J. Chem. Sci. (2020) 132:82
https://doi.org/10.1007/s12039-020-01789-y

REGULAR ARTICLE

A conjugated Schiff base-based chemosensor for selectively detecting mercury ion

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MS received 31 January 2019; accepted 7 March 2020

Abstract. A novel conjugated Schiff base-based chemosensor **DAP**, *N*′-((1E,2E)-3-(4-(dimethylamino)phenyl)allylidene)-3-nitrobenzohydrazide, has been synthesized. **DAP** showed significant selectivity toward mercury ion by color change of pale yellow to orange. Detection limit was 0.11 μM, which is lower than the value (2.48 μM) recommended by Health Canada. **DAP** could recognize and quantify mercury ion in real water samples. The binding mode of **DAP** and Hg^{2+} was demonstrated, based on Job plot, IR spectra and ESI-MS.

Keywords. Conjugated Schiff base; chemosensor; colorimetric; mercury.

1. Introduction

Selective and sensitive detection of various metal ions has received remarkable attention since exposure to metal ions could lead to a number of diseases to human being.[1–5] Mercury belongs to one of the heavy metals and is highly toxic to human body and environments.[6–8] Even small amount of mercury in the human body would cause critical diseases like digestive, brain damage, and chronic disease.[9–11] In spite of its severe effects, mercury is widely used at industrial fields including gold mining, batteries, paints and fuel combustion.[12–14] The use of mercury could lead to a big pollution in our environments.[15–17] Because of these destructive effects of mercury ion, detection of Hg^{2+} is essential.

Diverse types of analytical methods were developed to detect metal ions, for example, ICP-MS, ion selective membrane electrodes, and modified nanotube carbon electrode.[18–20] However, some techniques of them have several issues like expensive cost, complex instruments, and complicated processes.[21–23] On the other hand, colorimetric chemosensors have become an interesting field, since they have particular advantages like simple operation without exquisite method and low cost.[24–26]

Colorimetric chemosensors commonly include chromophores, such as bipyridines, Schiff bases, guanidine, azo and rhodamine dyes.[27–35] Among these moieties, conjugated Schiff bases are prominent due to their easy and simple synthesis, notable photophysical features, and strong interaction with various heavy metal ions.[21,36–40] With this in mind, we designed a new conjugated Schiff base colorimetric chemosensor by the reaction of 3-nitrobenzohydrazide and 4-(dimethylamino)cinnamaldehyde for the sensing of the heavy metal ions like mercury ion.

Herein, we describe design and application of a novel conjugated Schiff base-based chemosensor **DAP** for detecting mercury ion through color change. Sensor **DAP** could recognize only mercury ion in aqueous solution and showed obvious spectral and color changes of pale yellow to orange. With the Job plot, UV-vis titrations, ESI-MS and IR spectra, the binding structure and sensing mechanism for Hg^{2+}-2•**DAP** were suggested.

2. Experimental

2.1 *General information*

All chemicals were furnished from Sigma-Aldrich. All buffers (pH = 7) employed in the experiments were set to be

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Electronic supplementary material: The online version of this article (https://doi.org/10.1007/s12039-020-01789-y) contains supplementary material, which is available to authorized users.

Published online: 04 July 2020

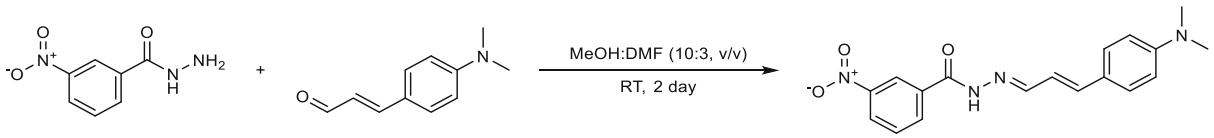
10 mM of Bis-Tris. A Varian spectrometer was adopted for ^{1}H and ^{13}C NMR measurements. A Thermo Finnigan instrument was used for producing ESI-MS data. UV-vis spectra were obtained with a Perkin Elmer spectrometer.

