

RESEARCH ARTICLE

Crown Ether Functionalized Barbituric Acid for Colorimetric Determination of Au³⁺

Tanaji R. Bhosale ^{1,*}, Sandeep B. Wategaonkar ², Prashant P. Patil ³, Prashant V. Anbhule ⁴, Madhukar B. Deshmukh ⁴

ABSTRACT: In this study, we report the successful synthesis of a novel benzo crown ether-functionalized barbituric acid derivative (receptor 3) as a colorimetric probe for detecting Au³⁺ ions in aqueous solutions. The receptor was synthesized using a straightforward Knoevenagel condensation between 4, 4'-diformyl dibenzo-15-crown-5 ether and barbituric acid in methanol at room temperature, yielding the product in good quantities. This receptor demonstrated excellent selectivity and sensitivity towards Au³⁺ ions over other common cations, enabling rapid visual detection without the need for advanced instrumentation. Upon interaction with Au³⁺, receptor 3 caused a distinct color change in the solution from colorless to violet, which could be observed with the naked eye. The detection limit of receptor 3 was calculated to be as low as 2.95×10⁻⁵ molL⁻¹, making it highly suitable for detecting trace amounts of Au³⁺ in real-world applications. The optical properties of the receptor were further characterized using various techniques, confirming its stability and efficacy in aqueous medium (DMSO+H₂O, 1:4 v/v). This newly developed sensor holds significant potential for practical applications, particularly in environmental monitoring and gold ion detection in industrial processes, offering a cost-effective and simple alternative to more complex detection methods.

Keywords: Formyl benzo crown ether, barbituric acid; chemo sensor; Au³⁺ cation; DMSO + H₂O (1:4, v/v).

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1. INTRODUCTION

The design and synthesis of new chemosensors for the recognition of transition metal cations is a vibrant field of research due to its broad spectrum of applications in clinical diagnostics, biological systems, and environmental monitoring [1-4]. Transition metal ions play significant roles in various physiological and pathological processes, and among these, gold ions have gained attention for their unique properties. Gold is an essential trace element known for its anti-inflammatory, anti-arthritis, tuberculostatic, and anti-

cancer activities [5]. Recent reviews have extensively covered diverse aspects of gold chemistry, such as its role in catalysis, selective oxidation, and its use in diagnostics and detection through nanoparticles [6]. Despite the growing recognition of gold's importance in various fields, only a few reports have focused on the selective detection of gold cations [7-8]. This limitation is particularly concerning given the increasing need for rapid, reliable, and sensitive methods to detect gold ions in various biological and environmental samples.

The traditional detection methods for gold species rely heavily on sophisticated instrumentation, such as atomic absorption spectrometry (AAS), graphite furnace atomic absorption spectrometry (GFAAS), inductively coupled plasma emission spectrometry (ICP-ES), inductively coupled plasma mass spectrometry (ICP-MS), and electrochemical assays [9]. These methods, while highly accurate, have several drawbacks, including high costs, the need for advanced and expensive equipment, and labor-intensive sample preparation. Moreover, the requirement for trained personnel further limits their accessibility, particularly in

¹ Department of Chemistry, D. Y. Patil Agriculture and Technical University, Talsande Kolhapur-416112, Maharashtra.

² Department of Chemistry, Kisan Veer Mahavidyalaya, Wai, 412803, Maharashtra, India.

³ Department of Technology, Shivaji University Kolhapur, Maharashtra, India.

⁴ Department of Chemistry, Shivaji University Kolhapur, Maharashtra, India.

*Authors to whom correspondence should be addressed:
trbhosal2013@gmail.com (T. R. Bhosale)

resource-constrained environments. In recent years, molecular sensors selective for gold cations, employing various fluorophores such as Rhodamine, BODIPY, fluorescein, and Naphthalimide, have emerged as alternatives [10]. However, these fluorometric methods still require specialized equipment and involve complex operational protocols [11].

