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Short Communication

A reusable test paper based on a simple salicylaldehyde derivate for the real-time detection of phosgene in gas phase

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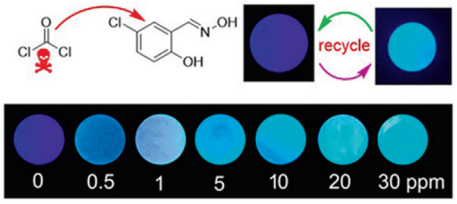
HIGHLIGHTS

- The chemosensor has a simple structure but excellent performance.
- The chemosensor showed high specificity and fast response to phosgene.
- The chemosensor loaded test paper was successfully used in the monitoring of phosgene vapor with cyclic utilization.

GRAPHICAL ABSTRACT



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ABSTRACT

Phosgene is an important organic activity intermediate as well as a poisonous gas. However, the widespread use and abuse of phosphene brings potential risks to public safety. So it is very important to detect phosgene quickly and reliably. Up to now, a lot of chemical sensors based on organoluminescent groups have been reported to monitor phosgene. However, most of them have complex molecular structures and cannot be recycled during detection. Herein, we developed a simple and effective fluorescent chemosensor using 5-chlorsalicylaldehyde as luminophor and azanol as recognition site. It exhibited significant fluorescence enhancement, excellent specificity and sensitivity. More importantly, the reusable test paper prepared by this chemosensor has been successfully used in the point-of-care testing of gaseous phosgene.

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1. Introduction

Phosgene ($COCl_2$), as an important organic activity intermediate, has been widely applied in organic synthesis, pesticide, medicine, dyestuff and other chemical products [1–5]. However, this colorless gas is not easily detected but is highly toxic due to its strong irritant and corrosive. The carbonyl groups of phosgene molecules are readily combined with the proteins and enzymes in lung tissues through acylation reaction, which can interfere with the normal metabolism of cells, damage cell membranes, alveolar epithelial cells and capillaries, thus leading to chemical pneumonia and pulmonary edema [6]. Exposure to low concentration of phosgene can cause a range of adverse symptoms, such as nausea, vomiting, dizziness, cough, retrosternal discomfort, wheezing and shortness of breath, and expectoration of blood [7]. Inhalation of high concentration of phosgene can cause respiratory failure and rapid death of the patients [8,9]. As a result, this cheap but highly toxic reagent was used as a chemical weapon in warfare and was responsible for many fatalities [10]. What's more, the widespread use of phosgene in industrial production brings the big risk of leakage, thereby casting a shadow over public safety. Therefore, it is very important to detect phosgene quickly and reliably.

Some detection techniques, such as electrochemical analysis, gas chromatography, and Raman spectroscopy have been developed for detecting phosgene [11–13]. Nevertheless, these test methods are often time-consuming and require tedious process, which seriously limits their application in on-the-spot detection

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of phosgene. Fluorescence analysis has been widely concerned because of its simple operation, visual operation and real time detection [14–16]. A lot of fluorescent probes and sensors have been reported and showed excellent detection performances for the monitor of phosgene [17–34]. However, these probes and sensors still have some shortcomings need to be overcome, for instance, complex and expensive synthesis, and being not reused or recycled during detection.

In this paper, we designed and synthesized a simple and effective fluorescent molecule (chemosensor 1) selecting 5-chlorsalicylaldehyde as luminophor and azanol as recognition site. It exhibited significant fluorescence enhancement, excellent specificity and sensitivity. More importantly, the test papers prepared by these fluorescent molecules realized the point-of-care testing of phosgene, and could be reused. Therefore, this chemosensor solved the perennial problem that the chemical sensor for phosgene is not reusable and cannot be detected in real time.