2.2 *Synthesis of **DAP** (N'-((1E,2E)-3-(4-(dimethylamino)phenyl)allylidene)-3-nitrobenzohydrazide)*

1×10^{-3} mol of 3-nitrobenzohydrazide and 1×10^{-3} mol of 4-(dimethylamino)cinnamaldehyde were dissolved in 3 mL of DMF. 10 mL of methanol was added into the solution, which was stirred for 2 days at 23 °C. After an orange powder was formed, it was filtered and rinsed with EtOH and ether (0.264 g, 78 %). ^{1}H NMR ($CD_3S(O)CD_3$, 400 MHz): 11.87 (s, 1H), 8.72 (s, 1H), 8.41 (d, J = 7.6 Hz, 1H), 8.32 (d, J = 8.0 Hz, 1H), 8.18 (d, J = 9.1 Hz, 1H), 7.81 (t, J = 8.0 Hz, 1H), 7.45 (d, J = 8.8 Hz, 2H), 6.94 (d, J = 16 Hz, 1H), 6.79 (m, 1H), 6.7 (d, J = 8.8 Hz, 2H), 2.94 (s, 6H), ^{13}C NMR ($CD_3S(O)CD_3$, 100 MHz): δ 160.52, 151.75, 150.79, 147.75, 140.64, 134.97, 134.05, 130.24, 128.55, 126.16, 123.50, 122.22, 120.20, 111.97, 39.80 ppm. ESI-MS: *m/z* calcd. for $C_{21}H_{25}N_5NaO_4^+$ ([**DAP** + *N,N*-dimethylformamide + Na^+]), 434.18; expd, 433.83.

2.3 *Colorimetric detection of Hg^{2+}*

DAP (3 μmol) was dissolved in 3 mL DMF (*N,N*-dimethylformamide) to produce 1 mM and 30 μmol of $Hg(NO_3)_2$ was dissolved in 3 mL DMF to produce 10 mM.



Scheme 1. Synthesis of sensor **DAP**.

