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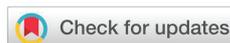
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Mini Review

Benzofuran and Naphthofuran based chemosensors for metal ion detection using fluorescence spectroscopy

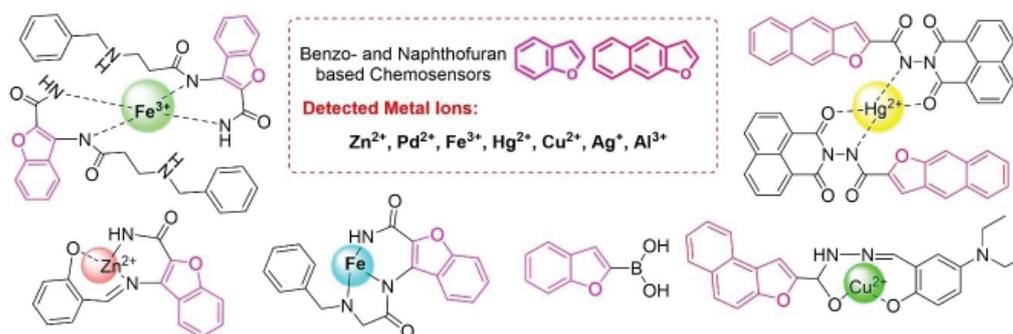
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Abstract

With the advancement in the field of agriculture and industrial regime, numerous metals such as lead, cadmium, mercury, zinc, copper, arsenic, etc. are released into the environment as effluent. These metal ions enter water bodies and generate many health issues. Considering their harmful impact on human lives, numerous fluorescent probes have been developed in recent years to detect the presence of these metal ions. The fluorescent probes are used owing to their ease of operation, good selectivity, and high sensitivity. The interaction between functional groups present in the structural framework of the probe and the empty orbitals of metal ions is responsible for the chemo selectivity of the probe. This report summarizes the organic probes which possess benzofuran and naphthofuran moieties and act as fluorescent chemosensors toward metal ions. The selectivity and sensitivity of these chemosensors along with the limit of detection have also been discussed.

Graphical abstract



Introduction

Metal ions play a significant role in our biological system. Every metal ion has its function to perform various biological and metabolic activities. All these metal ions are categorized as essential and non-essential metals [1]. The human body largely constitutes essential metal ions (Na⁺, K⁺, Ca²⁺, Mg²⁺)

often known as bulk elements while other metals such as Mn, Fe, Co, Cu, Zn, and Mo are present in trace amounts and are indispensable for humans life. Although metal ions are necessary for different biological processes, an appropriate range of concentration should be maintained in organisms. Both deficiency and excess of these metal ions can cause serious ailments in the human body [2,3]. Nonessential metals



or heavy metals (Cd, Pb, Hg, As, Cr, Ni, Al, Tl) are a threat to human life due to their toxic nature and non-biodegradability [4].

The World Health Organization (WHO) and Environmental Protection Agency (EPA) have strictly defined concentration limits of these metal ions in the human body [5,6]. Therefore, it becomes necessary to detect these metal ions at an early stage. For that purpose, various techniques have been developed to detect these metal ions like FAAS [7], AAS [8], ICP-MS [9], inductively coupled plasma emission spectrometry [10], and voltammetry [11]. However, the complicated instrumentation, skilled manpower, and costly operation have restricted their utilization in metal ion detection. As an alternative to these traditional methods, fluorescence spectroscopy is widely used due to its high selectivity and sensitivity, low cost, immediate response, easy visualization, and simple operational management [12]. As a result, several fluorescent probes known as chemosensors have been synthesized and explored for metal ion detection.

Chemosensors

Chemosensors are compounds that bear a binding site and define a mechanism of interaction between the analyte and the fluorophore. In the past few decades, diverse fluorescent chemosensors have been developed for metal ions detection [13,14]. The first fluorescent chemosensor was developed in 1867, by Goppelsroder, et al. This group reported a morin ring forming a strongly fluorescent chelate with Al^{3+} [15]. A chemosensing system generally consisted of a receptor and an active unit (chromophore/fluorophore) which can modify its properties upon interactions with the analyte and can react to the system by a “turn-on-off” mechanism in the presence of metal ions [16]. Most of the chemosensors which detect metal ions *via* fluorescence are generally based on the acceptor-fluorophore system, where the acceptor binds or chelates to the metal ion and the fluorophore translates this binding to fluorescence signal. Various moieties such as rhodamine dyes, amides, naphthalimides, pyrenes, azamacrocycles, Schiff bases, furan, and salicylaldehyde could serve as binding sites and have been used for the detection of metal ions [17-19].

