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Synthesis, Structural Characterisation, and Sensor Application of a Copper (II)–Glycine Coordination Complex

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تخليق وتوصيف الهيكلي وتطبيق الحساسات لمركب تناسقي من النحاس (*II*) والجليسين

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Abstract:
Optical chemical sensors are promising tools for identifying metal ions in a wide variety of samples. Among metal ions, copper (II) has drawn substantial attention due to its toxic properties and its extensive use in numerous industrial processes. In this study, glycine was investigated as a recognition ligand for the optical detection of Cu(II) in aqueous solution. In the Cu (II)–Glycine system, a maximum absorption was observed at 641 nm. The influence of pH on the optical response was examined across a pH range of 2–14. Optical sensing showed excellent linearity between absorbance and the Cu (II) concentration, with correlation coefficients (R2) of 0.9975, along with Cu (II) limits of detection (LOD) of 1 ×10-4 mol dm-3. The sensor system also exhibited outstanding photostability (RSD 0.02 %) and reproducibility under continuous illumination, with negligible interference from common metal ions. Validation with spiked lake water samples confirmed the reliability of the proposed ligand as selective, reproducible, and practical optical sensors for detecting Cu(II) in environmental analyses.

Keywords: Solution structure; Optical chemical sensor; UV–Vis; Selective.

الملخص
تعد أجهزة الاستشعار الكيميائية البصرية أدوات واعدة لتحديد أيونات المعادن في مجموعة واسعة من العينات. بين أيونات المعادن، حظي النحاس (II) باهتمام كبير بسبب خصائصه السامة واستخدامه المكثف في العديد من العمليات الصناعية. في هذه الدراسة، تم دراسة الجلايسين كرابطة تعرف للكشف البصري عن Cu(II) في المحلول المائي .في نظام Cu(II)–جليزين، لوحظ أقصى امتصاص عند 641نانومتر. تم فحص تأثير الرقم الهيدروجيني على الاستجابة البصرية عبر نطاق pH من 2.–14أظهر الاستشعار البصري خطية ممتازة بين الامتصاص وتركيز Cu(II)، مع معاملات ارتباط (R2) تبلغ 0.9975، بالإضافة إلى حدود كشف الجليسين (LOD) تبلغ 1 ×10-4 مول dm-3 .كما أظهرت الحساسات استقرارا ضوئيا ممتاز(RSD 0.02%) وقابلية تكرار تحت إضاءة مستمرة، مع تداخل ضئيل من أيونات المعادن الشائعة. أكد التحقق باستخدام عينات مياه البحيرة المتدفقة موثوقية الليغاند المقترح كحساسات بصرية انتقائية وقابلة للتكرار وعملية لاكتشاف Cu(II) في التحليلات البيئية.

الكلمات المفتاحية: بنية الحل، المستشعر الكيميائي البصري ، الاشعة فوق البنفسجية، انتقائي.

Introduction

Heavy metal ions represent significant environmental pollutants that may enter soil, water, and air via natural processes and anthropogenic activities [1]. Heavy metals are metallic elements characterized by high relative densities (typically >5 g/cm3) and considerable atomic masses [2]. Assessing heavy metals in aquatic environments is of great interest because of their dangerous effects on the ecosystem and on human health, which depend on the dose and toxicity [3].Although they may occur in biotic environments at trace levels, their presence can still produce pronounced toxicity. Among the most harmful heavy metals are mercury (Hg), cadmium (Cd), arsenic (As), chromium (Cr), thallium (Tl), and lead (Pb), since their detrimental effects can arise even at very low concentrations. In contrast, less toxic heavy metals such as iron (Fe), copper (Cu), zinc (Zn), cobalt (Co), and manganese (Mn)—which are essential for human physiology and are widely used in various enzymatic processes—are likely to create serious health risks only when their concentrations exceed established thresholds [4]. Regulatory bodies including the World Health Organisation (WHO) and the European Union's Water Framework Directive (EU WFD) recommend a maximum allowable copper concentration of 2 mg/L in drinking water, whereas the U.S. Environmental Protection Agency (EPA) sets a secondary maximum contaminant level of 1.3 mg/L, [5]. optical sensors with excellent selectivity and sensitivity have been developed to detect copper (II) ions. The proposed sensor is designed to quantify the luminescence generated by changes in the refractive index that result from interactions between light and materials [6]. Optical sensors, commonly referred to as opt(r)odes, constitute a category of chemical sensors in which electromagnetic (EM) radiation is employed to generate the analytical signal through a transduction mechanism. The interaction between the radiation and the sample is evaluated by the change in a specific optical parameter, which is related to the analyte concentration (Blum, 1997). Typically, an optical sensor comprises a chemical recognition element a sensing component or receptor integrated with a transduction mechanism (**Figure 1**) [7].

