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Indonesian Journal of Fundamental and Applied Chemistry

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Synthesis and Characterization of Schiff Base Compound Benzaldehyde-2,4-Dinitrophenylhydrazone as a Carbonate Anion Sensor

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Abstract

Benzaldehyde-2,4-dinitrophenylhydrazone was synthesized via the condensation of benzaldehyde with 2,4- dinitrophenylhydrazine. The product was analyzed using UV–Vis and FT-IR spectroscopy, and its solvatochromic properties were investigated in DMF, acetone, and ethanol. Its application as a carbonate anion sensor was evaluated under optimized conditions. Schiff base formed orange crystalline solids with a 91.86% yield. UV-Vis spectra showed maximum absorption wavelength at 260 nm (π-π*) and 390 nm (n-π*). FT-IR analysis proves the presence of azomethine band (HC=N) at 1618 cm^{-1} and the shift of the N-H stretching band region from 3325 to 3284 cm^{-1}. Among the solvents studied, DMF exhibited the highest solubility and color stability, acetone showed the greatest absorbance but poor stability, and ethanol showed low solubility with the formation of a precipitate. Functioning as a carbonate anion sensor, the Schiff base exhibited a noticeable color change from yellow to red, along with a bathochromic shift from 390 nm to 495 nm (π→π*), reaching its maximum response after 30 minutes.

Keywords: Schiff base, benzaldehyde, 2,4-dinitrophenylhydrazine, carbonate anion sensor

Article Info

Received 12 September 2025
Received in revised 5 October 2025
Accepted 7 October 2025
Available online 29 October 2025

Abstrak (Indonesian)

Benzaldehida-2,4-dinitrofenilhidrazon disintesis melalui reaksi kondensasi antara benzaldehida dengan 2,4-dinitrofenilhidrazin. Karakterisasi basa Schiff benzaldehida-2,4-dinitrofenilhidrazon dilakukan menggunakan spektrofotometer UV-Vis, FT-IR, dan dilakukan uji solvatokromik dengan berbagai pelarut seperti DMF, aseton, dan etanol. Pengaplikasian basa Schiff sebagai sensor anion karbonat dilakukan pada waktu optimum. Produk basa Schiff ditandai dengan terbentuknya kristal padat berwarna oranye dengan persen hasil sebesar 91,86%. Spektrum UV-Vis dari basa Schiff terdapat serapan maksimum pada panjang gelombang 260 nm yang menunjukkan transisi elektronik dari π ke π* dan panjang gelombang 390 menunjukkan transisi elektronik dari n ke π*. Analisis FT-IR membuktikan keberadaan pita azometin (HC=N) pada 1618 cm^{-1} dan terjadinya pergeseran daerah pita regangan N-H dari 3325 menjadi 3284 cm^{-1}. Basa Schiff pada pelarut DMF memberikan warna paling stabil dengan kelarutan terbaik, sedangkan aseton memiliki absorbansi tertinggi tetapi kurang stabil karena warna cepat memudar, dan etanol menunjukkan kelarutan terendah dengan masih adanya endapan. Pengaplikasian basa Schiff sebagai sensor anion karbonat pada waktu yang optimum menghasilkan warna dari kuning ke merah serta mengalami pergeseran panjang gelombang dari 390 nm ke 495 nm sehingga terjadi transisi elektronik dari π–π* dengan waktu terbaik yaitu 30 menit.

Kata Kunci: basa Schiff, benzaldehida, 2,4-dinitrophenylhydrazin, sensor anion karbonat

INTRODUCTION

The name 'Schiff base' is derived from the German chemist Hugo Schiff, who in 1864 first reported the compound formed from the reaction of a primary amine with a carbonyl compound [1-4]. Schiff bases, also referred to as imine or azomethine (C=N) groups, are formed by the condensation of primary amines with aldehydes or ketones, in which the original C=O group is substituted by a C=N–R group. The imine group (R^1–HC=N–R^2) can interact with metal ions through nitrogen or oxygen atoms that possess lone electron pairs [5-8]. Schiff base ligands are known to be able to interact with inorganic ions through the formation of complex compounds which are characterized by color changes or wavelength shifts [9]. Among all identified organic sensors, Schiff base ligands are widely recognized as some of the most thoroughly investigated chemosensors for detecting both cations and anions [2].