Benzofuran and naphthofuran molecules are the ring-fused furan derivatives (Figure 1). These moieties are expected to be fluorescent in nature due to the presence of a highly conjugated and electron-rich system. The presence of oxygen atoms and extended π -system in the aromatic skeleton of the benzofuran and naphthofuran molecules make them electron-rich molecules and such molecules show significant interaction with the electron-deficient metal ions. Naphthofuran molecules have a large aromatic system as compared to benzofuran molecules and possess better absorption and emission spectra. Many reports are available in the literature having benzo- and naphthofuran-derived molecules, which are fluorescent in nature and exhibit chemoselectivity for metal ion detection. However, to the best of our knowledge, no review is available in the literature listing such molecules and showcasing their significance in designing novel chemosensors and in the effective interaction of probes with electron-deficient metal

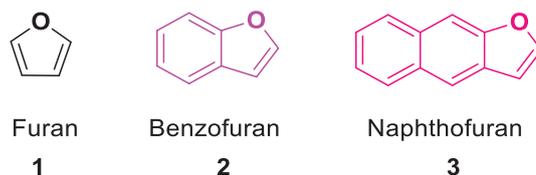


Figure 1: Representative structures of Furan, Benzofuran and Naphthofuran.

ions. Thus, in this review, we have reported such benzo- and naphthofuran-based fluorescent chemosensors and discussed their interaction mechanism and selectivity for metal ion detection.

Benzofuran based chemosensors

Oter, et al. in 2007 synthesized a benzofuran-based fluorescent dye molecule (BFK, 4) which was explored as a sensing agent in the optical sensor design. The photoluminescence property of this molecule was tested in various solvents along with the polymer matrix. In the polyvinylchloride (PVC) matrix, it displayed a large Stoke shift value which allowed the emitted fluorescence photons to be easily distinguished from the excitation photons. It led to the generation of a very low background signal and permitted the use of BFK dye in the construction of fiber optic sensors. This PVC-based optical sensor was prepared from PVC, plasticizer, BFK dye, potassium tetrakis (4-chlorophenyl) borate and THF, which was explored as a sensing film. This dye-doped membrane showed remarkable quenching toward the Fe^{3+} ion as BFK dye is supposed to form a non-fluorescent complex with Fe^{3+} . The selective quenching of Fe^{3+} ion by membrane was mainly accountable to the person's Hard Soft Acid Base (HSAB) principle which states that hard acids prefer to bind with a hard base and soft acids prefer to bind with a soft base. The O- and N- donor atoms on the BFK dye were in the category of hard Lewis base which was probably responsible for binding with Fe^{3+} . As an optic sensor, it can be used as an alternative to fluorescent Fe^{3+} indicators excited with UV light (Figure 2) [20].

In 2009, Kandaz, et al. synthesized fluorescent chemosensor 2,9,16,23-tetrakis-{6-(benzofuran-2-carboxylate)-hexylthio}-phthalocyanine Zinc (II) (ZnPcBzF, 5), which showed distinct changes in absorbance and fluorescence spectra upon treatment with silver ion $Ag(I)$ solution. Upon excitation at 616 nm, the fluorescence emission of ZnPcBzF (5) was decreased with the addition of silver ions. It indicated the formation of the silver ion complex *via* the sulfur atom on the sensor, which later altered the fluorescence emission of the phthalocyanine (Pc) core. The reason for the quenching of fluorescence emission was probably the intermolecular aggregation induced by silver ion which brought phthalocyanine units close enough to each other and interacted with the π -electrons of the Pc core (Figure 3) [21].

