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Synthesis and Spectral Characterization of N-[(2-CARBOXYPHENYL-3- DIAZENYL SALICYLIDINE) -2-AMINO BENZOIC ACID]

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ABSTRACT: Schiff bases are known to be a versatile class of compounds, and their applications ranging from simple analytical agents to anticancer agents are well established[1-3]. A diazodye has been synthesized from a salicylaldehyde derived Schiff base is reported in this work. The dye synthesized is subjected to spectral characterization. Further its utility to be explored.

KEYWORDS: Schiff bases, diazodye, reflux, ethanolic solution,

I.INTRODUCTION

Schiff bases

Schiff bases are condensation products of primary amines and carbonyl compounds and they were discovered by a German chemist, Nobel Prize winner, Hugo Schiff in 1864[1] .Structurally, Schiff base (also known as imine or azomethine) isan analogue of a ketone or aldehyde in which the carbonyl group (C=O) has been replaced by an imine or azomethine group[2].

Schiff base ligands are essential in the field of coordination chemistry,especially in the evelopment of complexes of Schiff bases because these compounds are potentially capable of forming stable complexes with metal ions[3]

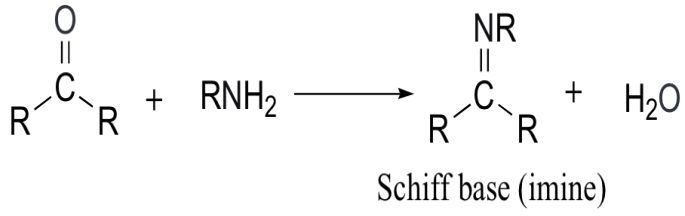

Fig. 1. General structure of Schiff bases

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A large number of Schiff base complexes are characterized by an excellent catalytic activity in a variety of reactions at high temperature (>100ºC) and in the presence of moisture. In recent years, there have been numerous reports of their use in homogeneous and heterogeneous catalysis[5,6]

Schiff bases and their metal complexes are increasingly being used as catalysts in various biological systems, polymers and dyes. Moreover, it is confirmed that these compounds can act as enzyme preparations[7].Due to the excellent selectivity, sensitivity and stability of Schiff bases for specific metal ions such as Ag(II), Al(III), Co(II), Cu(II),Gd(III), Hg(II), Ni(II), Pb(II), Y(III) and Zn(II), a large number of different Schiff base ligands have been used as cation carriers in potentiometric sensors.

Studies in terms of catalytic properties of Schiff bases exhibit the catalytic activity in the hydrogenation of olefins. One of the more interesting applications of these compounds is the possibility to use them as effective corrosion inhibitors. This phenomenon is the spontaneous formation of a monolayer on the surface to be protected.

The interest in metal complexes in which the Schiff bases play a role as the ligands are increasing as evidenced by the number of publications appearing annually (approximately 500)[8]. So much interest in imines can be explained by the fact that they are widely distributed in many biological systems and they are used in organic synthesis and chemical catalysis, medicine, pharmacy and chemical analysis, as well as new technologies[9].

ANTIFUNGAL PROPERTIES

Exploration and development of more effective antifungal agents is necessity, and the individual Schiff bases are considered to be promising antifungal medicines. Some of them, such as imine derivatives of quinazolinones possess antifungal properties against candida albicans, Trichophyton rubrum, T. mentagrophytes, Aspergillus niger and Microsporum gypseum.

BIOCIDAL PROPERTIES

Schiff bases obtained by the synthesis of o-aminobenzoic acid and β-keto esters have found biocidal use against S.epidermidis, E. coli, B.cinerea and A. niger[2].By contrast, Schiff bases of isatin derivatives are used in the destruction of protozoa and parasites[10].

ANTIVIRAL PROPERTIES

The use of vaccines may lead to the eradication of pathogens known viruses, such as smallpox, poliomyelitis (polio), rubella. Although there are many therapeutic ways to work against viral infections, currently available antiviral agents are not fully effective, which is likely to cause a high rate of mutation of viruses and the possibility of side effects. Salicylaldehyde Schiff bases derived from 1-amino-3-hydroxyguanidine tosylate are good material for the design of new antiviral agents[4].

Isatin Schiff base ligands are marked by antiviral activity, and this fact is very useful in the treatment of HIV[11].

