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Synthesis and Characterization of Schiff Bases Derived from the Condensation of Vanillin, Isovanillin, 5-Nitrosalicylaldehyde, 3,4,5-Trimethoxybenzaldehyde, and Indole-3-Carboxaldehyde with 3,3′,5,5′-Tetramethylbenzidine

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ABSTRACT

Schiff bases are an important class of organic compounds characterized by the presence of an azomethine –C=N– functional group, formed by the condensation reaction between primary amines and carbonyl compounds. In this study, a series of Schiff base derivatives were synthesized using selected aromatic aldehydes and primary amine under controlled laboratory conditions. The synthesis was performed by condensation reaction in presence of suitable solvents, and the resulting compounds were purified through recrystallization.

The synthesized Schiff base derivatives were characterized using Fourier Transform Infrared Spectroscopy (FTIR) to confirm the formation of the azomethine linkage and to identify other functional groups present in the molecules. The appearance of a characteristic absorption band in the region of 1570-1585 cm^{-1} corresponding to the -C=N- stretching vibration confirmed the successful formation of Schiff bases. Additional peaks corresponding to aromatic, unsaturated-C=C- group, and other substituents further supported the proposed structures.

This study demonstrates a simple and effective approach for the synthesis of Schiff base derivatives and highlights the significance of FTIR spectroscopy as a reliable tool for structural characterization. The synthesized Schiff bases may have potential applications in medicinal chemistry, coordination chemistry, and material science.

Keywords: Schiff Base; Azomethine Group; Condensation Reaction; FTIR Spectroscopy; Aromatic Aldehydes; Primary Amines; Indole-3-Carboxaldehyde; Vanillin; Isovanillin; Tetramethylbenzidine.

1. Introduction

Schiff bases are an important class of organic compounds containing the azomethine (–C=N–) functional group, typically formed through the condensation of primary amines with aldehydes or ketones. This reaction proceeds with the elimination of water and is widely employed due to its simplicity, efficiency, and high yield. The structural diversity of Schiff bases, arising from the wide variety of amines and carbonyl compounds available, makes them highly significant in applied chemistry [1,2,3]. In this study, a series of Schiff base derivatives were synthesized using 3,3′,5,5′-tetramethylbenzidine (TMB) as the amine component and various substituted aromatic aldehydes such as vanillin (4-Hydroxy-3-Methoxybenzaldehyde), isovanillin (3-Hydroxy-4-Methoxybenzaldehyde), 5-Nitrosalicylaldehyde, 3,4,5-trimethoxybenzaldehyde, and indole-3-carboxaldehyde. Schiff bases prepared from aromatic diamines like 3,3′,5,5′-tetramethylbenzidine can form bis-imine structures, contributing to increased conjugation and stability. In this work, five different Schiff bases were synthesized through condensation reactions between selected amines and substituted aromatic aldehydes. The synthesized compounds were characterized using Fourier Transform Infrared Spectroscopy (FTIR) to confirm the formation of imine linkages and to analyze their functional group features. This study aims to explore the synthesis and spectroscopic characterization of structurally diverse Schiff bases.

1.1. Study Objectives

1) To develop novel Schiff bases, via condensation reaction of amines with aldehydes.

2) To focus on optimizing conditions of synthesis in order to improve the yield.

3) To determine the physical properties of the Schiff base like colour, melting point etc.

4) To characterise the structure of synthesised Schiff base using instrumental method like FTIR.

5) To verify the formation of the azomethine -C=N- linkage through FTIR.

2. Literature

The presence of various substituents such as hydroxyl -OH, methoxy $–OCH_3$, nitro $–NO_2$, and indole moieties significantly influences the electronic properties, reactivity, and potential applications of these compounds. These variations may enhance their utility in fields such as medicinal chemistry, where Schiff bases are known to exhibit antimicrobial, antioxidant, anti-inflammatory, and anticancer activities. Furthermore, their ability to act as ligands in coordination chemistry makes them valuable for the preparation of metal complexes with diverse applications [4,5,6,7].

