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Selective Optical Chemosensors of Fe³⁺ Ions using 1*H*-Indole-2,3-dione

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Abstract: 1*H*-Indole-2,3-dione has attracted many attentions in the field of organic synthesis due to its easy modification with other organic compounds. On the other hand, 1*H*-indole-2,3-dione compound could also be used as chemosensor agent because it contains amide and carbonyl functional groups, which are able for binding as well as chelating metal ions. In the present work, we reported that 1*H*-indole-2,3-dione compound exhibits high sensing capability as well as high selective detection of Fe³⁺ ions through enhancement of its absorption peak at ultraviolet region. Particularly, a series of 0.5 mM of metal ions (Na⁺, K⁺, Ba²⁺, Ca²⁺, Co²⁺, Cu²⁺, Hg²⁺, Mg²⁺, Mn²⁺, Ni²⁺, Pb²⁺, Zn²⁺, Au³⁺, Al³⁺, Fe³⁺ and La³⁺) in distilled water was simply mixed with 0.05 mM of 1*H*-indole-2,3-dione in distilled water. Afterward, their UV-Vis spectral changes were recorded by using UV-Vis spectrophotometer. It was found that 1*H*-indole-2,3-dione itself gave two main absorption peaks at 242 and 302 nm. When the Fe³⁺ ions were added, the intensity of both absorption peaks was remarkably increased. From the UV-Vis titration experiment, limit of detection value was found to be 17.8 μM while limit of quantitation was found as 59.3 μM. This finding shows that the 1*H*-indole-2,3-dione gives a promising platform for fluorescent chemosensor agent for sensing of Fe³⁺ ion.

INTRODUCTION

Host-guest chemistry remains as an active research trends over the past several years [1-4]. Among them, nitrogen heterocyclic compounds have been extensively employed for their unique physicochemical properties [5]. Isatin or 1*H*-indole-2,3-dione was first obtained from the oxidation of indigo dye by acid oxidants such as nitric and chromic acids. The chemical structure of 1*H*-indole-2,3-dione is displayed in Figure 1. The 1*H*-indole-2,3-dione has been applied in many fields such as anticancer [6], antibacterial [7], corrosion [8], and chemosensor agent [9,10]. Furthermore, 1*H*-indole-2,3-dione has been utilized as raw material for organic synthesis because the phenyl ring, carbonyl and amide groups can be modified to form Schiff base, *N*-Mannich base, and other complex compounds [11-13].

Chemosensor from 1*H*-indole-2,3-dione derivatives as ligands, nanomaterials and nanocomposites were often reported due to their selectivity and sensitivity [2, 15]. Besides that, as nanomaterial and nanocomposite, 1*H*-indole-2,3-dione derivatives have advantages such as high thermal stability, large pore size and high surface area [16,17].

Several researchers have used 1*H*-indole-2,3-dione derivatives for sensing Al^{3+} [16], Hg^{2+} [17], Fe^{3+} [18] and Cd^{2+} [19] ions. Dhara *et al.* in 2014 synthesized Schiff base derivative from 1*H*-indole-2,3-dione rhodamine scaffold and tested as a chemosensor to detect Al^{3+} ions [16]. This chemosensor showed high sensitivity in DMSO/ H_2O (1:9 v/v) solvent. However several synthesis routes are necessary to synthesize 1*H*-indole-2,3-dione rhodamine scaffold. Lashgari *et al.* (2017) have reported that the grafting of commercial 1*H*-indole-2,3-dione with SBA-15 with (3-chloropropyl)trimethoxysilane as a linker and measured their emission with spectrofluorometer [17]. These nanocomposites were dispersed in water and used for sensing Hg^{2+} ions. It is well-known that the homogeneity from nanocomposite chemosensor is the main drawback for reproducible sensing method. Hence, it is necessary to develop a sensing method which employs a homogeneous system. Moreover, to the best of our knowledge, commercially available 1*H*-indole-2,3-dione as a chemosensor has not been evaluated previously for sensing metal ions. Whereas, 1*H*-indole-2,3-dione is promising to be applied as a chemosensor due to the presence of carbonyl and amide groups as well-known metal ion binding sites [20,21]. Therefore, in the present work, we reported the investigation of sensing Fe^{3+} ions in aqueous media using 1*H*-indole-2,3-dione as a chemosensor by evaluation of its selectivity and sensitivity.

