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UV–Visible Spectrophotometric Determination of Ni(II) Using a Schiff Base Ligand Derived from 1,5-Diphenylcarbazone and Aniline

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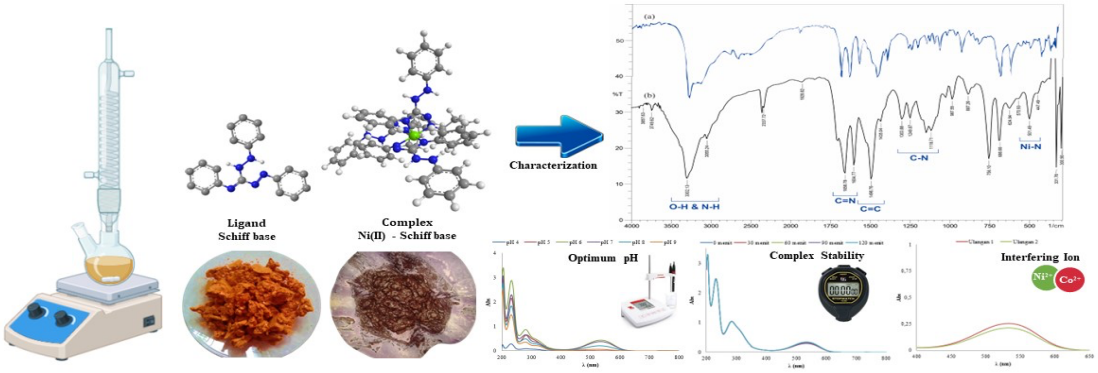
GRAPHICAL ABSTRACT



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ABSTRACT

UV–Vis spectrophotometric method for the determination of Ni(II) was developed based on complex formation with a Schiff base ligand synthesized from 1,5-diphenylcarbazone and aniline. The ligand was obtained in 64.98% yield and exhibited absorption bands at 239 and 278 nm, confirming the formation of an azomethine. Complexation at a metal to ligand molar ratio of 1:3 produced a Ni(II)–Schiff base complex with a maximum absorption wavelength (λ_{max}) at 532-535 nm in the visible region. The complex exhibited optimum performance at pH 6 and remained stable for 120 min. FTIR analysis supported coordination through a shift of the C=N stretching band to lower wavenumbers and the appearance of Ni–N vibrations. Comparative evaluation demonstrated that the crystallized complex provided higher analytical sensitivity at low concentration than the non-crystallized form. Statistical validation using Student's *t*-test at the 95% confidence level indicated no statistically significant effect of Co(II) on the absorbance response ($t_{cal} < t_{tab}$). This enhanced sensitivity, obtained through controlled crystallization, is the principal advantage of the proposed approach over conventional direct spectrophotometric complexation methods. Overall, the developed system provides a simple, reproducible, and analytically reliable method for the determination of Ni(II) in aqueous media.

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1. INTRODUCTION

Nickel (Ni) is recognized as a significant heavy metal contaminant in aquatic environments due to its persistence in natural waters and its tendency to bioaccumulate in aquatic organisms [1, 2]. Moreover, nickel poses considerable ecological and toxicological risks in areas influenced by anthropogenic activities [3], particularly those related to mining [4], electroplating [5], alloy manufacturing [6], and battery production [7]. Although nickel is required in trace amounts for certain biological functions [8], excessive exposure has been linked to adverse health effects, including respiratory disorders [9], allergic reactions [10], and carcinogenicity [11]. These environmental and health concerns necessitate reliable and accessible analytical methods for accurate nickel determination.

Nickel determination is commonly performed using advanced instrumental techniques, such as AAS and ICP-MS, due to their high sensitivity [12, 13]. Nevertheless, their routine application is often constrained by high operational and maintenance costs [14], the requirement for sophisticated instrumentation, and labor-intensive sample preparation procedures involving hazardous reagents [15]. These limitations reduce their practicality for routine monitoring, particularly in laboratories with restricted analytical resources. In this context, UV–Vis spectrophotometry remains an attractive alternative due to its simplicity [16], cost-effectiveness [17], and accessibility for the analysis of transition-metal ions [18]. The analytical performance of UV–Vis methods strongly depends on the formation of stable and intensely coloured metal-ligand complexes, which enhance sensitivity and improve selectivity through well-defined absorption characteristics [19, 20].