Carbonate anion (CO_3^{2-}) are of great interest for research due to their broad roles in the environment, physiological systems, and industry [10]. Among the various anions commonly present in the environment, the detection and quantification of carbonate are very important because of its abundance and widespread presence in natural waters and soil environments [11]. The main source of carbonate comes from the hydrolysis of carbon dioxide into carbonic acid and its subsequent transformation into carbonate and bicarbonate, which are involved in rock weathering, mineral precipitation, ocean acidification, and climate change [12].

The main source of carbonate is the hydrolysis of carbon dioxide into carbonic acid and its subsequent transformation into carbonate and bicarbonate, which are involved in rock weathering, mineral precipitation, ocean acidification, and climate change. In industry, carbonate is used in the production of glass, paper, toothpaste, detergents, and dyes [13]. However, excess carbonate in industrial waste can have negative impacts, being corrosive, and at high doses may cause health problems [14,15]. Therefore, it is urgently necessary to develop a simple, cost-effective carbonate chemosensor with the potential for real sample testing without interference from endogenous substrates.

Several methods have been established for anion detection, including electrochemical techniques, chromatography, spectroscopy, and the use of chemosensors [16]. Among these methods, colorimetry is a simple, rapid, and economical technique that allows for visual detection [17]. Although its sensitivity is lower than fluorescence, the colorimetric method remains superior due to its high selectivity, low cost, and ease of interpretation [18].

Benzaldehyde is one of the aromatic aldehydes, compounds that often has higher stability due to the resonance effect of the aromatic ring that helps stabilize the double bond of the azomethine group (C=N) [19]. Due to their extensive conjugation, aromatic aldehydes are more stable than aliphatic aldehydes, which tend to be less stable and prone to polymerization [14]. Meanwhile, the compound 2,4-dinitrophenylhydrazine acts as an amine and is a hydrazine compound which, when reacted with aldehydes or ketones, will produce hydrazone derivative compounds. Hydrazone derivatives are characterized by compounds that have an azomethine functional group (C=N) which acts as an anion sensor [17,20].

In recent years, several studies have demonstrated the efficacy of hydrazone-derived Schiff bases as sensors for other anions, such as cyanide, acetate, fluoride, and sulfide [10,15,16,18]. In recent years, hydrazone-based compounds have gained popularity because they are readily accessible, easy to synthesize, modular, resistant to hydrolysis, and feature the versatile N–C=N group, making them suitable for applications across diverse fields [21]. However, research specifically developing Schiff base- based sensors for detecting carbonate anions is still very limited. This is despite the fact that the azomethine (C=N) and amine (-NH) groups in hydrazone derivatives have the potential to interact with carbonate through hydrogen bonding or deprotonation between the (NH) group and the anion [22].

MATERIALS AND METHODS

Materials

Benzaldehyde (Merck), 2,4dinitrophenyl-hydrazine (Merck), glacial acetic acid (Merck), ethanol (96% Merck), ethanol p.a. (100% Merck), Dimethylformamide (DMF) (Merck), acetone (Merck), n-hexane (Merck), sodium carbonate (Na_2CO_3) (Merck), potassium bromide (KBr) (Merck), and distilled water.

Synthesis of Schiff base benzaldehyde-2,4- dinitrophenylhydrazone (BDPH)

Schiff base compounds are synthesized through the reaction of 2,4-dinitrophenylhydrazine (0.01 mol) and benzaldehyde (0.02 mol) in ethanol with the addition of a small amount of glacial acetic acid as a catalyst. The mixture was heated under reflux at 60 °C for 3 hours, then cooled to room temperature. The precipitate was filtered, washed with ethanol, and dried

in an oven at 60 °C for 3 hours to obtain a pure product [23,24].

