermometer and magnetic stirrer and 60 ml methanol was added into the flask as a solvent. Reaction mixture was heated at boiling temperature of methanol and bis(4-aminophenyl)ether was solved. A solution of 2-hydroxy-1-naphthaldehyde (0.516 g, 0.003 mol) in 20 ml methanol was added into the flask. Reaction was maintained for 3 h under reflux (Scheme 1). The precipitated Schiff base filtered, recrystallized from acetonitrile and dried in a vacuum desiccator (yield: 87%).

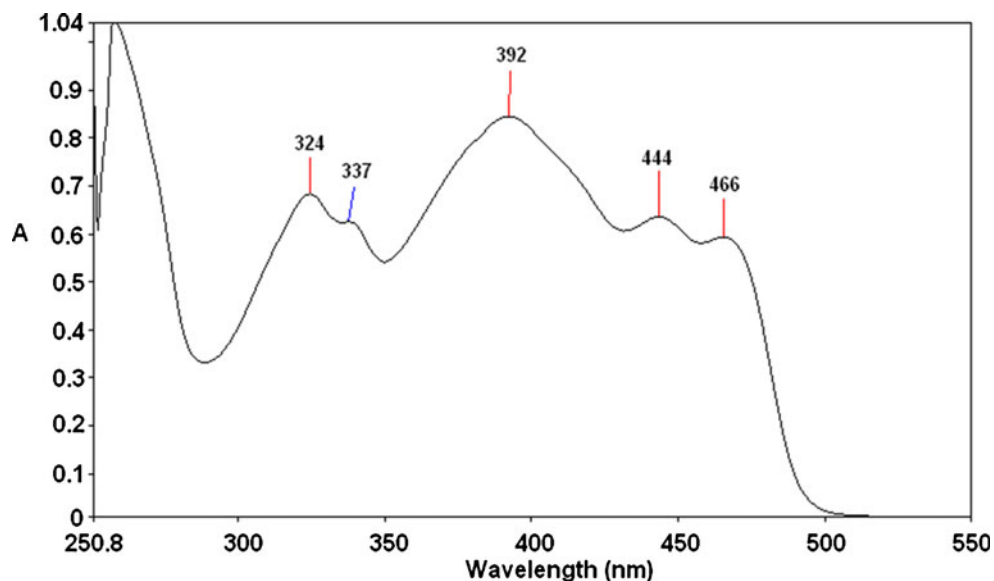
### Characterization techniques

The solubility tests were done in different solvents by using 1 mg sample and 1 ml solvent at 25 °C. The infrared and ultraviolet-visible spectra were measured by Perkin Elmer FT-IR Spectrum one and Perkin Elmer Lambda 25, respectively. The absorption spectrum was recorded in methanol at 25 °C. The optical band gap ( $E_g$ ) value of 2-HNA was also calculated from its absorption edge [21]. The

