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Synthesis and Characterization of Schiff Bases from 1-Amino Naphthalen-2-Ol

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Abstract: *Schiff bases are a significant class of organic compounds exhibiting diverse applications in medicinal chemistry, catalysis, and material science. This study presents the synthesis and characterization of novel Schiff bases derived from 1-amino-2-naphthol through its condensation with various aromatic aldehydes under optimized reaction conditions. The synthesized compounds were characterized using spectroscopic techniques to confirm their structural properties. It is potential as effective antimicrobial agents. These Schiff bases also hold industrial significance and exhibit diverse biological properties. The synthesized compounds were structurally confirmed using thin-layer chromatography (TLC), melting point analysis, and spectral data, which were consistent with reported literature. This method provided excellent product yield, minimized solvent usage, and reduced reaction time, making it a sustainable and efficient approach for synthesizing Schiff bases. The findings support the advancement of greener methodologies in organic synthesis and highlight the significance of these compounds in pharmaceuticals and industrial applications.*

Keywords: Schiff bases, 1-amino-2-naphthol, synthesis, characterization, spectroscopy, azomethine, bioactive compounds, catalysis

I. INTRODUCTION

Schiff base are organic compounds possessing azomethine group (-C=N-)which resulted from condensation of amine with aldehyde or ketone Azomethines play pivotal role as key intermediates inorganic synthesis particularly of heterocyclic compounds , Schiff bases are reported to show a variety of interesting biological activities, including antifungal, antimicrobial, anticancer and antitumor activities

Schiff's bases, named after Hugo Schiff, are formed when, under specific conditions, any primary amine interacts with an aldehyde or a ketone. Structurally a Schiff's base is an aldehyde or ketone nitrogen analogue in which the imine or the azomethane group has been substituted for the carbonyl group, and it has an ability to modulate the activities of many enzymes involved in metabolism. The pharmacophore potential of this group is due to its ability to form complex compounds with bivalent and trivalent metals found at the active core of various enzymes involved in metabolic reactions. Azomethane pharmacophore is used in the production of new bioactive molecules.

Schiff's base has attracted a lot of attention due to its broad range of promising applications such as antibacterial activity3, anti-fungal [4], anticancer 5,antidepressant 6, antioxidant, andanalgesic[7].

Matarand co-workers have reported the synthesis of 3, 3-diaminodipropylamine, and different derivatives of benzaldehyde having excellent bactericidal and fungicidal properties 8. Teran and co-workers have reported a series of new Schiff's base from 4-aminoantipyrine with excellent antibacterial, anti-fungal, leishmanicidal, and antioxidant activities for clinical applications 9. The goal of the present research is to develop novel Schiff's bases for potential applications as antimicrobial agents. Herein, we report the preparation of Schiff's base derived from 1-(4-amino-4 phenyl) 3-phenyl triazene-1-ol), Aldehyde with reasonably good antibacterial and antifungal activities.

Santosh Kumar and coworker synthesized Schiff base from 4-amino benzene sulphonamide and substituted aromatic aldehydes. Muhammad Aqeel Ashraf and co-worker, reported a series of Schiff bases from 2-amino-Benzthiazole, 4-amino-Salicylic acid and 4-aminophenol.[22]

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Muhammad Aslam, et all synthesized Schiff base by mixing of Aminophenol with 4-chloroacetophenon or 4-hydroxyacetophenone and the reaction mixture was refluxed for 3 h with stirring at 70oC after adding 3-4drops of conc.H2SO4. 4-Chloro-2-oxo-2H-chromene-3-carbaldehyde was made to react with different anilines in rectified spirit to yield a series of Schiff bases of the type 4-chloro-3-(substituted-phenylimino) methyl)-2H-chromen-2-one reported by S. Bairagi et al. Bag et al have synthesized a series of Schiff bases of benzidene with series of substituted aromatic aldehydes and examined the mercuration reaction.