In contrast, colorimetric chemosensors, which allow naked-eye detection of metal cations, offer several advantages. These include high sensitivity, rapid response time, simplicity of operation, and lower costs [12-14]. Such sensors are particularly appealing for real-time and in-field detection, where the availability of advanced laboratory equipment is limited. Our research focuses on developing such colorimetric sensors to address the pressing need for straightforward yet effective metal ion detection techniques. Barbituric acid derivatives, which have demonstrated diverse applications as fluorescent probes [15], in dye-sensitized solar cells [16], and as agents for treating hepatitis C [17], have also proven valuable in designing coordination and supramolecular compounds [18]. Additionally, barbituric acid derivatives are known for their role as reactive oxygen scavengers [19], further underscoring their versatility and potential in sensor design.

In light of these factors, and as part of our ongoing efforts in metal cation recognition [20-23], we aim to develop an efficient, selective, and sensitive colorimetric sensor specifically for Au³⁺ ions in aqueous media. Here, we report the synthesis and application of a new benzo crown ether functionalized barbituric acid-based chemosensor, designed for the naked-eye detection of Au³⁺ ions. The sensor was synthesized via a Knoevenagel condensation reaction between 4, 4'-diformyl dibenzo-15-crown-5 ether and barbituric acid in methanol under ambient conditions, yielding a product with excellent efficiency. This benzo crown ether functionalized barbituric acid derivative serves as a simple, reliable, and highly selective colorimetric chemosensor for Au³⁺ ion recognition in aqueous media. Notably, the receptor demonstrated exceptional sensitivity and selectivity towards Au³⁺ ions over other metal ions, making it a promising candidate for practical applications in gold ion detection.

2. EXPERIMENTAL DETAILS

2.1. Materials

Materials used in experiments were of analytical grade without further purification unless specified. Deionized water was used throughout the sensing experiments. The solution of metal ions was prepared from chloride salts of Ca²⁺, Fe²⁺, Co²⁺, Ni²⁺, Cu²⁺, Zn²⁺, Cd²⁺, Pb²⁺, Hg²⁺ and Au³⁺ ions with 10⁻³ molL⁻¹ in deionized water to evaluate the cations binding interaction with compound. Compound **3** was dissolved in DMSO to obtain the stock solution of receptor **3** (10⁻³ molL⁻¹) and then diluted with deionized

water to prepare the analytical solution (2.0×10⁻⁴ M).

2.2. Chemicals

All the chemicals used were of analytical grade and were used without any further purification. The progress of the reaction was monitored by thin-layer chromatography (TLC) (silica gel 60 F254, Merck, Germany). FT-IR spectra were recorded on JASCO Spectrometer with scanning from 4000 to 450 cm⁻¹. The ¹H NMR and ¹³C NMR were recorded on Bruker 300 AVANCE II in DMSO-d₆ as a solvent and tetramethyl silane (TMS) as an internal standard. Mass spectra were recorded on Agilent 6540 a Performa spectrometer equipped with an electron spray ionization interface. Elemental analysis was performed on EURO Vector Elemental Analyzer. Melting points were determined in open capillary tube and are uncorrected. The absorption spectra were recorded at room temperature on SPECORD 210 plus Analytic Jena spectrophotometer using of 1.0 cm quartz cell.

