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Spectrophotometric Detection of Nickel(II) Using Dimethylglyoxime as a Selective Optical Sensor

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ABSTRACT

Owing to their remarkable selectivity, sensitivity, and rapid detection capabilities, optical sensors have garnered significant scholarly attention for the identification of various metal ions. Given its toxicological implications and pervasive application across numerous industrial processes, nickel(II) has become a focal point of research among metal ions. In recent years, a variety of optical chemical sensors exhibiting exceptional sensitivity and selectivity have been developed specifically for the detection of Ni(II). In the present study, a dimethylglyoxime(DMG) optical chemical sensor is employed to quantify trace amounts of Ni(II) using spectrophotometric techniques under controlled pH conditions. resulting in well-defined Ni(II) complex with characteristic absorption maxima at 568 nm. Optical sensing showed excellent linearity between absorbance and Ni(II) concentration, with correlation coefficients (R^2) of 0.9952. and limits of detection (LOD) of 1×10^{-4}. DMG has emerged as an effective effective reagent for the detection of Ni(II) ions in a range of aqueous samples functioning as an optical chemical sensor.

1 Introduction

The advancement of selective chemical sensors has garnered significant scholarly interest over the last twenty years due to their potential applications in environmental surveillance, as they offer a swift, precise, and cost-effective means of analysis Jain et al [1997]. The efficacy of chemical sensors that respond selectively to specific ions across various matrices such as soils, industrial effluents, pharmaceuticals, and clinical analyses is firmly established. This methodology is characterized by its rapidity, precision, cost-effectiveness, and the requirement of minimal, non-destructive sample volumes for analysis Singh et al [2003]. Nickel is recognized as a deleterious element that is capable of inducing malignancies in the nasal and pulmonary regions, as well as causing dermatitis, asthma, and impairments of the central nervous system. Consequently, in light of the pressing necessity for the selective detection of nickel(II) in a variety of industrial, environmental, and alimentary matrices, numerous recent publications have emerged in the scientific literature Azizi et al [2006].In 1990,the International Agency for Research on Cancer (IARC) designated nickel compounds as group 1 carcinogens to humans. Consequently, an array of methodologies has been recently documented for the quantification of nickel(II) ion concentrations in diverse biological, industrial, and food samples Jiang et al [2012]. Nickel, one of the most widespread heavy transition metals,It also constitutes a crucial element in sustaining the specific functionality of numerous enzymes engaged in the metabolism of carbohydrates, amino acids, and lipids in both prokaryotic and eukaryotic organisms . Nickel ions, when present at low concentrations, serve as vital trace elements; however, they may manifest toxicological

Corresponding author: E-mail: rashd.elferjani@uob.edu.ly effects under conditions of excess Shen et al [2017]. Therefore, the assessment of nickel levels is of paramount importance due to its toxicological properties and its ubiquitous occurrence in the environment. The quantification of trace nickel within aquatic and environmental matrices presents significant challenges attributable to various factors, particularly its low concentrations and the influence of matrix effects Henden et al [2012]. Spectroscopy constitutes a methodological approach that quantitatively assesses the interactions between molecules and electromagnetic radiation Upstone et al [2006]. Transition metal complexe exhibit a captivating array of color. The electronic spectra associated with these complexes can yield significant insights pertinent to their bonding characteristics and structural configuration Huheey et al [2006].

The ultraviolet and visible spectra of coordination compounds involving transition metals are characterized by electronic transitions occurring between the d orbitals of the metallic constituents. Such transitions are commonly designated as d-d transitions, as they predominantly engage molecular orbitals that are chiefly of d character associated with the metal. Dimethylglyoxime(DMG) is presently recognized as the preeminent reagent for the quantitative analysis of nickel Adams et al [1918] .Dimethylglyoxime serves as one of the most frequently employed ligands in the coordination of nickel ions, facilitating the formation of nickel dimethylglyoxime complexes owing to its remarkable sensitivity and stability. This characteristic has been effectively utilized in the identification of nickel ions in natural water sources Cardoso et al [2009]. Furthermore, given that dimethylglyoxime exhibits solubility in ethanol, it can be uniformly amalgamated with an ethanolic solution of Nafion Ferancova et al [2017].In optical sensor for Ni(II) requires a ligand with high sensitivity and selectivity for Ni(II). In the design of such a ligand DMG in the **Fig 1**, it is recognised that Ni(II) prefers to coordinate to oxigen donor ligand rather than nitrogen donor ligands.