Amino acids possess functional groups with the capacity to interact with divalent metal ions, a factor that plays an important role in promoting and sustaining their functionalities [8,9] .Copper (Cu(II)) is an essential trace element for humans and participates in many enzymatic reactions that are necessary for vital metabolic pathways and physiological functions [10]. Copper (Cu(II)) is the third most abundant ion among heavy metals and occurs across a variety of matrices, including water, rock, and soil sediments. It is an essential element not only for humans but also for virtually all living organisms [11]. Disrupted copper homeostasis has similarly been linked to several neurodegenerative disorders, including Wilson's disease, Alzheimer's disease, Parkinson's disease, and Menkes disease [12] . Glycine is the smallest proteinogenic amino acid and is classified as a non-essential amino acid. It contains an amino group and a carboxyl group and supports important functions in protein synthesis and multiple metabolic pathways. It includes a carbon atom bonded to two hydrogen atoms, an amino group, and a carboxyl group. It was first proposed as a neurotransmitter in the mammalian spinal cord in 1965. Glycine can be readily synthesized by the enzyme serine hydroxymethyl transferase from Serine can also be produced endogenously [13] . Under typical conditions, the concentration of glycine in human serum lies between 200–400 μM [14] . Glycine functions as an essential substrate for the synthesis of several biologically important biomolecules and compounds, including glucose, creatinine, porphyrin, and purine nucleotides, as well as neurotransmitters. It is also a component of bile acids and glycocholic acid. It contributes to the formation of proteins, the tripeptide glutathione, and detoxification reactions [15] . Glycine is a non-essential amino acid synthesized by animals, microorganisms, and plants. Glycine is also of critical importance for the synthesis of macromolecules such as hemoglobin and myoglobin. Schematic overview of glycine metabolism. Glycine may be formed from serine through serine hydroxymethyl transferase, or from ammonia, carbon dioxide, and tetrahydrofolate. The last step is catalysed by glycine synthetase, which is part of the glycine cleavage system in the liver. Alternatively, glycine can be acquired from the diet. THF: tetrahydrofolate; MTHF: methylene tetrahydrofolate (**Figure 2**) [16] . In general, glycine is generated from choline, serine, hydroxyproline, and threonine through interorgan metabolism, with the kidneys and liver serving as the main organs involved. Glycine has essential functions in the metabolism and nutrition of many mammals and humans[17] . A variety of analytical methods have been established to detect metal ions, including anodic stripping voltammetry, electrochemical analysis, atomic absorption spectrometry (AAS), fluorescence sensing, inductively coupled plasma atomic emission spectrometry (ICP-AES), cold vapour atomic absorption spectroscopy, and inductively coupled plasma mass spectrometry (ICP-MS). Although these techniques provide high sensitivity and accuracy, their routine application is often limited by high instrumentation costs and operational complexity. In contrast, spectrophotometric methods remain widely preferred for the analysis of aqueous metal ions because they are simple, economical, and easy to operate [18]. The primary aim of this study was to examine Glycine as a recognition ligand for the optical detection of Cu (II) in aqueous solution. The research focuses on designing and fabricating novel optical chemosensors with high sensitivity, optimized for the selective detection of Cu (II) ions.

Material and methods

All chemicals and reagents used in this study were of analytical grade and were employed without any further purification. Glycine was obtained from Sigma-Aldrich. The remaining chemicals were also purchased from Sigma and used without additional purification. Stock solutions of the metal ions were prepared from analytical-grade reagents, and their concentrations were verified using titrimetric methods.[19].