2. Experimental section

2.1. Instruments and reagents

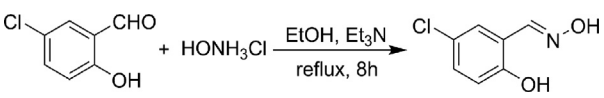
NMR spectra were acquired on a NMR spectrometer (Bruker, 500 MHz) in DMSO d_6 solution using TMS as the internal standard. HRMS was performed on a LCMS (Shimadzu, 8080). Absorption spectra were measured on spectrophotometer (Lambda 365). Fluorescence spectra were measured on a fluorescence spectrophotometer (Hitachi, F700). All reagents, including triphosgene, diethyl chlorophosphate (DCP), acetylchloride, dimethylaminozoyl chloride, trifluoroacetic acid (TFA), $SOCl_2$, 2, 4-dinitrobenzene sulfonyl chloride, $POCl_3$, HCl, triethylamine (TEA), oxalyl chloride, paratoluensulfonyl chloride (PTSC), oxalyl hydrazine, bisacrylamide, and various solvents (AR), were purchased from market suppliers, and were used without additional treatment. In order to ensure the safety of the experiment, triphosgene was selected to replace phosgene, and reacted with triethylamine (TEA) to release phosgene in CH_2Cl_2 solution.

2.2. Synthetic procedure

5-chlorsalicylaldehyde (0.31 g, 2 mmol), hydroxylammonium chloride (0.14 g, 2 mmol) and Et_3N (0.5 mL) were dissolved in absolute EtOH (15 mL), and the mixture was stirred and refluxed for 8 h (Scheme 1). Then the grey-green transparent crystal (chemosensor 1) could be obtained after volatilizing solvent at room temperature. Yield, 0.28 g (81.9%). ^{1}H NMR (500 MHz, DMSO d_6) δ: 11.49 (s, 1H), 10.31 (s, 1H), 8.28 (s, 1H), 7.52 (s, 1H), 7.26–7.24 (d, J = 10 HZ, 1H), 6.93–6.91 (d, J = 10 HZ, 1H). ^{13}C NMR (125 MHz, DMSO d_6) δ: 155.14, 145.93, 130.42, 126.64, 123.40, 120.69, 118.34. HRMS: 172.1112 ([M+H]$^+$), calculated for $C_7H_6ClNO_2$: 171.0087.

2.3. Spectral measurements

The stock solutions of chemosensor 1 and various analytes were prepared in CH_2Cl_2 containing 0.1% TEA (v/v), and were diluted to the required concentration for testing, respectively. All the spectral tests were performed at room temperature. The excitation and emission wavelengths were 350 nm and 410 nm, respectively. The slid width of fluorescence spectra was 2.5 nm/5 nm.



Scheme 1. Synthesis of chemosensor 1.

2.4. Preparation of test paper

The ordinary filter paper in the laboratory was cut into round pieces with a diameter of 1.6 cm. These round strips were soaked in CH_2Cl_2 solution of chemosensor (1 mM) for 1 min. Then, they were taken out for drying. The dry strips were soaked again, and repeated for 3 times.

10 μL of various concentrations (0, 3.38, 6.75, 33.75, 67.5, 135, 202.5 mM) of triphosgene in CH_2Cl_2 were transferred into seven reagent bottles (5 mL), respectively. After the addition of 10 μL TEA, the bottle caps with the test pieces inside were immediately placed to seal the reagent bottles. Different amounts (0, 0.5, 1, 5, 10, 20, 30 ppm) of gaseous phosgene were obtained assuming all the triphosgene was involved in the reaction.

3. Results and discussion

3.1. Absorption spectra of chemosensor 1 toward phosgene

UV–vis spectra of chemosensor 1 (50 μM) with increasing triphosgene (0 – 210 μM) were firstly measured in CH_2Cl_2 (containing 0.1% TEA, v/v). Chemosensor 1 exhibited two main absorption bands located at 315 nm and 350 nm, respectively. Along with the increase of triphosgene content (0, 30, 60, 90, 120, 150, 180, 210 μM), the absorbance of spectral band at 315 nm gradually trailed off, while absorbance at 350 nm steadily improved (Fig. 1). The change of UV–vis absorption spectra indicates that the chemosensor can react with triphosgene in a certain concentration range.