Schiff base ligands are widely employed as complexing agents in spectrophotometric metal analysis owing to their facile synthesis by condensation reactions using inexpensive starting materials [21]. Furthermore, these ligands exhibit a high degree of structural tunability [22] and strong coordination ability toward transition-metal ions via azomethine (–C=N–) groups and additional donor atoms [23, 24]. Numerous Schiff base systems have also been reported to exhibit relatively low inherent toxicity and compatibility with green synthetic approaches [25], supporting their suitability for routine and environmentally oriented analytical applications. Recent studies have demonstrated the versatility of Schiff base ligands in the synthesis and spectroscopic characterization of nickel(II) complexes [26]. Previous reports on the synthesis and characterization of Schiff base metal complexes for Fe(II) highlight the applicability of such ligands [27, 28], while indicating the limited exploration of analogous systems for nickel analysis.

Although Schiff base ligands have been widely investigated in coordination chemistry and metal complex characterization, their systematic application in UV–Vis spectrophotometric nickel determination remains comparatively limited. In particular, the influence of post-synthesis treatment, such as crystallization, on the analytical performance of Ni(II)–Schiff base complexes has not been comprehensively evaluated in routine UV–Vis spectrophotometric analysis. Against this background, the present study aims to develop and systematically assess a UV–Vis spectrophotometric method for the quantification of nickel(II) based on a Schiff base ligand derived from 1,5-diphenylcarbazone and aniline, with specific emphasis on comparing the analytical performance of crystallized and non-crystallized complexes.

2. EXPERIMENTAL METHODS

This work was carried out to establish and assess a UV–Vis spectrophotometric method for the determination of Ni(II) using a Schiff base ligand. The experimental procedures involved the preparation of reagents, synthesis of the ligand, formation of Ni(II)–ligand complexes by non-crystallization and crystallization approaches, and systematic evaluation of analytical parameters. All experiments were performed under controlled and consistent conditions to ensure methodological reliability and reproducibility.

2.1. Materials and Instrumentation

All reagents used in this work were of analytical grade and were used as received without further purification. The chemicals comprised 1,5-diphenylcarbazone, aniline, ethanol, hydrochloric acid (HCl), sodium hydroxide (NaOH), distilled water, nickel(II) nitrate hexahydrate, and cobalt(II)

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chloride hexahydrate. Supporting materials included Whatman No. 42 filter paper and aluminum foil.

Laboratory glassware, including volumetric flasks, volumetric pipettes, beakers, and measuring cylinders, was used during the experiments. Equipment included a magnetic stirrer, a reflux apparatus, a hot plate, a vacuum pump, a Büchner funnel, a desiccator, and a pH meter (Ohaus). Functional group characterization was conducted using a Fourier Transform Infrared (FTIR) spectrometer (Shimadzu), whereas absorbance measurements were obtained using a UV–Vis spectrophotometer (Cary 100).

2.2. Synthesis and Analytical Methods

2.2.1. Synthesis of Schiff Base Ligand

The Schiff base ligand was prepared from 1,5-diphenylcarbazone and aniline at a 1:1 molar ratio, following a previously reported method [27]. Both compounds were dissolved in ethanol and mixed until a homogeneous solution formed. Hydrochloric acid was added as a catalyst, followed by stirring at ambient temperature for 30 min and refluxing for 2 h at 75-80 °C. The reaction mixture was subsequently cooled to ambient temperature, affording a crystalline product, which was retrieved by vacuum filtration, rinsed with distilled water, and desiccated until mass constancy was achieved. The obtained ligand was dissolved in ethanol to prepare a 0.5 mM solution, which was analyzed by UV–Vis spectrophotometry.

2.2.2. Synthesis of Ni(II)–Schiff Base Complex

Complex Formation by Non-Crystallization

Stock solutions of Ni(II) ions and the Schiff base ligand were prepared at a concentration of 10 mM. Complex formation was achieved by mixing the two solutions at a 1:3 molar ratio [27]. The resulting solution was subsequently diluted to a working concentration of 5 mM using a distilled water-ethanol mixture (1:3).