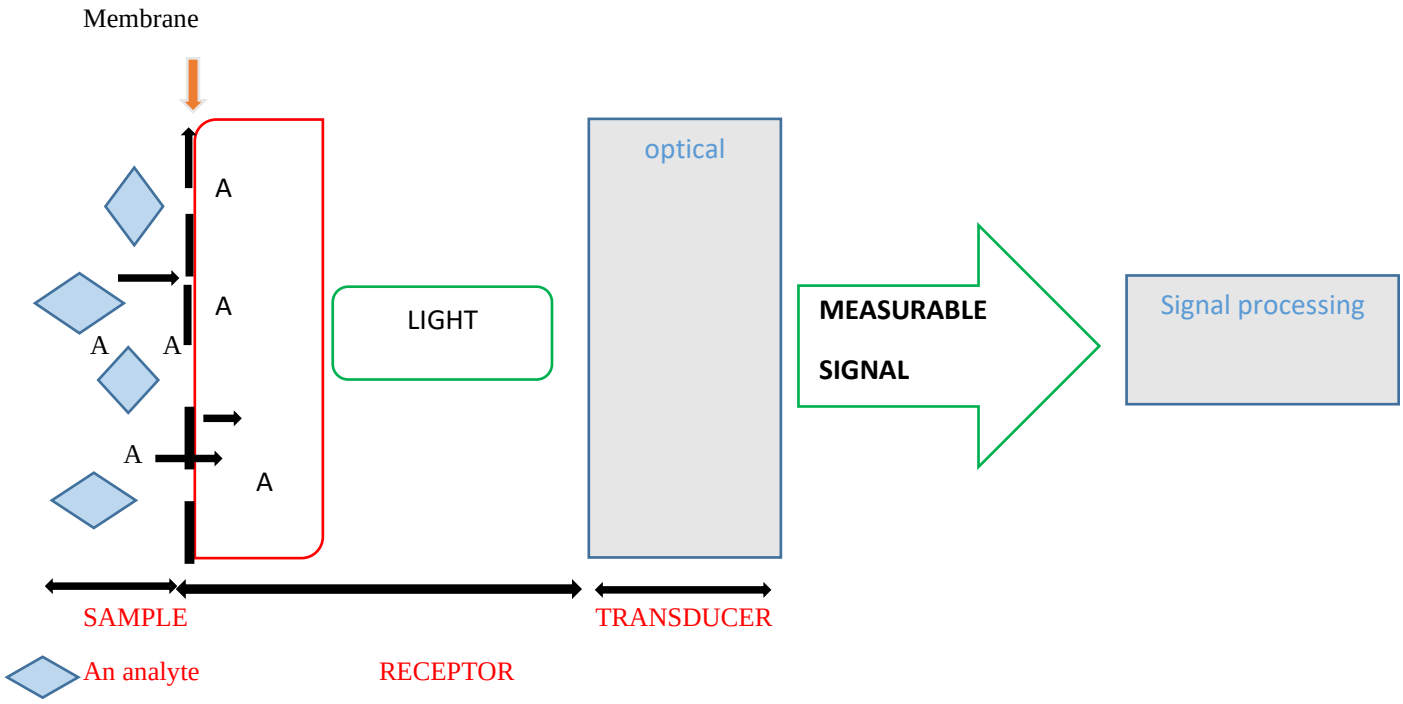

Figure 1 Schematic representation of the composition and function of an optical chemical sensor.

Measurements

The electronic spectra of the Cu (II) complexes were obtained using a The SPECORD R 210 PLUS spectrophotometer (Analytik Jena, Germany), which was set to measure over the wavelength range from 200 to 800 nm. All solutions were prepared freshly, with deionized water used for the metal and the ligand. The concentrations of both the ligand and the metal were held at 0.01 M. Measurements were carried out in aqueous media across the pH range of 2.00 to 12.1, using 10 mm pathlength cuvettes in the wavelength regions of 250 to 800 nm. An uncoordinated ligand was used as the blank reference [20].