2.3. Synthesis of 4,4'-diformyl dibenzo-15-crown-5 ether (1) Duff formylation of dibenzo-15-crown-5:

The mixture of dibenzo-15-crown-5 (0.95g, 3 mmol), trifluoroacetic acid (5 mL) and hexamethylene tetraamine (1.68 g, 12 mmol) was stirred at 90 °C under nitrogen for 24 hrs. The mixture was then extracted with 25 ml benzene and dried over magnesium sulphate. The concentration of the benzene extract under vacuum gave a brown oil, which on cooling solidified to white crystalline solid (1), Gives white solid in the yield of 0.76g; (68%), M.P. 76-78 °C; IR :2924 (aldehydic C-H), 2731 (Fermi resonance), 1682 (C=O), 1585 (C=C) cm⁻¹; ¹H-NMR (300 MHz, CDCl₃) δ ppm: 3.80 (s, 4H), 4.19-4.22 (t, 4H), 4.39-4.45 (t, 4H), 7.06-7.48 (m, 6H), 9.78 (s, 1H); ¹³C-NMR (DMSO-d₆) δ ppm: 67.08, 67.23, 67.46, 68.75, 69.05, 69.14, 69.19, 69.36, 112.98, 113.16, 113.35, 113.52, 113.66, 126.40, 126.55, 126.81, 130.24, 130.28, 130.39, 130.50, 141.06, 149.06, 149.23, 149.33, 149.39, 153.19, 154.48, 154.69, 154.78, 167.45 and 190.89 ppm. ESI-MS: calculated for C₂₀H₂₀O₇ [M]⁺: 372.12; found: 373.1207 [M+H]⁺.

2.4. Synthesis of hexahydrodibenzo[b,h][1,4,7,10,13]pentaacyclopentadecine-3,13-diyl)bis(methanylylidene) bis(pyrimidine-2,4,6(1H,3H,5H)-trione) (3)

A mixture of 4,4'-diformyl benzo crown ether (1 mmol) and barbituric acid (2 mmol) in methanol (5 mL) were stirred at a room temperature. After few minutes the faint yellow solution colored becomes dark yellow. The suspension of the flask to stirred overnight at room temperature. The solid product obtained was filtration and washed times with cold methanol (3×5 mL) to get yellow crystalline product as a brown solid in yield of 69%; Yield 69%, M.P. 290 °C; m. p.

290 °C; IR : 1137 (C-O-C), 1653 (C=O), 3045 (NH), 3107(NH), cm⁻¹; ¹H-NMR (300 MHz, DMSO-d₆) δ ppm: 4.17-4.49 (m, 12H, OCH₂), 7.05-7.19 (m, 2H, Ar-H), 7.89 (d, *J* = 3 Hz, 2H, Ar-H), 8.24-8.36 (m, 4H), 11.14 (s, 2H, N-H), 11.27 (s, 2H, N-H); ¹³C-NMR (75 MHz, DMSO-d₆) δ ppm: 67.33, 68.92, 113.23, 113.65, 115.67, 125.68, 125.87, 126.47, 130.12, 132.21, 147.65, 147.89, 149.28, 150.64, 154.24, 156.11, 162.66, 164.43, 167.60, 168.25 ppm. LC-MS calculated for C₂₈H₂₄N₄O₁₁ [M]⁺:592.51; found:593.1562 [M+H]⁺.