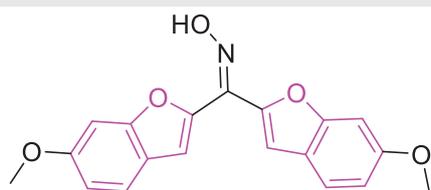
In 2017, Higashi, et al. synthesized Benzofuran-2-boronic acid (6), which was explored as an effective chemosensor for the detection of Pd^{2+} . It showed a significant rise in the emission spectra upon treatment with Pd^{2+} , giving a “turn-on” response. High selectivity for Pd^{2+} was displayed even in the presence of other competitive metal ions with a low detection



limit of 9.8 nM. Upon addition of Pd²⁺ to commercially available benzofuran-2-boronic acid, it got converted into a highly fluorescent benzofuran dimer (7) under basic conditions, thus acting as a potential chemosensor for the detection of Pd²⁺ ions (Figure 4) [22].

In 2018, Jang, et al. synthesized a benzofuran functionalized, ((E)-3-((2-hydroxybenzylidene)-amino)-benzofuran-2-carboxamide) chemosensor (HBC, 10) by the condensation reaction of 3-aminobenzofuran-2-carboxamide (8) and salicylaldehyde (9) for the selective detection of Zn²⁺. No interference of other ions was observed in the turn-on spectra of the HBC-Zn²⁺ complex and its detection limit was found to be 1.08 μM. From the theoretical analysis, it was observed that the first transition in probe 10 occurred from HOMO to LUMO due to the intramolecular charge (ICT) transition. When Zn²⁺ metal ion interacted with probe 10 it decreased the energy difference between HOMO-LUMO orbitals due to the chelation-enhanced-fluorescence (CHEF) effect. It reduced the non-radiative transition and resulted in the HBC-Zn²⁺ complex exhibiting turn-on spectra (Figure 5). The recyclability of probe HBC with Zn²⁺ using EDTA was also established which gave reversible fluorescence variation upon alternate successive addition of Zn²⁺ and EDTA [23].

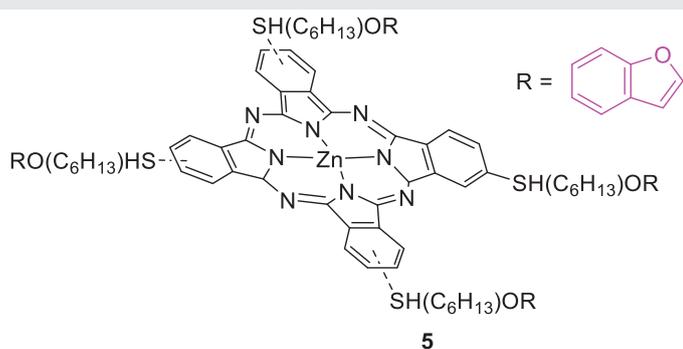
In 2019, Yue, et al. synthesized and developed {7-(dibutylamino)-3-methyl-1H, 3'H-spiro [chromeno-[2,3-c]-pyrazole-4,1'-[2]-benzofuran]-3'-one} (DHMP, 12) as highly selective chemosensor toward the copper (II) ion sensing in an aqueous medium (Figure 6). DHMP probe was found to be pH dependent as its selectivity toward copper metal was due to the protonation of the nitrogen atom (Figure 7). In a neutral medium, equilibrium occurred between two forms 12 and 15; but in an acidic medium, the DHMP molecule got



bis(7-methoxybenzofuran-2-yl)ketoxime (BFK)

4

Figure 2: Chemical structure of Bis-(7-methoxybenzofuran-2-yl)-ketoxime (BFK, 4).



5

Figure 3: Chemical structure of chemosensor 5.

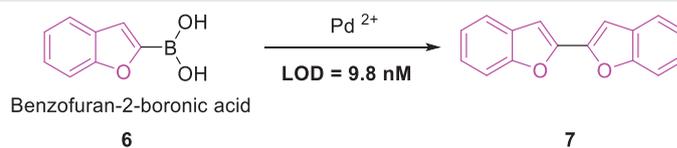


Figure 4: Proposed interaction mechanism of Benzofuran-2-boronic acid with Pd²⁺.