Cu(II) Optimisation Reaction of the Sensors

To assess the complexation behavior of the Cu (II) complexes and the optimal sensing conditions, their UV–visible absorption spectra were measured in aqueous solution. Spectrophotometric monitoring of the complex formation between Cu (II) and the synthesized ligand Glycine was performed at room temperature. Under constant ionic strength, ligand solutions were titrated with different amounts of Cu (II) ions, and the absorption spectra were recorded after each addition. To determine the optimal detection wavelength for each ligand, the wavelength corresponding to the maximum absorbance (λ_{max}) was calculated. To establish the optimal pH for complex formation, the effect of pH on absorption intensity was evaluated over a pH range of 2 to 14. All measurements were carried out at 25 °C after adjusting the pH using diluted HCl and NaOH solutions. The stoichiometric ratio of Cu (II) to ligand in the complex for each ligand was determined using Job's method of

continuous variation. While maintaining a constant total molar concentration, the mole fraction of Cu (II) was systematically varied. By plotting the absorbance values against the mole fraction of Cu (II), the complex's stoichiometry was determined at the maximum of the curve. To assess the sensor's selectivity, the effects of potentially interfering ions such as Ca (II), Cd (II), Co (II), and Ni (II) on Cu (II) complexation were examined as well using a fixed Cu (II) concentration. The response time and stability of the sensing process were evaluated by monitoring the time dependent absorbance changes that occurred after the addition of Cu (II).

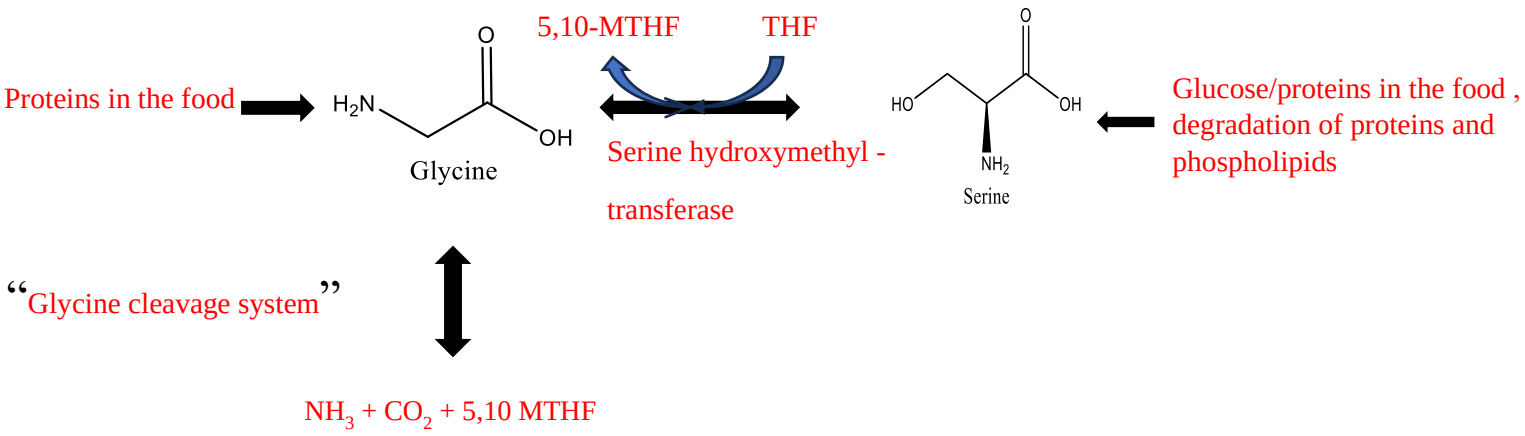

Figure 2 Overview of Glycine biosynthesis and degradation pathways.

Results and discussion

The occurrence of the electronic absorption transition in the compounds is indicated by the emergence of absorption peaks for glycine, Cu (II), and the Cu (II)–glycine complex in 0.01 M deionized water. The wavelength range of this absorption spectrum spans 200–800 nm. The UV–vis spectra of glycine, Cu (II), and the Cu (II)–Glycine complex display three bands. The formation of the complex is further supported by the amino acid UV–vis spectra (**Figure 3**), which present ligand absorption spectra of similar character that are shifted to lower wavelengths, along with an increase in intensity or the appearance of new peaks attributable to the d–d transition.

