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SYNTHESIS, CHARACTERIZATION AND ANTIMICROBIAL ACTIVITY OF SCHIFF BASE FROM BENZALDEHYDE AND PARA-TOLUIDINE USING GOOSEBERRY EXTRACT

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Abstract-Schiff's bases constitute a class of pharmaceutical and medicinally important molecules. The conventional methods for the synthesis of Schiff's bases require long reaction times and use of organic solvents.Conventionally, synthesis of Schiff base is carried out with or without acid catalyst by refluxing the mixture of aldehyde (or ketone) and an amine in organic medium. The utilization of green chemistry techniques are dramatically reduces chemical wastes and reaction time as recently hasbeen proven in several organic synthesis and chemical transformations. The objective ofthe present research consists of green methodologies for synthesis of Schiff base. Thus, the present study involves the use of gooseberryextract, as natural acid catalysts for the synthesis of Schiff base from benzaldehyde and *p*-toluidine. The Schiff base synthesised from gooseberry extract is characterized by UV-Visible and FT-IR spectral techniques. The antimicrobial efficacy of this Schiff base is tested against bacteria and fungi and it shows slight activity on *Staphylococcus aureus*, *Klebsiella pneumonia*, *Escherichia coli*, *Pseudomonas aeruginosa* and *Aspergillus flaves*.Compared with traditional methods, this methodis more convenient, low cost, simple to run, provide higher yield and shows maximum efficiencywith reduced reaction time.
Keywords: Green synthesis; Gooseberry extract; Schiff base; Characterization; 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 addconsiderably 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 easierseparations 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 [4,5]. The widespread applications of different fruit juices are due to their non-toxic, safe, inexpensive and environmentally benign nature [6].

Schiff bases 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 [7]. Schiff bases 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 [8-10]. Schiff bases have been associated with various significant catalytic and photochromic properties [11]. They form an interesting class of ligands that has enjoyed popular use in the coordination chemistry of transition, inner transition and main group elements.

Schiff bases are condensation products of primary amines with carbonyl compounds and they were first reported by Hugo Schiff in 1864. The common structural feature of these compounds is the azomethine group with a general formula $RN{=}CH{-}R_1$, where R and R_1 are alkyl, aryl, cycloalkyl or heterocyclic groups which may be variously substituted. Schiff bases that contain aryl substituents are substantially more stable than alkyl substituents. Schiff bases of aliphatic aldehydes are relatively unstable and readily polymerizable, while those of aromatic aldehydes have effective conjugation and stability [12]. The formation of a Schiff base from an aldehydes or ketones is a reversible reaction and generally takes place under acid or base catalysis, or upon heating.In recent years, environmentally benign green synthetic methods have received considerable attention for the synthesis of Schiff base. Many researchers have reported various eco-friendly methods for the synthesis of Schiff basesand the reported methodologies have some disadvantages such as prolonged reaction time, the high reactiontemperatures, an excess of costly dehydrating reagents/catalysts, moisture sensitive catalysts, and special apparatus [13,14].

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Based on the literature survey, the present work focus on the green synthesis of Schiff base from benzaldehyde and *para*-toluidine using gooseberry extract. Gooseberry is commonly known as amla and it belongs to the family Phyllanthaceae, one of the important herbal drugs used in Ayurvedic system of medical preparations against a variety conditions such as liver injury, atherosclerosis and diabetes [15,16]. Gooseberry is highly nutritious and is one of the richest sources of vitamin-C, amino acids and minerals. It contains several chemical constituents like tannins, alkaloids and phenols. Among all hydrolysable tannins, Emblicanin A and B, gallic acid, ellagic acid are reported to possess biological activity. The Schiff base synthesised from gooseberry extract is characterized by UV-Visible and FT-IR spectral techniques. The synthesised Schiff baseshows slight activity on *Staphylococcus aureus*, *Klebsiella pneumonia*, *Escherichia coli*, *Pseudomonas aeruginosa* and *Aspergillus flaves*.