Complex Formation by Crystallization

An alternative synthesis route involving crystallization was employed to synthesize the Ni(II)–Schiff base complex using the same molar ratio of 1:3. The Ni(II) solution was combined with the Schiff base ligand solution and stirred until complete homogenization was achieved, followed by reflux at 75-80°C for 2 h. The mixture was then cooled to ambient temperature to induce crystallization. Formed crystals were retrieved by vacuum filtration and desiccated until mass constancy was achieved. The isolated crystals were redissolved to prepare a 10 mM complex solution, which was further diluted to a working concentration of 0.05 mM for spectrophotometric measurements.

2.2.3. Determination of Analytical Parameters

All analytical parameters were determined for both the non-crystallized and crystallized Ni(II)–Schiff base complexes.

2.2.4. Determination of Optimum pH and Wavelength

Optimization of pH was performed by adjusting the pH of the 0.05 mM complex solution within the range of 4-9 using $2x10^{-4}$ M HCl and $2x10^{-4}$ M NaOH, and the pH was measured with a pH meter. The optimum wavelength was determined by measuring the absorbance of the complex solution and selecting the wavelength that produced the maximum absorbance.

2.2.5. Determination of Complex Stability

Complex stability was assessed by monitoring changes in absorbance at the optimum pH and wavelength over 0-120 min, with measurements taken at 30 min intervals.

2.2.6. Determination of Interfering Ion Effect

The influence of interfering ions was investigated by introducing Co(II) ions from a 10 mM stock solution into the Ni(II)–Schiff base complex solution. The mixture was diluted to 0.05 mM using a distilled water-ethanol mixture (1:3), and absorbance was recorded under optimum conditions. The results were compared with those obtained in the absence of Co(II) using a *t*-test.

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3. RESULTS AND DISCUSSIONS

This section presents the experimental results of this study, covering the synthesis and characterization of the Schiff base ligand and its Ni(II) complexes, as well as their analytical behavior. Spectroscopic data are examined to relate the observed absorption features to ligand formation and metal-ligand coordination. In addition, the effects of preparation method, solution pH, time stability, and the presence of interfering ions are systematically analyzed to assess the reliability of the Ni(II)–Schiff base complex for spectrophotometric determination.

3.1. Schiff Base Ligand Formation and Characterization

The Schiff base ligand was prepared by a condensation pathway between 1,5-diphenylcarbazone and aniline using a 1:1 molar ratio in ethanol under acidic conditions. The reaction mixture was initially stirred at ambient temperature and subsequently refluxed at approximately 79 °C for 2 h to facilitate the formation of the azomethine (C=N) linkage. After cooling to ambient temperature, the product crystallized and was isolated by filtration, then dried in a desiccator. The ligand was obtained with a yield of 64.98%. Such a yield is reasonable for Schiff base condensation reactions, which are known to be reversible and sensitive to residual moisture.

In addition, partial product loss during crystallization and washing steps may contribute to the observed moderate yield. Reported yields for Schiff base ligands prepared through similar condensation approaches are generally within a comparable range [27], although variations in substituent structure and reaction conditions may influence the overall yield.

Nevertheless, the synthesized ligand was obtained in sufficient quantity and purity for further complexation and analytical studies. UV–Vis spectroscopy of the ligand solution (0.5 mM) revealed two principal absorption bands at 239 nm and 278 nm. The absorption at 239 nm is associated with $\pi \rightarrow \pi^*$ excitation associated with the aromatic framework [28], whereas the band at 278 nm arises from an $n \rightarrow \pi^*$ excitation attributed to the lone pair electrons of the azomethine nitrogen [29]. The presence of this azomethine-related band provides clear spectroscopic evidence for Schiff base formation. Its occurrence at a slightly shorter wavelength than the theoretical value reflects the influence of the semipolar solvent environment.