3. RESULTS AND DISCUSSION

3.1. Synthesis of Benzo Crown Ether Functionalized Barbituric Acid (Receptor 3)

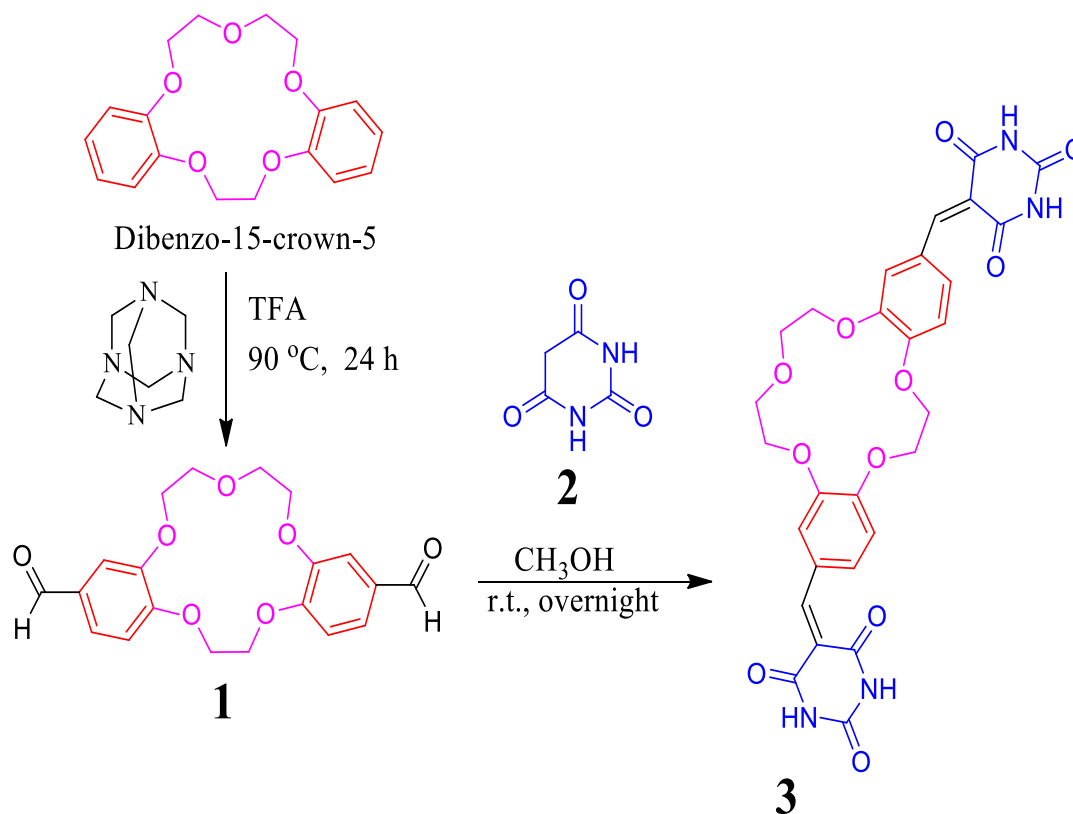
The synthesis of the target molecule, 4,4'-diformyl dibenzo-15-crown-5 ether (1), was achieved through Duff formylation. This process involved the reaction of dibenzo-15-crown-5 ether with trifluoroacetic acid and hexamine under a nitrogen atmosphere at 90°C for 15 hours. After cooling, the reaction mixture was extracted using 25 mL of benzene and dried over magnesium sulfate. Concentration of the benzene extract under vacuum produced a brown oily liquid, which solidified

on cooling into a white crystalline solid. This solid was identified as the desired compound (1). The subsequent synthesis of the benzo crown ether functionalized barbituric acid (receptor 3) was accomplished through a Knoevenagel condensation reaction between 4,4'-diformyl dibenzo-15-crown-5 ether and barbituric acid in methanol at room temperature, yielding receptor 3 in good yield (Scheme 1).

The reaction scheme in Scheme 1 clearly shows the two-step process: (1) the di-formylation of dibenzo-15-crown-5 ether via Duff formylation to obtain the diformyl derivative and (2) the Knoevenagel condensation of this diformyl derivative with barbituric acid to produce the benzo crown ether functionalized barbituric acid receptor (3). This strategy is efficient and provides good yields of the final product under ambient conditions.

3.2. Characterization of Receptor 3

The synthesized receptor 3 was fully characterized using Fourier-transform infrared spectroscopy (FT-IR), nuclear magnetic resonance (NMR), and mass spectrometry (MS). FT-IR analysis revealed significant bands at 3107–3045 cm⁻¹, corresponding to -NH stretching, at 1653 cm⁻¹, corresponding to C=O stretching, and at 1137 cm⁻¹, corresponding to C-O-C groups, confirming the presence of the functional groups necessary for selective ion recognition.



Scheme 1. Synthesis of target molecule **3**: firstly di-formylation of dibenzo-15-crown-5 using Duff formylation followed by Knoevenagel condensation of 4,4'-diformyl benzo crown ethers (**1**) with barbituric acid (**2**).