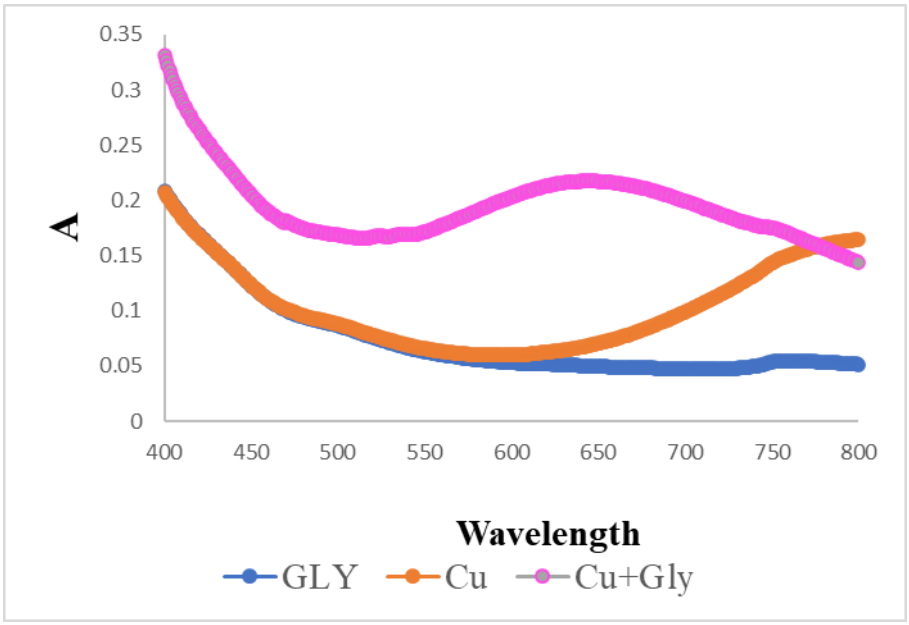

Figure 3 UV-Vis absorption spectra of Glycine solution 0.01M, Cu (II) solution 0.01M, and Cu (II) with Glycine, phosphate buffer pH 10.08, with a λ_{max} of 641 nm.

The effect of pH has been investigated across a range from 2 to 14, using a 0.25 M sodium hydroxide solution. In the acidic pH region, the absorption values of Glycine were minimal, as illustrated in (**Figure 4**), and no absorption peak was observed . Upon increasing the pH, the absorption values exhibited a gradual rise, culminating in the highest absorption value of 0.2174 at pH 12.08. The maximum absorption intensity was observed at a wavelength of 641 nm, with absorbance values varying at this wavelength across different pH levels [21].

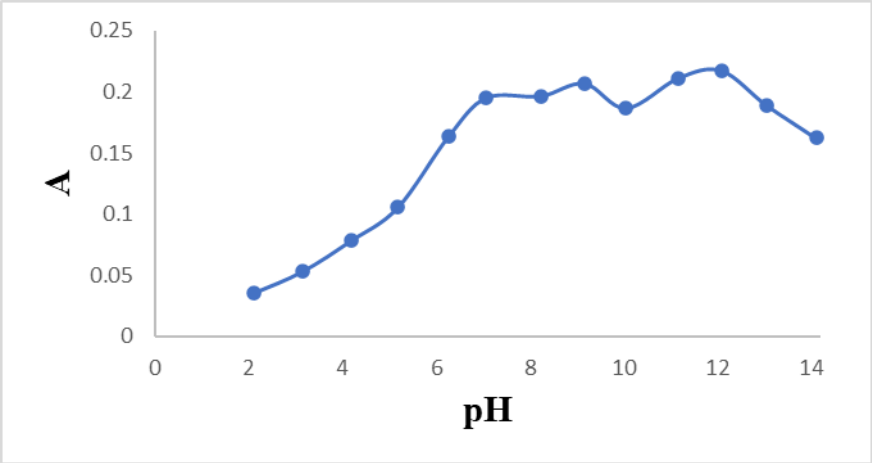

Figure 4 The effect of working pH on the absorbance of Glycine upon reaction with 0.01 M Cu (II) solution.

By recording absorbance at a fixed wavelength of 641 nm, the influence of glycine concentration on the formation of the Cu (II)-Glycine complex was meticulously investigated. As illustrated in (**Figure 5**), the optimal absorbance for the Cu (II)-Glycine complex demonstrated a proportional increase with glycine concentrations, within the range of 1×10^{-4} M to 0.2 M. Preliminary studies indicated a significant enhancement in the absorbance signal alongside increasing concentrations; however, this reaction gradually stabilised at higher levels, achieving saturation at 0.08 M. An amplified signal was observed with the application of greater concentrations of Glycine, facilitating more extensive interactions between Glycine ions and Cu (II) in the proximal phase. Ultimately, the absorbance signal reached a plateau as Cu (II) ions fully occupied the majority of Glycine binding sites.