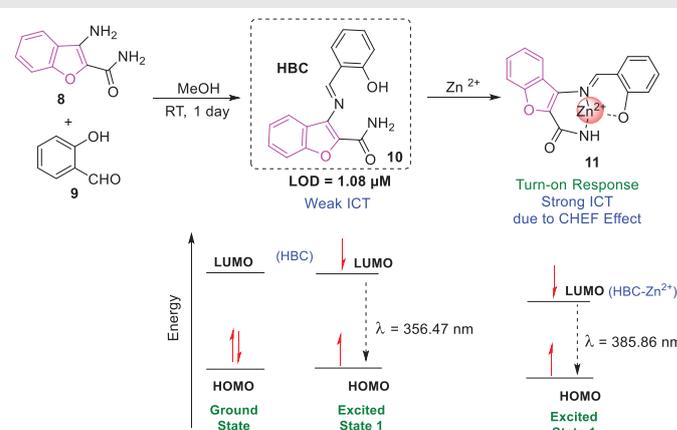


Figure 5: Synthesis of HBC and proposed interaction mechanism of HBC with Zn²⁺.

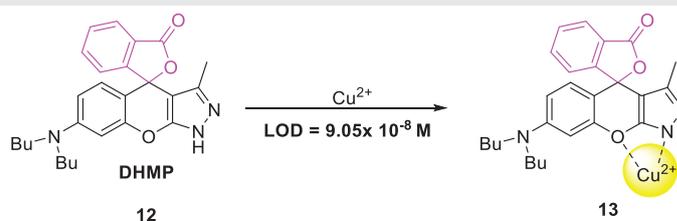


Figure 6: Proposed interaction mechanism of DHMP with Cu²⁺.

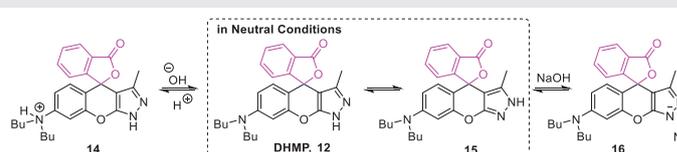


Figure 7: Protonation and deprotonation mechanism of DHMP.

protonated at the dibutylamino nitrogen atom (14), and in the basic medium deprotonation of pyrazole ring occurred to form sodium salt (16) (Figure 7). Chemosensor showed selectivity under strongly acidic conditions (pH range 1.1-2.1) and strong alkaline conditions (pH range 13.1-13.9). No significant emission intensity was found in the pH range of 2.9 to 11.9. Thus, the chemoselectivity of probe DHMP was observed to be strongly pH-dependent due to the protonation and deprotonation mechanism of dibutyl amino nitrogen and pyrazole ring [24].

In 2019, Madhu, et al. synthesized a benzofuran-based chemosensor 3-(3-((4-methylbenzyl)-amino)-propanamide)-benzofuran-2-carboxamide (BAA, 17) which showed a 'turn-on' response with high selectivity and sensitivity towards Fe³⁺ ions in DMSO/H₂O solution (9:1). Due to the paramagnetic nature of Fe³⁺, most of the reported iron sensors showed a 'switch-off response'. Probe BAA showed



an intramolecular charge transfer process in the Free State which was evident *via* theoretical analysis of HOMO and LUMO orbitals performed by the authors. Upon the addition of Fe^{3+} ion, the ICT process was diminished, and a strong CHEF effect was observed which displayed turn-on spectra. In addition to this, the HOMO-LUMO gap was dropped from 4.02 eV in the case of BAA to 1.62 eV in the case of $[\text{BAA}]_2\text{-Fe}^{3+}$ complex which confirmed the strong binding of sensor BAA with Fe^{3+} and stable complex formation. Furthermore, chemosensor BAA showed 'ON-OFF-ON' fluorescence signals upon alternate addition of Fe^{3+} and EDTA after several cycles. Upon the addition of EDTA, the decrease in fluorescence intensity was observed due to the formation of a strong complex between EDTA and Fe^{3+} , which further suggested that the sensor BAA could be used as a reversible chemosensor for Fe^{3+} (Figure 8) [25].