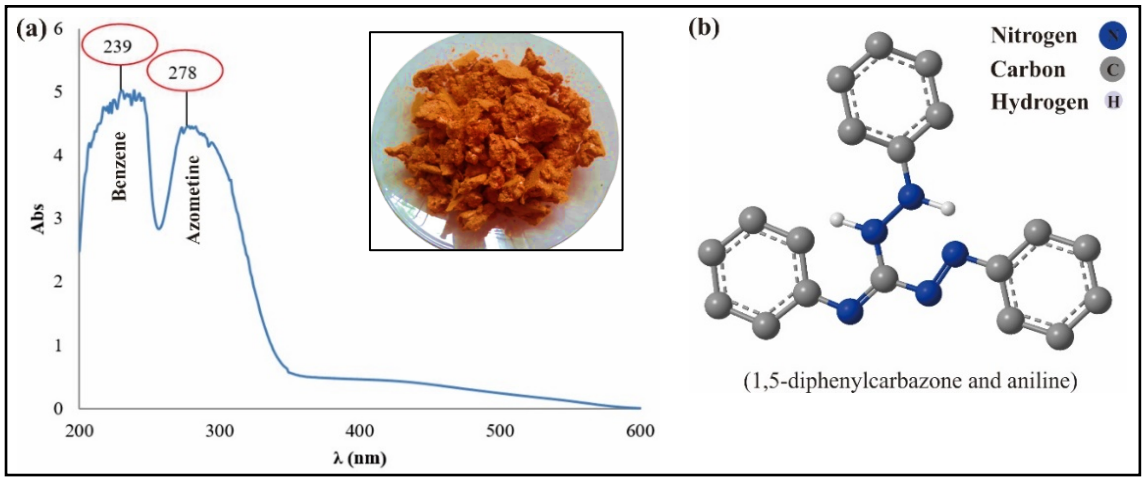

Figure 1. (a) UV–Vis spectrum of Schiff base ligand, (b) Molecular structure of Schiff base ligand.

FTIR spectroscopy was employed to identify the functional groups associated with Schiff base formation. The FTIR spectrum of the ligand (Figure 4) shows a broad absorption located in the 3500-3200 cm^{-1} region, which corresponds to N–H stretching vibrations, with possible overlapping contributions from O–H stretching due to hydrogen bonding or residual moisture.

A characteristic absorption signal associated with the azomethine (C=N) stretching vibration at 1650-1600 cm^{-1} region, confirming the formation of the Schiff base linkage. Additional absorption signals located in the 1600-1500 cm^{-1} region correspond to aromatic C=C stretching vibrations, whereas signals appearing in the 1350-1250 cm^{-1} range are assigned to C–N stretching vibrations of the Schiff base framework [27]. These FTIR features are consistent with those reported for azomethine-based ligands and confirm the successful synthesis of the Schiff base ligand.

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3.2. Ni(II)–Schiff Base Complex Formation and Characterization

Following ligand synthesis, coordination with Ni(II) ions was carried out using a metal to ligand molar ratio of 1:3. Two preparation approaches were employed: direct complex formation in solution and complex formation followed by crystallization, to assess whether the preparation method influences the spectroscopic characteristics and analytical performance of the resulting complexes.

3.2.1. Complex Formation by Non-Crystallization

When the Ni(II) solution was combined with the ligand solution, a reddish-brown coloration developed immediately, indicating rapid coordination in the solution phase. The UV–Vis spectrum of the non-crystallized complex (5 mM) revealed a distinct absorption maximum at 532 nm [30]. This band is absent in the spectrum of the free ligand. It is attributed to an electronic transition associated with the coordination of the azomethine nitrogen to the Ni(II) center. The appearance of this visible region absorption confirms the formation of a Ni(II)–Schiff base complex by non-crystallization.