K. Mounika, B. Anupama and co-worker, prepared some Schiff basses by treating of 3-ethoxy salicylaldehyde and 2-amino benzoic in ethanol. Synthesis and pharmacological studies of novel Schiff bases of 4-Hydroxy -6-carboxyhydrazino benzofuran and their metal complexes were reported by Gopal Krishna Rao et al.

Vijay Aanandhi et al have reported the synthesis of a series of 1-(5-substituted-2-oxoindolin-3-ylidene)-4-(substituted-pyridin-2yl)thiosemicarbazide derivatives. These compounds were screened for in vitro antibacterial and antifungal activity against B.subtilis ,S. aureus, E.coli, P. aeruginosa, C. albicans, and A. niger. All the compounds were reported to exhibit moderate to good antibacterial and antifungal activity. Metal complexes of Schiff bases derived from 2-furancarboxaldehyde and o-phenylenediamine and 2-thiopheneacarboxaldehyde and 2-aminothiophenol was reported by Gehad Geindy et al, These authors have reported the ligand dissociation as well as the metal-ligand stability constants for these complexes. The synthesized ligands, in comparison to their metal complexes were also screened for their antibacterial activity against bacterial species, Escherichia coli, Pseudomonas aeruginosa and Staphylococcus Pyogones as well as fungi (Candida). The activity data reveal that the metal complexes are found to be more potent antibacterial than the parent Schiff base ligand against one or more bacterial species.

Z.H. Chohan and S. Mushtaq synthesized A series of biologically active pyrazine derived Schiff base ligands have been synthesized by the condensation reaction of 2-aminopyrazine with salicylaldehyde and acetamidobenzylaldehyde. Then their Co(II), Ni(II) & Zn (II) complexes have been prepared. The biological evaluation of the simple uncomplexed ligand in comparison to their complexes have been determined against bacterial strains namely Escherichia coli, Staphylococcus aureous and Pseudomonas aeruginosa.

Natarajan Raman et al [36] have reported the synthesis of a novel 14-membered macrocyclic Schiff base derived from 3-cinnamalideneacetanalide and o-phenylenediamine which acts as a tetradentate and strongly conjugated ligand to form a cationic solid complex with Cu(II)/Ni(II)/Co(II) and /Zn(II). The ligand and the complexes were characterized by the usual spectral and analytical techniques. The antimicrobial tests were also recorded and gave good results in the presence of metal ions in the ligand system. S. Bawa and coworker synthesized A series of 4-substituted-emoni methyltetrazolo[1,5-a]quinoline with appropriate aromatic amine by refluxing in dioxane. They have been evaluated for their anti-inflammatory and antimicrobial activities.

Today coordination chemistry comprises a large body of inorganic chemistry research. It is mainly the chemistry of metal complexes and has fascinated and inspired the chemists all over the world. There is an ever increasing academic, commercial and biochemical interest on the metal complexes of organic chelating ligands. This has resulted in the emergence of associated fields like organometallic chemistry, homogeneous catalysis and bioinorganic chemistry. Among the chelating ligands, Schiff bases have attracted the attention of chemists due to the ease of preparation and complexation.

Mohammed and co-worker synthesized Schiff's bases by condensation of acrolein with 2-aminophenol, 2-aminophenol with cinnamaldehyde and similarly cinnamaldehyde with phenylene diamine. The prepared Schiff's bases were identified by UV-Vis, IR, CHN analysis and NMR spectroscopy. The main aim of this work was to use Schiff bases as inhibitors for corrosion of carbon steel in acidic media 0.5 N HCl. The rate of corrosion was measured by electrochemical and weight loss methods. They found that Schiff bases inhibited the corrosion prominently, as they have ability to form spontaneously a monolayer on the surface to be protected. This tendency is shown by Schiff bases, as they contain unoccupied π^* Orbitals, which enable electron backdonation from metal d orbitals and thereby stabilize the existing metal-inhibitor bond

Badami and co-workers developed an efficient green approach for the synthesis of Schiff bases of 1-amino-2-aryl-3-oxo-1,2,4-triazoles using Mg(ClO4)2 as catalyst followed by the reaction with chloroacetyl chloride in solvent-free conditions to yield the azetidinones with excellent yields. The synthesized compounds were evaluated for the extent of

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penetration into biological membranes (clogP), drug-likeliness and finally drug score was calculated and also screened for antitubercular and antimicrobial activities. MIC values of the final compounds were evaluated respectively and promising results were obtained for the compounds as depicted by the Osiris property explorer and some compounds have exhibited excellent activity against tubercular strain H37Rv. The developed protocol is simple, efficient and the easy to operate.