ANTIMALARIAL PROPERTIES

Malaria is a disease which when is neglected causes serious health problems. Human malaria is largely caused by four species of the genus Plasmodium (P. falciparum, P. vivax, P. ovale and P. malariae). The search for new drugs, vaccines and insecticides for the prevention or treatment of this disease is a priority. Schiff bases are interesting compounds, which could be part of antimalarial drugs.

ANTICANCER PROPERTIES

Some Schiff bases have a high antitumor activity. Imine derivatives of N-hydroxy-N'-aminoguanidine block ribonucleotide reductase in tumor cells, so that they are used in the treatment of leukemia[12].

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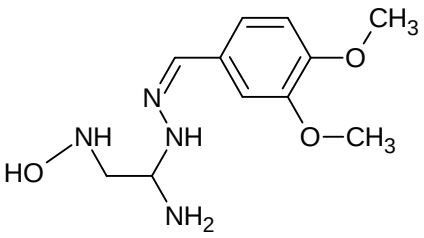

Fig.2 An antitumor guanidine Schiff base

OTHER SCIENTIFIC APPLICATIONS

- Photo- and thermochromic properties of Schiff bases as well as their biological activity make them applicable in modern technology.
- They are used in optical computers, to measure and control the intensity of the radiation, in imaging systems, as well as in the molecular memory storage, as organic materials in reversible optical memories and photodetectors in biological systems[13].
- Due to photochromic properties, Schiff compounds could behave as photostabilizers, dyes for solar collectors, solar filters. They are also exerted in optical sound recording technology.
- worthy of interest in the properties associated with Schiff rules include: properties of liquid crystal, chelating ability, thermal stability, optical nonlinearity and the ability to create the structure of a new type of molecular conductors using electrical properties to proton transfer.
- Because of their thermal stability Schiff bases can be used as stationery phase in gas chromatography.

II. SIGNIFICANCE OF THE SYSTEM

Azo compounds were the most important class of substances which received scientific attention. They are highly stable over temperature and pH when compared to other natural dyes. Schiff base ligands are essential in the field of coordination chemistry, especially in the development of complexes of Schiff bases because these compounds are potentially capable of forming stable complexes with metal ions. Studies in terms of catalytic properties of Schiff bases exhibit the catalytic activity in the hydrogenation of olefins. Since both the Schiff base and the diazo dyes are having extensive conjugation in their structures, they can be exploited as fluorescence markers and / or chemo sensors. The diazo dye can be investigated for its application as an acid base indicator.The electrochemical properties of the Schiff base can be studied by modifying electrodes with the Schiff base or its polymer. As the first step of foresaid possibilities, the present work attempts to synthesize and characterize the Schiff base and the diazo derivative of it.

III. METHODOLOGY

Salicyladehyde (S.D.Fine Chemicals, India) , Antharanilic acid (Sigma Aldrich) and absolute ethanol were of Analytical Grade and used as such. Other reagents used namely, NaCl, $NaNO_2$ and Con.HCl was of General Reagent grade. Doubly distilled water was used throughout the preparation.

Stage 1: SYNTHESIS OF 2-(N-SALICYLIDIENE AMINO) BENZOIC ACID

To a stirred ethanolic solution of anthranilic acid (0.02 mol, 2.75 g), salicylaldehyde solution in ethanol (0.02 mol, 2.13 ml) was added, and stirring was continued for fifteen more minutes. A bright orange precipitate of the Schiff base was formed almost in quantitative amount (4.68 g, 96 % yield). The precipitate was recrystallized from 90% alcohol.

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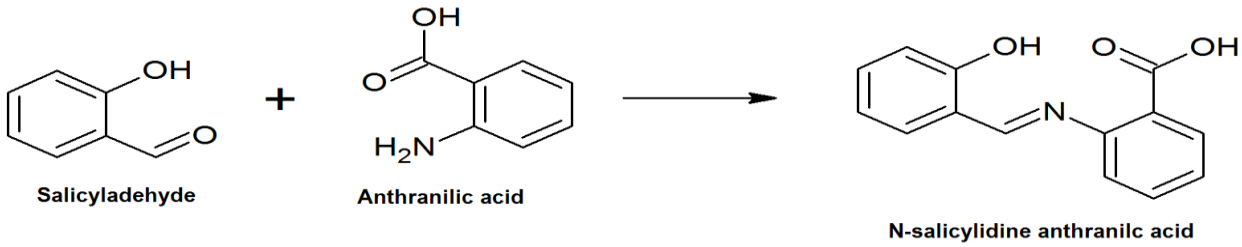