3.2. Sensitivity and linear range of chemosensor 1 toward phosgene

In CH_2Cl_2 solution (containing 0.1% TEA, v/v), the fluorescence intensity of chemosensor 1 (50 μM) was very weak, and had a very low fluorescence quantum efficiency (Φ_F = 1.4%). The fluorescence signal was barely visible under a 365 nm ultraviolet lamp. However, the fluorescence emission of chemosensor 1 strengthen gradually along with the titration of a series of triphosgene (0, 15, 30, 45, 60, 75, 90, 105, 120, 135, 150, 165, 180, 195, 210, 225, 240 μM) (Fig. 2a). And a strong blue fluorescence (Φ_F = 18.3%) could be observed under a 365 nm ultraviolet lamp when the concentration of triphosgene was 240 μM (Fig. 2a Inset). What's more, a good linear relationship between the fluorescence intensity of the chemosensor (50 μM) and the concentration of triphosgene

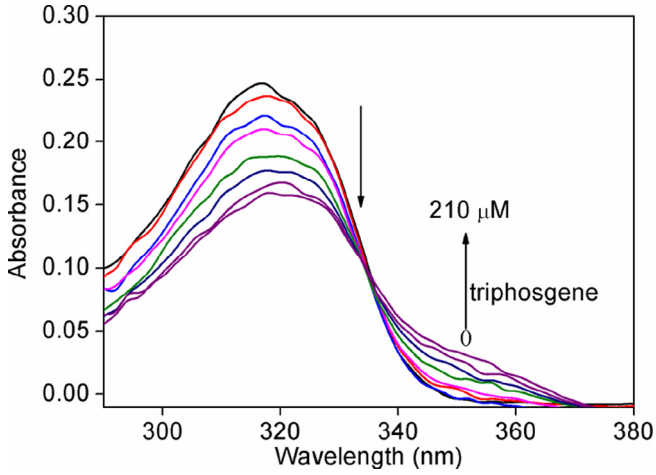


Fig. 1. UV–vis spectra of chemosensor 1 (50 μM) in CH_2Cl_2 (containing 0.1% TEA, v/v) along with the addition of triphosgene (0–210 μM).

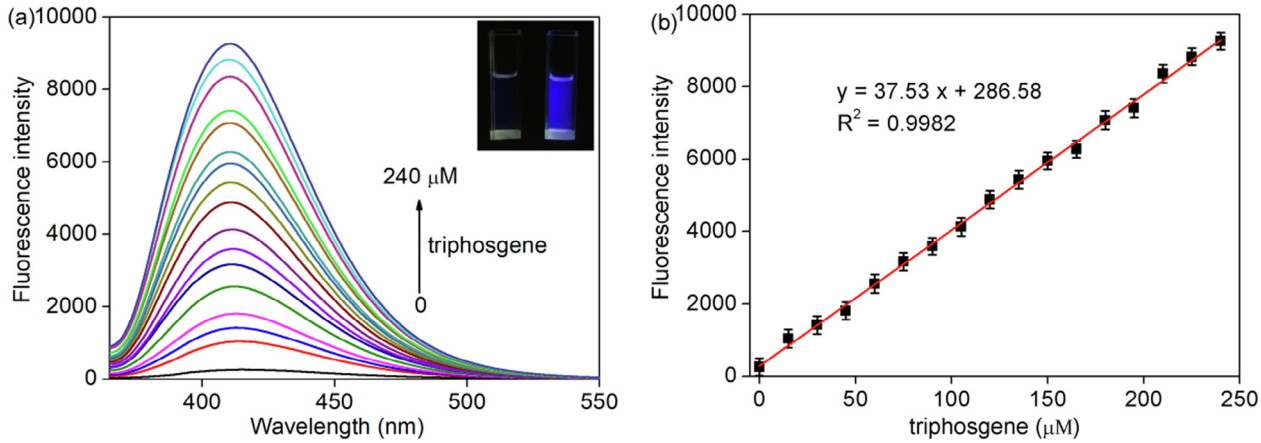


Fig. 2. (a) Fluorescence spectra of chemosensor 1 (50 μM) in CH_2Cl_2 (containing 0.1% TEA, v/v) along with the addition of various amounts of triphosgene. (b) Linearity between the fluorescence intensity (410 nm) of chemosensor 1 (50 μM) and concentration of triphosgene (0–240 μM).