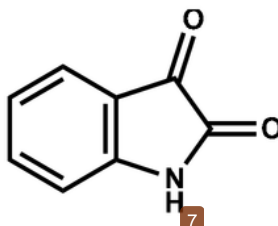


FIGURE 1. The structure of 1*H*-indole-2,3-dione

EXPERIMENTAL SECTION

General

1*H*-indole-2,3-dione (97%) and all other metals in chloride/nitrate salts were purchased from Sigma-Aldrich and were used as they are. The absorbance spectra were measured using spectrophotometer JASCO V-760 while the fluorescence spectra were measured using spectrofluorometer JASCO FP-8500ST. All measurements were performed using a quartz cuvette as a sample holder with 1 cm width.

Procedure

Optical Properties Study of 1H-Indole-2,3-dione

1*H*-indole-2,3-dione was dissolved in distilled water to obtain 0.1 mM concentration. After that, UV-Vis spectrum of 1*H*-indole-2,3-dione as the ligand were measured in the range of 200-800 nm. Fluorescent spectrum of this compound was particularly recorded using 3D measurement for each excitation-emission to the intensity in the range of 200-800 nm for every 10 nm.

The Spectroscopy Study of 1H-Indole-2,3-dione

Metal ions in chloride and nitrate salts were dissolved in distilled water for obtaining each metal ions stock solutions in 1 mM concentration. The spectroscopy study of 1*H*-indole-2,3-dione was carried out by contacting 1 mM of 1*H*-indole-2,3-dione (0.15 mL) with various of metal ion solution (0.15 mL) and distilled water to obtain the final volume of 3 mL. All absorbance spectra were recorded by using spectrophotometer UV-Vis.

Absorbance Spectral Titrations Study of 1H-Indole-2,3-dione

The UV-Vis titrations were conducted to calculate the limit of detection (LOD) and also limit of quantification (LOQ). The stock solution of 0.05 mM of the 1*H*-indole-2,3-dione was prepared by dissolving in distilled water.

Similarly, a 100 mM stock solution of Fe^{3+} was prepared separately in distilled water. The Fe^{3+} ions concentration was varied starting from 0.01, 0.025, 0.05, 0.075, 0.1, 0.25, 0.5 to 0.75 and 15 mM. Both solutions were mixed to obtain a solution with 3 mL as the total volume of the mixture, and then the changes in their UV-Vis spectra were recorded.

The Selectivity Study of *1H*-Indole-2,3-dione

The selectivity study of *1H*-indole-2,3-dione was performed to evaluate the capability of *1H*-indole-2,3-dione to detect Fe^{3+} ions in the presence of other metal ions. The 0.5 mM of Fe^{3+} ions solution, 0.5 mM of other metal ions and 0.5 mM *1H*-indole-2,3-dione solution in distilled water were mixed for this evaluation. The absorbance spectrum of the mixture was measured by UV-Vis spectrophotometer.