2. Experimental Section

Fresh and ripened gooseberry fruit were obtained from the local market. Benzaldehyde and *p*-toluidine used for the synthesis of Schiff base were procured from Merck. Double-distilled deionized water was used for the preparation of the gooseberry extract.

2.1. Preparation of gooseberry extract

Ripened gooseberry 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 Schiff base.

2.2 Synthesis of Schiff base from gooseberry extract

The equimolar amount of benzaldehyde (0.1 mol) with *p*-toluidine (0.1 mol) was taken in a beaker. Add 1ml of gooseberry extract to the mixture and then kept for 5-10 minutes. Then the mixture was stirred for 10minutes at room temperature, pale yellow solid crude product was formed. After completion of the reaction, the product was washed with distilled water and purified by recrystallization with minimum amount of ethanol. The recrystallized sample was characterized by UV-Visible and FTIR spectral techniques.

2.3. Characterization Techniques

The absorption spectrum of Schiff basein ethanol was carried out using Shimadzu UV-1800 spectrophotometer. FTIR analysis of the Schiff base in ethanol was carried out through the potassium bromide (KBr) pellet (FTIR grade) method in 1:100 ratio and spectrum was recorded using Shimadzu IR Affinity-1. Fourier transform infrared spectrophotometer with the range of 4000-400 cm^{-1} at the resolution of 4 cm^{-1}.

2.4 Antimicrobial Activity

Antimicrobial activities of synthesized Schiff base against five bacteria and two fungi cultures of *Staphylococcus aureus*, *Klebsiella pneumonia*, *Escherichia coli*, *Pseudomonas aerogenisa*, *Aspergillus flaves* and *Candida albicans* were assayed by Kirby-Bauer discs 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 Schiff base 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 natural catalyst like gooseberry extract in the synthesis of biologically active Schiff base from benzaldehyde and *p*-toluidine is reported in this section. The synthesised Schiff base is characterized by UV-Visible and FTIR spectral analysis. The reaction for the formation of Schiff base 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 Schiff bases.

Scheme 1 Synthesis ofSchiff base from benzaldehyde and p-toluidine

3.1 Absorption Spectral Analysis

The formation of Schiff base using gooseberry extract is preliminary confirmed by UV-Visible spectrophotometric analysis. The absorption spectrum of Schiff base is carried out in ethanol. The Schiff base shows an absorption maximum at 296 nm (**Fig.1**). The higher energy bandappearing at 296 nm is attributed to π-π^* transition of the azomethine group [17].