Characterization of the Schiff bases is essential to confirm the azomethine linkage and to identify functional groups present in the molecules. Infrared (IR) spectroscopy is a powerful technique for this purpose. The formation of Schiff bases is typically confirmed by the characteristic –C=N– stretching vibration in the region of 1570-1585 cm^{-1}. Additional absorption bands corresponding to functional groups –OH, $–NO_2$, $–OCH_3$, and aromatic rings provide further structural confirmation [8,9,10,11].

3. Methodology

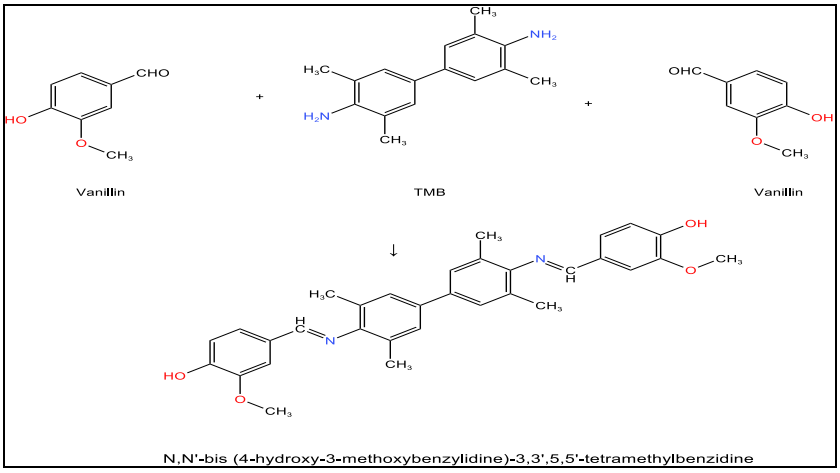
3.1. Materials and Methods

All the chemicals 3,3′,5,5′-Tetramethylbenzidine TMB, Vanillin, Isovanillin, 5-Nitrosalicylaldehyde, 3,4,5-Trimethoxybenzaldehyde and Indole-3-carboxaldehyde were sourced from Loba Chemie, and Otto Kemi. All chemicals were used as received without further purification. FTIR was obtained using the Potassium Bromide (KBr) pellet technique done on FT/IR-4600typeA.

3.2. Experimental

3.2.1. Synthesis of N, N’-bis(vinylidene)-3,3',5,5'-tetramethylbenzidine

In a round bottom flask, vanillin (0.02mol) was dissolved in ethanol. A solution of 3,3’,5,5’-tetramethyelbenzidine (0.01mol) in ethanol was added to the above solution. The reaction mixture was heated at reflux for 3-4 hours. After cooling to room temperature, the resulting solid was collected by filtration, washed with ethanol and dried yielding N, N’-bis(vinylidene)-3,3',5,5'-tetramethylbenzidine (Scheme 1). The product was obtained with a good yield of 92.31 % yield. The colour of the compound observed was yellowish orange and melting point was determined to be 210^oC.



Scheme 1. Synthesis of N, N'-bis(vinylidene)-3,3',5,5'-tetramethylbenzidine. Structure was drawn using King Draw software.

