

Bilimbi Fruit Extract Mediated Synthesis of Aldimine from Vanillin and Para Nitroaniline and Its Antimicrobial Activity

K.R. Thiruthika, J. Antony Jethisha, S. Sindhu. B. Stephy Bose, W. Vibishma, J.V. Vijayadharshini and *Sheeba Daniel

Department of Chemistry, Holy Cross College (Autonomous), Nagercoil - 629004.

Affiliated to Manonmaniam Sundaranar University, Tirunelveli - 627012.

*Corresponding Author - Email: sheebadaniel@holycrossngl.edu.in

ABSTRACT

Aldimines constitute a class of pharmaceutical and medicinally important molecules. The conventional methods for the synthesis of aldimines require long reaction times and use of organic solvents. The aim of the present studies to synthesise an aldimine from vanillin and p-nitroaniline using bilimbi fruit extract. The aldimine synthesised from bilimbi fruit extract is characterized by UV-Visible and FT-IR spectral techniques. The antibacterial activity of the synthesised aldimine is tested against three bacteria and it shows slight activity on Escherichia coli, Bacillus subtilis and Pseudomonas aeruginosa. Compared with traditional methods, this method is simple, more convenient, eco-friendly and shows maximum efficiency with reduced reaction time.

Keywords: Green synthesis, Bilimbi fruit extract, Aldimine, Antimicrobial activity

1. Introduction

Green chemistry approach is an eco-friendly approach and has tremendous application for the synthesis of various organic compounds and key intermediates in recent past. This technique involves as an alternative reaction media to replace hazardous and expensive solvents routinely used in organic synthesis [1]. Organic reactions under solvent-free conditions have gained in popularity in recent years, since the majority of solvents are either toxic or flammable and add considerably to the cost of an overall synthesis. These solvent-free reactions usually need shorter reaction time, simpler and more efficient work up procedures, more improved selectivities and easier separations and purifications than conventional solvents [2]. Recently fruit juice is known to be potential organic solvents for the synthesis of compounds of pharmaceutical interest [3]. Fruit juice is being used on regular basis in various organic transformation reactions [4]. The widespread applications of different fruit juices are due to their non-toxic, safe, inexpensive and environmentally benign nature.

Aldimines are important intermediates for the synthesis of various bioactive products and they are used as fundamental materials for the synthesis of various Schiff base ligands which are used as chiral auxiliaries in asymmetric synthesis [5]. Aldimines have been reported to show a variety of biological actions by virtue of the azomethine linkage, which is responsible for various antibacterial, antifungal, herbicidal and clinical activities [6,7]. Based on the

literature survey, the present work focusses on the solvent free synthesis of aldimine from vanillin and *p*-nitroaniline using bilimbi fruit extract. Bilimbi extract contains several chemical constituents like vitamins, tannins, alkaloids, terpenoids and polyphenols. The aldimine synthesised from bilimbi fruit extract is characterized by UV-Visible and FT-IR spectral techniques. The synthesised aldimine shows slight activity on selected bacteria *Escherichia coli*, *Bacillus subtilis* and *Pseudomonas aeruginosa*.

2. Materials and Methods

Fresh and ripened bilimbi fruit were obtained from the local market. Vanillin and *p*-nitroaniline used for the synthesis of aldimine were procured from Merck. Double-distilled deionized water was used for the preparation of the bilimbi fruit extract.

2.1. Preparation of bilimbi fruit extract

Ripened bilimbi was used for the preparation of the extract. 25 g of this ripened fruit was thoroughly washed with distilled water, dried and cut into small pieces. Grind the pieces by a pestle and mortar and the resulting extract was filtered using Whatmann filter paper. The filtrate was collected and then centrifuged for about 8,000 rpm for about 10 minutes. The supernatant extract was collected and used for the synthesis of aldimine.

2.2 Synthesis of aldimine from bilimbi extract

The equimolar amount of vanillin (0.1 mol) and *p*-nitroaniline (0.1 mol) was taken in a beaker. Add 1 mL of bilimbi extract to the mixture and stirred at room temperature. The pale-yellow product was formed immediately after the addition of the extract. The product aldimine was washed with distilled water and purified by recrystallization with minimum amount of ethanol. The recrystallized sample was characterized by UV-Visible and FT-IR spectral techniques.

2.3 Antimicrobial Activity

Antimicrobial activities of synthesized aldimine against three bacteria *Escherichia coli*, *Bacillus subtilis* and *Pseudomonas aeruginosa* were assayed by Kirby-Bauer diffusion method. These antimicrobials were grown in LB broth for 24 h. Approximately 20 mL of molten and cooled Muller Hinton agar was poured into the Petri dishes. The tested organisms were swapped over the agar medium and the aldimine containing disks were kept over the medium using sterile forceps. Antimicrobial activity was evaluated by measuring the zone of inhibition for the test organisms. The diameters of zones were measured to the nearest millimetre with vernier calipers.

3. Results and Discussion

The role of bilimbi extract in the synthesis of biologically active aldimine from vanillin and *p*-nitroaniline is reported in this section. The synthesised aldimine is characterized by UV-Visible and FT-IR spectral analysis. The reaction for the formation of aldimine is shown in **Scheme 1**. This solvent-free approach is non-polluting and does not employ any toxic materials, quantifying it as a green approach for the synthesis of aldimines.