was found in a wide concentration range of triphosgene (0 – 240 μM). And the linear equation was set up as y = 37.53x + 286. 58 (R^2 = 0.9982) (Fig. 2b). The limit of detection (LOD) also could be calculated as 4.0 nM using LOD = 3σ/S, where 3 is SNR, σ represents RSD (5.1%) of fluorescence intensity of chemosensor 1, and S is the slope (37.53) of the liner equation. These results indicate that this chemosensor has turn on fluorescence response to phosgene, which can be used to detect phosgene quantitatively in a wide range of phosgene concentration with a good LOD.

3.3. Selective identification of chemosensor 1 towards phosgene

The selectivity of chemosensor 1 (50 μM) towards phosgene was further investigated in the presence of various analytes (240 μM) including phosgene, triphosgene, $SOCl_2$, $POCl_3$, acetyl chloride, dimethylaminozoyl chloride, 2,4-Dinitrobenzenesulfonyl chloride, TFA, HCl, oxalyl chloride, paratoluensulfonyl chloride, oxalyl hydrazine, bisacrylamide, DCP. Phosgene could significantly increase the fluorescence intensity of the chemosensor. Triphosgene (without TEA) could only cause fluorescence improvement to a small extent. Nevertheless, none of other analytes could change the fluorescence intensity of the chemosensor significantly (Fig. 3a). In addition, this chemosensor was selective to phosgene with little interference from other analytical objects. The maximum fluorescence intensity (410 nm) of the chemosensor with phosgene did not decrease with the coexistence of other analysis objects (Fig. 3b). Therefore, the chemosensor has excellent specific recognition ability to phosgene.

3.4. Response time of chemosensor 1 towards phosgene in solution

The response time of chemosensor 1 towards phosgene was assessed by the change of fluorescence intensity over time in CH_2Cl_2 solution containing 0.1% TEA, v/v. The maximum fluorescence intensity of the chemosensor was very weak and remained stable for 44 min. On the contrary, when the chemosensor was treated with 240 μM of triphosgene, the fluorescence intensity at 410 nm gradually increased and reached a stable value after 30 min. Although the reaction process of chemosensor 1 towards phosgene took 30 min, the difference in fluorescence intensity before and after addition of phosgene was well differentiated after a few minutes (Fig. 4).

3.5. Detection of gaseous phosgene by test paper

In order to realize the quick detection for phosgene, chemosensor 1 was fixed on the common filter paper to develop a simple and convenient test paper. The test paper stained with chemosensor 1 displayed faint dark blue fluorescence under 365 nm ultraviolet light. However, when they were exposed to different concentra-