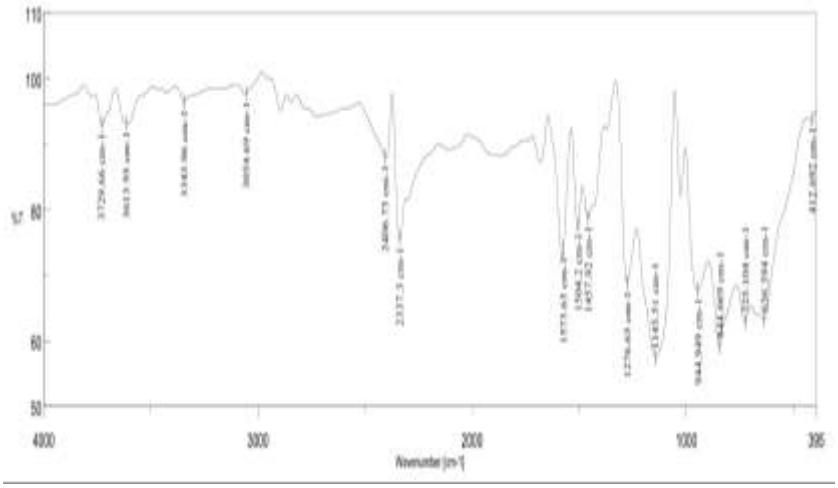


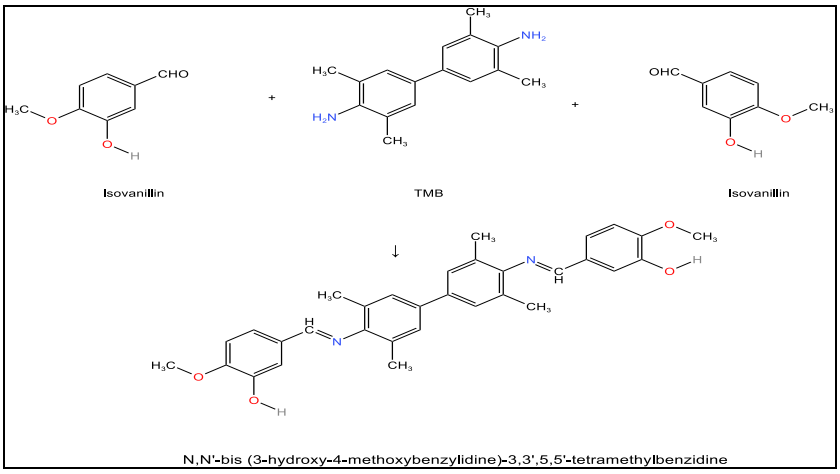
Figure 1. Fourier Transform Infra-Red Spectrum of N, N'-bis(vinylidene)-3,3',5,5'-tetramethylbenzidine scanned from wavenumber 395 cm^{-1} to 4000 cm^{-1} using FT/IR-4600typeA model.

In Fig 1. a broad absorption band observed at 3343 cm^{-1} is due to the phenolic O–H stretching vibration, of the hydroxyl group derived from vanillin. The weak band at 3054 cm^{-1} corresponds to aromatic -C–H stretching band. The formation of Schiff base is confirmed by the appearance of a strong band in the region 1573cm^{-1}, due to the azomethine -C=N- stretching vibration. It confirms the condensation reaction between the amine groups of 3,3′,5,5′-tetramethylbenzidine and the aldehyde group of vanillin. The presence of aromatic ring vibrations is further supported by band at 1504 cm^{-1}, corresponding to -C=C- stretching of the benzene rings. The peak at 1457 cm^{-1} is attributed to aromatic ring. The band observed at 1276 cm^{-1} is attributed to -C–N stretching of the imine linkage. Additionally, strong absorptions in the region 1145 cm^{-1} are assigned to -C–O stretching vibrations of the phenolic and methoxy groups. Importantly, the absence of the aldehyde -C=O stretching band around ~1700 cm^{-1} and the disappearance of primary amine -N–H stretching bands ~3400–3500 cm^{-1} further confirm the formation of Schiff base ligand.

Overall, the FTIR spectral data confirms the successful synthesis of azomethine Schiff base derived from 3,3′,5,5′-tetramethylbenzidine and vanillin.