**Fig. 1** FT-IR spectrum of 2-HNA



**Fig. 2** UV-vis spectrum of 2-HNA

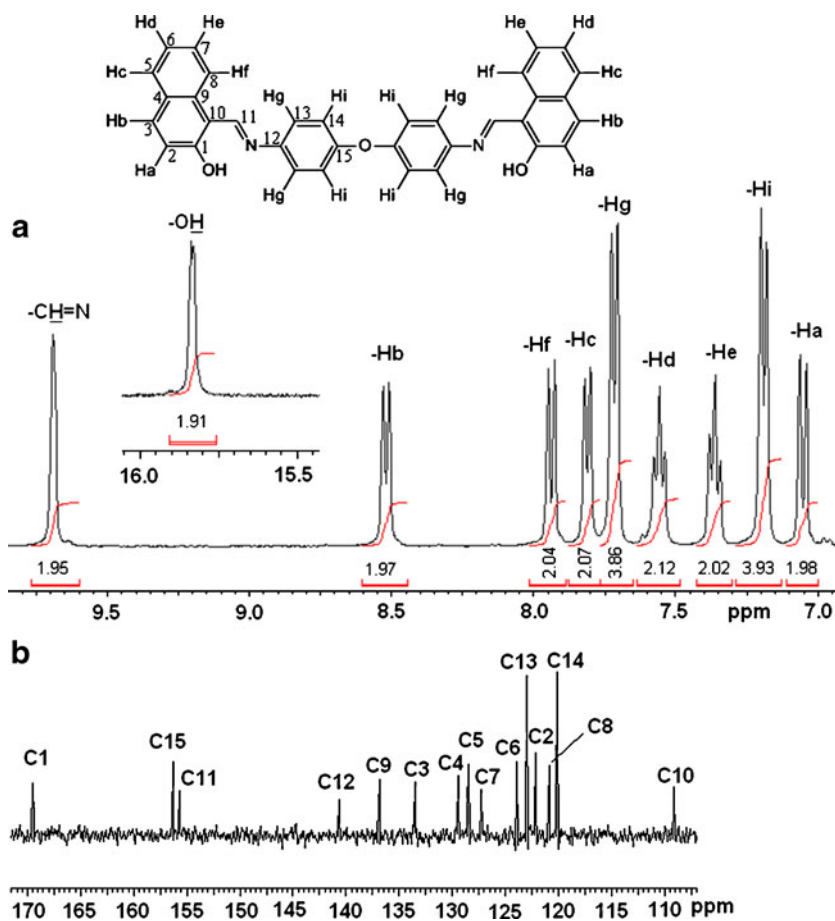


FT-IR spectrum was recorded using universal ATR sampling accessory (4,000–550  $\text{cm}^{-1}$ ).  $^1\text{H}$  and  $^{13}\text{C}$ -NMR spectra (Bruker AC FT-NMR spectrometer operating at 400 and 100.6 MHz, respectively) were obtained by using deuterated DMSO- $\text{d}_6$  as a solvent at 25  $^\circ\text{C}$ . The tetramethylsilane was used as internal standard.

Florescence measurements

A Shimadzu RF-5301PC spectrofluorophotometer was used in florescence measurements. Emission spectra of 2-HNA were obtained in different solvents with the concentration of  $2 \times 10^{-4}$  M 2-HNA. The optimal emission and excitation

**Fig. 3**  $^1\text{H}$ -NMR (a) and  $^{13}\text{C}$ -NMR (b) spectra of 2-HNA obtained in DMSO- $\text{d}_6$



**Table 1** Fluorescence spectra related data of 2-HNA

Solvent	<sup>a</sup> $\lambda_{\text{Ex}}$	<sup>b</sup> $\lambda_{\text{Em}}$	<sup>c</sup> $\lambda_{\text{max}} (\text{Ex})$	<sup>d</sup> $\lambda_{\text{max}} (\text{Em})$	<sup>e</sup> $I_{\text{Ex}}$	<sup>f</sup> $I_{\text{Em}}$	<sup>g</sup> $\Delta\lambda_{\text{ST}}$
DMF	484	507	484	498	112	117	14
DMSO	486	511	486	499	154	169	13
MeOH	493	530	493	531	324	187	37
THF	478	500	477	492	76	82	15
Acetone	475	498	475	491	68	72	16
Acetonitrile	–	–	–	–	–	–	–
Dichloromethane	–	–	–	–	–	–	–

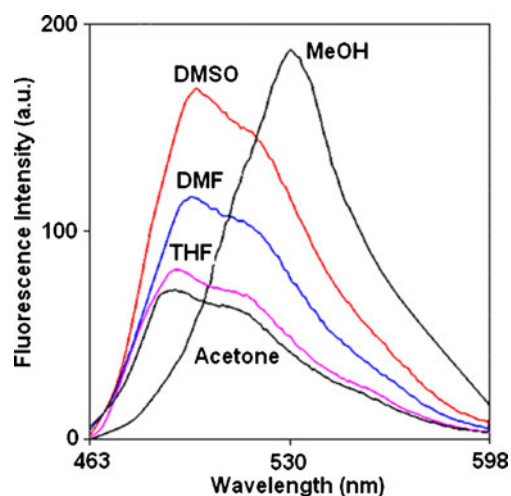
<sup>a</sup> Excitation wavelength for emission<sup>b</sup> Emission wavelength for excitation<sup>c</sup> Maximum emission wavelength<sup>d</sup> Maximum excitation wavelength<sup>e</sup> Maximum excitation intensity<sup>f</sup> Maximum emission intensity<sup>g</sup> Stoke's shift