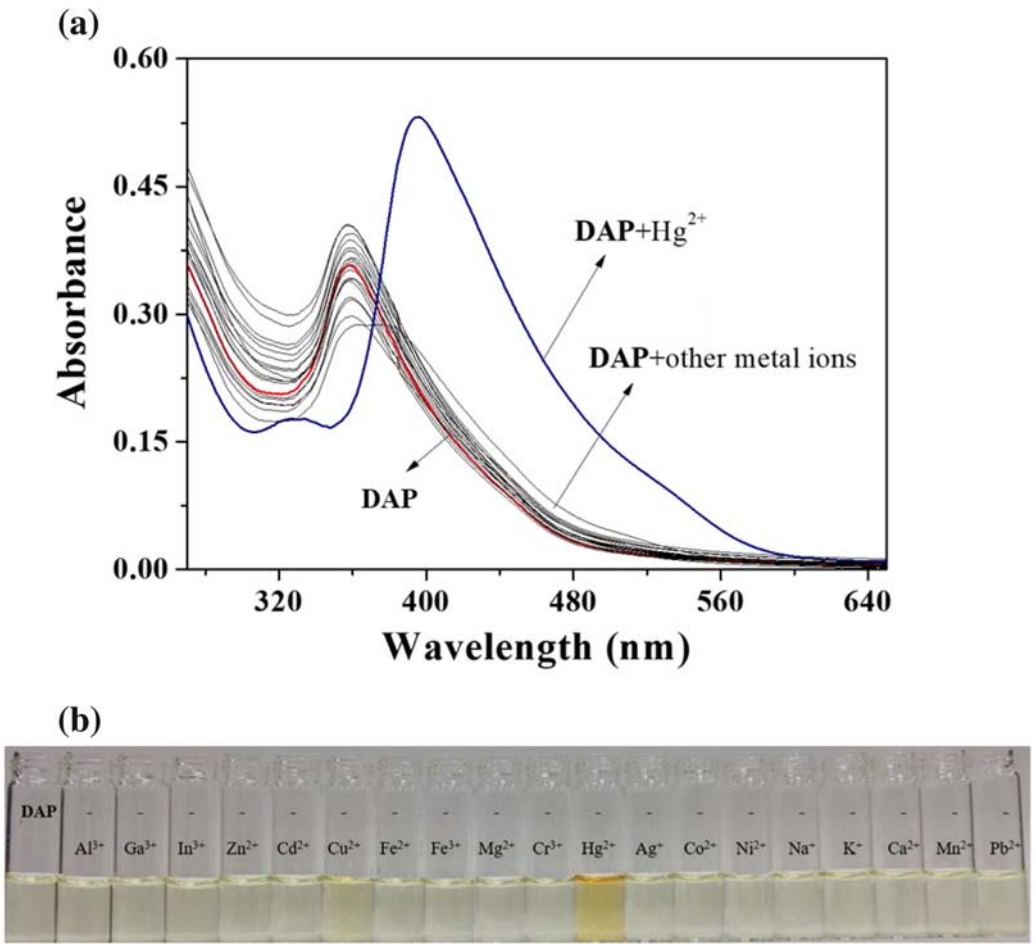


Figure 1. (a) UV-vis variations of **DAP** (2×10^{-5} M) upon addition of varied cations (0.5 equiv) in buffer/DMF (98:2). (b) Color changes of **DAP** (2×10^{-5} M) upon addition of varied cations (0.5 equiv) in buffer/DMF (98:2).

2.4 *UV-vis titration*

The prepared **DAP** solution (60 μL) was put into 2.94 mL of buffer to produce 20 μM. After blending it, 0.3–3.3 μL of the 10 mM $Hg(NO_3)_2$ was gradually put into the solution. The absorption spectra were recorded using a UV/Vis spectrometer.

2.5 *Job plot*

6–54 μL of **DAP** solution (1×10^{-3} M) was transferred to 3 mL buffer in each cuvette. 5.4–0.6 μL of mercury solution (1×10^{-2} M) was put into the diluted **DAP** solution. After 20 min, the absorption spectra were recorded using a UV/Vis spectrometer.

2.6 *Competitive experiment*

Metal solutions (30 μmol) of $NaNO_3$, $Fe(ClO_4)_2$, $AgNO_3$, $Fe(NO_3)_3$, $Zn(NO_3)_2$, $Cd(NO_3)_2$, $Cu(NO_3)_2$, $Ga(NO_3)_3$, $Mg(NO_3)_2$, $In(NO_3)_3$, $Hg(NO_3)_2$, $Co(NO_3)_2$, $Ni(NO_3)_2$, $Ca(NO_3)_2$, $Mn(NO_3)_2$, $Pb(NO_3)_2$, $Al(NO_3)_3$, $Cr(NO_3)_3$ and KNO_3 were prepared in DMF (3 mL). 3.0 μL of each metal was put into buffer, respectively. Then, 3.0 μL of mercury solution was mixed with each cuvette. Lastly, 60 μL of **DAP** solution was added to make 3 mL solution. The absorption spectra were measured using a UV/Vis spectrometer.

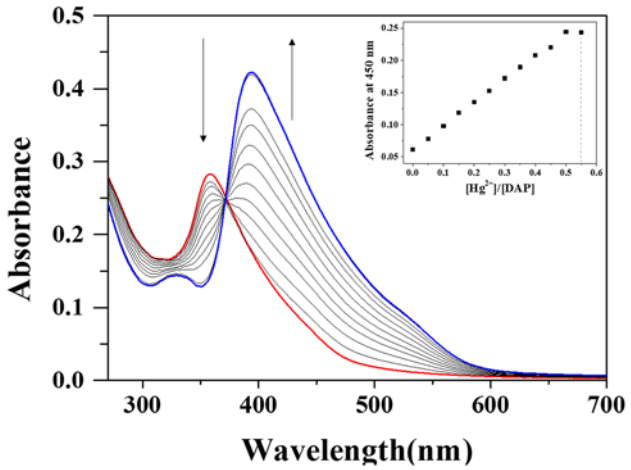


Figure 2. UV-vis variations of **DAP** (2×10^{-5} M) with different concentrations of Hg^{2+} ions in buffer/DMF (98:2).