1-amino-2-hydroxy-3-(4-nitrophenylazo)- 4-naphthalene sulfonic acid with salicylaldehyde. Later on, they have synthesized copper (II), nickel (II), and zinc (II)complexes of the Schiff base ligands and characterized by spectroscopic methods, magnetic measurements, elemental, and thermogravimetric analysis [26]. Kalhor and group has developed a simple and efficient method for the synthesis of some novel Schiff bases via the reaction of aromatic aldehydes with2-aminobenzimidazole by using catalytic amount of metal nitrates in an organic solvent at room temperature. Some advantages of this protocol were its very good yields, use of available catalysts, simple workup procedure, and short reaction times. Ni $(NO3)_2$ $.6H_2O$was employed as a catalyst for the preparation of Schiff bases containing benzimidazole moiety by reaction of 2-aminobenzimidazole and aromatic aldehydes. The attractive features of the developed procedure are its good conversions, easy workup, and short reaction times, making it a useful practical method for the synthesis of Schiff bases [27]

From above literature survey promoted us to synthesize some important Schiff bases from 1-amino-2- naphthol derivatives. Basically 1-amino-2-naphthol derivative synthesized from aniline. Aniline undergoes diazotization and then coupling with 2- naphthol which on reduction with stannous chloride it gives derivatives of 1-amino-2- naphthol. Then this derivative reacted with different benzaldehyde and substituted benzaldehyde using fresh lemon juice in mortar and pestle.

II. MATERIALS AND METHODS

2.1 Materials

All the chemicals and reagents used were of Analytical grade and Laboratory grade. And used without further purification. 1-amino-2-naphthol, aromatic aldehydes, water, ethanol, round bottom flask, magnetic stirrer, glass rod, magnetic needle, etc.

2.2 Methods:

a. General Procedures

Melting points were determined in open capillary tubes and are uncorrected. The purities of the compounds were checked on silica-gel-coated Al plates.

b. Experimental Procedures:

i) Synthesis of Diazonium salt:

Aniline 4 (4.5 cm^3) was dissolved in concentrated hydrochloric acid (16 cm3) and distilled water (16 cm3). The reaction mixture was shake gently to dissolve any hydrochloride which might have separated and the solution was cooled to a temperature of 5 oC. Sodium nitrite (4 g) dissolved in 20 cm3 of water and 1 spatula of urea was added with constant stirring at a temperature of 0-5 oC. Diazotization was achieved by gradually adding the cold solution of sodium nitrite to a cold solution of aniline with constant stirring, making sure the temperature never exceeds 5 oC.

ii) Coupling Reaction

A solution of 2-naphthol was prepared by dissolving 2-naphthol (5 g) in 45 cm3 of 10 % NaOH in a 250 cm3 beaker with constant stirring. This was followed by slow addition of the cold diazonium salt solution and the reaction mixture was further cooled below 5 oC by placing it in an ice bath and by direct addition of crushed ice (25 g). A red-orange colour and red-orange crystal develops and eventually separate. The reaction mixture was further allowed to stand in an ice bath for 30 minutes with constant stirring, after which it was filtered through a Buchner funnel and washed with water. The residue was air dried for 3 day