The proton NMR (¹H-NMR) spectra further confirmed the structure of receptor 3. Signals for the methylene oxy (-OCH₂-) groups were observed between δ 4.17–4.49 ppm, while the aromatic protons exhibited signals between δ 7.07–8.24 ppm. The vinylic protons, crucial for interaction with metal ions, were detected at δ 8.35–8.36 ppm, while the four -NH protons appeared as two broad singlets at δ 11.14 and 11.27 ppm, consistent with the barbituric acid moiety. Carbon-13 NMR (¹³C-NMR) spectra provided additional confirmation, with the methylene oxy (-OCH₂-) carbon signals occurring between δ 67.33–68.92 ppm, and aromatic ring carbon signals observed at δ 113.23–150.64 ppm. Mass spectrometry analysis of receptor 3 yielded a molecular ion peak at m/z = 593.1562, corresponding to the molecular weight of the compound, further confirming its identity and purity.

3.3. Colorimetric "Naked-Eye" Detection of Au³⁺ Ions

One of the primary objectives of this study was to develop a simple, efficient, and selective colorimetric chemosensor for the detection of gold (Au³⁺) ions in aqueous media. The "naked-eye" detection method is particularly advantageous due to its simplicity, allowing for on-site detection without the need for specialized instruments or trained personnel. As shown in Figure 1, receptor 3, when dissolved in a ~100% aqueous solution, exhibited selective color changes upon the addition of various metal ions such as Ca²⁺, Fe²⁺, Co²⁺, Ni²⁺, Cu²⁺, Zn²⁺, Cd²⁺, Pb²⁺, Hg²⁺, and Au³⁺. Among these, only Au³⁺ ions induced a distinct and rapid color change from colorless to violet, making receptor 3 highly selective for Au³⁺ ions.

The selective colorimetric response of receptor 3 towards Au³⁺ ions is likely due to a specific interaction between the Au³⁺ cation and the receptor's functional groups, particularly the crown ether moiety and the barbituric acid unit. This interaction leads to a change in the electronic structure of the receptor, which is visible as a color change. In contrast, other metal ions tested did not induce any noticeable color change, further demonstrating the selectivity of receptor 3 for Au³⁺ ions.

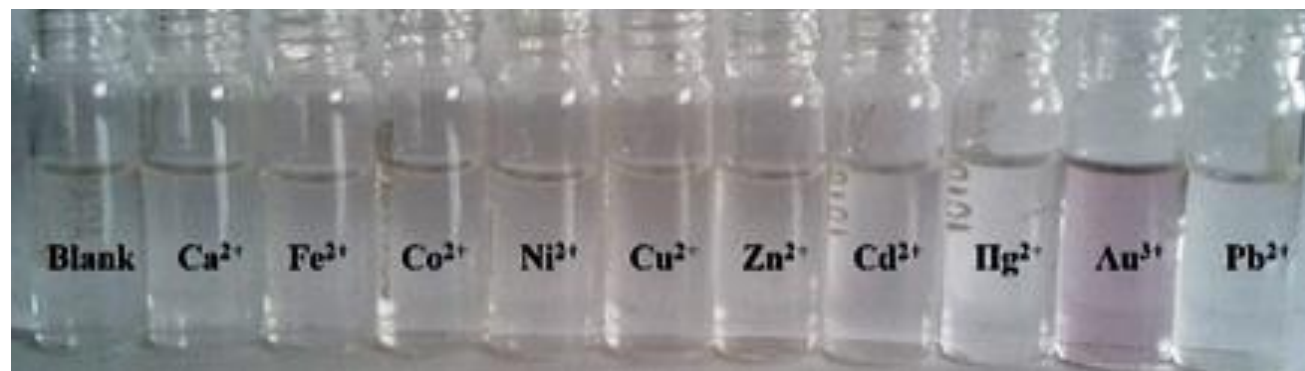


Fig. 1. Photograph of the color change is observed by addition of 1 mL of various metal cations ($1 \times 10^{-3} \text{ mol L}^{-1}$) to the solution of the receptor 3 ($10^{-3} \text{ mol L}^{-1}$) in DMSO+H₂O (1:4, v/v).