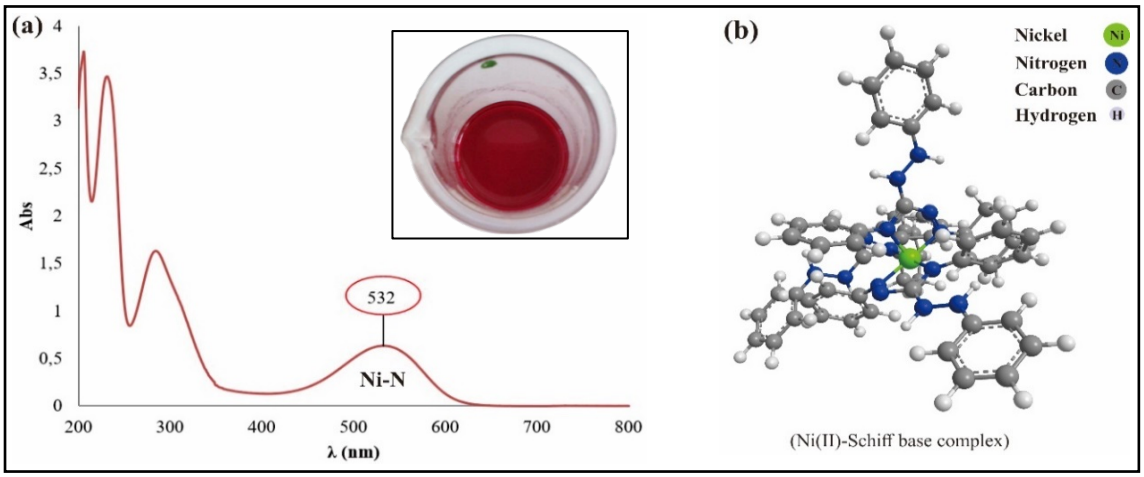

Figure 2. (a) UV–Vis spectrum of Ni(II)–Schiff base complex by non-crystallization, (b) Molecular structure of Ni(II)–Schiff base complex.

3.2.2. Complex Formation by Crystallization

In the crystallization approach, the mixture was refluxed and then cooled under controlled conditions, yielding a brownish-purple crystalline material with a yield of 57.11%. The moderate yield may be attributed to material loss during recrystallization and purification, processes commonly associated with solid-phase isolation. Yields within a comparable range have been reported for other Schiff base metal complexes prepared through similar condensation and isolation procedures [27].

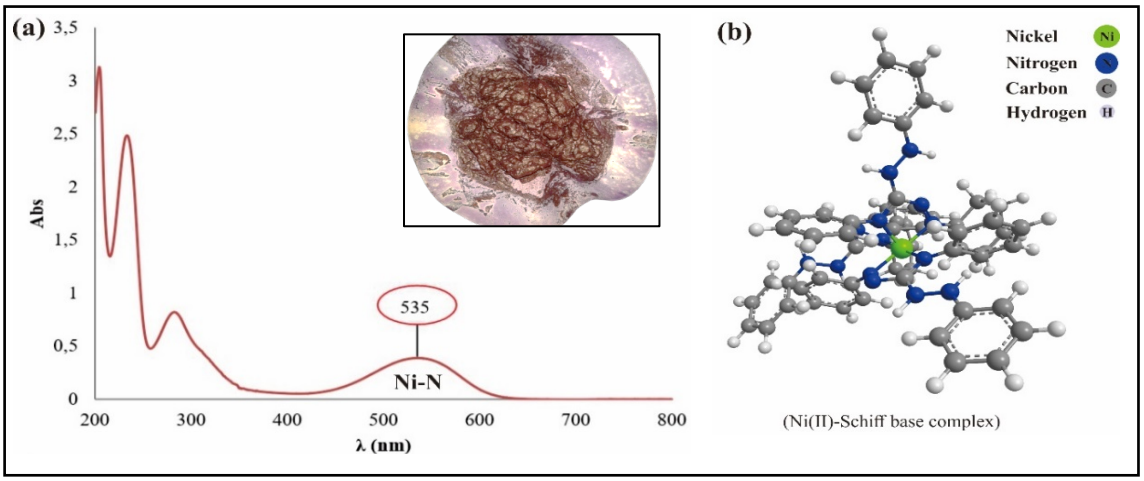

Figure 3. (a) Ni(II)–Schiff base complex by crystallization, (b) Molecular structure of Ni(II)–Schiff base complex.

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Nevertheless, the crystallized complex was obtained in sufficient quantity and purity for subsequent analytical evaluation. Upon dissolution and UV–Vis analysis at 0.05 mM, the complex displayed a maximum absorbance at 535 nm [30]. This value lies within the same visible region as that observed for the non-crystallized complex, indicating that both preparation methods generate the same chromophoric species.

FTIR spectra of the Ni(II)–Schiff base complex (Figure 4) exhibit distinct differences relative to the free ligand, indicating changes in ligand vibrational modes upon coordination. The C=N stretching signal shifts to lower wavenumbers, appearing in the 1600-1550 cm^{-1} region, indicating a reduction in azomethine bond order induced by coordination of the nitrogen donor atom to the Ni(II) ion. New absorption bands are observed in the low-wavenumber region of 550-450 cm^{-1}, which are attributed to Ni–N stretching vibrations [27]. Based on these spectral features, Ni(II) coordination is dominated by azomethine nitrogen coordination, consistent with the proposed structure of the Ni(II)–Schiff base complex.