In 2020, Zhu, et al. synthesized Schiff base [3,3-bis((4-hydroxyphenyl)-2-benzofuran-1-one-isoquinoline-1-hydrazine) (19) from phenolphthalein and isoquinoline hydrazide. Upon excitation at 377 nm, probe 19 displayed an extremely weak fluorescence emission profile (emission at 479 nm) with a very low quantum yield value of 0.0012. This was caused by the lone pair electrons of the nitrogen atom in the Schiff base on reaching the chromone moiety which suppressed the fluorescence *via* the photoinduced electron transfer (PET) effect. But upon the addition of Al^{3+} , the fluorescent intensity of probe 19 was increased gradually up to 700-fold and the quantum yield reached 0.032 due to the generation of the CHEF effect that diminished the PET effect [26]. Thus, the chemosensor showed distinguished spectra with Al^{3+} but no other metal showed an interference effect except Cu^{2+} metal which quenched the fluorescence of 19 due to its paramagnetic nature Figure 9.

In 2022, Madhu, et al, synthesized benzofuran glycinamide-based chemosensor, 3-(2-((4-fluorobenzyl)-amino)-acetamido)-benzofuran-2-carboxamide (BGA, 21) *via* multistep reaction for the selective and sensitive detection of Fe^{3+} ions. Chemosensor BGA showed a "turn-off" fluorescence response towards Fe^{3+} ion even in the presence of other metal ions. Probe BGA had a large electron density on the glycinamide moiety instead of the benzofuran scaffold in the HOMO orbital and possessed intramolecular charge transfer spectra (ICT). When Fe^{3+} ion interacted with BGA, the energy level of LUMO decreased which changed the photo behavior of the probe BGA. Due to this phenomenon, the ICT process was diminished, and a strong chelation effect occurred causing the quenching of BGA fluorescence. The reusability of probe BGA was further accepted by repeated alternate addition of EDTA and Fe^{3+} giving "turn off-on-off" spectra. Fe^{3+} responsive chemosensor BGA showed high selectivity and sensitivity with a very low detection limit of 43 nM (Figure 10) [27].

Naphthofuran based chemosensors

Though several naphthofuran-based molecules have been synthesized and utilized in different fields such as medicinal chemistry, catalysis, semiconducting devices, etc. [28-35]; only sporadic reports are available in literature exploring such molecules in the chemosensing field using fluorescence spectroscopy.



Figure 8: Proposed interaction mechanism of BAA with Fe^{3+} .

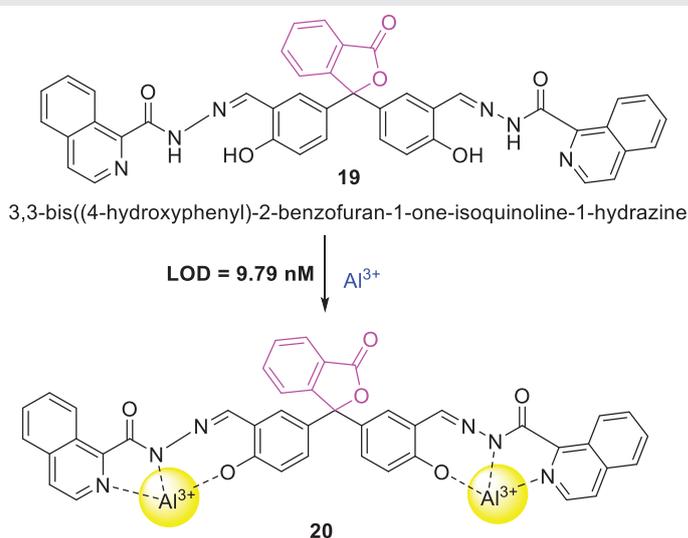


Figure 9: Proposed interaction mechanism of 19 with Al^{3+} .

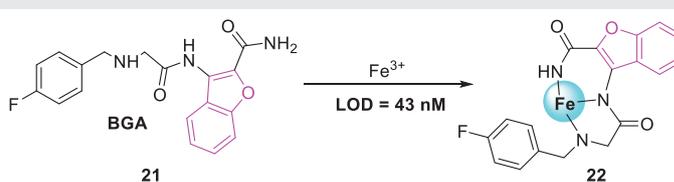


Figure 10: Proposed interaction mechanism of BGA with Fe^{3+} .