2 Materials and Methods

All chemicals and reagents utilized in this study were of analytical grade and were employed without any additional purification. Dimethylglyoxime(DMG) was procured from Sigma-Aldrich, with its purity confirmed through chromatographic and potentiometric analyses. All remaining chemicals were sourced from Sigma and were applied without further purification. The stock solutions of metal ions were formulated from analytical grade reagents, and their concentrations were verified through titrimetric methods Antony et al [1990].

2.1. Measurements

The electronic spectra of the Ni(II) complexes were recorded utilizing The SPECORD R 210 PLUS spectrophotometer (Analytik Jena, Germany), configured to operate within the wavelength spectrum of 200 to 800 nm. All solutions were freshly prepared employing deionized water for metal and ethanol for ligand. The concentrations of both the ligand and metal were maintained at 0.03 M. Measurements were conducted in aqueous solutions within the pH range of 2.00 to 12.1, utilizing 10 mm pathlength cuvettes across the wavelength regions of 250 to 800 nm. An uncoordinated ligand served as the blank reference.

2.2 Ni(II) optimisation reaction of the sensor

All chemicals and reagents used in this study, including $NiCl_2$. $6H_2O$,L_Dimethylglyoxim (DMG), deionised water, ethanol, and phosphate buffer, (BDH or Aldrich). Standard Ni(II) solution of 0.2 M was prepared by dissolving an adequate amount of 2.592g in 100 ml deionised water. (DMG) standard stock solution of 0.14 M was prepared by dissolving required amount of 0.812g in 50 mL ethanol. The influence of the concentration of DMG on Ni(II) was investigated by amalgamating an identical concentration of Ni(II) solution with varying quantities of L-Dimethylglyoxime DMG at diverse concentrations within the spectrum of 1×10^{-3} to 0.14M. Subsequent to this, all samples were subjected to analysis via a UV-visible double-beam spectrophotometer for the purpose of measuring absorption. The reproducibility of the sensor's response was assessed by executing seven independent trials utilizing a 0.03M Ni(II) concentration. An evaluation of the photostability of the sample was performed. The concentration of DMG employed for this investigation was established at 0.05M. This identical concentration was maintained for a duration of six hours, during which the absorption intensity was documented at thirty-minute intervals. The absorption spectrum for each mixture was recorded utilizing a UV-visible spectrophotometer. The effects of various foreign ions were examined separately, utilising differing quantities of interfering ions to ascertain Ni(II) concentration at 0.03 M. The interference levels of Cu(II), Fe(III), and Co(II) ions were evaluated in both the presence and absence of these interfering ions, employing an optimisation strategy.

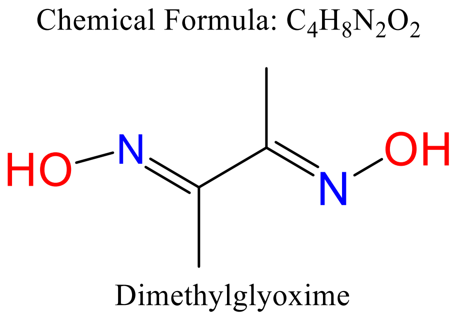


Fig. 1. Chemical structure of dimethylglyoxime (DMG)

3- Results and Discussion

The elucidation of the absorption peaks associated with Dimethylglyoxime DMG, Ni (II), and the Ni (II)-DMG complex in a 0.03 M deionized aqueous medium indicates the manifestation of electronic absorption transitions of these compounds. The wavelength range of this absorption spectrum spans from 200 to 800 nm. Within the UV-vis spectra of DMG, Ni (II), and the Ni (II)-DMG complex, three distinct bands are observable. The UV-vis spectra depicted in **Fig 2** of the amine group reveal analogous ligand absorption characteristics that are shifted towards lower wavelengths, accompanied by an increase in intensity or the emergence of peaks due to charge-transfer transitions, thereby corroborating the formation of the complex.