RESULTS AND DISCUSSION

Optical Properties Study

The photophysical properties of *1H*-indole-2,3-dione were also systematically examined by using spectrofluorometer and spectrophotometer UV-Vis. The UV/visible absorption spectrum and 3D fluorescent spectrum were shown in Figure 2a-b. The UV-Vis spectrum in Figure 2a shows four absorption peaks at 208, 242, 302, and 418 nm. Absorption maxima at 208, 242, and 302 nm correspond to the $\pi \rightarrow \pi^*$ transition of the aromatic ring, $\pi \rightarrow \pi^*$ carbonyl C-2, and $\pi \rightarrow \pi^*$ carbonyl C-3, respectively. A weak absorption band at 418 nm correspond to the nitrogen and oxygen lone pair electrons ($n \rightarrow \pi^*$ transition) [22]. The 3D fluorescent spectrum of *1H*-indole-2,3-dione in Figure 2b shows the blank area in the region 200-800 nm for the emission and excitation wavelength. This result indicates that *1H*-indole-2,3-dione in distilled water does not have fluorescence phenomena due to the formation of aggregation (Aggregation-caused Quenching) [23]. The aggregation caused by overlaps in two π -conjugated systems (π - π stacking) between benzene ring from two *1H*-indole-2,3-dione molecules. From the aforementioned above, in this work, the sensing method of *1H*-indole-2,3-dione was evaluated through UV-Vis spectrometry method.

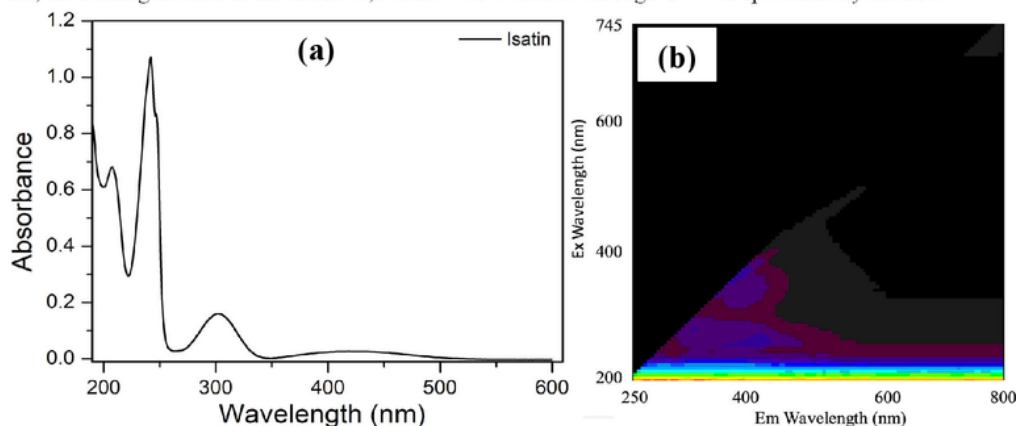


FIGURE 2. (a) The UV-Vis absorption and (b) the fluorescent 3D spectra of *1H*-indole-2,3-dione 0.05 mM

The Spectroscopy Studies

The spectroscopy studies were conducted to evaluate the sensing capability of *1H*-indole-2,3-dione as a chemosensor. Particularly, the selectivity profile of *1H*-indole-2,3-dione sensor against different metal ions were also investigated. The UV-Vis spectral changes of the *1H*-indole-2,3-dione solution after addition of various metal ions solution, such as Na^+ , K^+ , Ba^{2+} , Ca^{2+} , Co^{2+} , Cu^{2+} , Hg^{2+} , Mg^{2+} , Mn^{2+} , Ni^{2+} , Pb^{2+} , Zn^{2+} , Au^{3+} , Al^{3+} , Fe^{3+} and La^{3+} are shown in Figure 3a. As shown in the Figure 3a, it is interesting to note that only Fe^{3+} ions were able to induce strong absorption peaks at 242 and 302 nm due to complexation phenomenon between *1H*-indole-2,3-dione at carbonyl group with Fe^{3+} ions.

After the addition of metal ions, the color of the solution was changed from pale-yellow to bright yellow as shown in Figure 3b. Hence, the changes in UV-Vis spectrum of 1*H*-indole-2,3-dione solution for sensing Fe³⁺ ions were well supported by its color as bright yellow.

