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Antipyrine based Schiff bases as Turn-on Fluorescent sensors for Al (III) ion

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ABSTRACT

Two new fluorescent sensors C1 and C2 with 4-aminoantipyrine unit have been prepared and characterized. Their complexation behaviour and binding mode towards Al^{3+} and other metal ions have been studied by UV–vis, fluorescence spectrometric and HRMS methods. The ^1H NMR titrations were carried out to explore the nature of interaction between receptor and aluminum ion. These sensors are successfully applied in highly acidic and neutral pH medium with the fastest response time (<5 sec). The fluorescence color change could be easily detected by the naked eye under a UV lamp.

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1. Introduction

Aluminum is the most abundant element in the earth's crust after oxygen and silicon and is the second most widely used metal after iron for the manufacture of electrical equipments, automobiles, packaging materials, water purification, clinical drug and building construction etc. [1,2]. Recent studies are shown that the deposition of aluminum in bone and the nervous system in human body can cause neurotoxicity in high dosage [3]. It plays an important role in the pathology of Parkinson's, Alzheimer's and dialysis diseases [4–7]. In plants, higher concentration of aluminum affects the growth of root and seed [8,9]. Thus, the monitoring of aluminum is essential in environment, medicine, foodstuff, etc.

In recent years, many analytical methods have played a role in the detection of Al^{3+} ion and others, including ion selective electrodes [10–22], voltammetric [23–33] and colorimetric sensors [34–38]. Recently, the fluorescent method has become popular due to its operational simplicity, high selectivity and sensitivity, real-time response and naked eye detection [39–48]. Due to its poor coordination ability compared to transition metals [49], only a few fluorescent sensors have been reported and on the other hand, most of the Al^{3+} sensors are difficult to synthesis and insoluble in aqueous solvents. Some of the antipyrine derivatives are detected as a fluorescent chemosensor towards ions [50].

In this paper, we designed and synthesized 4-aminoantipyrine based compounds (C1 and C2) in a simple approach as fluorescent sensors for Al^{3+} ion. The fluorescence intensities of the two sensors C1 and C2 are enhanced by mixing Al^{3+}, and show bright blue and bright green color respectively, which can be easily detected by the naked eye under UV lamp. The results indicated that the prepared 4-aminoantipyrine based sensors can show high sensitivity and selectivity towards Al^{3+} over other metal ions.

2. Experimental

2.1. Reagents and apparatus

4-aminoantipyrine, salicylaldehyde, 2-hydroxy-1-naphthaldehyde and metal salts (from Merck and Aldrich, India). The IR spectra were recorded on a Nexus FT-IR (Illinois, USA) spectrometer in the range 4000–400 cm^{-1} with KBr. The NMR spectra were recorded on a Bruker 500 MHz (USA), TMS as an internal standard, $CDCl_3$ and CD_3OD are taken as solvents. The mass spectra were recorded using Bruker-micrOTOF II (USA). The UV–vis absorption spectra were measured on a Shimadzu UV-2450 spectrophotometer (Japan) and the Fluorescent spectra were recorded on a Shimadzu RF-5301PC spectrofluorophotometer (Japan) instruments.

2.2. Synthesis and characterisation

The Schiff base ligands (C1 and C2) were prepared by the reported method [51–64] with some modifications (Scheme 1). The

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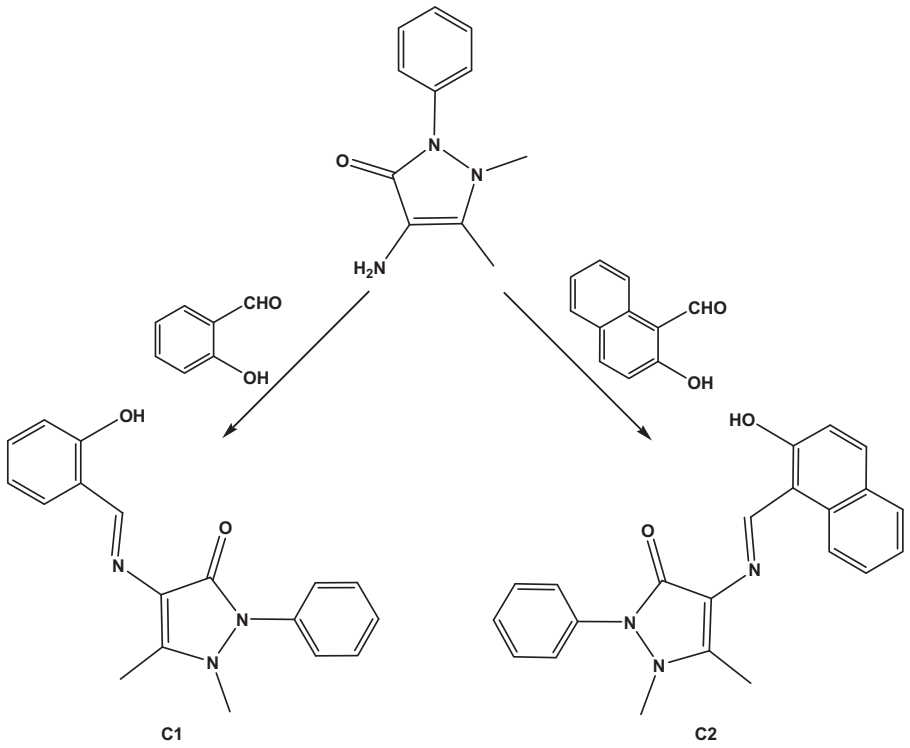

Scheme 1. Synthetic routes to C1 and C2.