Fig. 3. Synthesis of the Schiff base N-Salicylidiene anthranilic acid

Stage 2: SYNTHESIS N-[(2-CARBOXYPHENYL-3- DIAZENYL SALICYLIDINE) -2-AMINO BENZOIC ACID] from 2-(N-SALICYLIDIENE AMINO) BENZOIC ACID

Anthranilic acid (0.01 mol, 1.37 g) was dissolved in 4 ml of concentrated HCl and 4 ml of water, cooled in ice. The anthranilic acid was diazotized under cold condition (temperature less than 5^{o} C) by adding a cold aqueous solution of $NaNO_2$ in 5 ml of water with stirring followed by alkaline solution of Schiff base (0.01 mol, 2.41 g) in 3 ml of 10% NaOH. A reddish brown precipitate of the dye was formed which was washed with cold water and filtered under suction. The yield was about 62%.

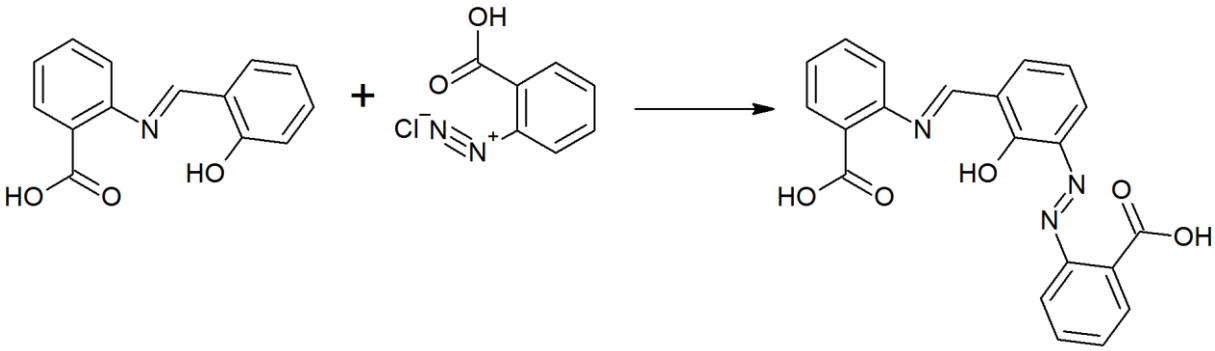

Fig. 4. Synthesis of diazo dye from the Schiff base and anthranilic acid

Synthesized compound was characterized by the spectral techniques of UV, IR and H1,C13 NMR spectra. For recording the UV-Visible spectra, a solution of the compound in ethanol (1x 10-4 M) was used. The spectrum was recorded by using a double beam UV spectrometer (ANALYTIKJENA – SPECORD 200 PLUS). FTIR spectrum of the sample was recorded by KBr pellet technique in Bruker α-T instrument. NMR spectrum was recorded in CDCl3 solution by using BRUKER NMR spectrometer at instrumentation facility VIT University, Vellore. Structures of the molecules are confirmed by comparing the spectra with standard literature.