Analysis using thin layer chromatography (TLC)

Approximately 50 mg of Schiff base was dissolved in a small amount of ethanol and then spotted onto a TLC plate along with reference compounds (2,4-dinitrophenylhydrazine, benzaldehyde, and Schiff base). The plate was eluted with mixed solvent n-hexane-acetone (9:1) and observed using UV at 254 nm. Differences in Rf values were used to identify the formation of new compounds, such as Schiff bases.

Characterization of Schiff bases using a UV-Vis Spectrophotometer

A total of 0.0028 g of Schiff base was dissolved in DMF to obtain a stock solution of 1×10^{-3} M, then diluted to 1×10^{-5} M. The solution was analyzed using a UV-Vis spectrophotometer in the wavelength range of 200 - 600 nm.

Characterization using Fourier transform infrared (FT-IR)

The Schiff base product Schiff base (BDPH) was mixed with KBr, ground, then pressed into pellets and analyzed using FT-IR in the wavenumber range 4000-500 cm^{-1} [25].

Solvatochromic test of Schiff base benzaldehyde- 2,4-dinitrophenylhydrazone

A total of 0.0028 g of benzaldehyde-2,4-dinitrophenylhydrazone Schiff base was dissolved in DMF and acetone to a concentration of 1×10^{-5} M, then the color change and maximum wavelength were observed using a UV-Vis spectrophotometer at 300–600 nm [26].

Application of Schiff base BDPH as a carbonate anion (CO_3^{2-}) sensor using a UV–Vis spectrophotometer

1.5 mL solution of Schiff base BDPH (1×10^{-5} M) in DMF was mixed with 50 µL of Na_2CO_3 (1×10^{-2} M), resulting in a final Schiff base concentration of (9.86×10^{-6}) M and a carbonate concentration of (3.23×10^{-4}) M. The mixture was allowed to stand for 5, 10, 30, and 60 minutes. After each contact time, the sample was analyzed using a UV-Vis spectrophotometer within the wavelength range of 300-600 nm. The absorbance was then recorded at the maximum wavelength, indicating the interaction between the Schiff base and carbonate anions.

RESULTS AND DISCUSSION

Synthesis of Schiff base benzaldehyde-2,4-dinitrophenylhydrazone

Schiff bases, or azomethines, are produced via the condensation of a primary amine with an aldehyde [27]. In this research, the prepared Schiff base was obtained from the reaction of benzaldehyde, an aromatic aldehyde with greater stability due to conjugation [26,28], and 2,4-dinitrophenylhydrazine (DNPH) as the primary amine and a hydrazone derivative. The product obtained was Benzaldehyde-2,4-dinitrophenylhydrazone (BDPH), appearing as orange-colored powder or crystals with a mass of 2.6274 g and a yield of 91.8%. The reaction between benzaldehyde and 2,4-dinitrophenylhydrazine leading to the formation of the Schiff base is illustrated in **Figure** 1.

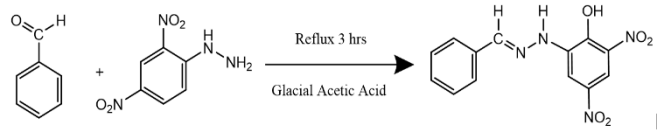

Figure 1. Reaction scheme of Schiff base formation from benzaldehyde and 2,4-dinitrophenylhydrazine [24]

The synthesis of the Schiff base BDPH requires the presence of an acid catalyst, namely glacial acetic acid. The primary role of the acid is to activate the carbonyl group. Acid-induced protonation of the aldehyde group in benzaldehyde enhances the polarity of the C=O bond, thus increasing the electrophilicity of the carbon atom. This enables the nitrogen atom in DNPH to perform a nucleophilic attack, which donates its lone pair of electrons to the carbonyl carbon, forming an addition intermediate (carbinolamine). The intermediate then dehydrates through the removal of a water molecule, producing a C=N bond (imine/azomethine). Thus, the carbonyl carbon of benzaldehyde functions as the electrophile, while the nitrogen atom of DNPH serves as the nucleophile [25].