3.2.2. Synthesis of N, N'-bis (isovanillin dene)-3,3',5,5'-tetramethylbenzidine

In a round bottom flask, Isovanillin (3-hydroxy-4-methoxybenzaldehyde) (0.02mol) was dissolved in ethanol. A solution of 3,3',5,5'-tetramethyelbenzidine (0.01mol) in ethanol was added to the above solution. The reaction mixture was heated at reflux for 3-4 hours. After cooling to room temperature, the resulting solid was collected by filtration, washed with ethanol and dried yielding N, N'-bis (isovanillin dene)-3,3',5,5'-tetramethylbenzidine (Scheme 2). The product was obtained with a good yield of 93.1%. The colour of the compound observed was orange and melting point were determined to be 205° C.



Scheme 2. Synthesis of N, N'-bis (isovanillin dene)-3,3',5,5'-tetramethylbenzidine. Structure was drawn using King Draw software.

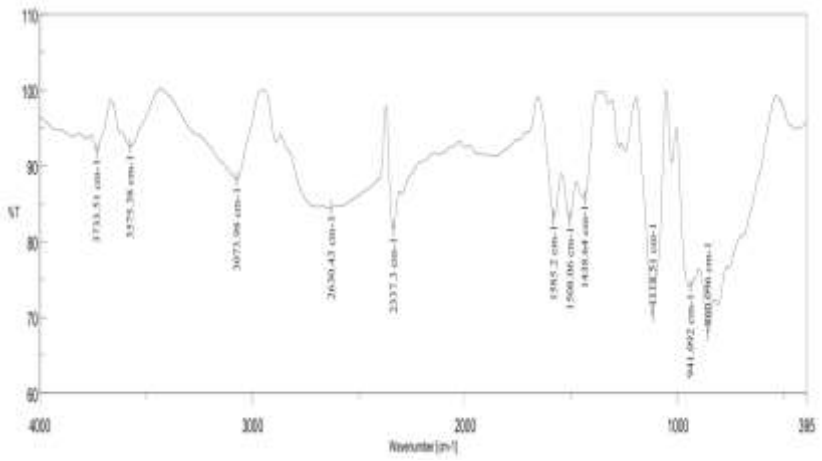


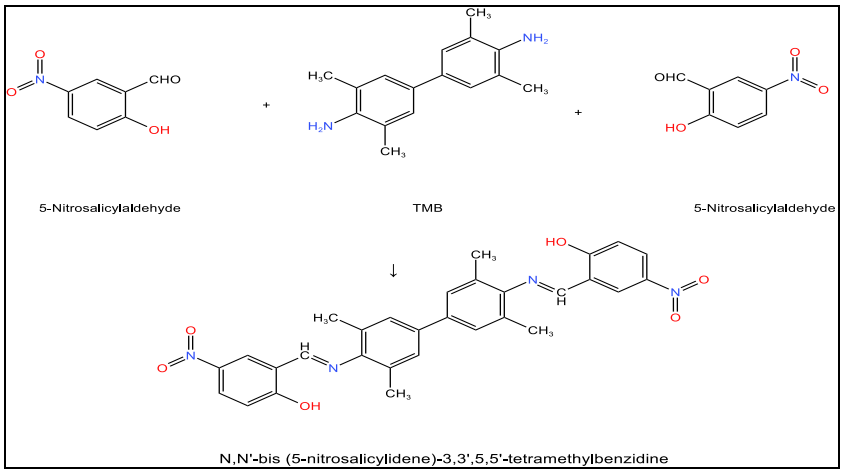
Figure 2. Fourier Transform Infra-Red Spectrum of N, N'-bis (isovanillin dene)-3,3',5,5'-tetramethylbenzidine scanned from wavenumber 395 cm^{-1} to 4000 cm^{-1} using FT/IR-4600typeA model.