Titration Study

The UV-Vis titration spectral changes of 1*H*-indole-2,3-dione in the presence of different concentrations of Fe³⁺ ions were recorded to calculate the LOD and LOQ [24]. As showed in Figure 4a, it was clearly showed that the addition of Fe³⁺ ions with higher concentration (0.01-1 mM) gave a gradual increment of the absorption intensity at 242 and 302 nm. Thus, from the calibration curve, a linear correlation between the concentration of Fe³⁺ and the absorption intensity was achieved as shown in Figure 4b. The linear equation of $dI/I_0 = 8.9643 [Fe^{3+}] + 0.132$ was obtained with 0.9985 as the correlation coefficient. It was found that the LOD and LOQ values were 17.8 and 59.3 μ M, demonstrating that 1*H*-indole-2,3-dione is a potential chemosensor agent to detect Fe³⁺ ions even at lower concentration (good sensitivity). Compared to other previous results, the LOD and LOQ of 1*H*-indole-2,3-dione is the best as shown in Table 1.

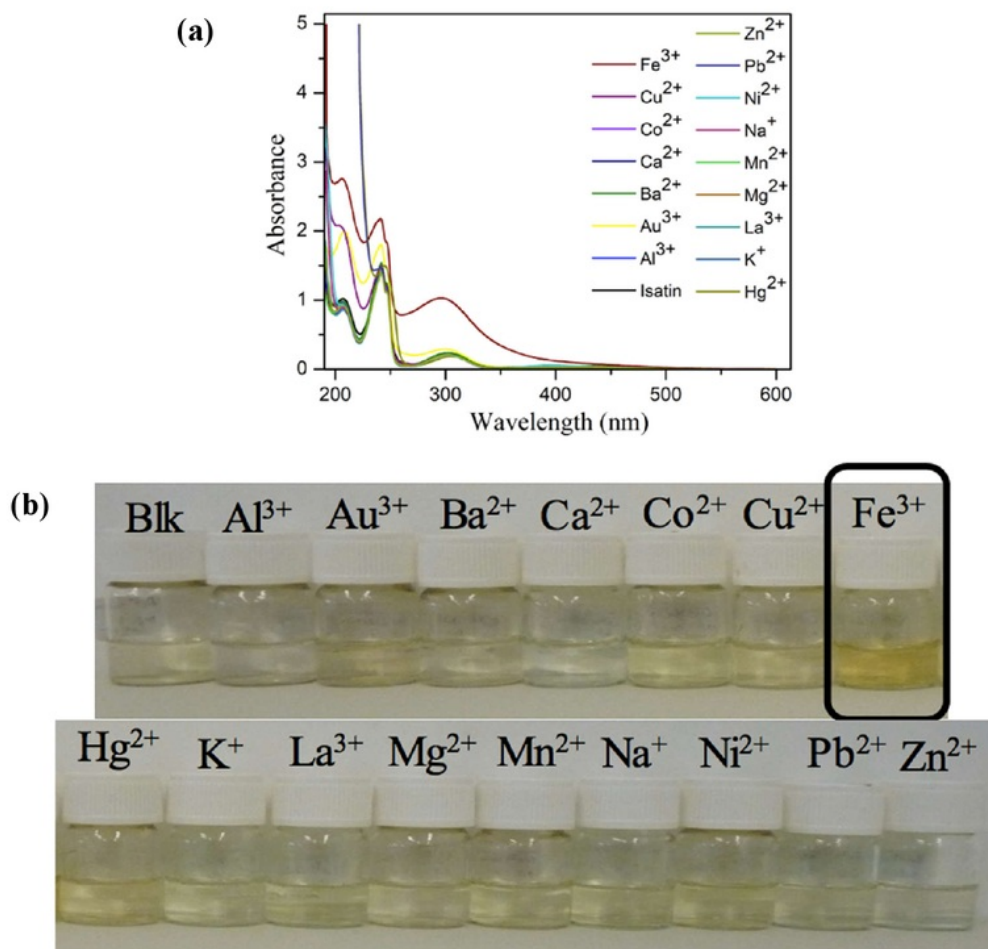


FIGURE 3. (a) The UV-Vis absorption spectral changes of 1*H*-indole-2,3-dione (0.27 mM) in distilled water in response to various metal ions with concentration of 0.5 mM and (b) their color changes upon addition of 10 equiv. of different metal