wavelengths in each solvent were determined. The effects of transition metal ions on quenching of emission spectra were investigated in 1:1 (v:v) MeOH / deionized water solutions each of which containing  $5 \times 10^{-2}$  M metal ion and  $4.91 \times 10^{-4}$  M 2-HNA. The changes of fluorescence intensities depending on the concentration of  $\text{Cu}^{+2}$  ion were determined using  $4.50 \times 10^{-4}$  M 2-HNA and  $\text{Cu}^{+2}$  ion with the concentration range of  $5 \times 10^{-2}$ – $5 \times 10^{-8}$  M in 1:1 (v:v) MeOH / deionized water mixtures.

## Results and discussion

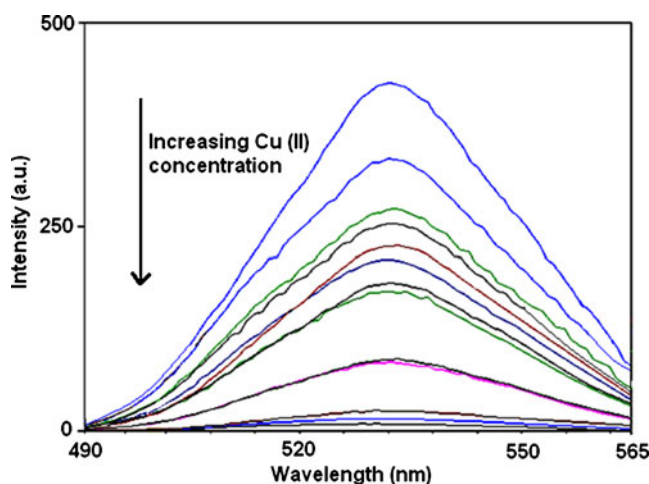
### Solubility and structure of 2-HNA

The synthesized Schiff base has yellow colored-powder form. 2-HNA is soluble in MeOH, EtOH, THF, DMF,

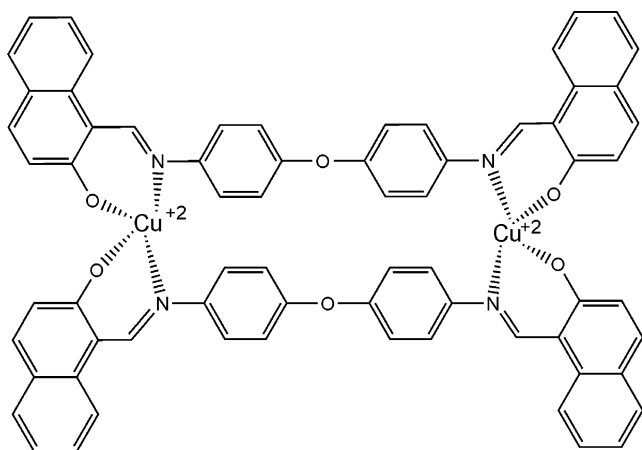


**Fig. 4** Emission spectra of 2-HNA in different solvents. Conditions: Slit:  $\lambda_{\text{Ex}}$  5 nm,  $\lambda_{\text{Em}}$  5 nm; concentration of 2-HNA:  $2 \times 10^{-4}$  mol/L

DMSO, acetonitrile, acetone, and chlorinated hydrocarbons such as chloroform and dichloromethane. But it's insoluble in water, benzene, toluene, hexane, and heptane. The FT-IR spectrum of 2-HNA is shown in Fig. 1. This spectrum confirms the proposed structure. After the condensation reaction the new peak at  $1,618 \text{ cm}^{-1}$  indicating imine ( $\text{CH}=\text{N}$ ) bond appears. Also,  $-\text{NH}_2$  peaks of bis(4-amino-phenyl)ether and  $-\text{C}=\text{O}$  peak of 2-hydroxy-1-naphtaldehyde disappear in the FT-IR spectrum of 2-HNA. The peak values of  $-\text{OH}$ , aromatic C-H, aromatic C=C, and C-O-C vibrations are seen at 3,290, 3,043, 1,542–1,493, and  $1,248 \text{ cm}^{-1}$ , respectively. These values agree with the literature data published for similar structures [19].