In Fig 2, the appearance of a strong absorption band at 1585 cm^{-1} is attributed to the characteristic azomethine -C=N- stretching vibration, which is the key evidence for Schiff base formation. The bands observed at 3733 cm^{-1} and 3575 cm^{-1} correspond to phenolic -O-H stretching, indicating the presence of hydroxyl groups from isovanillin. The peak at 3073 cm^{-1} is due to aromatic -C-H stretching band. The absorption at 1508 cm^{-1} is due to the aromatic -C=C stretching, and the band at 1438 cm^{-1} corresponds to methyl -CH_3 bending vibrations. Additionally, peaks at 1118 cm^{-1} are assigned to -C-O stretching vibrations of phenolic and methoxy groups. The band at 941 cm^{-1} is

associated with aromatic -C-H out-of-plane bending vibrations. The absence of aldehyde carbonyl -C=O band of isovanillin around 1700 cm^{-1} and the disappearance of primary amine -N-H stretching bands of TMB further confirm that the formation of the Schiff base compound.

3.2.3. N, N'-bis(5-nitro-salicylidene)-3,3',5,5'-tetramethylbenzidine

In a round bottom flask, 5-Nitrosalicylaldehyde (0.02mol) was dissolved in ethanol. A solution of 3,3',5, 5'-tetramethyelbenzidine (0.01mol) in ethanol was added to the above solution. The reaction mixture was heated at reflux for 3-4 hours. After cooling to room temperature, the resulting solid was collected by filtration, washed with ethanol and dried yielding N, N'-bis(5-nitro-salicylidene)-3,3',5,5'-tetramethylbenzidine (Scheme 3). The product was obtained with a good yield of 87.5% yield. The colour of the compound observed was reddish brown and melting point was determined to be 215° C.



Scheme 3. Synthesis of N, N'-bis(5-nitro-salicylidene)-3,3',5,5'-tetramethylbenzidine. Structure was drawn using King Draw software.

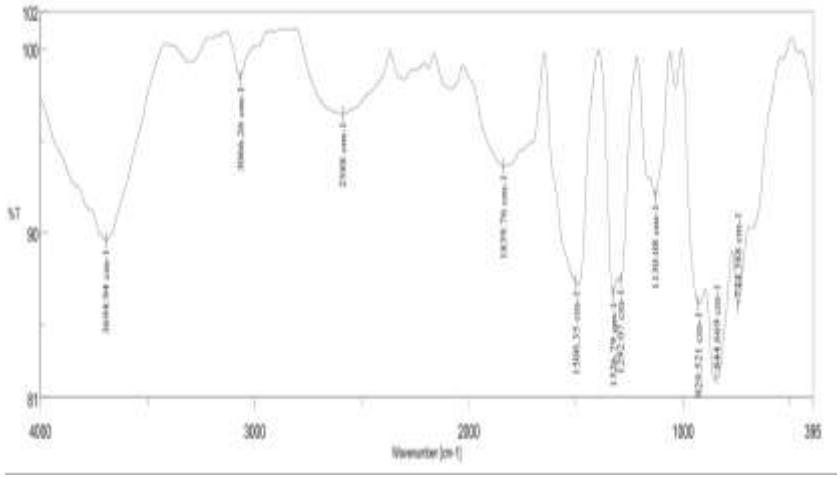


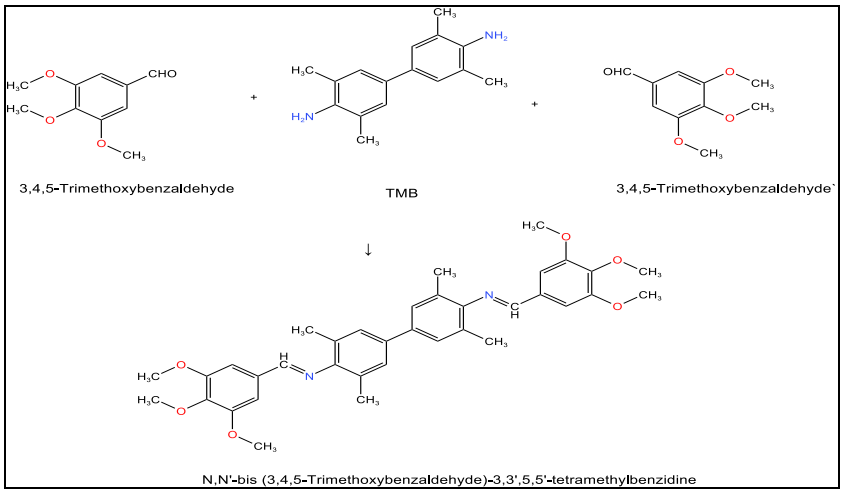
Figure 3. Fourier Transform Infra-Red Spectrum of N, N'-bis(5-nitro-salicylidene)-3,3',5,5'-tetramethylbenzidine scanned from wavenumber 395 cm^{-1} to 4000 cm^{-1} using FT/IR-4600typeA model.