2.7 *pH test*

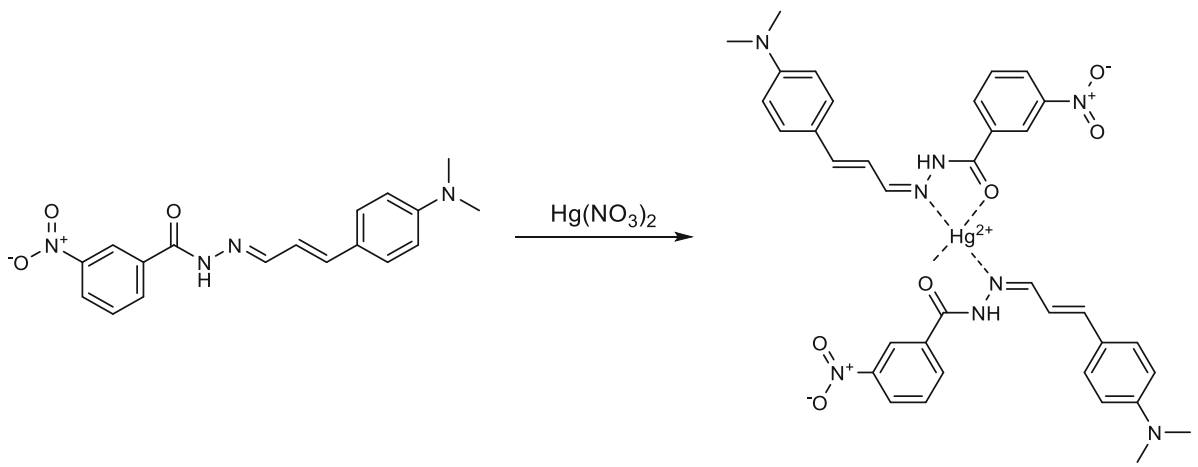
10 mM of Bis-Tris buffer was adjusted to pH 4–12 by adding HCl or NaOH. Each pH buffer was transferred into each cuvette and 60 μL **DAP** was added into the buffer solution. Absorption spectrum of **DAP** (20 μM) in the different pH buffer was recorded using a UV/Vis spectrometer. Next, 3 μL of mercury ion was put into the solutions and mixed well. The absorption spectra of the complex at each pH were measured using a UV/Vis spectrometer.

2.8 *Analysis of Hg^{2+}*

Drinking and tap water samples were selected for analysis of mercury ion in real water sample. 60 μL of **DAP** (1×10^{-3} M) was added into a mixture of 2.64 mL of water sample and 0.30 mL of 0.1 M Bis-Tris buffer. The absorption spectra were measured using a UV/Vis spectrometer.

3. Results and Discussion

We prepared a conjugated Schiff chemosensor, **DAP**, through condensation reaction of 3-nitrobenzohydrazide and 4-(dimethylamino)cinnamaldehyde



Scheme 2. Plausible structure of Hg^{2+}-2•**DAP**.

(Scheme 1). Compound **DAP** was analyzed by ^{1}H and ^{13}C NMR, and ESI-MS. The selective detection and sensing mechanism of mercury ion were studied and explained through UV-vis spectroscopy, FT-IR and ESI-MS.