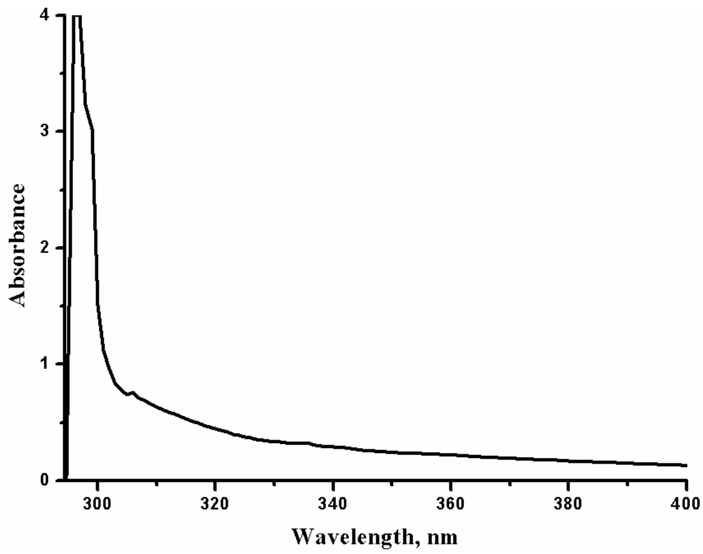

Fig. 1 UV spectrum of Schiff base synthesised from benzaldehyde and p-toluidine

3.2 FTIR Spectral Analysis

The FTIR spectrum of synthesised Schiff base shows bands at 3456, 2918, 2845, 1676, 1480, 1429, 1320, 1283, 1174, 927,808 cm^{-1} respectively (**Fig.2**). The FTIR spectrum of the synthesised Schiff base shows a band around 3456 $cm^{-1}$is due to the O–H stretching of the solvent ethanol, since the FTIR spectrum of Schiff base is taken in ethanolic solution. The presence of weak band at 2918 cm^{-1} and 2845 cm^{-1} corresponds to the C-H stretching of methyl group. The IR band at 1676 cm^{-1} is due to the presence of azomethinegroup, this confirms the formation of Schiff base.IR spectrum exhibits a weak band at 1480 and 1429 cm^{-1} is due to the stretching vibration of aromatic C=C.The band around 1320 cm^{-1} is the C-H bending of alkyl group. Absorption bands at 1283cm^{-1} and 1174 cm^{-1} is due to C-N stretching vibration. Aromatic C-H bending vibrations occur at 927 cm^{-1} and 808 cm^{-1} respectively. The absorption spectral data and FTIR analysis thus confirms the formation of Schiff base.

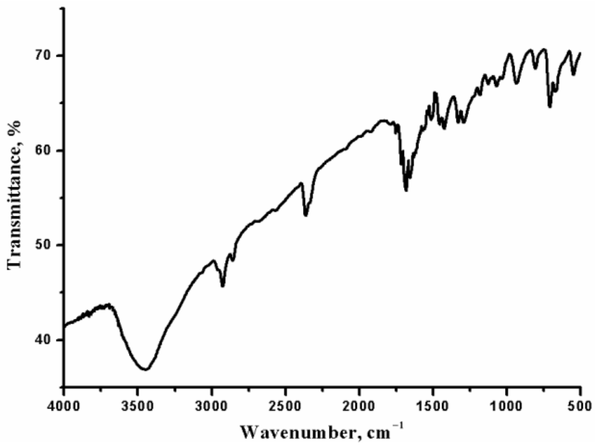

Fig. 2 FT-IR spectrum of Schiff base synthesised from benzaldehyde and p-toluidine

3.3 Antimicrobial activity of Synthesised Schiff base

Antimicrobial activity of the synthesized Schiff baseis tested against *Staphylococcus aureus*, *Klebsiella pneumonia*, *Escherichia coli*, *Pseudomonas aerogenisa*, *Bacillus cereusAspergillus flaves* and *Candida albicans*.It shows slight activity on *Staphylococcus aureus*, *Klebsiella pneumonia*, *Escherichia coli*, *Pseudomonas aeruginosa* and *Aspergillus flaves*(**Table 1**).Standard antibiotic disc Amikacin and Flucanazoleare used as the reference drug for the evaluation of antimicrobial activity.Thus the Schiff base synthesised from benzaldehyde and *p*-toluidine using gooseberry extract may have a potential use in the biomedical applications due to its antimicrobial activity.

Table 1 Antimicrobial activity of Schiff base synthesised from benzaldehyde and *p*-toluidine

Microbes	Activity
Escherichia coli	+
Klebsiella pneumonia	+

Pseudomonas aeruginosa	+
Staphylococcus aureus	+
Bacillus cereus	-
Candida Albicans	-
Aspergillusflaves	+

+ sign indicates slight activity, - sign indicates no activity

Conclusion

An eco-friendly route for the synthesis of Schiff baseform benzaldehyde and *p*-toluidine using gooseberry extract has been investigated. The role of natural catalyst like gooseberry extract in the synthesis of biologically active molecules has been well demonstrated. The synthesised Schiff base shows an absorption maximum at 296 nm.The IR band at 1676 cm^{-1} is due to the presence of azomethinegroup, this confirms the formation of Schiff base.This solvent-free approach is non-polluting and does not employ any toxic materials, quantifying it as a green approach for the synthesis of Schiff base. In addition to this, compared to traditional methods, this new method is clean, safe and more eco-friendly, involving mild reaction conditions and simple workup. The reaction conditions such as reaction time, use of hazardous solvents can be reduced by maintaining good yield of product.The antimicrobial activity results prove that the synthesized Schiff base can be used for the treatment ofdiseases caused by microbes.The biological activity of this compound will trigger more interest in thesynthesis of such compounds from the easilyavailable starting materials.

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