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III. EXPERIMENTAL RESULTS

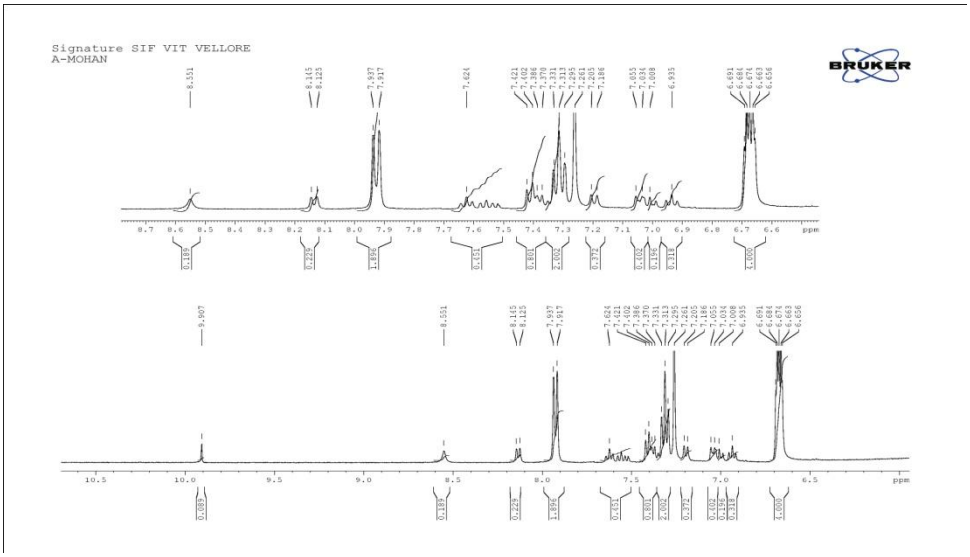

Fig 5: H1NMR of Schiff base N-Salicylidiene anthranilic acid

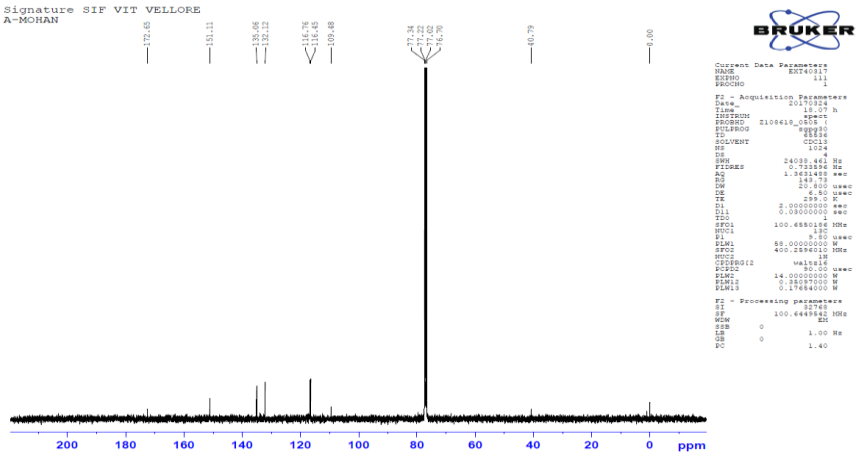

Fig 6: C13NMR of Schiff base N-Salicylidiene anthranilic acid

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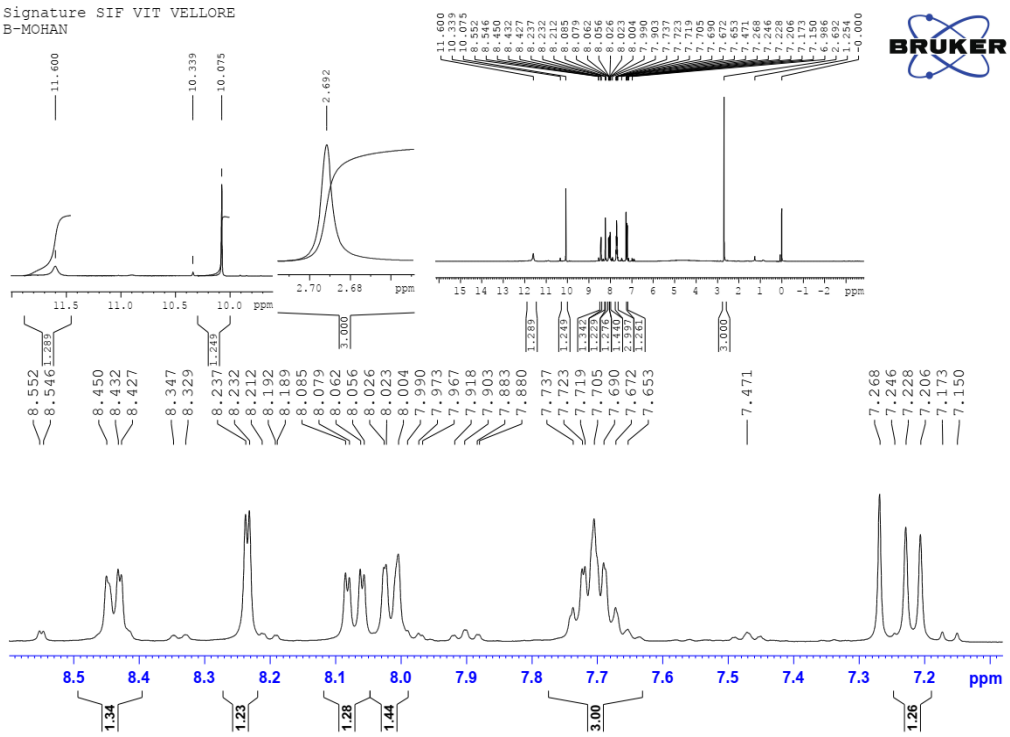