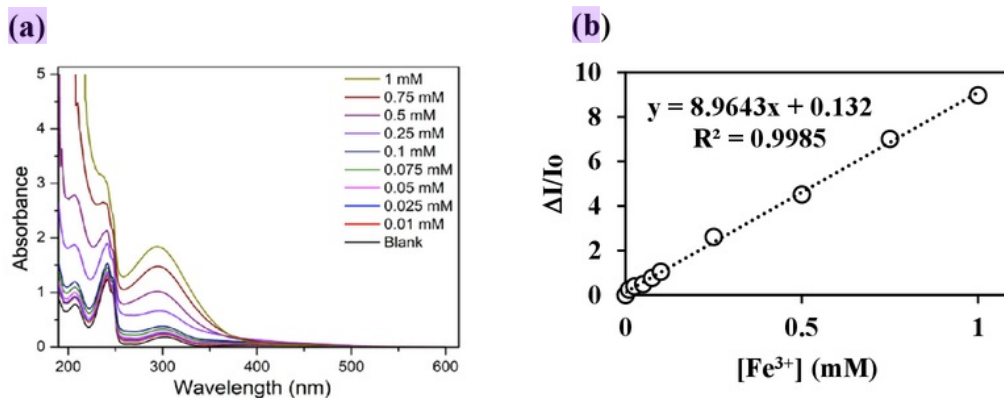


FIGURE 4. (a) The UV-vis absorption spectral changes of *1H*-indole-2,3-dione (0.05 mM) in distilled water upon gradual addition of Fe^{3+} ion with concentration 0.01, 0.025, 0.05, 0.075, 0.1, 0.25, 0.5, 0.75 and 1 mM and (b) the calibration curve plot of *1H*-indole-2,3-dione by Fe^{3+} ions presented in the form of $(\Delta I/I_0)$ vs $[\text{Fe}^{3+}]$

The Selectivity Study

The sensing capability of *1H*-indole-2,3-dione toward Fe^{3+} ions in the presence of other competing metal ions is pivotal to evaluate its performance. Therefore, the interference study was critical to be investigated by addition of Fe^{3+} ions with the presence of other metal ions into a *1H*-indole-2,3-dione solution. The absorption spectral changes of the mixture were recorded and shown as Figure 5 in its bar chart. As shown in Figure 5, the presence of other metal ions did not show remarkable interference to the absorption of change of *1H*-indole-2,3-dione toward Fe^{3+} ions. These results demonstrate that *1H*-indole-2,3-dione as a chemosensor is highly selective to Fe^{3+} ions even with the involving of interferences from other metal ions.