Analysis using thin layer chromatography (TLC)

The Schiff base formation was confirmed through analysis by Thin Layer Chromatography (TLC). Spots of the starting materials (2,4- dinitrophenylhydrazine and benzaldehyde), the synthesized product (Schiff base), and their mixture were applied onto a TLC plate. The chromatographic separation was carried out using mixed solvent n-hexane-acetone (9:1) as the mobile phase, and the spots were visualized under UV light at a specific wavelength. The results of the TLC analysis are shown in **Figure** 2.

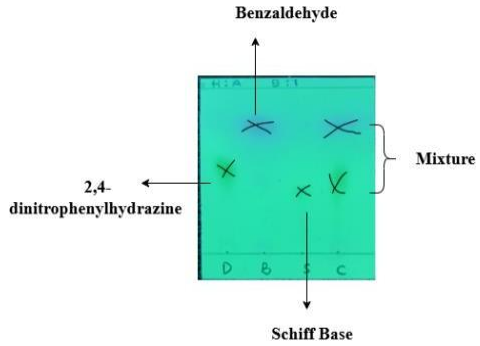

Figure 2. TLC detection results of the Schiff base the TLC analysis revealed distinct single

Spots for each compound with different Rf values: 2,4 dinitrophenylhydrazine (Rf = 0.4), benzaldehyde (Rf = 0.76), and the Schiff base (Rf = 0.36). In the case of the mixture, two spots corresponding to Rf values of 0.54 and 0.76 were observed. The differences in Rf values indicate distinct characteristics between the reactants and the product. The Schiff base exhibited a unique spot with a characteristic color, confirming the successful formation of the target compound in a relatively pure form.

Characterization of the Schiff base using UV–Vis spectrophotometry

UV-Vis spectrophotometric analysis was performed to observe the electronic transitions of the synthesized Schiff base. Measurements were carried out at a concentration of 1×10^{-5} M in DMF over the wavelength range of 200-600 nm. The resulting Schiff base spectrum is shown in **Figure** 3.

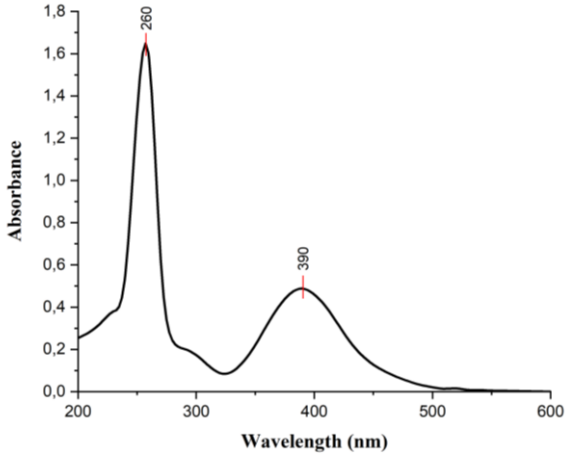

Figure 3. UV–Vis Spectrum of the Schiff base

The spectrum showed a π→π* transition at 260 nm, associated with the aromatic rings, and an n→π* transition at 390 nm, attributed to the lone pair electrons on the imine group (C=N). The n→π* transition appeared at a longer wavelength due to its lower energy requirement compared to the π→π* transition. These findings confirm the successful formation of a new Schiff base compound, characterized by distinct electronic properties compared to its starting materials [29].

Characterization using Fourier transform infrared (FT-IR)

Based on the IR spectral measurements of the Schiff base compound (**Figure** 4), a new peak was observed to shift from 3325 cm^{-1} to 3284 cm^{-1}, associated with the N-H stretching mode of the hydrazone group. The aromatic C-H functional group also showed a shift from 3086 cm^{-1} to 3097 cm^{-1}. Another absorption band appearing at 1330 cm^{-1} indicated the presence of a symmetrical $-NO_2$ functional group. Meanwhile, the absorption at 1618 cm^{-1}, associated with the -HC=N functional group, confirmed the successful formation of the Schiff base product.