Fig 7: H1NMR of diazo dye from the Schiff base and anthranilic acid

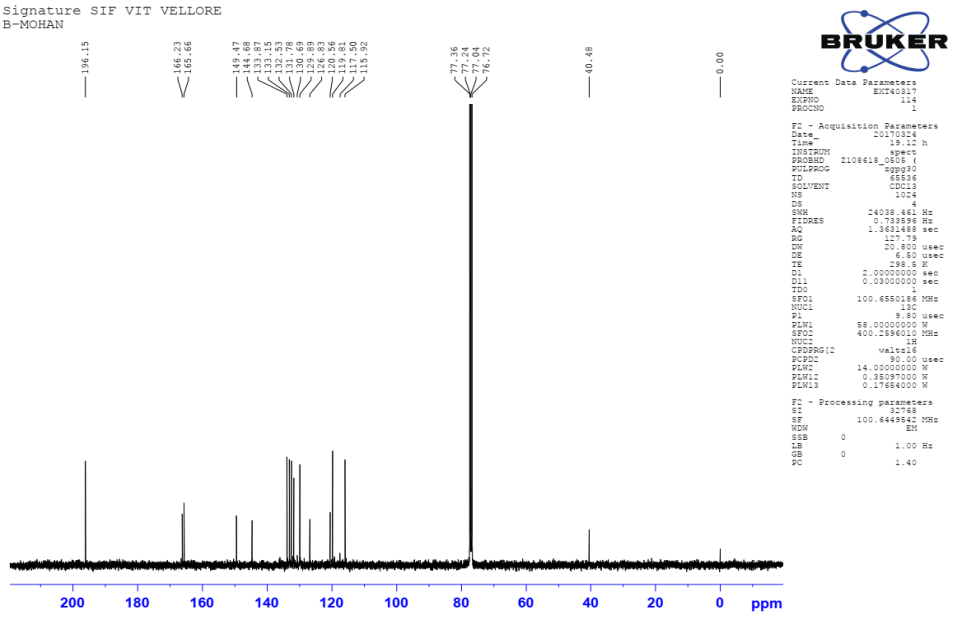

Fig 8: C13 NMR of diazo dye from the Schiff base and anthranilic acid

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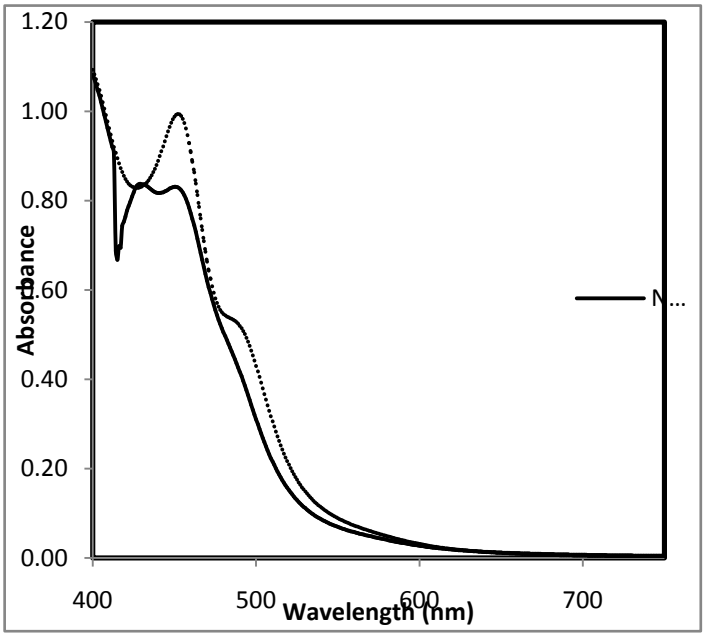