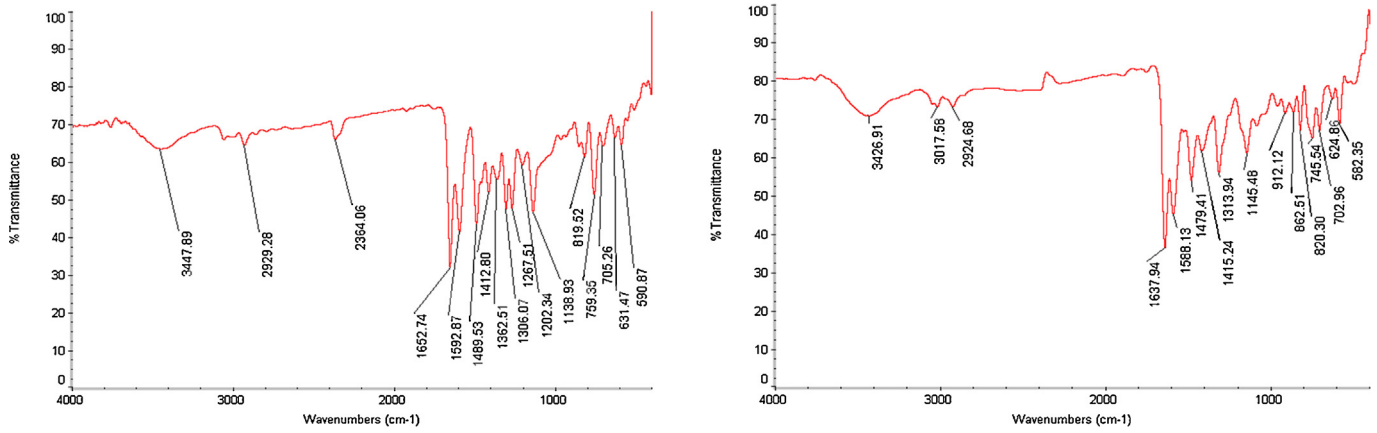

Fig. 1. FT-IR Spectrum of C1 and C2.

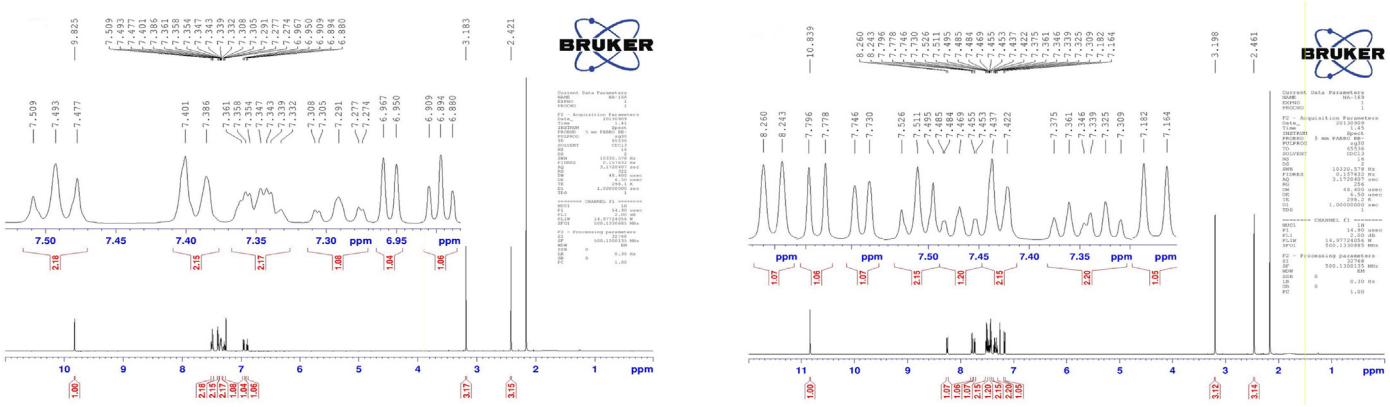

Fig. 2. ^{1}H NMR Spectrum of C1 and C2.

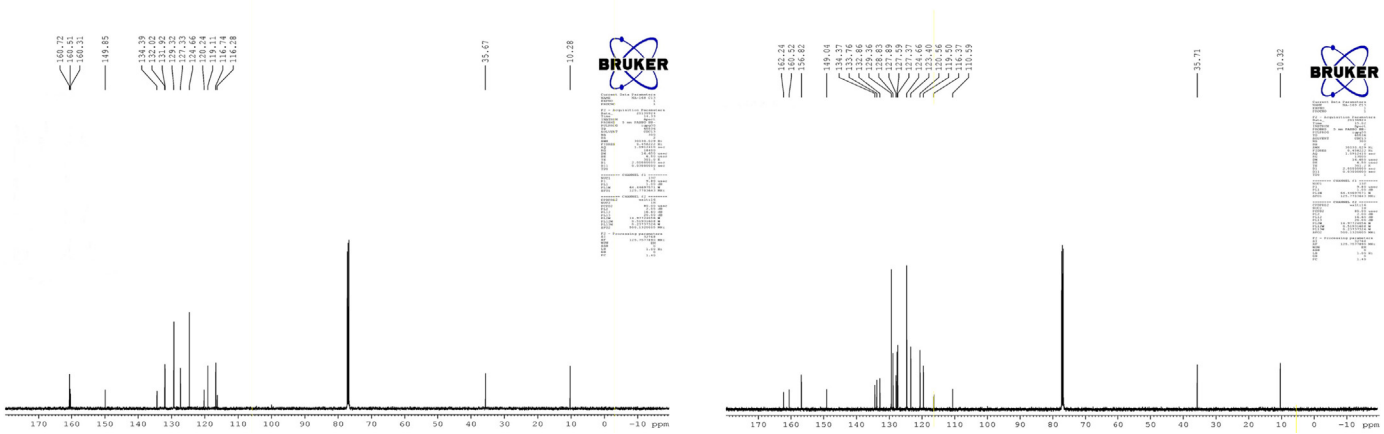

Fig. 3. ^{13}C NMR Spectrum of C1 and C2.