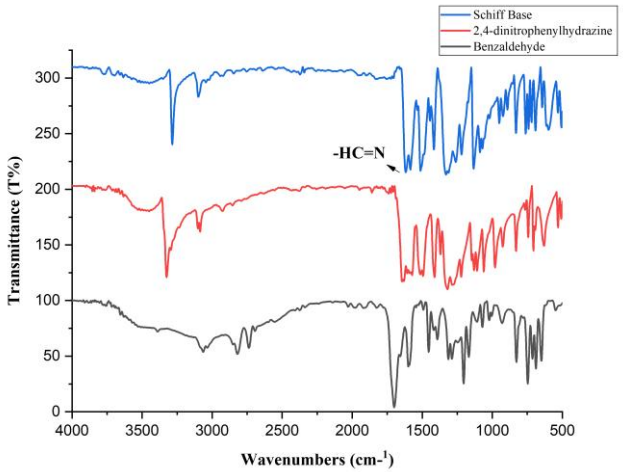

Figure 4. FT-IR Spectrum of the Schiff base

Solvatochromic test of Schiff base benzaldehyde- 2,4-dinitrophenylhydrazone

The solvatochromic test was conducted to determine the most suitable solvent for further analysis. The solvents employed with different polarity levels were DMF, acetone, and ethanol. This analysis was performed at a wavelength of 300-600 nm. The spectra of the resulting Schiff bases in various solvents are shown in **Figure** 5.

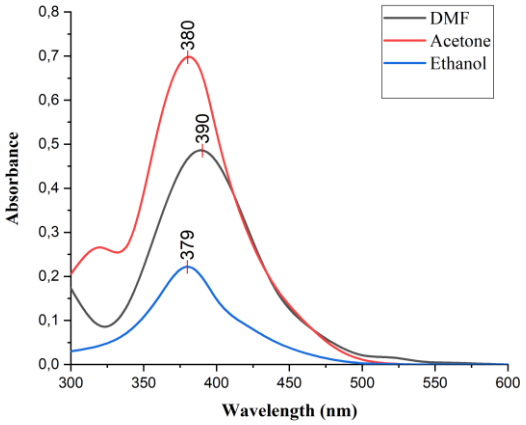

Figure 5. UV-Vis Solvatochromic spectrum of Schiff base BDPH in DMF, acetone, and ethanol.

The solvatochromic study of the Schiff base BDPH revealed that both solubility and UV-Vis spectral properties varied depending on the solvent used. The resulting solution colors in DMF, acetone, and ethanol were deep yellow, yellow, and pale yellow, respectively. The highest maximum wavelength (λ max) was observed in DMF, followed by acetone and ethanol, whereas the highest absorbance was recorded in acetone. However, the solubility of BDPH in acetone was less stable, leading to rapid fading of the solution color. According to [30], this occurs because the Schiff base is not optimally deprotonated (does not release protons) in acetone, resulting in less stable color changes. In contrast, DMF exhibited the best solubility with the deepest solution color, while ethanol showed the poorest solubility with undissolved residues remaining. Differences in solvent polarity influenced the bathochromic shift of the π–π* transition, which plays an essential role in the sensing performance of the compound. According to [22], the greater the wavelength shift of a solvent, the more effective it is for use as a sensor compound. Based on these findings, DMF was selected as the most suitable solvent for subsequent analyses.

Application of Schiff base BDPH as a carbonate anion (CO_3^{2-}) sensor using a UV–Vis spectrophotometer at the optimum contact time

This experiment aimed to evaluate the detection ability of the Schiff base benzaldehyde-2,4-dinitrophenylhydrazone (BDPH) before and after interaction with carbonate anions, as well as to determine the optimum time for BDPH to detect carbonate anions within different time intervals of 5, 10, 30, and 60 minutes

Table 1. Absorbance of Schiff base with carbonate anion at different time intervals

Time (Minutes)	Absorbance			Average
	1	2	3	
5	0.562	0.571	0.568	0.567
10	0.553	0.576	0.566	0.565
30	0.576	0.595	0.590	0.587
60	0.578	0.593	0.584	0.585

Based on **Table** 1, the Schiff base compound interacting with the carbonate anion (CO_3^{2-}) exhibited relatively stable absorbance values, with a slight increase observed at 30 minutes (0.587). At 60 minutes, the absorbance value (0.585) was not significantly different, indicating that the absorbance response tended to stabilize after 30 minutes. Further characterization was conducted using a UV–Vis spectrophotometer to investigate the electronic transitions occurring during the interaction process in DMF solvent. The wavelength spectrum resulting from this interaction is presented in the **Figure** 6.