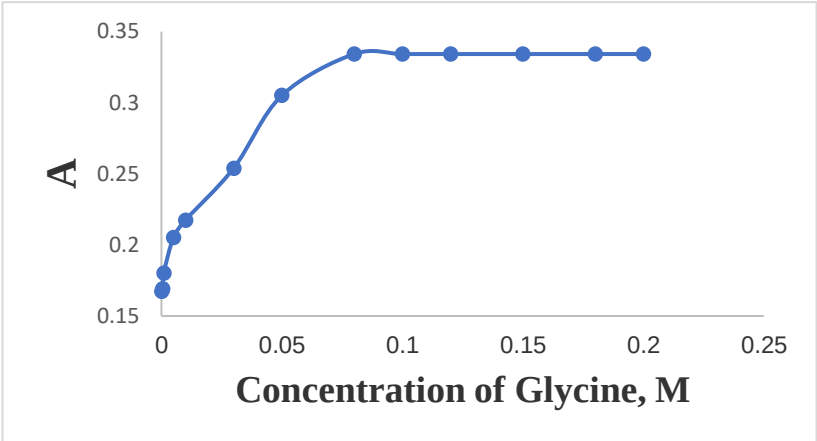

Figure 5 The effect of Glycine concentration on the response of the Cu (II)-Glycine complex when Cu (II) concentration used was fixed at 0.01 M

The detector was subjected to continuous exposure to the light source for a duration of six hours in order to evaluate the photostability of the Glycine solution, as reported in recent research conducted by Rashd et al. The results pertaining to photostability were deemed satisfactory, yielding a relative standard deviation (RSD) value of 1.06% [22] . The reaction conditions were optimised for laboratory temperature and light exposure. As illustrated in (**Figure 6**), a relative standard deviation (RSD) of 0.02% was recorded at a Glycine concentration of 0.08 M. This observation indicates that the detector phase within the solution remained stable and was not adversely affected by light exposure..

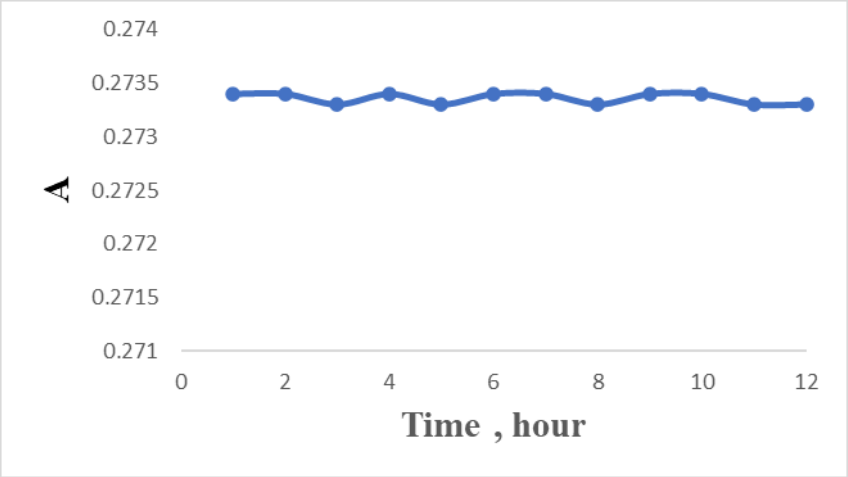

Figure 6 The photostability of the Glycine with concentration of 0.08 M against Time.

Seven measurements of the solution response were obtained through an experimental procedure involving various samples with differing concentrations of Cu (II) and Glycine at 0.01 M and 0.08 M, respectively, to conduct a repeatability study (**Figure 7**). This process resulted in a relative standard deviation (RSD) value of 0.07%. Furthermore, the optical pH sensor, characterised by a film thickness of 300 nm, facilitated a wide linear dynamic range for solution pH (5.02–8.54), demonstrated excellent repeatability with a minimal (RSD) below 1.5% over 20 consecutive cycles, and exhibited reversibility, with an RSD below 1.5% across the same number of cycles, as reported by Wenying Shi *et al* [23].