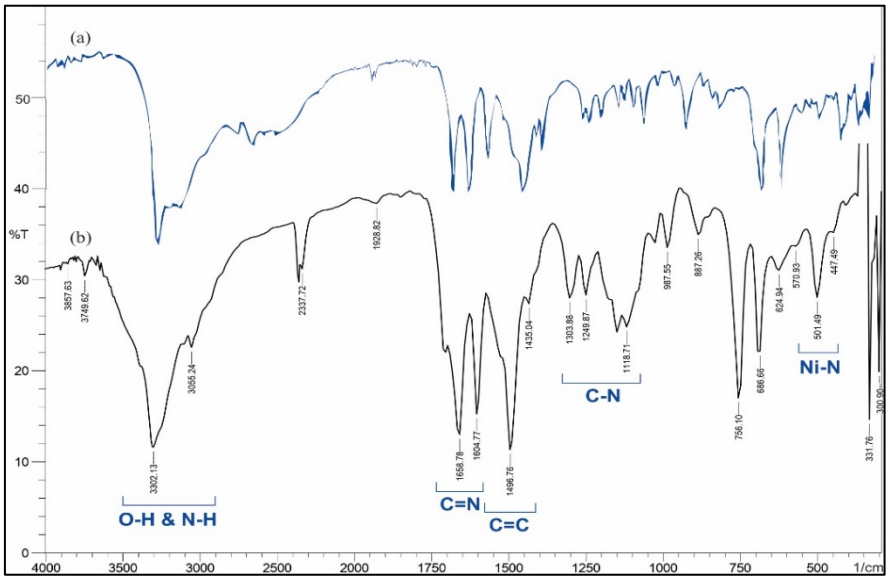

Figure 4. (a) FTIR spectra of the Schiff base ligand, and (b) Ni(II)–Schiff base complex.

3.3. Spectrophotometric Determination of Ni(II)–Schiff Base Complex

3.3.1. Effect of pH

The effect of pH on complex formation was investigated over the pH range of 4-9. For both crystallized and non-crystallized complexes, the maximum absorption wavelength remained within a narrow visible region ($\lambda_{max} \approx$ 532-535 nm), indicating that the electronic structure of the absorbing species is largely unaffected by pH within this range. Maximum absorbance was consistently obtained at pH 6. At lower pH values, decreased absorbance can be attributed to proton competition with ligand donor atoms and increased solubility of Ni(II) ions, which hinder effective coordination.

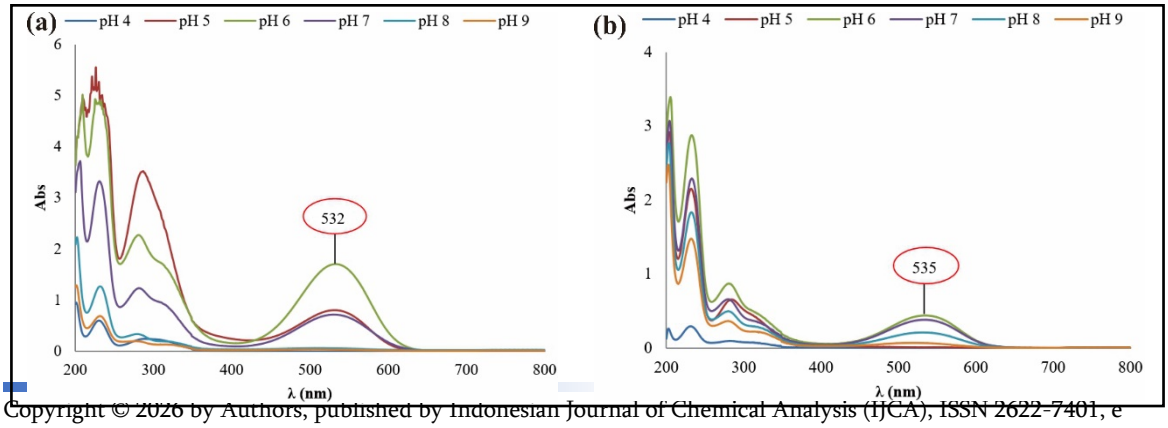

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Figure 5. Effect of pH variation on complex (a) by crystallization, (b) by non-crystallization.