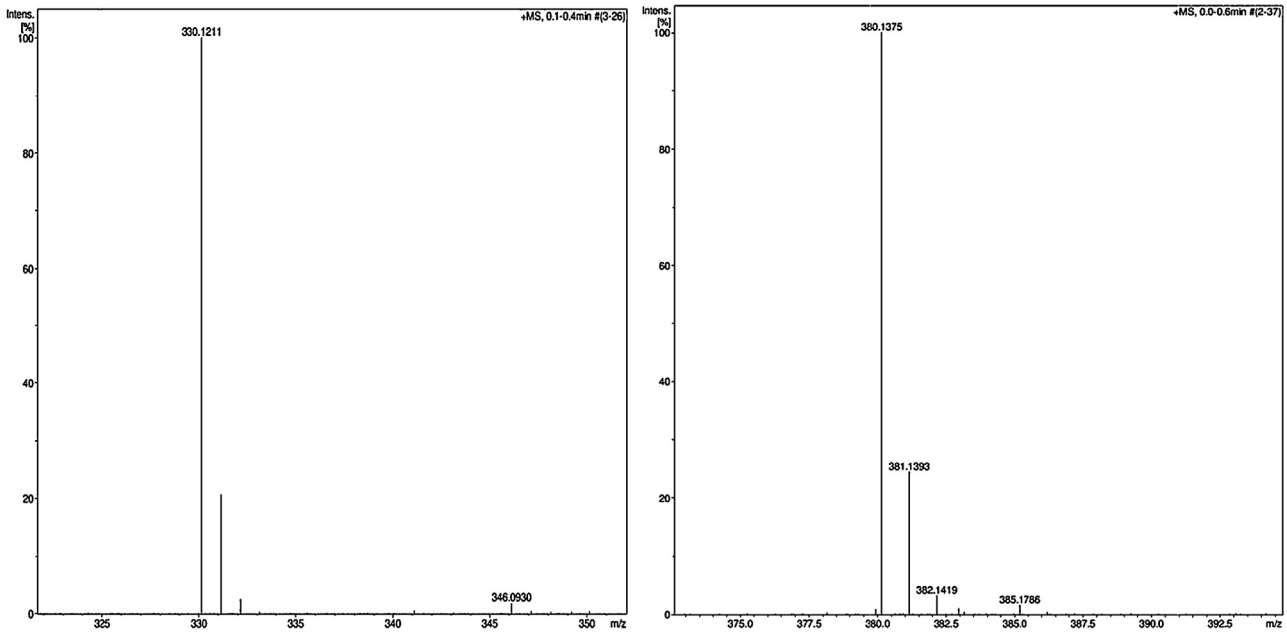

Fig. 4. HRMS Spectrum of C1 and C2.

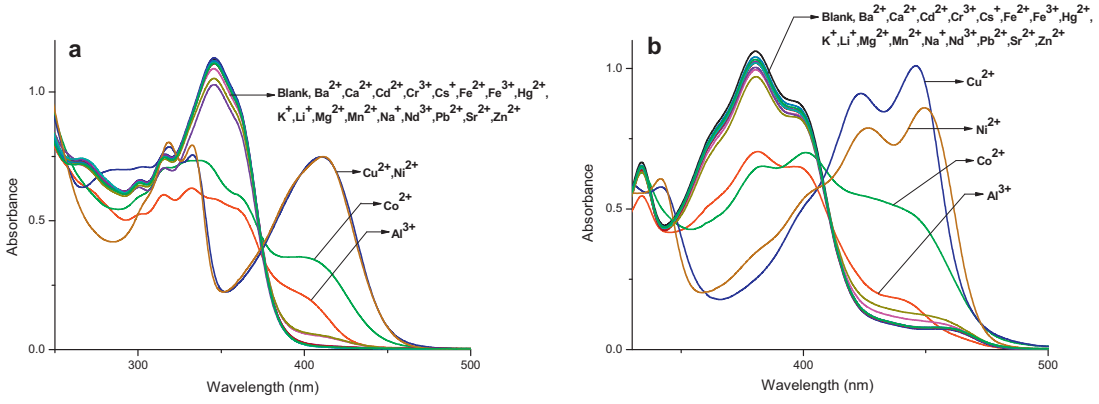

Fig. 5. UV–vis absorbance spectra of C1 (50 μM) (a) and C2 (50 μM) (b) in the presence of different metal ions (Al^{3+}, Ba^{2+}, Ca^{2+}, Cd^{2+}, Co^{2+}, Cr^{3+}, Cs^{+}, Cu^{2+}, Fe^{2+}, Fe^{3+}, Hg^{2+}, K^{+}, Li^{+}, Mg^{2+}, Mn^{2+}, Na^{+}, Nd^{3+}, Ni^{2+}, Pb^{2+}, Sr^{2+}, Zn^{2+}) (50 μM) in methanol solvent.