**Fig. 5** Emission spectra of 2-HNA in the presence  $\text{Cu}^{+2}$  ion with different concentrations (mol/L, from bottom to top):  $5 \times 10^{-2}$ ,  $2.5 \times 10^{-2}$ ,  $1.25 \times 10^{-2}$ ,  $5 \times 10^{-3}$ ,  $2.5 \times 10^{-3}$ ,  $1.25 \times 10^{-3}$ ,  $5 \times 10^{-4}$ ,  $2.5 \times 10^{-4}$ ,  $1.25 \times 10^{-4}$ ,  $5 \times 10^{-5}$ ,  $5 \times 10^{-6}$ ,  $5 \times 10^{-7}$ , and 0. Conditions: Slit:  $\lambda_{\text{Ex}}$  5 nm,  $\lambda_{\text{Em}}$  5 nm;  $\lambda_{\text{Ex}}$  280 nm,  $\lambda_{\text{Em}}$  531 nm; concentration of 2-HNA:  $4.5 \times 10^{-4}$  mol/L



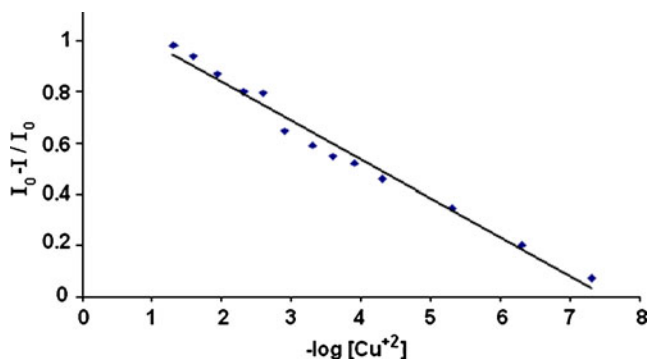
**Scheme 2** Possible structure of  $\text{Cu}^{+2}$  complex of 2-HNA formed after exposure to  $\text{Cu}^{+2}$  ion

The UV-vis spectrum of 2-HNA exhibits absorbances ( $\lambda_{\text{max}}$ ) at 258, 324, 337, 392, 444, and 466 nm (Fig. 2). The optical band gap value of 2-HNA is found as 2.34 eV. This is a smaller value than the previously reported similar structured bis(4-aminophenyl)ether Schiff bases [19]. The smaller band gap indicates easy electronic transitions from bulk to vacuum energy level of the compound and also high electrical conductivity as well as fluorescence property.

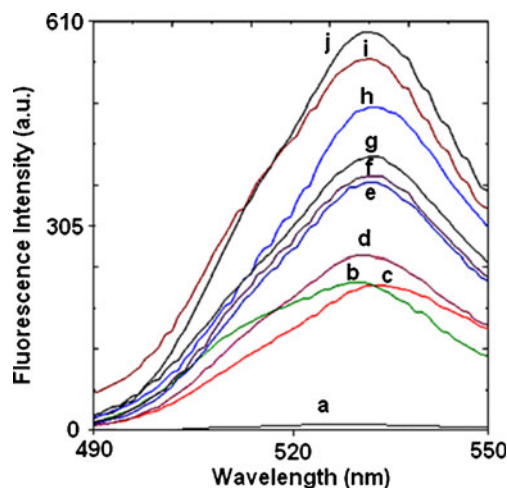
$^1\text{H-NMR}$  and  $^{13}\text{C-NMR}$  spectra of 2-HNA are given in Fig. 3a and b, respectively. The structure of 2-HNA is also confirmed by these spectra. NMR spectral data are summarized as follows:

$^1\text{H-NMR}$  (DMSO): 15.84 (s, 2H, -OH), 9.69 (s, 2H, -CH=N-), 8.52 (d, 2H, -Hb), 7.94 (d, 2H, -Hf), 7.81 (d, 2H, -Hc), 7.72 (d, 4H, -Hg), 7.56 (t, 2H, -Hd), 7.36 (t, 2H, -He), 7.19 (d, 4H, -Hi), 7.05 (d, 2H, -Ha).