3.4. UV-Vis Spectroscopy Analysis of Metal Ion Selectivity

To further investigate the selectivity of receptor 3 for Au³⁺ ions, UV-Vis absorption spectra were recorded for solutions of receptor 3 in the presence of various metal ions. As shown in Figure 2, the UV-Vis spectrum of receptor 3 in the presence of Au³⁺ ions exhibited a distinct absorption peak at 550 nm, corresponding to the interaction of Au³⁺ ions with receptor 3. In contrast, the presence of other metal ions did not produce any significant absorption peaks at this wavelength, indicating that receptor 3 selectively binds to Au³⁺ ions.

A bar graph of the absorbance values at 550 nm (Figure 2) further illustrates the selectivity of receptor 3 for Au³⁺ ions. The absorbance for Au³⁺ ions was significantly higher than that for other metal ions, confirming the strong and selective binding of receptor 3 to Au³⁺ ions. This high selectivity is attributed to the unique coordination chemistry between Au³⁺ and the benzo crown ether functionalized barbituric acid moiety, which is not as favorable for other metal ions.

3.5. Effect of Au³⁺ Ion Concentration on Colorimetric Response

To evaluate the sensitivity of receptor 3 to different concentrations of Au³⁺ ions, solutions of receptor 3 were prepared with varying concentrations of Au³⁺ ions (ranging from 0.1 to 0.9 mL of $1 \times 10^{-3} \text{ mol L}^{-1}$) while keeping the concentration of receptor 3 constant. As shown in Figure 3, the intensity of the violet color increased with increasing concentrations of Au³⁺ ions, demonstrating a clear concentration-dependent response.

The UV-Vis absorption spectra of receptor 3 at different concentrations of Au³⁺ ions (Figure 4) confirmed this observation, with a gradual increase in absorbance at 550 nm as the concentration of Au³⁺ ions increased. This correlation between absorbance and Au³⁺ ion concentration is further illustrated in Figure 5, which shows a linear relationship between absorbance and Au³⁺ ion concentration, indicating that receptor 3 can quantitatively detect Au³⁺ ions within this concentration range.

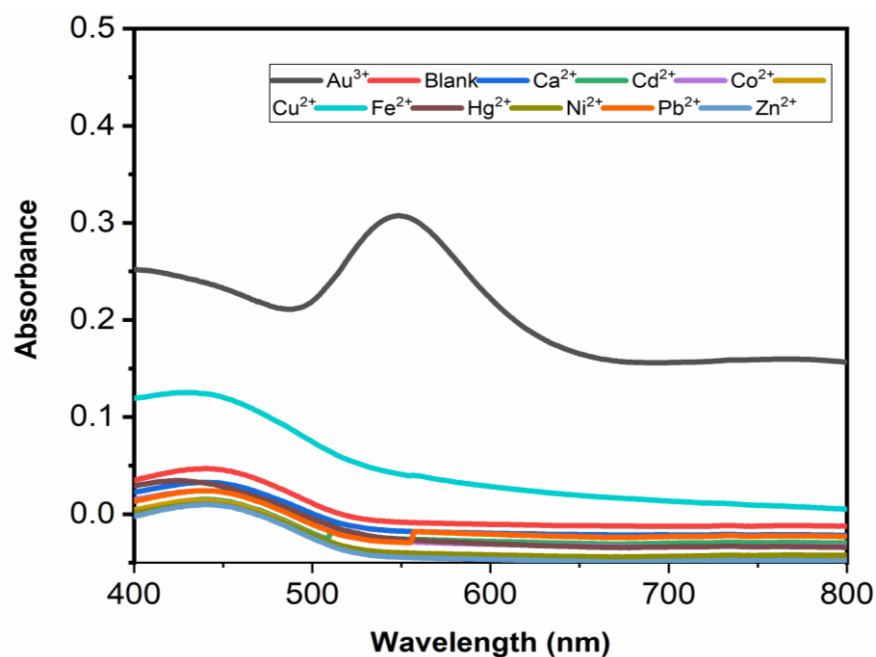


Fig. 2. UV-Vis absorption spectrum of (10^{-3} molL⁻¹) of receptor 3 and metal cation conc. 10^{-3} molL⁻¹ in DMSO+H₂O (1:4, v/v) solution at room temperature. In comparison the relative shifts of wavelengths with other metal cations, Au³⁺ ions, which does not show any influence.