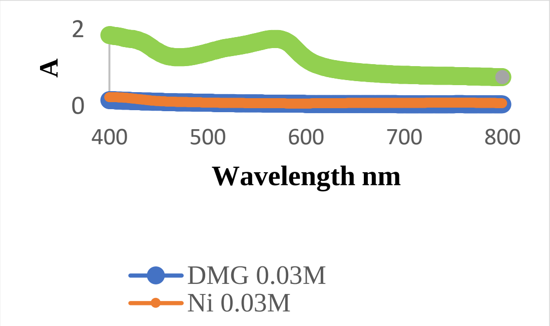


Fig. 2. UV–Vis absorption spectra of DMG solution 0.03M, Ni (II) solution 0.03M and Ni (II) with DMG.

The impact of pH on the formation of the complex was meticulously examined in a 0.20 M sodium hydroxide solution across a spectrum of pH values. **Fig 3** elucidates the pivotal significance of pH in relation to the stability and persistence of the resultant product. The absorption values demonstrated a noticeable upward trajectory trend within the acidic pH range .In contrast, the absorption values associated with pH were systematically quantified across a wavelength spectrum of 200-800 nm, elucidating a distinctive absorption peak at a pH level surpassing 7, with the peak absorption value reaching 4.259 at a pH of 9.09. Moreover, the highest absorption intensity for the ligand DMG complex was recorded at a wavelength of 568 nm, wherein the absorbance measurements exhibited variability in relation to the alterations in pH.

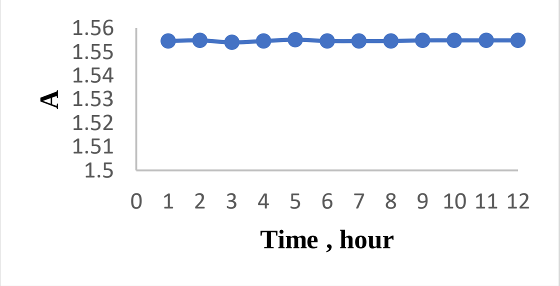
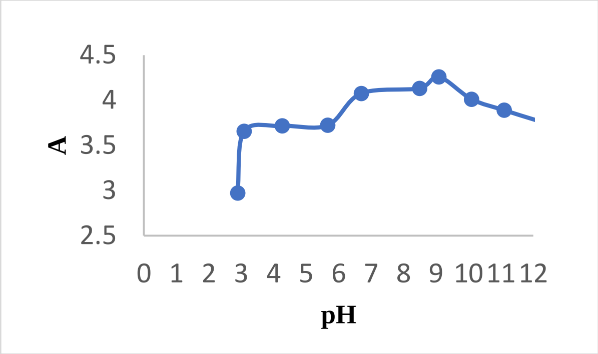


Fig. 3. The effect of working pH on the absorbance of DMG upon reaction with 0.03 M Ni (II) solution.

The influence of DMG concentration on the formation of the Ni (II)- DMG complex was rigorously examined by assessing absorbance at a fixed wavelength of 568 nm. The maximal absorbance for the Ni(II)-DMG complex solution was ascertained within a reagent concentration range extending from $1*10^{-3}$ to 0.14 M, as depicted in **Fig 4**. Within the concentration range of $1*10^{-3}$ M to 0.14 M, absorbance exhibited a direct increase corresponding to the rising concentrations of DMG. Preliminary analyses revealed a significant enhancement in the absorbance signal corresponding with elevated concentrations; nevertheless, this response progressively approached a state of equilibrium at higher levels, ultimately culminating in saturation at a concentration of 0.05 M.



Fie. 4. The effect of DMG concentration on the response of the Ni (II) DMG complex when Ni(II) concentration used was fixed at 0.03 M with buffer solution of pH 9.09.