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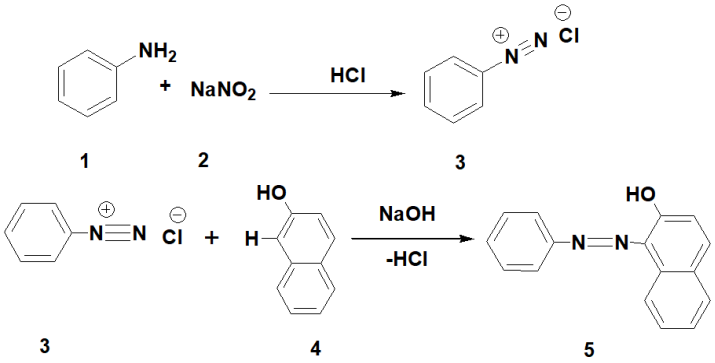
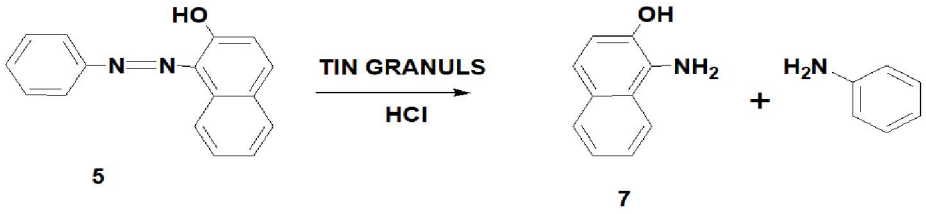


iii) Reduction Process

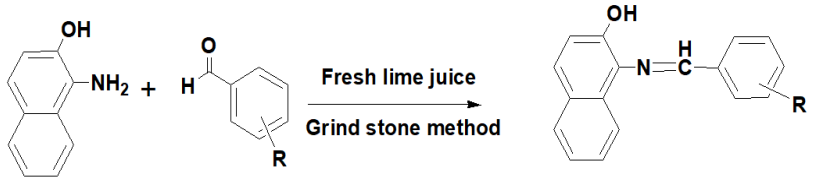
1-(1-Phenylazo)-2-Naphthol to 1-Amino-2-Naphthol Hydrochloride

The crude uncrystallized 1-(1-phenylazo)-2-naphthol 1 (8.00 g) was dissolved in a beaker containing 60 cm3 of methylated spirit. This was poured into a round bottom flask fitted with a reflux condenser. The mixture was boiled gently until most of the azo compounds have dissolved. A solution of Tin(II)chloride (20.0 g) dissolved in 60 cm3 of concentrated HCl was warmed to produce a clear solution which was added to the contents of the flask and refluxed for further 30 minutes, for the formation of slight dark colour precipitate which was poured into a beaker, placed in an ice bath of cooling process until the crystal of 1-amino-2-naphthol hydrochloride appeared. The crystal obtained were filtered and recrystallized using 2 cm3 of hot water which contains two drops of Tin(II)chloride solution in an equal weight of concentrated HCl then dried for 3 days in desiccator and the percentage yield determined. The yield of 1-amino-2-naphthol hydrochloride obtained was 72.2 %



iii) Synthesis of Schiff bases:

1-amino-2napthol and substituted benzaldehyde, and lime jwat were added into a 250 ml of beaker at room temperature. Then the mixture was stirred over a grindstone for 10-15minutes. After completion of the reaction, the solid product was collected by simple filtration and washed with water. The crude product was purified by recrystallization from ethanol. The obtained product was determined by tlc and melting point