At higher pH values, reduced absorbance is associated with the formation of nickel hydroxide species, which limits the availability of free Ni(II) ions for complexation [31]. Accordingly, pH 6 was selected as the optimum condition for subsequent spectrophotometric measurements.

3.3.2. Effect of Complex Stability

The time stability of the complexes was evaluated over 0-120 min under optimal conditions. For the crystallized complex 0.05 mM, absorbance at 535 nm remained within the range 0.2979-0.3400 throughout the measurement period. Similarly, the non-crystallized complex 5 mM showed stable absorbance values at 532 nm, ranging from 0.9018 to 1.0950. These results indicate that both complexes form rapidly and remain stable over the time scale relevant to spectrophotometric analysis, thereby ensuring reliable and reproducible measurements [32].

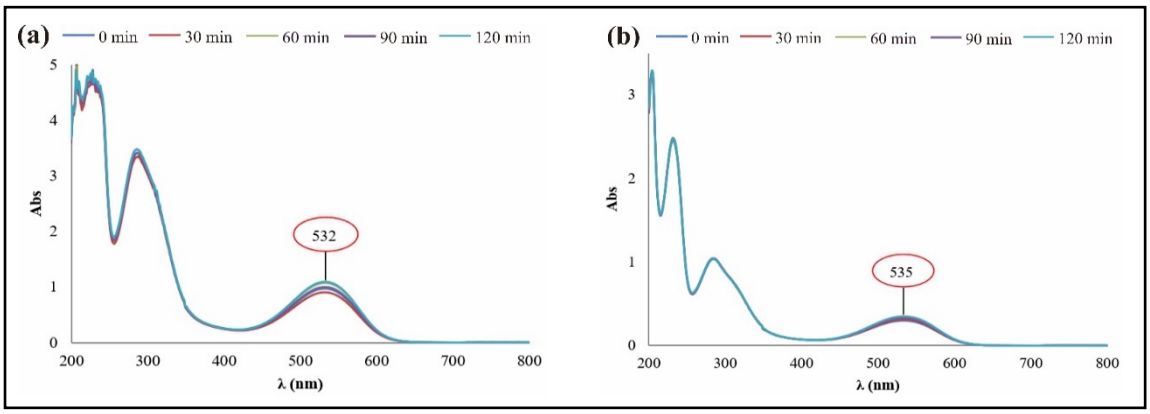

Figure 6. Effect of complex stability (a) by crystallization, (b) by non-crystallization.

3.3.3. Effect of Interfering Ion

The influence of interfering ions was examined using Co(II) as a potential competitor. After the addition of Co(II), the maximum absorption wavelength of the complexes remained unchanged, with λ_{max} values of 535 nm for the crystallized complex and 532 nm for the non-crystallized complex. The absence of any wavelength shift indicates that the same absorbing species dominates the spectra before and after the introduction of the interfering ion [33].

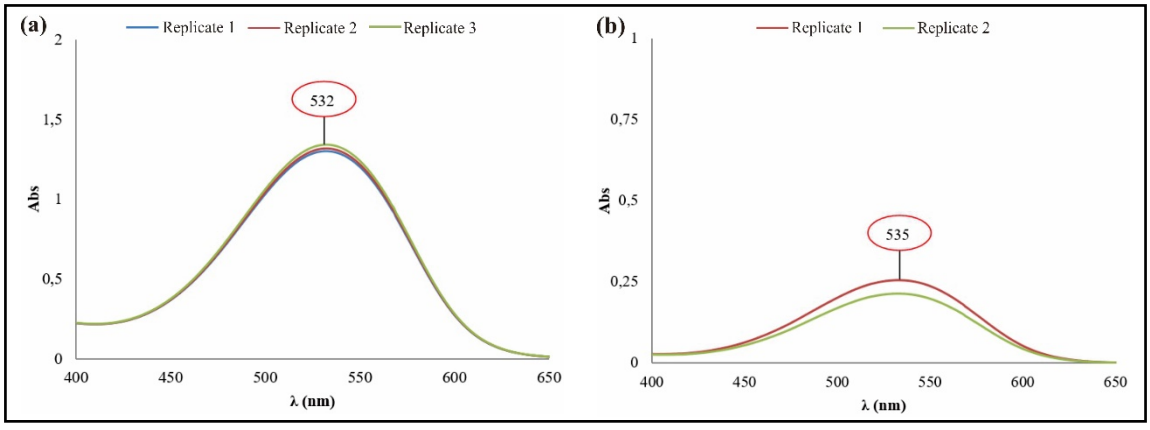

Figure 7. Effect of interfering ion complex (a) by crystallization, (b) by non-crystallization.