$^{13}\text{C-NMR}$  (DMSO): 169.49 (C1-*ipso*), 156.31 (C15-*ipso*), 155.72 (C11-H), 140.65 (C12-*ipso*), 136.87 (C9-*ipso*), 133.50 (C3-H), 129.45 (C4-*ipso*), 128.49 (C5-H), 127.23 (C7-H), 123.93 (C6-H), 122.95 (C13-H), 122.15 (C2-H), 120.89 (C8-H), 120.11 (C14-H), 109.15 (C10-*ipso*).



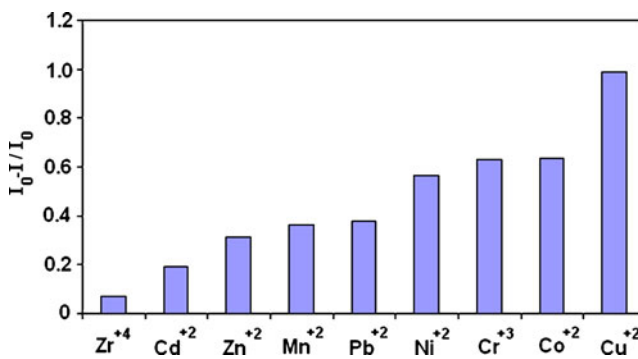
**Fig. 6** Linearized response of 2-HNA to  $\text{Cu}^{+2}$  ion



**Fig. 7** Emission spectra of 2-HNA with presence of  $5 \times 10^{-2}$  mol/L metal ions, (a)  $\text{Cu}^{+2}$ , (b)  $\text{Cr}^{+3}$ , (c)  $\text{Co}^{+2}$ , (d)  $\text{Ni}^{+2}$ , (e)  $\text{Pb}^{+2}$ , (f)  $\text{Mn}^{+2}$ , (g)  $\text{Zn}^{+2}$ , (h)  $\text{Cd}^{+2}$ , (i)  $\text{Zr}^{+4}$ , and (j) 2-HNA free metal ions. Conditions: Slit:  $\lambda_{\text{Ex}}$  5 nm,  $\lambda_{\text{Em}}$  5 nm;  $\lambda_{\text{Ex}}$  280 nm,  $\lambda_{\text{Em}}$  531 nm. Concentration of 2-HNA:  $4.91 \times 10^{-4}$  mol/L

#### Fluorescence characteristics of 2-HNA

The optimal emission and excitation wavelengths of 2-HNA in different solvents are determined and shown in Table 1. 2-HNA has no fluorescence effect in acetonitrile and dichloromethane. However it has remarkable emission and excitation intensities in the other solvents, as seen in Table 1. According to these results 2-HNA has the maximal emission intensity in methanol solution. Also, the Stoke's shift values ( $\Delta\lambda_{\text{ST}}$ ) in all employed solvents are calculated as in the literature [3] and shown in Table 1. Stoke's shift is important for a fluorescence sensor. The higher Stoke's shift value supplies very low background signals and resultantly allows the usage of the material in construction of a fluorescence sensor. As seen in Table 1, in comparison with the other solvents methanol solution of 2-HNA has quite higher Stoke's shift with the value of 37 nm, while in the other employed solvents it has  $\Delta\lambda_{\text{ST}}$  values between 13 and 16 nm. This indicates that the usage of 2-HNA as an ion selective



**Fig. 8** Relative intensity change of 2-HNA on exposure to  $5 \times 10^{-2}$  mol/L different transition metal cations

sensor is useful in methanol solution. The emission spectra of 2-HNA in different solvents are also shown in Fig. 4.

It's experimentally seen that absence of  $\text{Cu}^{+2}$  ion in MeOH-water solution of 2-HNA causes considerable quenching in fluorescence intensity. The changes in emission spectrum of 2-HNA solution in 1:1 (v:v) methanol/water mixture depending on different  $\text{Cu}^{+2}$  concentrations are also given in Fig. 5. According to these spectra when the  $\text{Cu}^{+2}$  concentrations are  $5 \times 10^{-2}$  and  $5 \times 10^{-7}$  mol/L the relative intensity changes ( $I_0-I/I_0$ ) are 0.981 and 0.204, respectively.