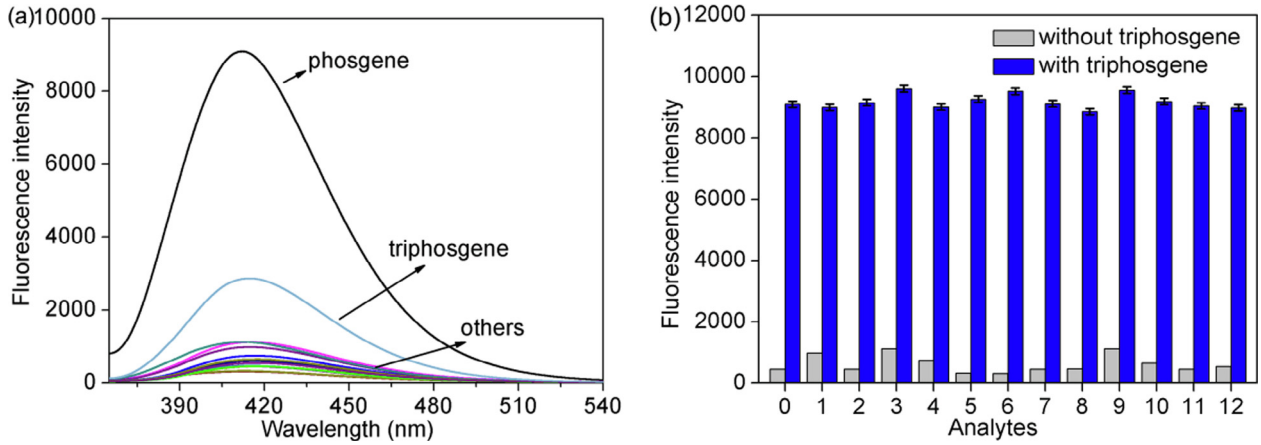


Fig. 3. (a) Selectivity and (b) specificity of chemosensor 1 (50 μM) to triphosgene (240 μM) and other analytes (240 μM). (1) $SOCl_2$, (2) $POCl_3$, (3) acetyl chloride, (4) Dimethylaminozoyl chloride, (5) TFA, (6) HCl, (7) 2,4-Dinitrobenzenesulfonyl chloride, (8) Oxalyl chloride, (9) paratoluensulfonyl chloride, (10) Oxalyl hydrazine, (11) bisacrylamide, (12) DCP.

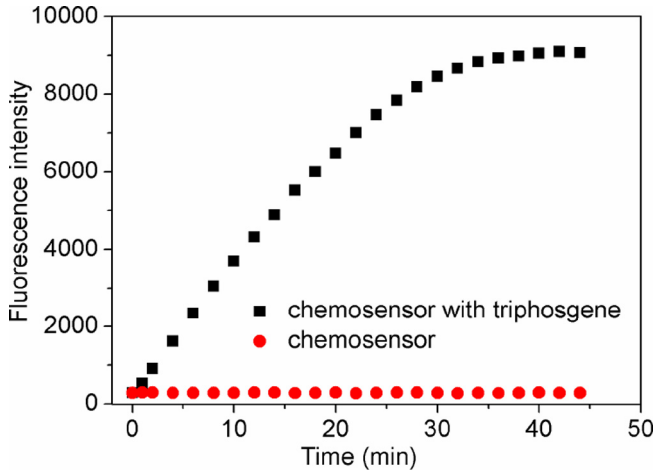


Fig. 4. Change in the fluorescence intensity (410 nm) of chemosensor 1 (50 μM) after the addition of triphosgene (240 μM).

tions (0.5, 1, 5, 10, 20, 30 ppm) of phosgene for only a few seconds, the fluorescence color of these test papers gradually changed to bright blue, accompanied by a significant increase of fluorescence (Fig. 5a). More importantly, when these test papers were removed from the phosgene environment for 1 min, the color and fluorescence intensity returned to normal. Furthermore, the test papers could still detected phosgene visually when they were reused ten times (Fig. 5b). These results show that the chemosensor can conveniently detect phosgene in real time, and can be reused many times.

3.6. Detection mechanism of chemosensor 1 towards phosgene

The different response rate of chemosensor 1 towards phosgene in solution and gas prompted us to further explore its detection mechanism by NMR and HR-MS spectra. After the reaction with phosgene in solution, the hydroxy hydrogen signal (—C=N—OH) at 10.31 ppm in ^{1}H NMR spectrum of the chemosensor 1 disappeared, and the other signal at 11.49 ppm of hydroxyl hydrogen (Ar-OH) moved to 10.79 ppm (Fig. S5), suggesting that the hydroxyl group (—C=N—OH) was involved in the reaction with phosgene. In addition, the molecular peak at 172.1112 ([M+H]$^+$) of chemosensor 1 in HR-MS disappeared, while a new peak at 369.2004 appeared (Fig. S6). The peak at 369.2004 should be ascribed to the molecular peak of compound 2. Based on these