In 2015, Jie, et al. synthesized naphthofuran carbonyl hydrazone derived Zn^{2+} coordinated complex (23) as a fluorescent chemosensor *via* a displacement approach. Upon treatment with Cu^{2+} , the chemosensor exhibited an excellent "turn-off" response. To know the binding mode of Cu^{2+} to probe 23, an NMR measurement experiment had been carried out which showed the disappearance of the -OH (δ 12.22) and -NH (δ 11.24) protons in the NMR spectra of probe 23. When Cu^{2+} ion interacted with probe 23, displacement of Zn^{2+} occurred, and turn-off spectra were observed due to the paramagnetic properties of Cu^{2+} . The chemosensor efficiently detected Cu^{2+} even in the presence of other metal ions with a detection limit of 2.3×10^{-7} M (Figure 11) [36].

In 2022, Guan, et al. synthesized a naphthofuran functionalized naphthalimide (25) chemosensor for the selective detection of highly toxic Hg^{2+} ions. The chemosensor 25 showed a "turn-off" response for Hg^{2+} ions with a low detection limit of 4.7×10^{-7} M, displaying the excellent stimuli-responsive property for Hg^{2+} in the presence of other metal ions. From the theoretical analysis of the HOMO-LUMO orbital and electron potential map of 25 and 26, it was concluded that



the turn-off spectra were observed due to the charge transfer between 25 and Hg^{2+} (Figure 12) [37].

Conclusion and future prospective

This report summarizes the organic molecules which possess benzofuran and naphthofuran moieties in their structural framework and act as efficient chemosensors toward metal ion detection. The presence of benzofuran and naphthofuran moieties make organic molecules electron rich that can interact with an empty orbital of metal ion effectively and produce significant emission spectra. The performance of these chemosensors has been listed in Table 1 along with their limit of detection.

Most of the listed chemosensors were found to be highly sensitive as LOD was achieved up to 9.79 nM. All the probes were found to be highly selective towards one specific metal ion. Some of them such as probes 10, 17, and 21 were able to show reusability due to their reversible interaction with metal ions. Thus the studies clearly depict the potential of these molecules in the chemosensing field. Unfortunately, not many chemosensors have been designed keeping in mind the structural benefits of benzofuran and naphthofuran skeleton. In this scenario, we believe that this article may give an opportunity to the chemists to design and develop novel benzo- and naphthofuran-based chemosensors which can be explored in near future more effectively for the detection of various metals.

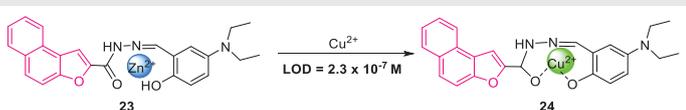


Figure 11: Proposed mechanism of 23 with Cu^{2+} .

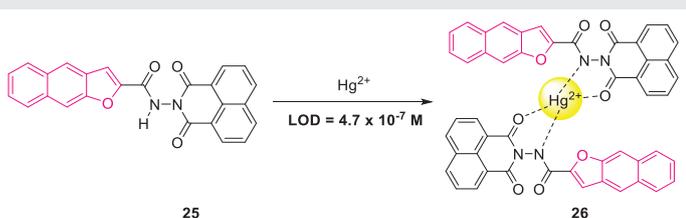


Figure 12: Proposed interaction mechanism of 25 with Hg^{2+} .

Table 1: Benzofuran and naphthofuran-based chemosensors along with their limit of detection and metal ion detection.

S. No.	Compounds	LOD	Metal ion detected	References
1.	6	9.8 nM	Pd^{2+}	Higashi, et al. 2017; [22]
2.	10	1.08 μM	Zn^{2+}	Jang, et al. 2018; [23]
3.	12	9.05×10^{-8} M	Cu^{2+}	Yu, et al. 2019; [24]
4.	17	0.067 μM	Fe^{3+}	Madhu, et al. 2019; [25]
5.	19	9.79 nM	Al^{3+}	Zhu, et al. 2020; [26]
6.	21	43 nM	Fe^{3+}	Madhu, et al. 2022; [27]
7.	23	2.3×10^{-7} M	Cu^{2+}	Jie, et al. 2015; [28]
8.	25	4.7×10^{-8} M	Hg^{2+}	Gau, et al. 2022; [29]

Acknowledgment

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