The reagent was subjected to continuous illumination from the light source for a duration of six hours to facilitate the photostability investigation of the DMG and Ni (II) solution depicted in **Fig 5**. The reaction was performed in a laboratory environment characterized by regulated temperature and lighting conditions. At a concentration of DMG measuring 0.05 M, a relative standard deviation (RSD) of 0.01% was determined. This finding implies that the reaction phase of the solution demonstrated stability under light exposure.

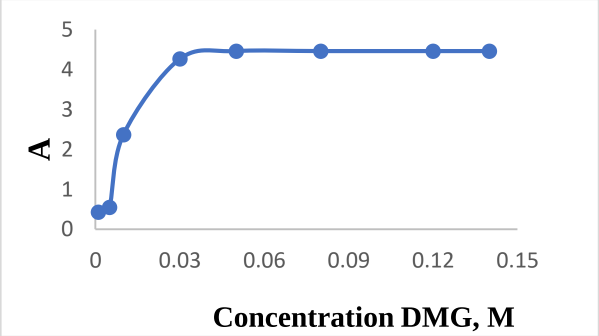


Fig. 5. The photostability of the DMG with concentration of 0.05 M against Time.

The reproducibility study, illustrated in **Fig 6**, focused on the solution's response and was conducted by obtaining seven measurements from distinct experimental batches, with each batch maintaining consistent concentrations of DMG 0.05M and Ni(II) at 0.03 M. This experimental approach yielded a relative standard deviation (RSD) of 0.003%. Moreover, an optical pH sensor, designed with a film thickness of 300 nm, displays a wide linear dynamic range for pH assessments of solutions, extending from 5.02 to 8.54.The data further illustrates a notable degree of repeatability, evidenced by a relative standard deviation (RSD) of less than 1.5% over the course of 20 consecutive cyclesas well as reversibility, whereby comparable RSD levels are upheld throughout multiple testing cycles Shi et al [2010].

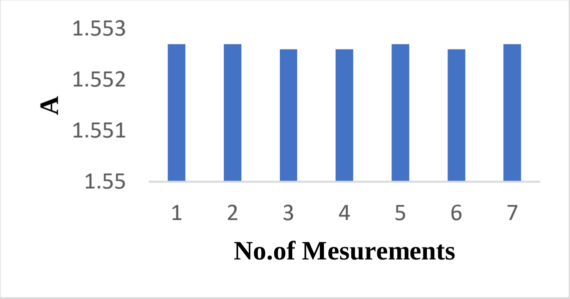
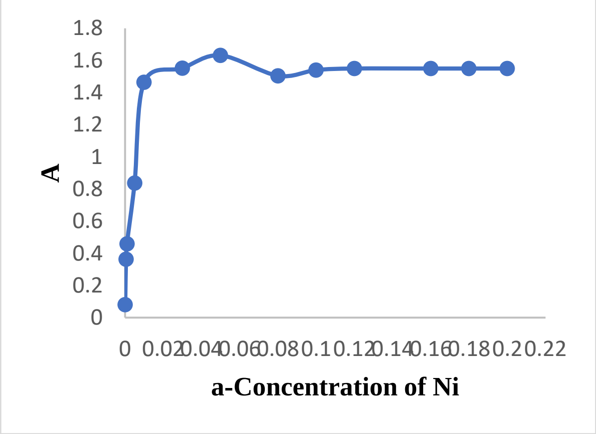


Fig. 6. The reproducibility of the reagent DMG 0.05 M Ni (II) analyte.

Fig 7 elucidates the response curve associated with the sensitive material. The signal recorded by the spectrophotometer exhibited a significant increase in correlation with the rise of Ni (II) concentrations during the initial phase of the study, which was subsequently followed by a gradual stabilization at heightened concentrations and eventual saturation at 0.12 M Ni(II). An elevated concentration of Ni (II) promoted a greater number of interactions between the reactant and analyte molecules, resulting in an intensified observed signal. Throughout these studies, the concentration of DMG was consistently maintained at 0.05 M.**Fig 7**(a) illustrates the variation in Ni (II) concentration across the range from $1*10^{-4}$M to 0.2 M.In conjunction,