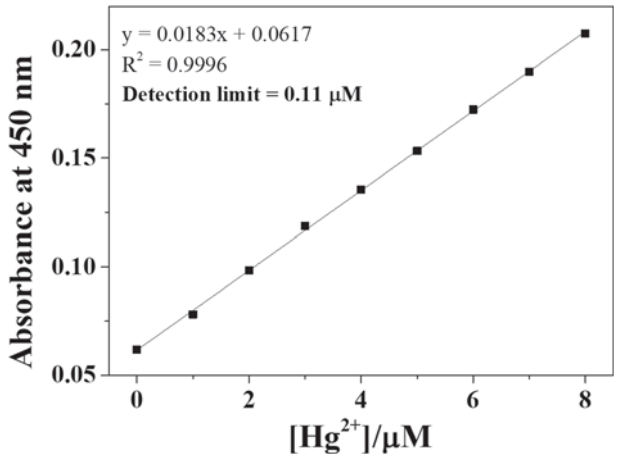


Figure 3. Detection limit of **DAP** (2×10^{-5} M) for Hg^{2+} based on variation of absorbance at 450 nm.

3.1 *Colorimetric sensing of Hg^{2+}*

The ability of **DAP** as an efficient colorimetric sensor was investigated in aqueous solution upon the addition of varied cations (Co^{2+}, Mn^{2+}, Ni^{2+}, Ga^{3+}, Fe^{3+}, Cd^{2+}, K^{+}, Cu^{2+}, Hg^{2+}, Fe^{2+}, Pb^{2+}, Mg^{2+}, Ag^{+}, Na^{+}, Zn^{2+}, Ca^{2+}, Cr^{3+}, Al^{3+} and In^{3+}) (Figure 1). Only mercury ion showed spectral and obvious color changes from pale yellow to orange; otherwise, there was no such change with other cations. These results suggested that we could use **DAP** for colorimetric detection of mercury ion.

To examine the chelating ability of **DAP** for Hg^{2+}, UV-vis titration was performed (Figure 2). Mercury ion solution was gradually put into **DAP** solution, and the additions led to the spectral changes increasing at 400 nm and decreasing at 350 nm. There was an isosbestic point at 372 nm, which means that **DAP** and mercury ion produced only one species. Job plot was depicted to analyze binding mode, and the result indicated that **DAP** bound to Hg^{2+} with a 2:1 mode (Figure S1). To propose the clearer binding form of **DAP** with Hg^{2+}, FT-IR experiments were conducted