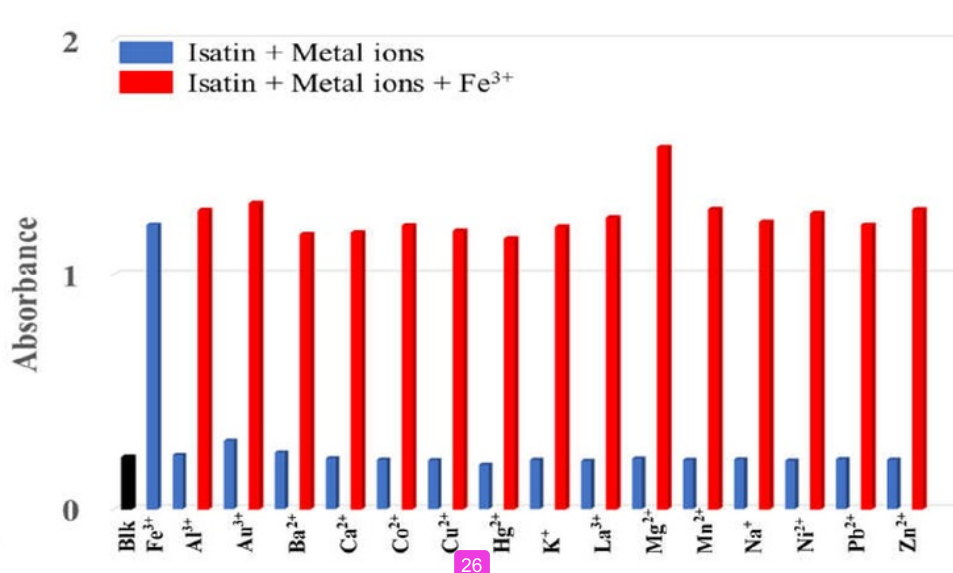
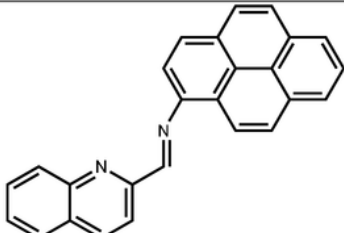
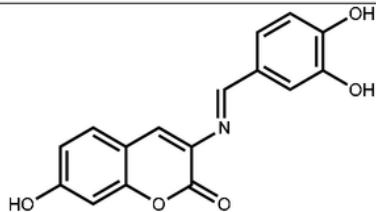
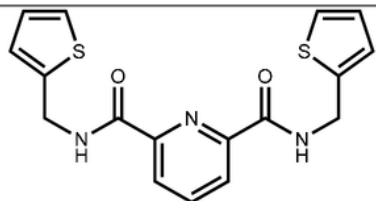
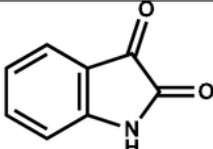


FIGURE 5. The competitive study of isatin with Fe^{3+} and the presence of other metal ions. black bar: absorption intensity of blank (*1H*-indole-2,3-dione 0.05 mM) in distilled water. Blue bar: absorption intensity of *1H*-indole-2,3-dione (0.05 mM) with other metal ions (0.5 mM) in distilled water. Red bar: absorption intensity of *1H*-indole-2,3-dione (0.05 mM) with Fe^{3+} ions (0.5 mM) and the addition of the respective competing cations (0.5 mM) in distilled water

TABLE 1. The comparison of the analytical performance between isatin-Fe³⁺ with other recently reported sensors

Chemosensor	Limit of Detection	Testing Media	Reff.
	32.2 μM	DMSO/H ₂ O (v/v=70:30)	[25]
	51.7 μM	HEPES buffer, pH 7.4, 1% DMSO	[26]
	8760 μM	MeOH	[27]
	17.0 μM	H ₂ O	Present work

CONCLUSION

In conclusion, 1*H*-indole-2,3-dione is potential to be used as a selective chemosensor for Fe³⁺ ions by utilizing a UV-Vis spectrophotometer for the evaluation of its absorption spectral changes. Among all evaluated monovalent, divalent and trivalent metal ions (Na⁺, K⁺, Ba²⁺, Ca²⁺, Co²⁺, Cu²⁺, Hg²⁺, Mg²⁺, Mn²⁺, Ni²⁺, Pb²⁺, Zn²⁺, Au³⁺, Al³⁺, Fe³⁺ and La³⁺), the a complex between 1*H*-indole-2,3-dione and Fe³⁺ ions could selectively increase the absorption band of the ligand at 242 and 302 nm at carbonyl absorption band. This interaction provides limit of detection in 17.8 μM and limit of quantification in 59.3 μM that are more sensitive than previously reported ones. Moreover, the solution appearance of the complex between 1*H*-indole-2,3-dione and Fe³⁺ ion gave a bright yellow color to support presence of the binding which was clearly visible to the naked eye for direct recognition. Such interaction does not significantly influence by the presence of other metal ions as interferences, which is remarkable for the development of chemosensors. Hence, these findings are pivotal for further investigation on design a selective chemosensor based on nitrogen heterocyclic moieties.

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