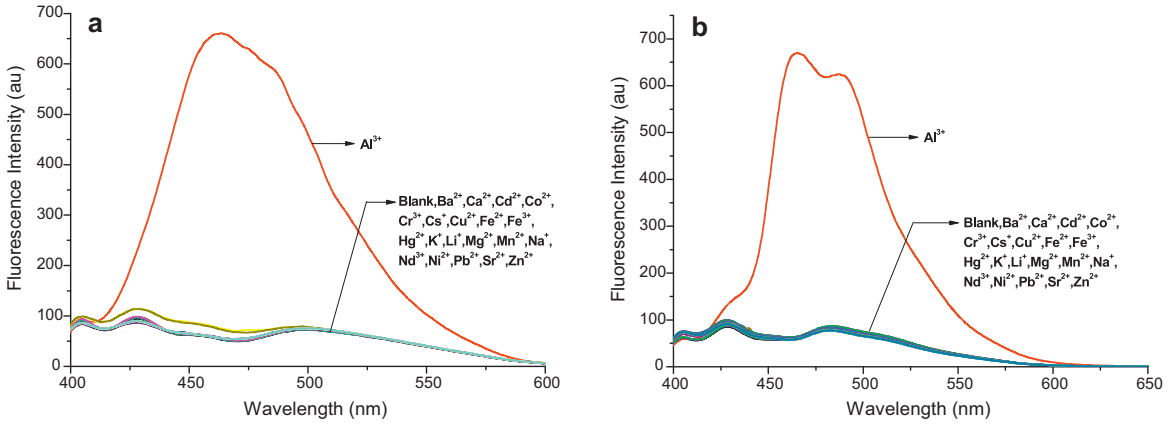

Fig. 6. Fluorescence emission spectra of C1 (20 μM) (a) and C2 (20 μM) (b) in the presence of different metal ions (Al^{3+}, Ba^{2+}, Ca^{2+}, Cd^{2+}, Co^{2+}, Cr^{3+}, Cs^{+}, Cu^{2+}, Fe^{2+}, Fe^{3+}, Hg^{2+}, K^{+}, Li^{+}, Mg^{2+}, Mn^{2+}, Na^{+}, Nd^{3+}, Ni^{2+}, Pb^{2+}, Sr^{2+}, Zn^{2+}) (20 μM) in methanol solvent.

ethanol containing the mixture of 4-aminoantipyrine (10 mmol) and aldehyde (10 mmol) was stirred at room temperature about 2–3 hours, the obtained solid was filtered, washed with cold ethanol, dried under vacuum and characterised by FT-IR, NMR and HRMS (Figs. 1–4).

1-phenyl-2,3-dimethyl-4-(N-2-hydroxybenzylidene)-3-pyrazolin-5-one (C1): Yield: 2.76 g (90%); color: light yellow solid; m.p. 218-220 °C; FT-IR (KBr), ν, cm^{-1}: 3448 (O – H), 2929, 759 (C – H), 1653 (C = O), 1593 (C = N), 1490 (C = C), 1139 (N – N); ^{1}H NMR ($CDCl_3$), δ, ppm (*J*, Hz): 2.42 (s, 3H), 3.18 (s, 3H), 6.89 (t, *J* = 7.5, 1H), 6.96 (d, *J* = 8.5, 1H), 7.29 (dt, *J* = 1.5, 7.5, 1H), 7.33 – 7.37 (m, 2H), 7.39 (d, *J* = 2.5, 2H), 7.49 (t, *J* = 8.0, 2H), 9.82 (s, 1H); ^{13}C NMR ($CDCl_3$), δ, ppm: 10.3, 35.7, 116.3, 116.7, 119.1, 120.2, 124.7, 127.3, 129.3, 131.9, 132.0, 134.4, 149.9, 160.3, 160.5, 160.7; HRMS calcd for $C_{18}H_{17}N_3O_2$ $(M + Na)^+$: 330.1218, found: 330.1211.

1-phenyl-2,3-dimethyl-4-(N-2-hydroxynaphthylidene)-3-pyrazolin-5-one (C2): Yield: 3.11 g (80%); color: yellow solid; m.p. 210-212 °C; FT-IR (KBr), ν, cm^{-1}: 3427 (O – H), 3018, 2925, 746 (C – H), 1638 (C = O), 1588 (C = N), 1479 (C = C), 1145 (N – N); ^{1}H NMR ($CHCl_3$), δ, ppm (*J*, Hz): 2.46 (s, 3H), 3.20 (s, 3H), 7.17 (d, *J* = 9.0, 1H), 7.34 (m, 2H), 7.43 (d, *J* = 7.5, 2H), 7.47 (dt, *J* = 1.0, 7.5, 1H), 7.51 (t, *J* = 8.0, 2H), 7.74 (d, *J* = 8.0, 1H), 7.79 (d, *J* = 9.0, 1H), 8.25 (d, *J* = 8.5, 1H), 10.84 (s, 1H); ^{13}C NMR ($CDCl_3$), δ, ppm: 10.3, 35.7, 110.6, 116.4, 119.5, 120.6, 123.4, 124.7, 127.4, 127.6, 127.9, 128.8, 129.4, 132.9, 133.8, 134.4, 149.0, 156.8, 160.5, 162.2; HRMS calcd for $C_{22}H_{19}N_3O_2$ $(M + Na)^+$: 380.1375, found: 380.1375.

2.3. UV–vis and Fluorescent measurements

UV–vis absorption and fluorescence emission spectra of sensors measured in 1.0 cm path length quartz cuvettes were measured using a Shimadzu UV-2450 spectrophotometer and a Shimadzu RF-5301PC spectrofluorophotometer. Absorption and emission spectra of the sensor (C1 and C2) in the presence of various metal ions (Al^{3+}, Ba^{2+}, Ca^{2+}, Cd^{2+}, Co^{2+}, Cr^{3+}, Cs^{+}, Cu^{2+}, Fe^{2+}, Fe^{3+}, Hg^{2+}, K^{+}, Li^{+}, Mg^{2+},Mn^{2+}, Na^{+}, Nd^{3+}, Ni^{2+}, Pb^{2+},Sr^{2+} and Zn^{2+}) were measured in methanol solvent in the concentration of 50 μM and 20 μM, respectively.

3. Results and discussion

The binding ability and mode of sensors (C1 and C2) towards Al^{3+} and other metal ions was measured through UV–vis, fluorescent spectrometry, naked-eye observation, HRMS and ^{1}H NMR experiments.