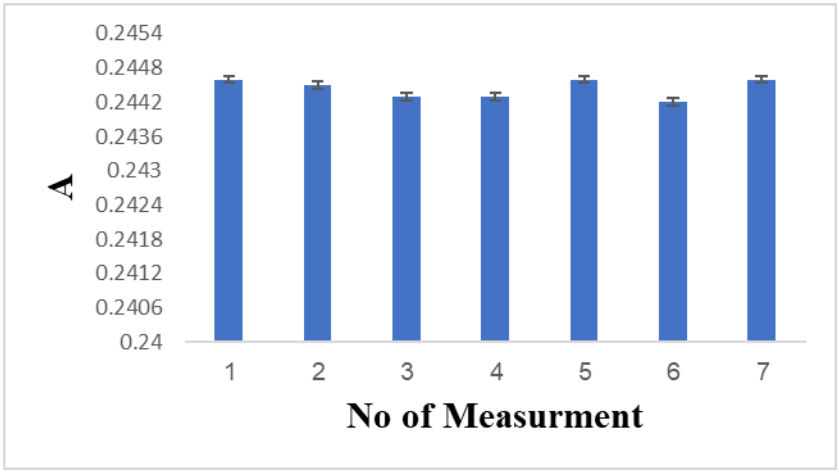

Figure 7 The reproducibility of the reagent Glycine 0.08 M Cu (II) analyte.

The graphical representation in (**Figure 8(a)** elucidates the response characteristics of the sensor. The figure demonstrates that the signal generated by the spectrophotometer increases significantly with rising concentrations of Cu (II). In the initial experiment, an increase in Cu (II) concentration resulted in the response stabilising and ultimately reaching saturation at 0.18 M Cu (II). Elevated Cu (II) concentrations facilitated stronger interactions between the reagent and analyte particles in the adjacent phase, thereby producing a more intense signal. Throughout these investigations, the concentration of Glycine was maintained at 0.08 M , while the Cu (II) concentration varied from 1×10^{-4} M to 0.26 M. The graphical representation indicates a linear relationship between Cu (II) concentration and absorption across the concentration range from 1×10^{-4} M to 0.08 M. (**Figure 8(b)** illustrates a linear correlation ($R^2 = 0.997$) between the relative absorbances.

Previous studies have documented the development of a multiparametric optical sensor employing a porous thin film composed of a chitosan–silica nanocomposite (CSNC), which facilitates efficient glucose detection at pathological concentrations[24]. The resulting sensor demonstrated exceptional sensitivity, selectivity, and reproducibility, achieving a limit of detection (LOD) of 0.76 mM. Furthermore, it sustained consistent performance across a broad dynamic range of 3–30 mM and was capable of quantifying glucose concentrations both below and above the physiological threshold of 17 Mm.

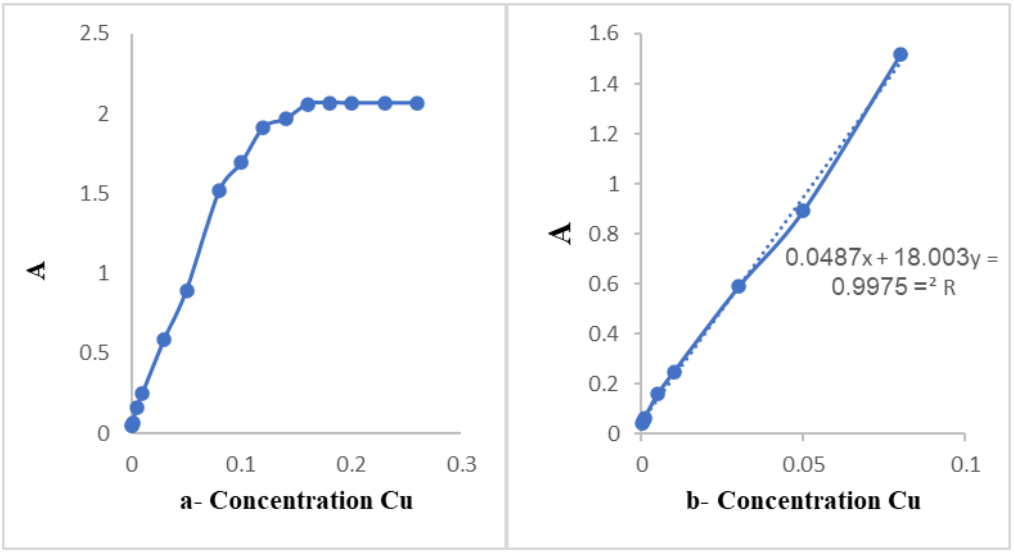

Figure 8 The response curve of the Glycine at different concentrations of Cu(II) (**a**) and is the linear dynamic range of the Cu(II) concentration (**b**).