The quenching in fluorescence of 2-HNA during exposure to  $\text{Cu}^{+2}$  cation indicates the complexation of 2-HNA with  $\text{Cu}^{+2}$ . The possible structures of the Schiff base complexes and its polymeric forms derived from bis(4-aminophenyl)ether have been previously published [19, 22]. According to the proposed structures 2-HNA has bidentate sides in both ends of the molecule. As a result the structure of the forming  $\text{Cu}^{+2}$  complex is expected to be like in the Scheme 2.

The dependence of relative intensity change with the concentration of  $\text{Cu}^{+2}$  ion is shown in Fig. 6. According to Fig. 6 when the concentration of  $\text{Cu}^{+2}$  ion increased from  $5 \times 10^{-2}$  mol/L to  $5 \times 10^{-8}$  mol/L the relative intensity change linearly decreases. The linear regression equation is as follow:

$$\frac{I_0 - I}{I_0} = 1.1449 + 0.1522 \log[\text{Cu}^{+2}]$$

where  $I_0$  is the emission intensity of  $\text{Cu}^{+2}$  free 2-HNA solution and  $I$  is the intensity of  $\text{Cu}^{+2}$  containing 2-HNA solution. The correlation coefficient is 0.99076. According to obtained results 2-HNA can be used as a new fluorescence sensor to detect the quantity of  $\text{Cu}^{+2}$  ion in any sample solution depending on the relative intensity change.

#### Selectivity studies

In order to determine the selectivity of the new  $\text{Cu}^{+2}$  sensor the influence of a number of transition metal cations were investigated. The experiments were performed in  $5 \times 10^{-2}$  mol/L  $\text{Pb}^{+2}$ ,  $\text{Mn}^{+2}$ ,  $\text{Cu}^{+2}$ ,  $\text{Zn}^{+2}$ ,  $\text{Co}^{+2}$ ,  $\text{Cd}^{+2}$ ,  $\text{Ni}^{+2}$ ,  $\text{Zr}^{+4}$ , and  $\text{Cr}^{+3}$  separate solutions each of which contain  $4.91 \times 10^{-4}$  M 2-HNA in 1:1 (v:v) methanol/deionized water mixture (Fig. 7). The results show that 2-HNA has quite fluorescence quenching in absence of  $\text{Cu}^{+2}$  ion. Yet, there is no remarkable spectral change on exposure to the other cations, which are the same concentrated. The relative intensity changes with the tested ions are also shown in Fig. 8.

The selectivity results show that 2-HNA can be easily complex with  $\text{Cu}^{+2}$  ion. Also, as seen in Figs. 7 and 8, the intensity of 2-HNA relatively decreases in presence of some other ions such as  $\text{Cr}^{+3}$ ,  $\text{Co}^{+2}$ , and  $\text{Ni}^{+2}$ . These decreases

may affect the quantitative measurement of  $\text{Cu}^{+2}$  ion in any sample if they presence with too high concentrations. However, the known samples like sea water usually contain little concentration of these cations those aren't expected to affect the quantitative measurement of  $\text{Cu}^{+2}$  ion. As a result, 2-HNA can be used as an alternative  $\text{Cu}^{+2}$  sensor by using the fluorescence technique.

#### Conclusion

A new tetradentate Schiff base 2-HNA was synthesized as a  $\text{Cu}^{+2}$  ion selective fluorescence sensor. Characterization of 2-HNA was made by FT-IR, UV-vis, and NMR techniques. Fluorescence intensities of 2-HNA in different solvents were determined. Stoke's shift values in the employed solvents showed that methanol was the best solvent to use 2-HNA as a  $\text{Cu}^{+2}$  sensor. The relative emission intensity changes ( $I_0-I/I_0$ ) of 2-HNA were determined in the presence of  $\text{Cu}^{+2}$  ion over the concentration range of  $5 \times 10^{-8}$  to  $5 \times 10^{-2}$ . A linearized change with a regression coefficient of 0.99076 was obtained. Selectivity studies were carried out with some other transition metal cations those have the same concentrations. It was found that 2-HNA has quite high selectivity to  $\text{Cu}^{+2}$  ion. As a result, 2-HNA can be used in quantity determination of  $\text{Cu}^{+2}$  ion in any aqueous solution such as sea or lake water using relative fluorescence change.

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