Fig 9: UV of Schiff base & diazo dye with N-Salicylidiene anthranilic acid

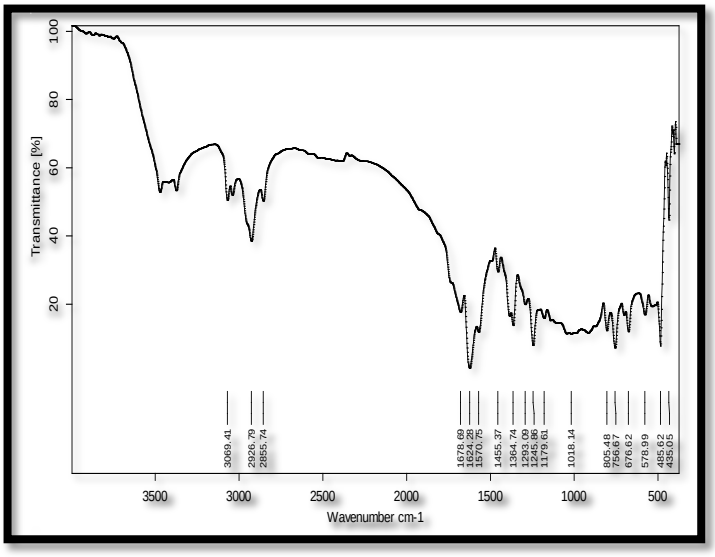

Fig 10: FTIR of Schiff base N-Salicylidiene anthranilic acid

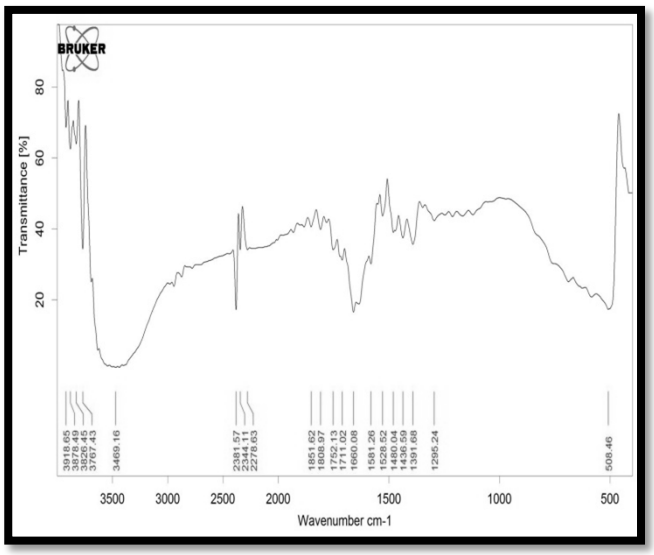

Fig11: FTIR of diazo dye from the Schiff base and anthranilic acid

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IV DISCUSSION

Schiff base of salicylaldehyde and anthranilic acid was synthesized by conventional reflux procedure. NMR spectra were theoretically predicted, compared with the experimental spectra and found to be in good correlation, confirming the expected products formation. The 1H NMR spectra of the azo based salicylaldehyde showed singlet peak that appeared at a range of δ 10.03-10.34 for aldehydic protons whereas the Schiff base showed sharp singlet peak at δ 8.74 which may be due to the presence of azomethine group. The absorption band in the range of 320-380nm shows the spectra of Schiff base N-Salicylidiene.The broad visible absorption band showed by adiazo is in the range of 450-480nm has been assigned to n-π* transition. The IR spectra of the synthesized compounds are studied in the range of 3500-400cm−1. The IR absorption band in the range of 1660-1711 cm−1 indicates the presence of carbonyl stretching of salicylaldehyde in all the azo compounds. The medium IR absorption band appeared in the the range of 2926- 3069 and 2926-2855 cm−1 which may be due to aldehydic hydrocarbons. The broad absorption bands seen in the range of 3469-3767 cm−1 may be due to presence of –OH stretching in all the compounds.

V. CONCLUSION AND FUTURE WORK

Schiff base of salicylaldehyde and anthranilic acid was synthesized by conventional reflux procedure.Purity of the synthesized compound was ascertained by running a paper chromatogram with benzene-ethanol (1:3) solvent system.Synthesized Schiff base was characterized by UV, IR , 1-H and 13-C NMR and the standard values got correlated with the sample peaks.The Schiff base was coupled with dizotized anthranilic acid to form a red brown diazodye.The dye was also characterized by the spectral methods like UV,IR NMR and Mass spectrometry.NMR spectra were theoretically predicted, compared with the experimental spectra and found to be in good correlation, confirming the expected products formation.

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