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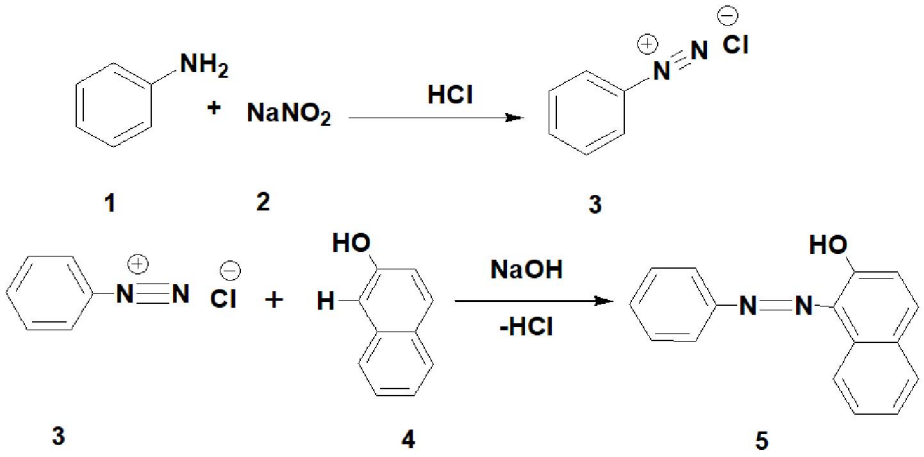
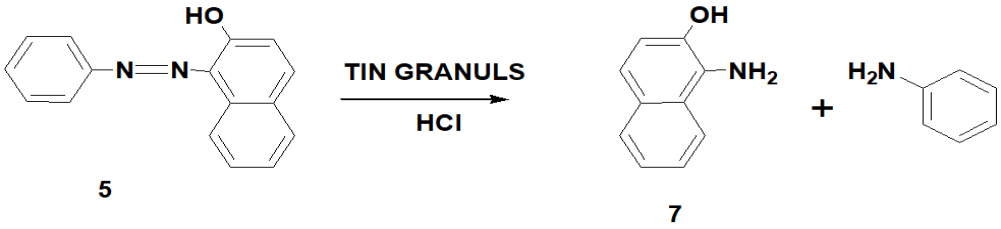
III. SCHEMATIC WORK

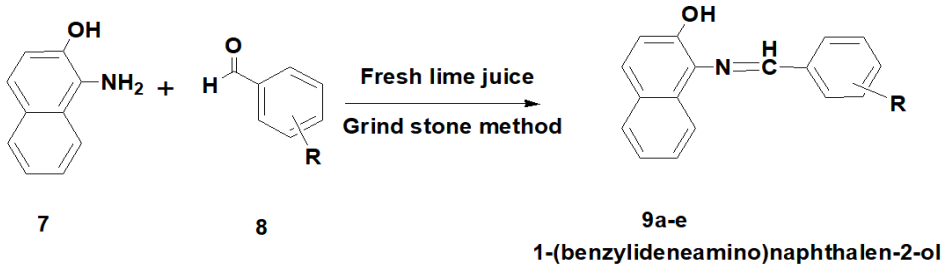
(A) Scheme 1. Synthesis of Azo dye using 2- Naphthol coupling



(B) Scheme 2. Synthesis of 1-amino-2-naphthol from Azo Dye



(C) Synthesis of 1-(benzylidene amino)phthalen-2-ol from 1-amino-2-Naphthol & different substituted aromatic aldehyde.



IV. RESULT AND DISCUSSION

Synthesis of Novel Schiff bases from aromatic aldehyde and 1-amino-2-napthol were studied by using fresh lemon juice as catalyst. (scheme 1).

4.1 Schematic work:

(A) Scheme 1. Synthesis of Azo dye using 2- Naphthol coupling

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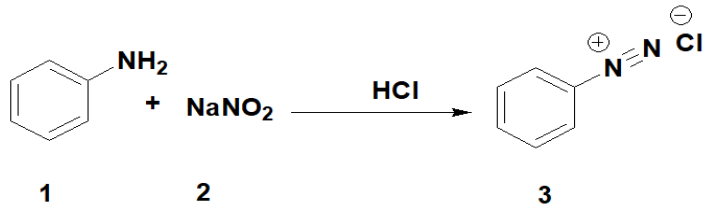
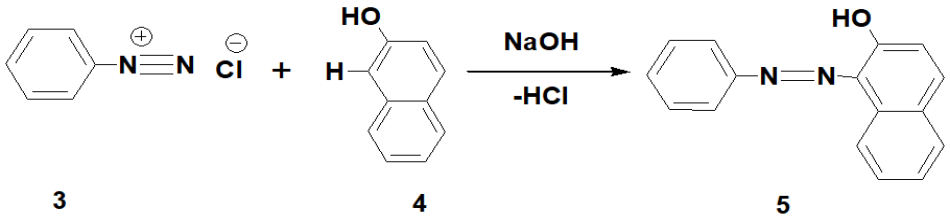
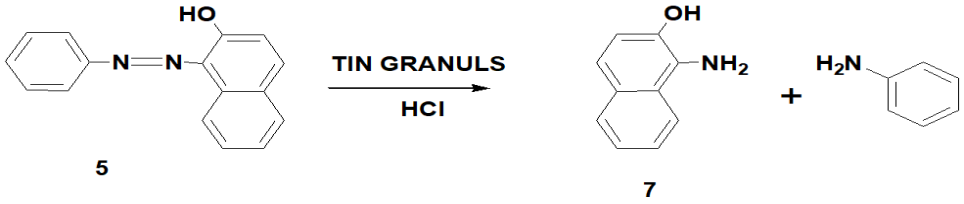