3.1. UV–vis spectral studies

The chemosensors (C1 and C2) were investigated by UV–vis absorption spectral behaviour in the presence of various metals in the 50 μM concentration of each component in methanol solvent. The free ligands C1 and C2 exhibited a main absorption band at about 345 nm and 380 nm, respectively. On the addition of metal ions to sensors, a new broad absorption band (mainly for Cu^{2+}, Ni^{2+}, Co^{2+} and Al^{3+} ions) was observed at 350–480 nm region (Fig. 5). At the same time the absorption band at 300–390 nm and 340–430 nm of receptors C1 and C2 respectively, have also been shifted to low intensity. It means these two sensors are responding to the above mentioned metal ions, there is no significant changes were observed when mixed with other metal ions such as Ba^{2+}, Ca^{2+}, Cd^{2+}, Cr^{3+}, Cs^{+}, Fe^{2+}, Fe^{3+}, Hg^{2+}, K^{+}, Li^{+}, Mg^{2+}, Mn^{2+}, Na^{+}, Nd^{3+}, Pb^{2+}, Sr^{2+} and Zn^{2+}.

3.2. Fluorescence emission studies

The fluorescence response of sensors C1 and C2 (20 μM) upon addition of various metal ions (20 μM) have been investigated in methanol. Receptor C1 and C2 alone displayed a very weak single fluorescence emission band at 498 nm and 484 nm respectively, with an excitation of 360 nm. Upon addition of various metals (Ba^{2+}, Ca^{2+}, Cd^{2+},Co^{2+}, Cr^{3+}, Cs^{+}, Cu^{2+}, Fe^{2+}, Fe^{3+}, Hg^{2+}, K^{+}, Li^{+}, Mg^{2+}, Mn^{2+},

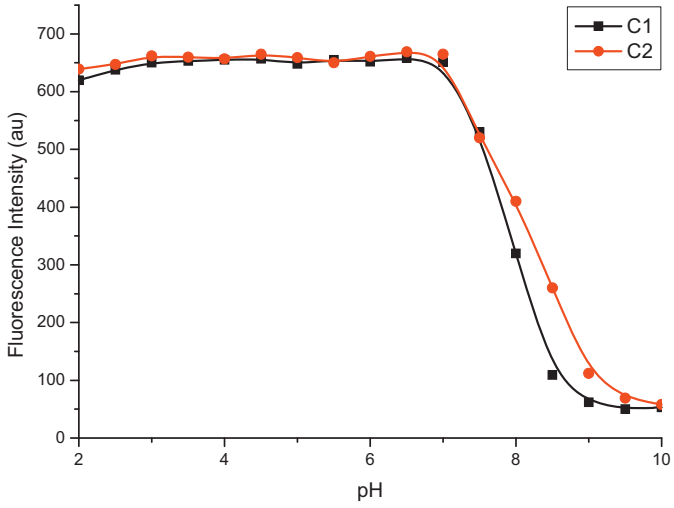

Fig. 7. The variation in fluorescence intensity with the pH of receptor (C1 and C2) (20 μM) in the presence of Al^{3+} (1.0 eq.) at 466 nm.

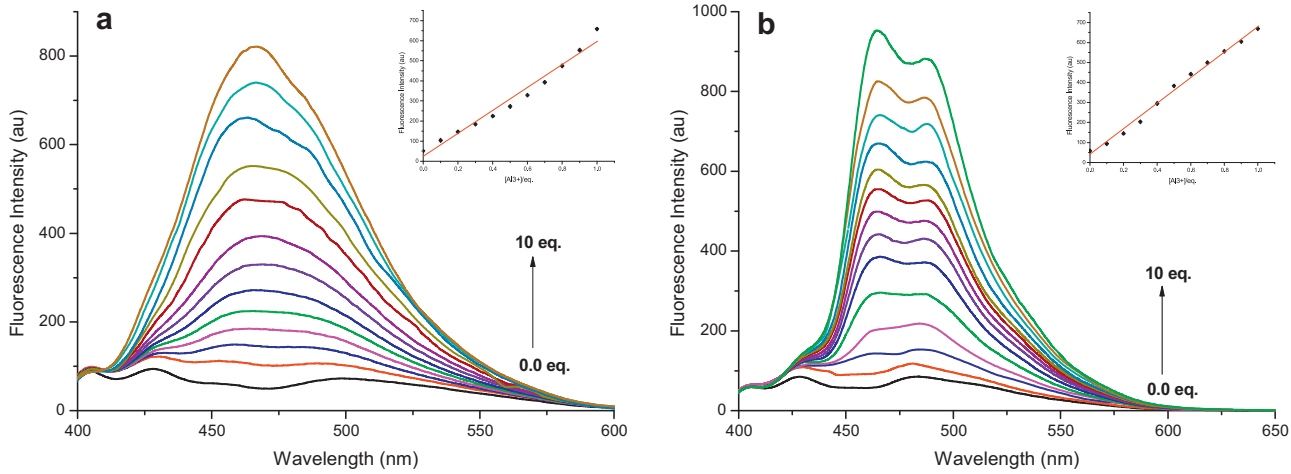

Fig. 8. Fluorescence emission spectra of (a) C1 (20 μM) and (b) C2 (20 μM) in methanol upon the addition of Al^{3+} ion (0.0, 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, 1.0, 2.0, 5.0 and 10.0 eq.) with an excitation of 360 nm. Inset shows the fluorescence change at 466 nm as a function of the amount of Al^{3+} ions.