In Fig 3, a strong band at 1500 cm^{-1} is assigned to the azomethine -C=N- stretching vibration, indicating successful condensation with imine formation. The broad band at 3694 cm^{-1} corresponds to phenolic –OH stretching, often

involved in intramolecular hydrogen bonding. Aromatic C–H stretching appears at 3066 cm^{-1}. The nitro group – NO_2 shows characteristic symmetric stretching bands at 1326 cm^{-1}. The peak at 1292 cm^{-1} is attributed to -C-O stretching vibration. These bands collectively confirm the formation of Schiff base ligand.

3.2.4. Synthesis of N, N'-bis(3,4,5-trimethoxybenzylidene)-3,3',5,5'-tetramethylbenzidine

In a round bottom flask, 3,4,5-Trimethoxybenzaldehyde (0.02mol) was dissolved in ethanol. A solution of 3,3',5,5'-tetramethyelbenzidine (0.01mol) in ethanol was added to the above solution. The reaction mixture was heated at reflux for 3-4 hours. After cooling to room temperature, the resulting solid was collected by filtration, washed with ethanol and dried yielding N, N'-bis(3,4,5-trimethoxybenzylidene)-3,3',5,5'-tetramethylbenzidine (Scheme 4). The product was obtained with a good yield of 98.2% yield. The colour of the compound observed was orange and melting point were determined to be 200°C.



Scheme 4. Synthesis of N, N'-bis (3,4,5-trimethoxybenzylidene) -3,3',5,5'-tetramethylbenzidine. Structure was drawn using King Draw software.