This study systematically examined the possible interference caused by different ions in the determination of Cu (II). (**Figure 9**) shows the magnitude of interference observed for several foreign ions—specifically Cd (II), Co (II), Ni (II), and Ca (II)—using a molar ratio of 1:1 for Cu (II) to each foreign ion. Even so, the value obtained for Ca(II) was markedly lower. When these results are compared with those reported for Cu(II), it becomes clear that these foreign ions do not interfere with the detection of Cu (II).

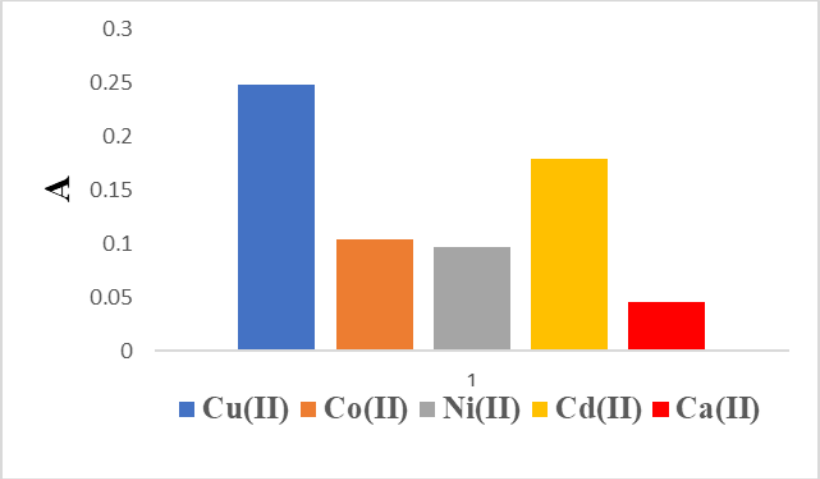

Figure 9. The degree of interference of the Glycine at 1:1 molar ratio of Cu (II): foreign ion**.**

Furthermore, an experiment was conducted employing the sensor developed in this study to measure the concentration of Cu (II) in lake water samples, aimed at validating the device's performance, particularly in the analysis of real samples. The sensor's readings were evaluated against the established flame Atomic Absorption Spectroscopy (AAS) method, demonstrating concordance with the standard procedure.

Table 1: Determination of Cu(II) using the standard flame AAS method and the optical Cu(II) sensor based on Glycine.

SN.	Standard Cu(II) solution (ppm)	Cu(II) determined by flame AAS (ppm)	Cu (II) determined by optical sensor (ppm)
1	Real Sample **Glycine**	**0.118M**	**0.02M**

Conclusion

In this study, a mixed amine/carboxylic-based ligand, glycine, was successfully assessed as an optical chemical sensor for the detection of Cu (II) ions. After complexation, the systems produced distinct spectral responses, characterized by strong absorption bands in the visible region and excellent linear correlations between absorbance and the Cu (II) concentration. The sensors displayed low detection limits (LOD $1*10^{-4}$), high reproducibility, and outstanding photostability, thereby supporting their analytical reliability. Comparative analysis indicates that the derivative yields slightly higher sensitivity and lower detection limits, while also providing improved photostability and enhanced operational robustness. The sensors exhibit favorable photostability and reproducibility, with low relative standard deviation values of 0.02% and 0.07%, respectively. Selectivity experiments confirmed negligible interference from competing metal ions, and validation with real samples demonstrated excellent recovery values (0.02M) for lake water specimens, in agreement with the results obtained by flame AAS. Overall, amino-acid derivatives show strong promise as optical Cu (II) sensors, With practical value for environmental monitoring and analytical chemistry, their stability, selectivity, and reproducibility make them promising candidates for the development of portable, field-deployable optical sensing devices.

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Compliance with ethical standards

Disclosure of conflict of interest

The authors declare that they have no conflict of interest.

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