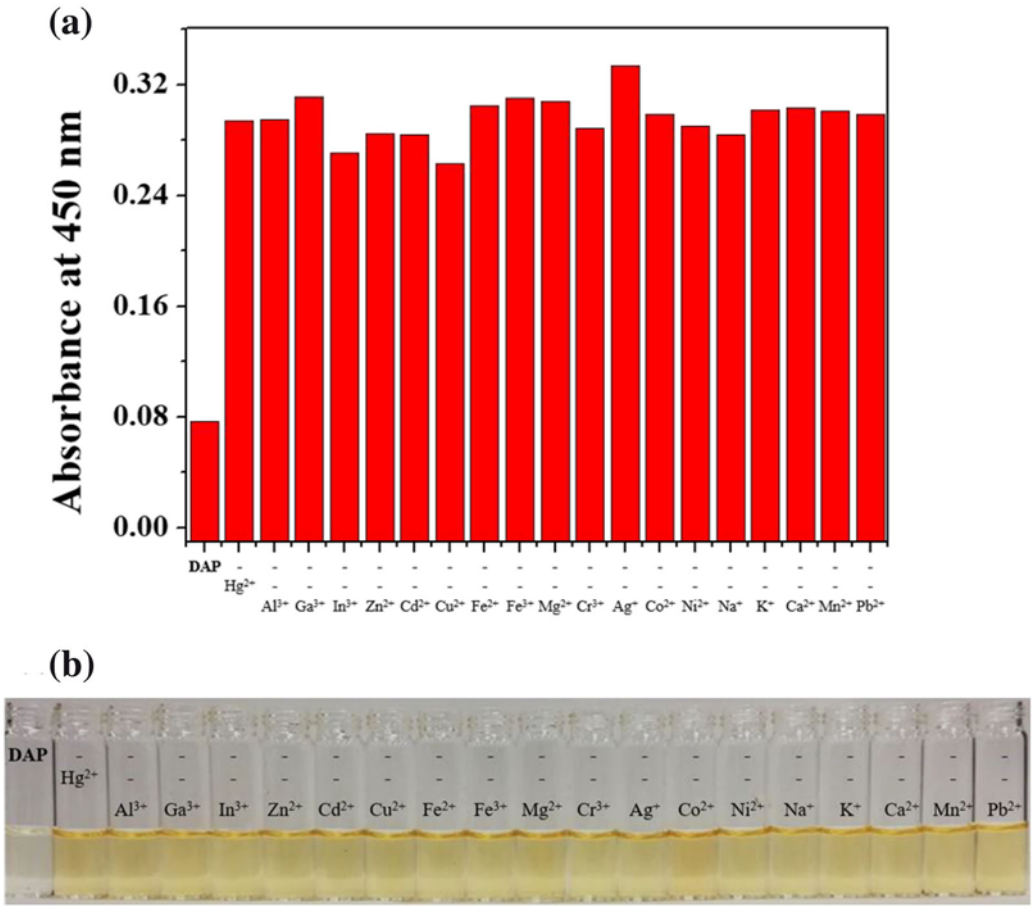


Figure 4. (a) Competitive selectivity of **DAP** (2×10^{-5} M) to Hg^{2+} (0.5 equiv) with varied cations (0.5 equiv). (b) Color changes of **DAP** (2×10^{-5} M) to Hg^{2+} (0.5 equiv) with varied cations (0.5 equiv).

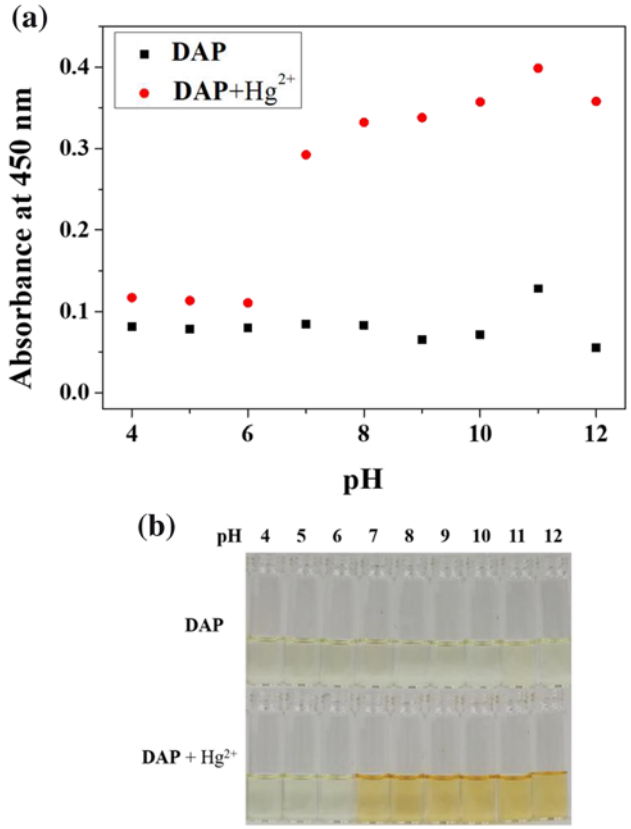


Figure 5. (a) Absorbance (450 nm) of **DAP** and **DAP** with Hg^{2+} in different pH. (b) Color change of **DAP** and **DAP** with Hg^{2+} in different pH.

Table 1. Analysis of Hg^{2+}.[a]

Sample	Hg(II) added (μM)	Hg(II) found (μM)	Recovery (%)	R.S.D. (n=3) (%)
Drinking water	0.00	0.00		
	3.00	3.08	102.7	0.64
Tap water	0.00	0.00		
	3.00	3.21	107.0	2.89

[a]Conditions: [**DAP**] = 20 μM in buffer/DMF (98:2, pH 7.0).