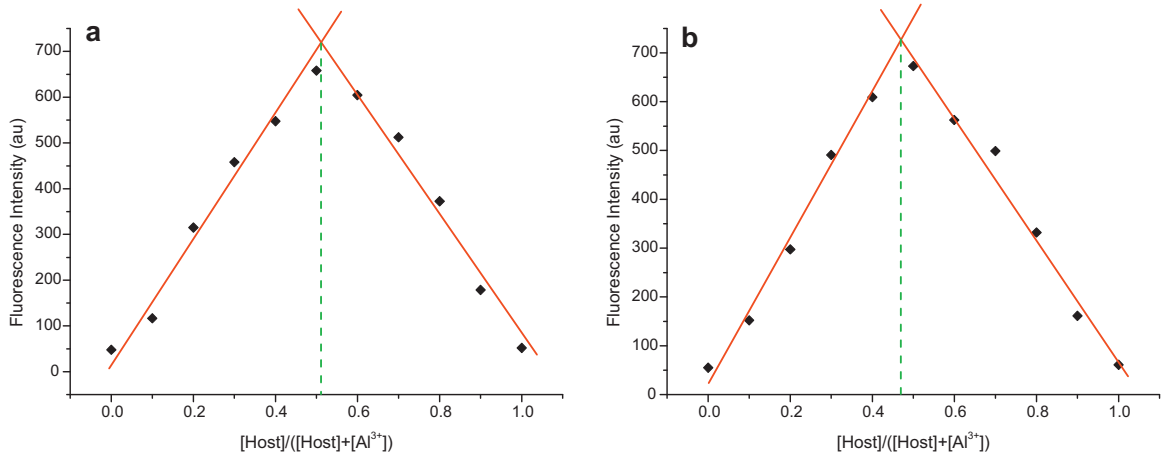

Fig. 9. Job's plot for C1 (a) and C2 (b) by fluorescence method. Total concentration of receptor and metal is 20 μM.

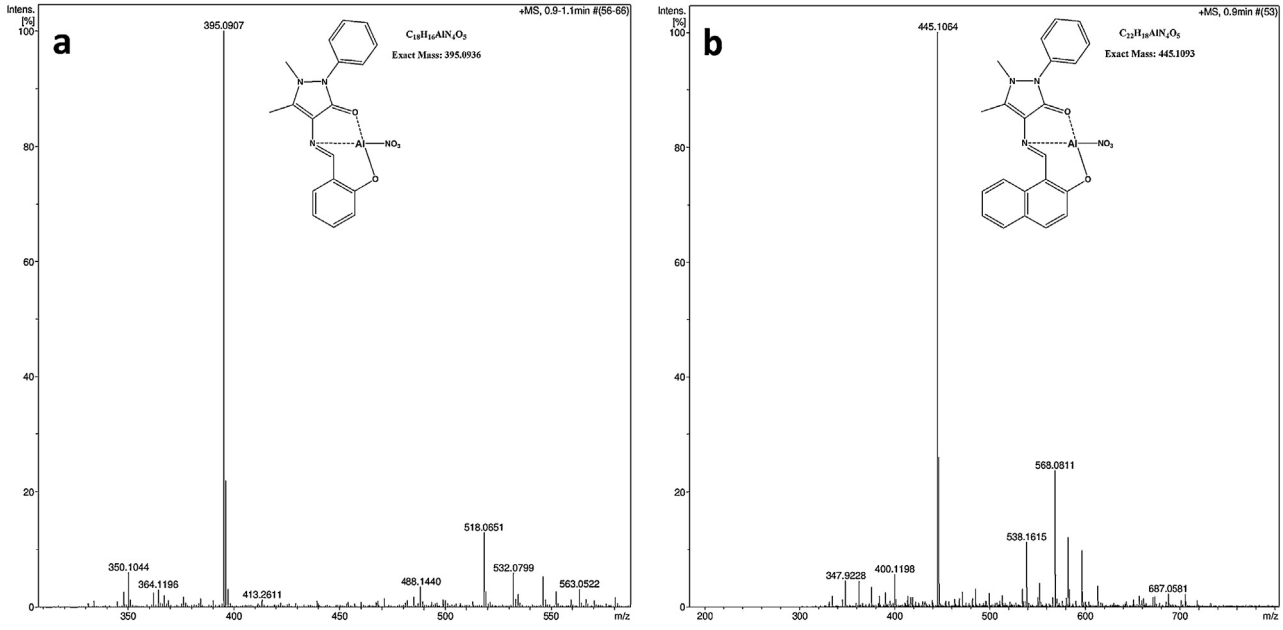

Fig. 10. HRMS spectrum of (a) C1 and (b) C2 upon addition of $Al(N0_3)_3.9H_2O$ (1 eq.) in MeOH.