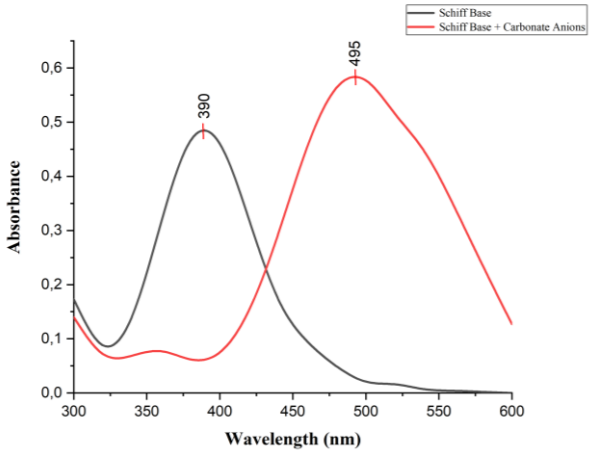

Figure 6. Maximum adsorption wavelength of of Schiff base interacted with carbonate anion (CO_3^{2-}).

According to **Figure 6**, the chemosensor demonstrated the ability to detect carbonate anions (CO_3^{2-}) through a color change from yellow to red. According to [11], when the active group interacts with the analyte in this case, carbonate anions it can induce hydrogen bonding and deprotonation. Deprotonation is a process of electron delocalization within the π-bond, which leads to a color change from the compound's original color.

The Schiff base VDPH, when interacting with CO_3^{2-} anions, exhibited a maximum absorption wavelength at 495 nm. This indicates a bathochromic

shift in the wavelength from 390 nm to 495 nm with a π→π* electronic transition after interaction with CO_3^{2-} anions, where the response reached stability at 30 minutes. According to [22], the shift in the absorption peak wavelength spectrum can be attributed to the presence of electron-withdrawing groups such as the nitro group (NO_2) in the sensor compound. The electron-withdrawing group, in this case NO_2, can increase the polarization of N-H and enhance the hydrogen bonding capability of the sensor compound. The nitro group is able to delocalize electrons from the amine (-NH) toward the nitro group, thereby decreasing the electron density of the amine group and increasing its hydrogen bond donor ability. This condition enhances the strength of interaction between the sensor compound and the anion. According to [11], carbonate ions also act as bases that deprotonate the -NH/azomethine group, thereby enhancing electron delocalization within the π-conjugated system. This delocalization reduces the HOMO-LUMO energy gap, resulting in a spectral shift toward longer wavelengths (bathochromic shift).

CONCLUSION

The Schiff base compound Benzaldehyde-2,4-dinitrophenylhydrazone (BDPH) was successfully synthesized in a high yield of 91.86% as orange crystals. Characterization by UV-Vis spectroscopy indicated absorption maxima at 260 nm (π–π*) and 390 nm (n–π*), while FT-IR confirmed the presence of the azomethine group (-HC=N) at 1618 cm^{-1} along with a shift of the -NH band from 3325 cm^{-1} to 3284 cm^{-1}. Solvatochromic analysis identified DMF as the optimum solvent, and the application of BDPH as a carbonate anion sensor proved to be effective, as indicated by a distinct color change from yellow to red, together with a bathochromic shift from 390 nm to 495 nm, which reached relative stability at 30 minutes with a maximum absorbance of 0.587.

ACKNOWLEDGMENT

The author would like to thank Sriwijaya University for granting permission and fully supporting the author in conducting this research in the Inorganic Chemistry Laboratory.

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