(Figure S2). The stretching vibration band at 3201 cm^{-1}, that can be assigned to amide−NH absorption of **DAP**, shifted to 3062 cm^{-1} in the presence of Hg^{2+}. In addition, the absorptions of the amide C=O and imine C=N groups were moved from 1658 to 1597 and 1601 to 1562 cm^{-1}, respectively. These results indicated that the imine nitrogen atom and amide oxygen atom of sensor **DAP** might coordinate to Hg^{2+}. As depicted in Figure S3, the ESI-MS showed a peak at *m/z* of 877.75, which was well matched with [2·**DAP**-H^++Hg^{2+}] complex [calcd, 877.24]. Combining all the results observed together, we depicted the plausible structure of Hg^{2+}-2•**DAP** (Scheme 2).

Detection limit and association constant (*K*) were calculated based on UV-vis titration. Detection limit of 0.11 μM was obtained, which is lower than the standard (2.48 μM) suggested in Health Canada (Fig. 3).[41] In addition, this value (0.11 μM) is the second lowest among those formerly reported by colorimetric mercury sensors in a near-perfect aqueous media (Table S1).[42–50] Li's equation was used for calculating *K*, and the value was 2.0×10^9 M^{-2} which is within scope of those formerly reported for colorimetric mercury chemosensors (10^3–10^{10}) (Figure S4).

To examine the capability of **DAP** as a mercury ion sensor, we conducted competitive experiment with various metal ions (0.5 equiv) such as Co^{2+}, Mn^{2+}, Ni^{2+}, Ga^{3+}, Fe^{3+}, Cd^{2+}, K^+, Cu^{2+}, Fe^{2+}, Pb^{2+}, Mg^{2+}, Ag^+, Na^+, Zn^{2+}, Ca^{2+}, Cr^{3+}, Al^{3+} and In^{3+} (Figure 4). None of the coexistent cations inhibited the detection of Hg^{2+}. Thus, **DAP** could operate as a competent probe capable of distinguishing Hg^{2+} among various cations.

The ability as a practical mercury sensor was investigated through pH test from pH 4 to 12 (Figure 5). **DAP** did not show a notable color change at different pH, while Hg^{2+}-2•**DAP** exhibited orange color from pH 7 to 12. Thus, **DAP** could be used as a mercury sensor at neutral or basic environments. In addition, we examined the sensing ability of **DAP** toward mercury ion in real water samples. Drinking and tap water samples were chosen for the experiments, and quantification of Hg^{2+} was replicated three times. As shown in Table 1, sensor **DAP** could successfully recognize Hg^{2+} in real water sample. Hence, sensor **DAP** might be used as a practical chemosensor for detecting mercury ion in real environment.

4. Conclusion

A new conjugated Schiff base-based chemosensor **DAP** was synthesized and characterized for the detection of Hg^{2+}. **DAP** showed an obvious color change only with Hg^{2+} in aqueous solution. **DAP** could detect Hg^{2+} without any interruption when other metal ions coexisted. Detection limit of 0.11 μM was obtained, which is lower than the standard suggested in Health Canada. Meaningfully, the value is the second lowest among those formerly reported by using colorimetric mercury sensors in a near-perfect aqueous media. In addition, quantification and recognition of mercury ion by **DAP** was successful in real water samples. Sensing mechanism of Hg^{2+} by **DAP** was demonstrated through FT-IR, ESI-mass and UV-vis titration. Therefore, we expect that **DAP** could be

employed as an excellent mercury chemosensor via a colorimetric method.

Supplementary Information (SI)

Supplementary data (Table S1 and Figures S1–S4) associated with this article can be found. Supplementary Information is available at www.ias.ac.in/chemsci.

Acknowledgements

NRF (National Research Foundation of Korea) (2018R1A2B6001686) are thankfully acknowledged.

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