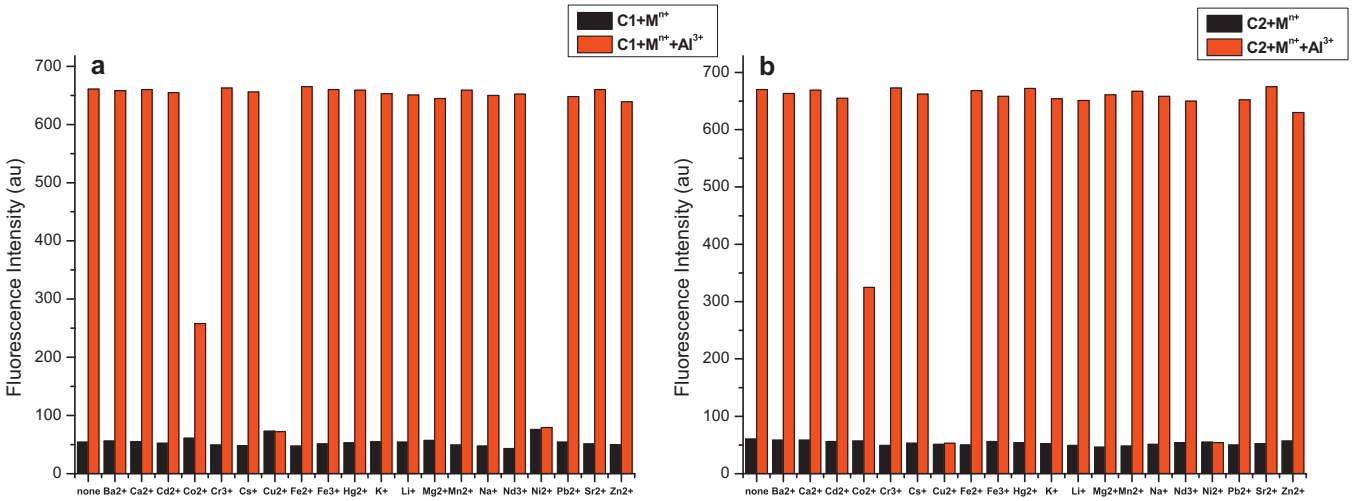

Fig. 11. Selectivity of the C1 (a) and C2 (b) toward Al^{3+} and other metal ions. In these experiments, the fluorescence measurement was taken for 20 μM concentration of C1 (a) and C2 (b) at λ_{ex} = 360 nm in methanol at room temperature with various metal ions (1.0 eq.) and in the absence (black bars) and presence (red bars) of 1.0 eq. Al^{3+} ion.

Na^+, Nd^{3+}, Ni^{2+}, Pb^{2+}, Sr^{2+} and Zn^{2+}) no significant changes were observed (Fig. 6). But on addition of Al^{3+}, receptors C1 and C2 exhibited a prominent fluorescence enhancement accompanied by a blue shift of 32 nm from 498 to 466 nm and 18 nm from 484 to 466 nm, respectively, indicating, that the receptors C1 and C2 exhibit "off-on" mode with high sensitivity towards Al^{3+} over other metal ions which are used. Using a UV lamp, the receptors C1 and C2 in the presence of Al^{3+} showed a dramatic colour changes from colourless to bright blue and bright green respectively, within 5 seconds, which could easily be detected by the naked-eye. At the same time, the addition of other metal ions did not show any significant colour change.

The interaction between receptor (C1 and C2) and Al^{3+} ion was investigated at a pH range from 2.0 to 10.0. This experiment was carried out at a fixed concentration of receptor-Al^{3+} is 20 μM in methanol. As shown in Fig. 7, the fluorescence intensity at 466 nm almost did not change with the pH value at acidic and neutral conditions (pH <7.0). However, the fluorescence intensity decreased gradually from pH 7.0–10.0, due to the formation of salt. For further studies, the pH of solvent kept constant at 6.5.

Furthermore, the fluorescence response of receptors (C1 and C2) to various concentrations of Al^{3+} (0–10 eq.) was investigated. Upon addition of Al^{3+}, the fluorescence intensity centred at 466 nm of receptor gradually increased and remained steady when 1 eq. Al^{3+} was added, indicating the formation of a 1:1 bonding mode between chemosensor and Al^{3+} (Fig. 8). In addition, a Job plot obtained from the emission data showed the 1:1 stoichiometric complexation (Fig. 9). Also, the high resolution mass spectrum of receptors upon addition of 1 eq. of Al^{3+} indicated the formation of the receptor-Al^{3+} complex with 1:1 ratio (Fig. 10).

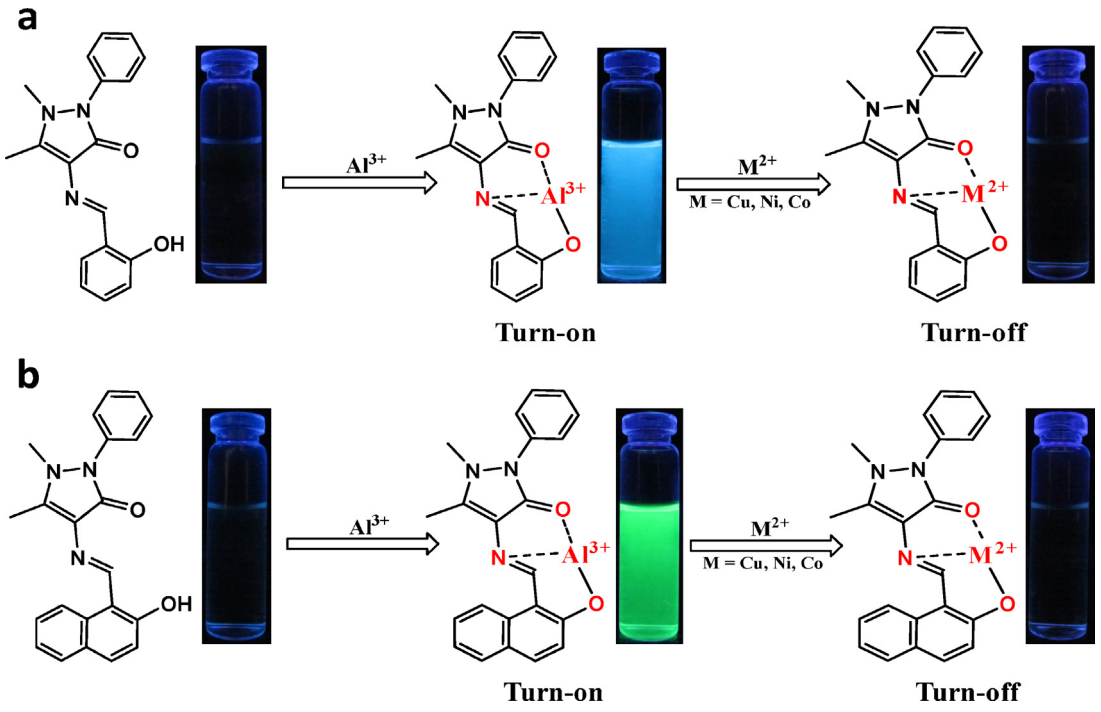

Fig. 12. The fluorescence emission changes of sensors C1 (a) and C2 (b) (20 μM) with 1.0 eq. of Al^{3+}, and in the presence of other metal ions (Cu^{2+}, Ni^{2+}, Co^{2+}) excited by a commercially available UV lamp (λ_{ex} = 365 nm).