Scheme 1 Synthesis of aldimine from vanillin and *p*-nitroaniline

3.1 Absorption Spectral Analysis

The formation of aldimine from vanillin and *p*-nitroaniline using bilimbi fruit extract is preliminary confirmed by UV-Visible spectrophotometric analysis. The absorption spectrum of aldimine is carried out in ethanol. The aldimine synthesised from vanillin and *p*-nitroaniline shows absorption bands at 232, 278, 308 and 392 due to π - π^* and n - π^* transitions (**Fig. 1**). The higher energy band appearing at 278 nm is attributed to π - π^* transition of the azomethine group [8].

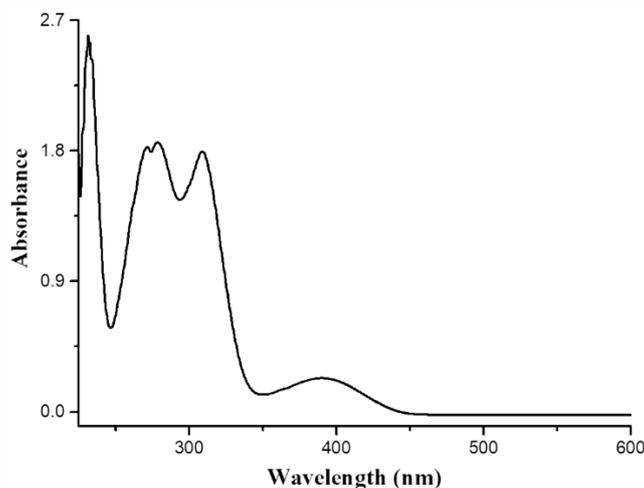


Fig. 1 UV spectrum of aldimine from vanillin and *p*-nitroaniline

3.2 FT-IR Spectral Analysis

The FT-IR spectrum of aldimine synthesised from vanillin and *p*-nitroaniline using bilimbi extract shows absorption bands at 3213, 3091, 2921, 2843, 2360, 1610, 1509, 1417, 1284, 1155, 1036, 961, 869, 759, 696, 548 and 474 cm^{-1} respectively (**Fig. 2**). The band at 3213 cm^{-1} is due to the O-H stretching of the hydroxyl group. Weak band at 3091 cm^{-1} is due to the stretching vibration of aromatic C-H group. The presence of weak bands at 2921 and 2843 cm^{-1} correspond to the C-H stretching of alkene and the aromatic system. The weak absorption

band at 2360 cm^{-1} is due to the presence of atmospheric carbon dioxide. The IR band at 1610 cm^{-1} is due to the presence of azomethine group, this confirms the formation of aldimine. The band at 1509 cm^{-1} represents the N-O stretching frequency of the nitro group. IR spectrum exhibits weak band at 1422 cm^{-1} due to the stretching vibration of aromatic C=C bond. The bands at 1284 and 1155 cm^{-1} indicate the C-O stretching of methoxy group. The band at 1036 cm^{-1} is due to ring stretching. Aromatic C=C and aliphatic C-H bending vibrations occur at 961 , 869 , 759 , 696 , 548 and 474 cm^{-1} respectively. The absorption spectral data and FT-IR analysis thus confirms the formation of aldimine.

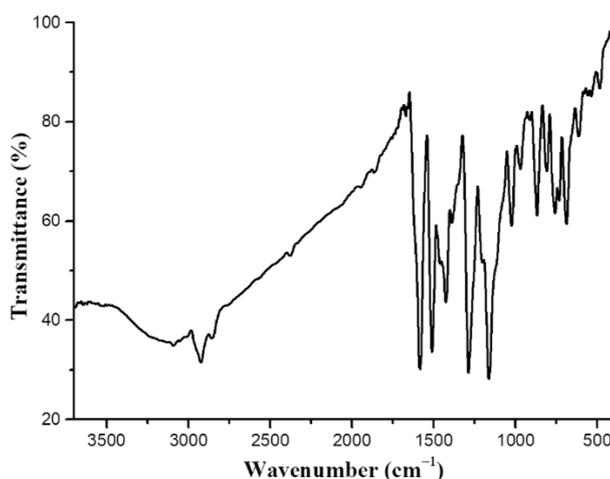


Fig. 2 FT-IR spectrum of aldimine synthesised from vanillin and *p*-nitroaniline

3.3 Antimicrobial activity of Synthesised Aldimines

Antimicrobial activity of the synthesized aldimine is tested against *Escherichia coli*, *Bacillus subtilis* and *Pseudomonas aeruginosa*. It shows slight activity on the selected microorganisms (**Table 1**). Standard antibiotic disc streptomycin is used as the reference drug for the evaluation of antibacterial activity. Thus, the synthesized aldimine from vanillin and *p*-nitroaniline using bilimbi fruit extract may have a potential use in the biomedical applications due to its antimicrobial activity.

Table 1 Antimicrobial activity of aldimine synthesised from vanillin and *p*-nitroaniline

Microbes	Zone of Inhibition (mm)	
	Control	Activity
<i>Escherichia coli</i>	27	8
<i>Bacillus subtilis</i>	30	10
<i>Pseudomonas aeruginosa</i>	20	7

Conclusion

An eco-friendly route for the synthesis of aldimine from vanillin and *p*-nitroaniline using bilimbi fruit extract has been investigated. The role of natural catalyst like bilimbi fruit extract in the synthesis of biologically active molecules has been well demonstrated. The synthesised aldimine shows an absorption maximum at 278 nm. The IR band at 1610 cm⁻¹ is due to the presence of azomethine group, this confirms the formation of aldimine. This solvent-free approach is non-polluting and does not employ any toxic materials, quantifying it as a green approach for the synthesis of aldimine. The synthesised compound shows slight antibacterial activity. The biological activity of this compound will trigger more interest in the synthesis of such compounds from the easily available starting materials.

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