Fig. 3. Photograph of change color observed when addition of Au³⁺ ions increase from 0.1 to 0.9 mL and keeping the concentration of **3** constant.

The detection limit for Au³⁺ ions using receptor **3** was determined to be 2.95×10^{-5} mol L⁻¹, which is comparable to, or better than, other colorimetric sensors reported in the literature. This low detection limit, coupled with the selective colorimetric response, makes receptor **3** an ideal candidate for practical applications in detecting trace amounts of Au³⁺ ions in aqueous environments.

3.6. Mechanism of Au³⁺ Ion Detection

The selective and sensitive detection of Au³⁺ ions by receptor **3** can be attributed to the unique combination of functional groups present in the receptor molecule. The benzo crown

ether moiety is known for its ability to complex with metal ions through coordination interactions, while the barbituric acid unit provides additional binding sites for metal ions through hydrogen bonding and electrostatic interactions. In the case of Au³⁺ ions, it is likely that the Au³⁺ cations coordinate with both the crown ether and barbituric acid groups, leading to a conformational change in the receptor that results in the observed color change.

Additionally, the high selectivity of receptor **3** for Au³⁺ ions over other metal ions may be due to the larger size and higher charge density of the Au³⁺ cation compared to other metal ions, which allows for stronger interactions with the receptor. This hypothesis is supported by the fact that other metal ions, such as Ca²⁺, Fe²⁺, and Co²⁺, did not induce any

noticeable color change or significant absorption in the UV-Vis spectrum.

3.7. Practical Implications and Future Prospects

The development of receptor 3 as a selective and sensitive colorimetric sensor for Au³⁺ ions has significant practical implications. The ability to detect Au³⁺ ions using a simple "naked-eye" colorimetric method makes this sensor ideal for

on-site detection in environmental and industrial settings. Moreover, the low detection limit of receptor 3 for Au³⁺ ions enables the detection of trace amounts of gold, which is important for monitoring gold pollution in water sources and for applications in gold mining and refining industries.

Future work could focus on improving the sensitivity of receptor 3 by modifying its structure to enhance its binding affinity for Au³⁺ ions. Additionally, the development of portable devices or test strips based on receptor 3 could further increase the utility of this sensor for field applications.