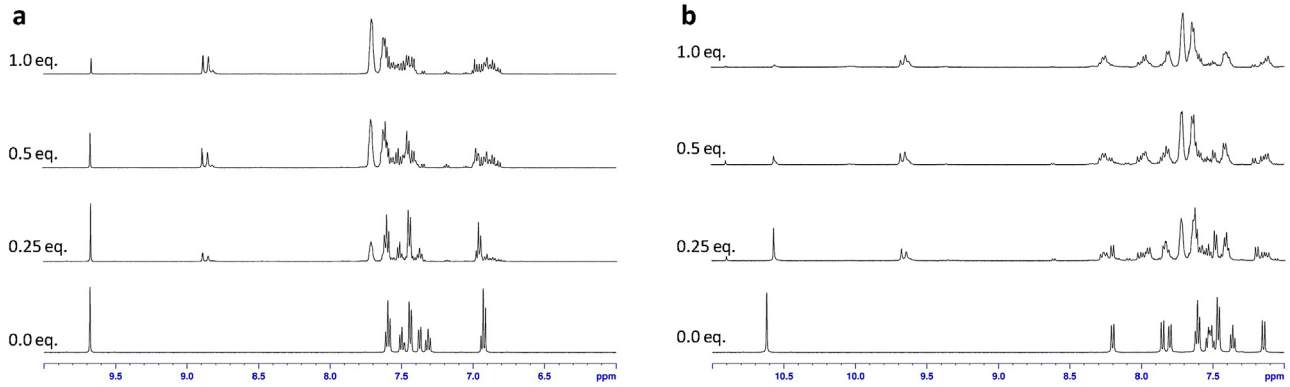

Fig. 13. ^{1}H NMR (500 MHz) spectra of receptor (a) C1 and (b) C2 with Al^{3+} (0.0–1.0 eq.) in CD_3OD.

The selectivity of C1 and C2 (20 μM) for Al^{3+} over other metal ions (1.0 eq.), was investigated by the competition experiments. As shown in Fig. 11, the fluorescence response of C1 and C2 toward Al^{3+} in the presence of various metal ions was investigated, and the results indicated that Cu^{2+}, Ni^{2+} and Co^{2+} could interfere in the interaction between receptor (C1 and C2) and Al^{3+}. Indicating that the binding abilities of Cu^{2+}, $Ni^{2+}$and Co^{2+} are stronger than that of Al^{3+} toward Schiff bases. Upon addition of 1.0 eq. of Al^{3+} in the presence of other metal ions (Ba^{2+}, Ca^{2+}, Cd^{2+}, Cr^{3+}, Cs^{+}, Fe^{2+}, Fe^{3+}, Hg^{2+}, K^{+}, Li^{+}, Mg^{2+}, Mn^{2+}, Na^{+}, Nd^{3+}, Pb^{2+}, Sr^{2+} and Zn^{2+}), 11-fold enhancement of the fluorescence intensity was observed, which is large enough to determine Al^{3+} from other metal ions. We could also directly observe the fluorescence changes of receptors (C1 and C2) before and after addition of Al^{3+}, with and without other sensed metal ions under UV lamp. As depicted in Fig. 12, by addition of other metal ions (Cu^{2+}, Ni^{2+} and Co^{2+}) quenches the fluorescence of the receptor-Al^{3+} complex, due to the more binding ability towards Cu^{2+}, Ni^{2+} and Co^{2+} ions. It means, the reported sensors provide simultaneous detection of Al^{3+} and other metal ions (Cu^{2+}, Ni^{2+}, Co^{2+}).

3.3. ^{1}H NMR titration

The binding mode of receptor (C1 and C2) towards Al^{3+} was confirmed by the ^{1}H NMR titrations using CD_3OD as a solvent. As depicted in Fig. 13, the proton of imine moiety at about 9.68 ppm (of C1) and 10.62 ppm (of C2) was shifted up field to 8.87 ppm and 9.67 ppm, respectively, followed the addition of Al^{3+}, due to interrupt the intramolecular hydrogen bonding between the phenolic hydroxyl group and the nitrogen of the imine moiety by the addition of Al^{3+}. The imine proton signal at δ 10.62 almost completely disappeared, when 1.0 eq. of Al^{3+} was added to receptor C2, indicating that the receptor interacts with Al^{3+} and form stable complex with 1:1 stoichiometry. On the other hand, the protons of phenylene were shifted downfield with the addition of Al^{3+}, which indicated that the structure of receptors became more rigid after coordination with Al^{3+}. It indicated that the phenolic hydroxyl group and nitrogen atom of the imine moiety participated in complexing with Al^{3+}.

4. Conclusion

The proposed sensors (C1 and C2) exhibit good selectivity and sensitivity toward Al^{3+} ion over other tested metal ions. Moreover, the sensory system in methanol as well as in water shows bright blue (for C1) and bright green (C2) colour with Al^{3+} under a UV lamp, which can be easily identified by the naked eye. Thus, the reported sensors have the ability to serve as a practical sensor for detection of Al^{3+} ion in both environment and biological samples.

Acknowledgements

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