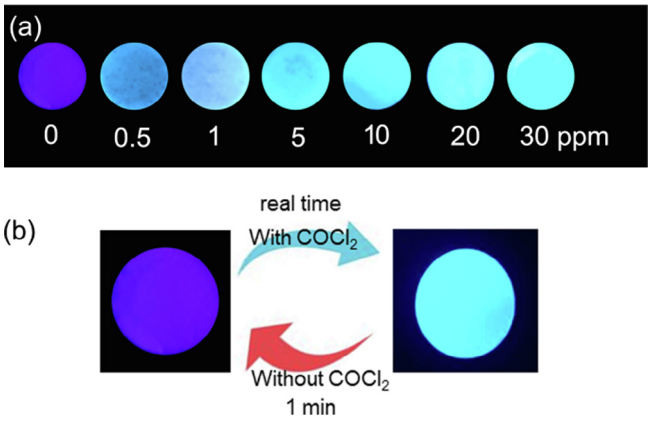


Fig. 5. (a) Color change of chemosensor test papers upon exposure to various amounts of phosgene vapor. (b) A reversible process for detecting phosgene with test papers.

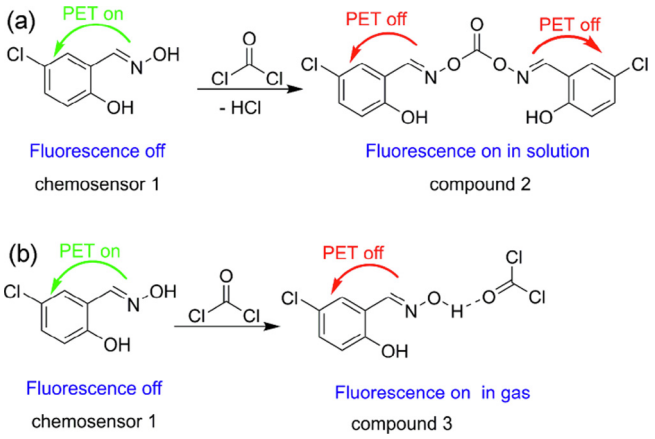


Fig. 6. The proposed detection mechanisms of chemosensor 1 towards phosgene in solution (a) and gas phase (b).

experimental results, the detection mechanism was suggested. In solution, the PET process of C=N double bond to benzene ring dissipated the energy of the excited chemosensor molecule, hence chemosensor 1 showed very weak fluorescence. Under the action of phosgene, the chemosensor molecule was further transformed into compound 2 slowly, and the PET process was inhibited (Fig. 6a). As a result, the fluorescence of the chemosensor significantly enhanced. On the test paper, the chemosensor molecule could quickly form compound 3 with phosgene by the hydrogen-bonding interaction, which could destroy the PET process within the chemosensor molecule, thus the fluorescence of chemosensor 1 increased rapidly. However, compound 3 formed by hydrogen bond was unstable, and quickly became chemosensor 1 in the absence of phosgene (Fig. 6b). Therefore, the detection of phosgene by chemosensor 1 in solution had a long equilibrium time and the fluorescence signal was irreversible, but had a short response time and could be recycled on the test paper.

4. Conclusions

In brief, a simple chemical sensor was developed through the Schiff base condensation reaction between 5-chlorosalicylaldehyde and hydroxylamine. It has been proven to be capable of reliably detection of phosgene with good selectivity and a low LOD of 4 nM in solution. More importantly, this chemical sensor could be stained on common filter paper to make test strip, realizing the rapid and reversible detection of gaseous phosgene. This study provides an idea for the design and development of reversible phosgene probe.

CRediT authorship contribution statement

Jinbiao Zhu: Investigation, Formal analysis, Methodology. **Xinyue Mu:** Investigation, Formal analysis, Methodology. **Shiqing Zhang:** Investigation, Formal analysis, Methodology. **Liqiang Yan:** Conceptualization, Investigation, Formal analysis, Methodology, Writing - original draft, Supervision, Project administration. **Xiongzhi Wu:** Conceptualization, Formal analysis, Methodology, Resources, Supervision.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary material

Supplementary data to this article can be found online at https://doi.org/10.1016/j.saa.2021.119485.

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