Fig 7(b) delineated a linear relationship between absorbance and Ni (II) concentration within the range of 0.0001 M to 0.01 M, demonstrating a linear regression characterized by a coefficient of determination of $R^2 = 0.9952$. Nickel foam, modified through the incorporation of hollow sphere $NiCO_2O_4$ particles, was effectively synthesized via the hydrothermal technique utilizing nanosphere SiO_2 particles as the rigid templates for the hollow architecture. The examination of the $NiCo_2O_4$-modified nickel foam as an electrode for glucose detection in a sodium hydroxide solution demonstrated a significant linearity in the anodic currents (R^2=0.99) within the concentration spectrum of 0 – 2.5 µM, accompanied by an estimated limit of detection of 0.18 µM, the proposed sensor exhibits substantial applicability for the detection of real samples utilizing blood plasma, thereby rendering it exceptionally appropriate for practical application. The findings suggested that the material holds significant potential for the continued advancement of the nickel-based sensor Eprilia et al [2024].

Furthermore, the influence of various ions on the determination of Ni(II) was meticulously investigated in this study. **Fig 8** encapsulates the extent of interference quantified for several extraneous ions, including Fe(III), Co(II), and Cu(II), at a molar ratio of 1:1 between Ni(II) and the foreign ion. The data illustrated in the curve indicates that the measured values for Fe(III) and Co(II) ions were relatively comparable, whereas the Cu(II) ion exhibited a markedly reduced value.



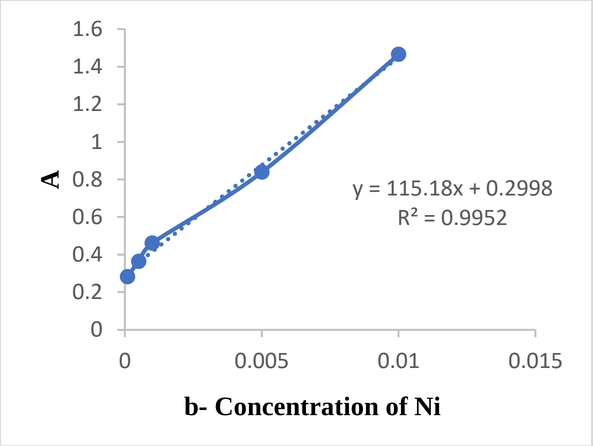


Fig. 7. The response curve of the DMG at different concentration of Ni (II) (a)and is the linear dynamic range of the Ni (II) concentration (b).

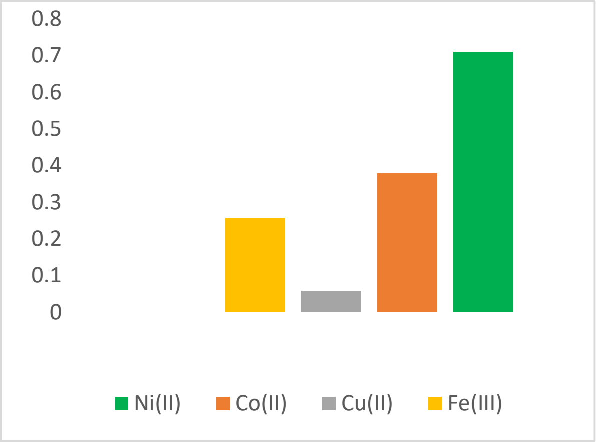


Fig. 8. The degree of interference of the valine at 1:1 molar ratio of Ni (II): foreign ion.

3 Conclusions

In summary, optical chemical sensors have demonstrated remarkable potential as highly sensitive analytical tools. DMG was observed to form various Ni(II) complexes in the solution, DMG was investigated in developing an optical sensing material for Ni(II) has been examined in this study. The colour intensity of the adduct increases proportionally with the increase in substrate concentration. The sensor has good photostability and reproducibility features. The solution used for operation is maintained at pH 9.09. The reproducibility study showed a good RSD value of 0.003%. The photostability test gave an RSD of 0.01% indicated no significant photobleaching upon exposure to ambient light, Fe(III), Co(II), and Cu(II) with their effect during Ni(II) determination was also examined in this work; the result showed these ions did not interfere during Ni (II) detection by using DMG.

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Conflict of 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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