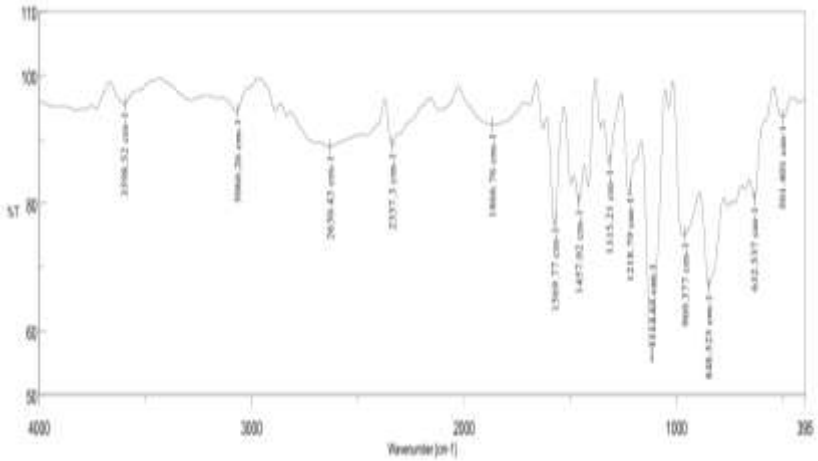


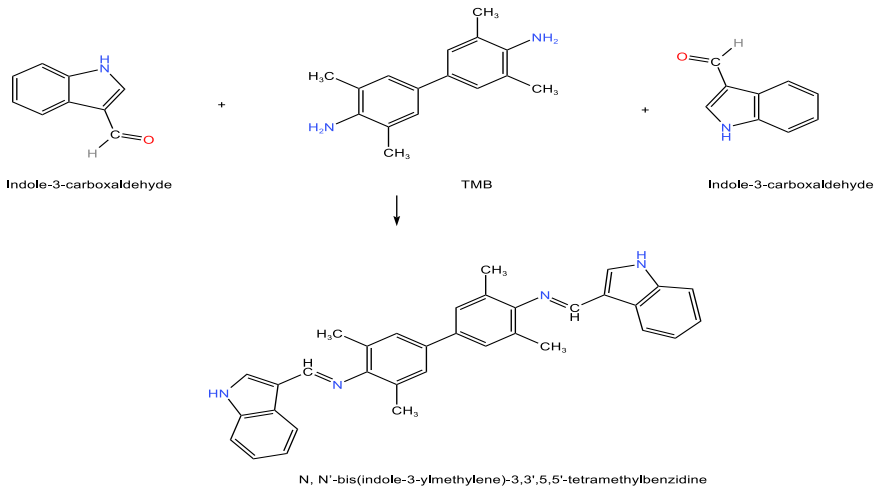
Figure 4. Fourier Transform Infra-Red Spectrum of N, N'-bis (3,4,5-trimethoxybenzylidene) -3,3',5,5'-tetramethylbenzidine scanned from wavenumber 395 cm^{-1} to 4000 cm^{-1} using FT/IR-4600typeA model.

In Fig 4, a strong absorption band at 1569 cm^{-1} is attributed to the azomethine –C=N– stretching vibration, indicating successful condensation and imine formation. Aromatic -C–H stretching appears at 3066 cm^{-1}. The

presence of methoxy substituents is confirmed by -C–O stretching bands in the region at 1218 cm^{-1}, along with -C–N vibration at 1315 cm^{-1}. The peaks below 1000 cm^{-1} are due to out-of-plane bending vibrations of substituted benzene rings. The disappearance of the aldehyde -C=O band around ~1700 cm^{-1} further supports Schiff base formation.

3.2.5. Synthesis of N, N'-bis(indole-3-ylmethylene)-3,3',5,5'-tetramethylbenzidine

In a round bottom flask, Indole-3-carboxaldehyde (0.02mol) was dissolved in ethanol. A solution of 3,3',5,5'-tetramethyelbenzidine (0.01mol) in ethanol was added to the above solution. The reaction mixture was heated at reflux for 3-4 hours. After cooling to room temperature, the resulting solid was collected by filtration, washed with ethanol and dried yielding N, N'-bis(indole-3-ylmethylene)-3,3',5,5'-tetramethylbenzidine (Scheme 5). The product was obtained with a good yield of 95.9% yield. The colour of the compound observed was brown and melting point was determined to be 150° C.



Scheme 5. Synthesis of N, N'-bis(indole-3-ylmethylene)-3,3',5,5'-tetramethylbenzidine. Structure drawn using King Draw software.