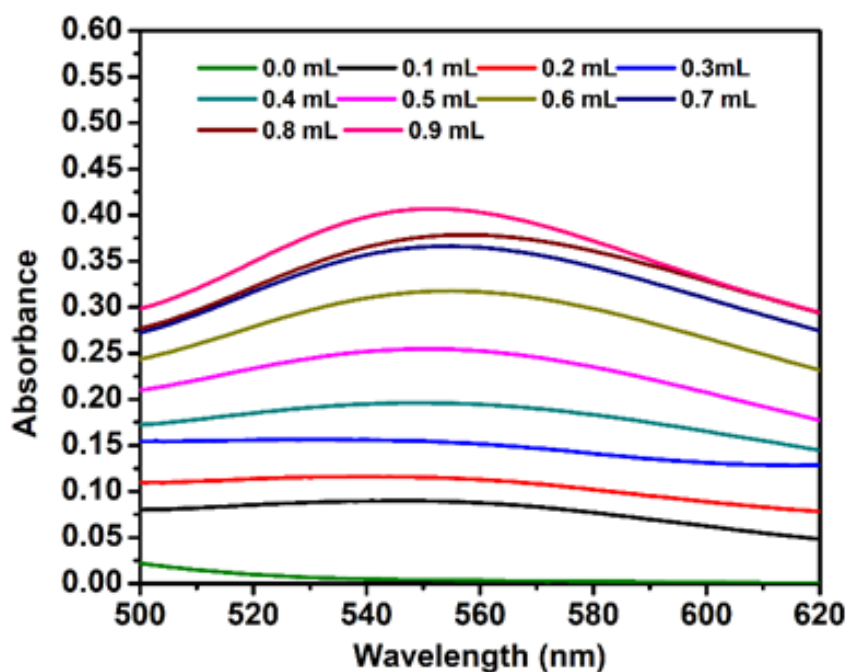


Fig. 4. Absorption spectra of receptor 3 (conc. 1×10^{-3} mol L⁻¹) at different concentrations of Au³⁺ ions.

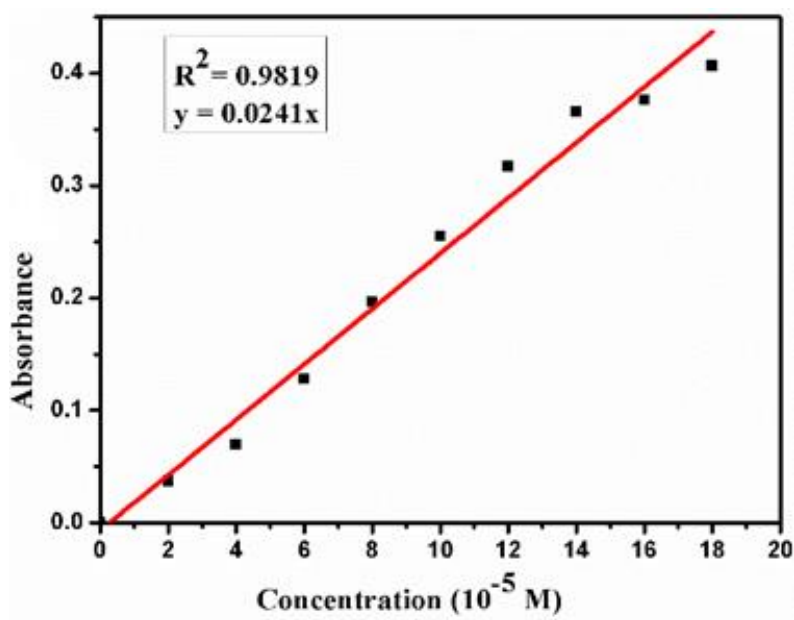


Fig. 5. A graph showing the linearity of absorbance of receptor 3 solution (10^{-3} mol L⁻¹) with increasing concentration of Au³⁺ ions from 0.1- 0.9 mL (10^{-3} mol L⁻¹).

4. CONCLUSION

In conclusion, we successfully designed and synthesized a novel naked-eye colorimetric sensor, receptor 3, for the selective detection of Au³⁺ ions. The sensor demonstrated remarkable selectivity and sensitivity toward Au³⁺ ions in a DMSO-water (1:4, v/v) solution. Upon interaction with Au³⁺, the solution of receptor 3 exhibited a noticeable color change from colorless to violet, which could be easily detected by the naked eye without the need for advanced instrumentation. This distinct colorimetric response was specific to Au³⁺ ions, as other metal cations did not induce any significant color change, further highlighting the sensor's selectivity. The UV-Vis spectroscopic studies confirmed the sensor's high affinity for Au³⁺, showing a characteristic absorption peak at 550 nm. Additionally, the sensor exhibited a linear response to increasing concentrations of Au³⁺ ions, with a detection limit as low as $2.95 \times 10^{-5} \text{ molL}^{-1}$, indicating its potential utility in practical applications for detecting gold ions in aqueous environments. Given its simplicity, cost-effectiveness, and high selectivity, sensor 3 presents a promising tool for real-time, on-site monitoring of Au³⁺ ions, especially in environments where sophisticated analytical instruments may not be readily available. The results demonstrate the feasibility of developing more efficient and practical sensors for heavy metal detection in environmental, biological, and industrial contexts. Further exploration could involve extending this design to detect other metal ions or pollutants, broadening the scope of its applications.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests.

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