(B) Scheme 2. Synthesis of 1-amino-2-naphthol from Azo Dye



(C) Synthesis of 1-(benzylidene amino)phthalen-2-ol from 1-amino-2-Naphthol & different substituted aromatic aldehyde.

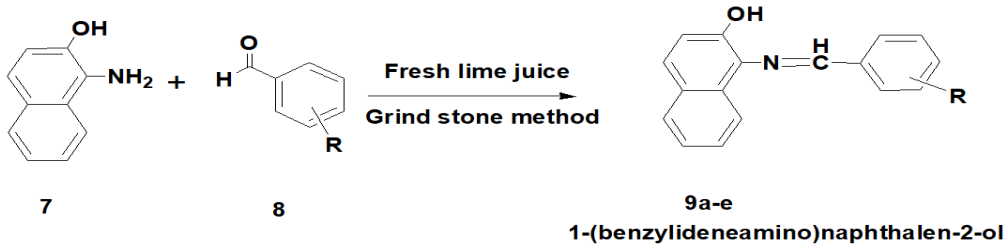
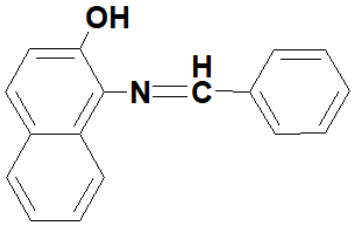


Table1: Physical data of Novel 1-(benzylidene amino) phthalen-2-ol

Sr. No.	R	Melting point	Percentage yield	Time required in Minutes
9a		172°C	65	15min.

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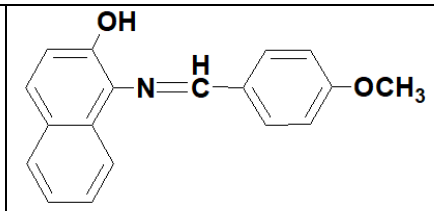
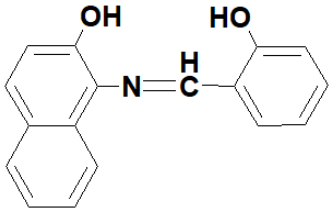
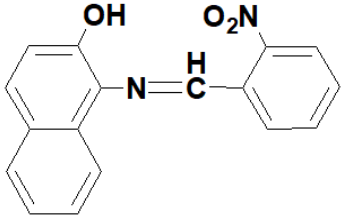
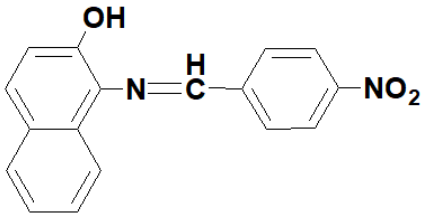
9b		113°C	80	10min.
9c		137°C	56	10min.
9d		122°C	65	10min.
9e		147°C	78	12min.

The compounds were prepared in good yield by synthesis of Novel schiff bases by aromatic aldehyde and 1-amino-2napthol using fresh lemon juice as catalyst in water as a solvent. Better yields are obtained using vinegar as catalyst and time duration of reaction is also less. Work up of reaction is also easy. The compounds synthesized and their yields are presented in Table 1.

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