3.3.4. Evaluation of Analytical Response

Comparable optimum pH, complex stability time, and resistance toward Co(II) interference were observed for both crystallized and non-crystallized Ni(II)–Schiff base complexes. Nevertheless, a distinction emerges in their analytical responses at low concentration. Under identical measurement

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conditions, the non-crystallized complex produced only a weak absorbance at 0.05 mM, whereas the crystallized complex yielded a discernible and stable signal at the same concentration. Accordingly, subsequent measurements were conducted at 5 mM for the non-crystallized complex and at 0.05 mM for the crystallized complex. The dependence of absorbance on concentration follows the Beer-Lambert relationship:

$$A = \mathcal{E}.b.c \quad (1)$$

Where A denotes absorbance, $\mathcal{E}$ designates the molar absorptivity, b signifies the optical path length, and c refers to the concentration of the absorbing species. Based on the Beer-Lambert relationship, the molar absorptivity ($\mathcal{E}$) values were calculated using a 1 cm optical path length. The non-crystallized complex exhibited a molar absorptivity of 291.24 $L.mol^{-1}.cm^{-1}$ at 5 mM, whereas the crystallized complex showed a significantly higher value of 1174 $L.mol^{-1}.cm^{-1}$ at 0.05 mM. The approximately fourfold increase in ε confirms that crystallization enhances the intrinsic analytical sensitivity of the Ni(II)–Schiff base complex. Because the optical path length remains constant under identical experimental conditions and the same chromophoric system is involved, absorbance is directly proportional to concentration:

$$A \propto c \quad (2)$$

The crystallized complex exhibited markedly higher analytical sensitivity than the non-crystallized form, particularly at low concentration levels. Under identical experimental conditions, the crystallized system generated a clearly measurable and stable absorbance signal, whereas the non-crystallized complex produced only a weak response. This observation confirms that crystallization enhances the intrinsic analytical performance of the Ni(II)–Schiff base complex.

The interference study further supports this observation. Although absorbance intensity increased following Co(II) addition, a *t*-test at the 95% confidence level indicated that the difference was not significant. For the crystallized complex, the average absorbance increased from 0.0587 to 0.2329 ($t_{cal} < t_{tab}$; 7.424 < 12.706; $n = 2$, $df = 1$), while for the non-crystallized complex it decreased from 1.4562 to 1.3200 ($t_{cal} < t_{tab}$; 2.187 < 4.303; $n = 3$, $df = 2$). These results indicate that crystallization enhances analytical sensitivity while preserving the stability and selectivity of the Ni(II)–Schiff base complex [34, 35].

TABLE I. Evaluation of The Ni(II)–Sciff Base Complexes Analytical Parameters

Complex	λ (nm)	Optimum pH	Optimum Stability (min)	$t_{cal} < t_{tab}$
Crystallized	535	6	120	7.424 < 12.706
Non-crystallized	532	6	120	2.187 < 4.303

4. CONCLUSIONS

A UV–Vis spectrophotometric method for Ni(II) determination was developed using a Schiff base ligand synthesized from 1,5-diphenylcarbazone and aniline. The ligand formed a stable Ni(II) complex at a 1:3 metal-to-ligand molar ratio, exhibiting a characteristic visible absorption band at 532–535 nm. Optimum performance was achieved at pH 6, and the complex remained stable for at least 120 min, providing a reliable time window for analysis. Evaluation of Co(II) as a potential interfering ion resulted in no observable shift in the maximum absorption wavelength, indicating that the principal absorbing species remained predominant under the tested conditions. A clear difference in analytical sensitivity was observed between the crystallized and non-crystallized complexes. The crystallized complex exhibited higher molar absorptivity and generated a stable, measurable signal at lower concentration. Statistical analysis at the 95% confidence level confirmed a significant difference between the two preparation approaches. These findings demonstrate that controlled crystallization enhances analytical sensitivity. Within the scope of this study, the proposed method represents a viable and practical alternative for the determination of Ni(II) by UV–Vis spectrophotometry.

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