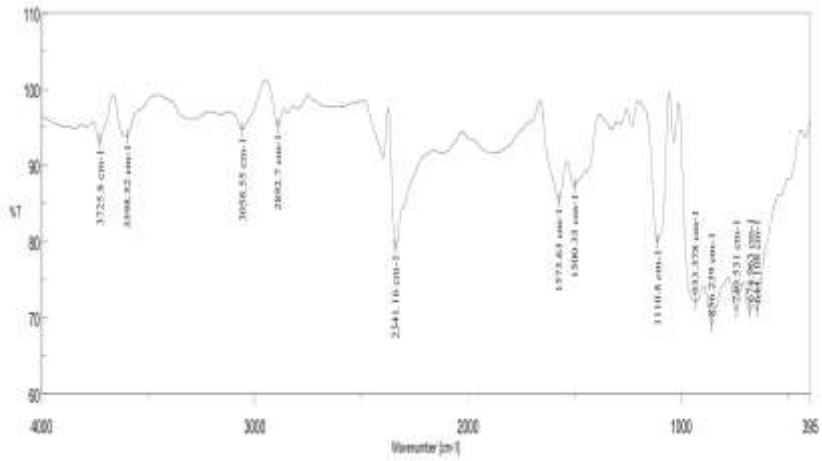


Figure 5. Fourier Transform Infra-Red Spectrum of N, N'-bis(indole-3-ylmethylene)-3,3',5,5'-tetramethylbenzidine scanned from wavenumber 395 cm^{-1} to 4000 cm^{-1} using FT/IR-4600typeA model.

In Fig 5, a characteristic strong band around at 1573 cm^{-1} corresponds to the azomethine -C=N- stretching vibration confirming imine formation. The indole N-H stretching appears as a broad band at 3725 cm^{-1}, while aromatic C-H stretching is observed at 3058 cm^{-1} and aliphatic C-H stretching at 2892 cm^{-1}. Band at 1500 cm^{-1} region are attributed to aromatic C=C stretching vibrations. Additional peaks at 1110 cm^{-1} indicate -C-N stretching, and bands below 900 cm^{-1} correspond to out-of-plane bending of substituted aromatic rings. The absence of the aldehyde -C=O band around ~1700 cm^{-1} confirms successful condensation to form the Schiff base.

4. Result and Discussions

The synthesis of Schiff bases was confirmed by infrared spectral analysis. The FTIR spectrum of the synthesized compounds exhibited significant changes in comparison to the starting materials primary amine and aldehyde.

The characteristic carbonyl -C=O stretching band of the parent aldehyde, typically observed in the region 1700–1750 cm^{-1}, was absent in the IR spectra of the synthesized compounds, indicating the use of the carbonyl group during the condensation reaction. Additionally, the N–H stretching vibrations of the primary amine, generally appearing as two bands in the region 3300–3500 cm^{-1}, were either completely absent or significantly reduced in intensity, further supporting the involvement of the amine group in the reaction. A new band appeared in the region 1570-1585 cm^{-1}, which is due to the azomethine –C=N– stretching vibration, confirming the formation of the Schiff base linkage [12,13].

5. Conclusion

The IR spectral data indicates the successful synthesis of Schiff bases through the condensation reaction of primary amines with aldehydes. The disappearance of the carbonyl -C=O and amine -N–H stretching bands, along with the appearance of the characteristic azomethine -C=N- band, provides evidence of the imine functional group formation [14,15].

Thus, IR spectroscopy confirms that the reaction proceeded as expected, in formation of the desired Schiff base structures with high certainty.

6. Future Recommendations

1. The synthesised Schiff base can be checked for its antimicrobial activity against Gram-positive bacteria Staphylococcus aureus, Gram-negative bacteria E-coli and fungal strain Candida albicans using agar well diffusion method.
2. Minimum Inhibitory Concentration (MIC), can be determined to check for the antimicrobial potency.
3. Molecular docking analysis can be performed to investigate the interaction of the synthesized compounds with target proteins, to know the varying binding affinities, through hydrogen bonding, hydrophobic forces etc.
4. Fusing Schiff bases with heterocycles such as pyrazole, imidazole to create hybrid compounds with improved biological potential.
5. To carry further optimization and advanced studies to explore the full potential of these compounds in medicinal chemistry.

Declaration

Source of Funding

This study did not receive any grant from funding agencies in the public, commercial, or not- for-profit sectors.

Competing Interests Statement

The authors declare no competing financial, professional, or personal interests.

Consent for publication

The authors declare that they consented to the publication of this study.

Authors' contributions

All the authors made an equal contribution in the Conception and Design of the work, Data collection, Drafting the article, and Critical revision of the article.

Availability of data and materials

Authors are willing to share data and material according to the relevant needs.

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