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Schiff base ligand of p-Toluidine and Salicylaldehyde: Synthesis, characterization and metal ion sensing

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Abstract
At first synthesis of Schiff base ligand by using p-toluidine & salicylaldehyde has been prepared at a certain pH by using dry ethanol as a solvent. Then its characterization by UV-Vis, 1H-NMR and fluorescence spectra are investigated and discussed. The UV-vis absorption spectra and fluorescence spectra are investigated in THF and methanol solvent respectively. Investigation of metal ion (Al3+) sensing has been done by adding different metal ion concentration and check fluorescence.

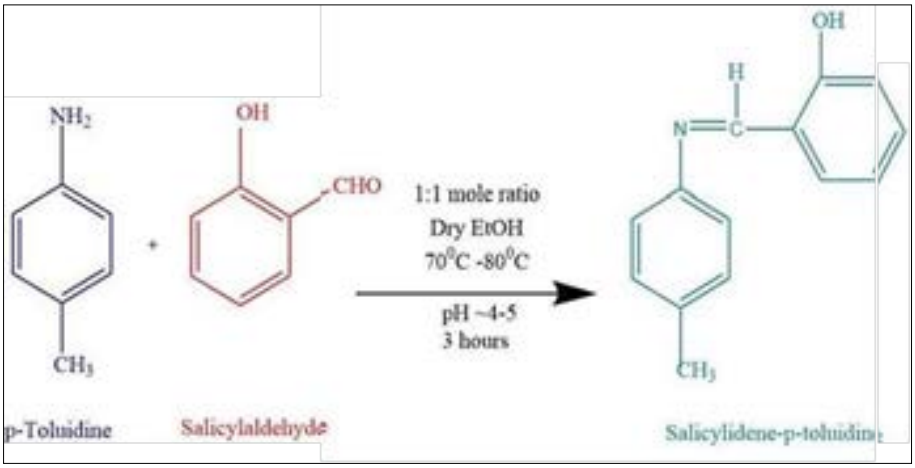
Keywords: Schiff bases, p-Toluidine, salicylaldehyde, UV, Fluorescence, 1H-NMR spectroscopy

Introduction
In the field of organic chemistry, Schiff bases have gained significant attention due to their diverse applications. The synthesis of Schiff bases involves the reaction of a primary amine with an aldehyde or ketone, resulting the formation of an imine bond. One such important Schiff base is formed by reaction of p-toluidine and salicylaldehyde.
Schiff base have drawn attention due to their fascinating properties such as colour, biological activity and coordination behavior with metal ions. The synthesis of Schiff bases can be accomplished using various methods, including condensation reactions. In this article, we will discuss the synthesis of a Schiff base formed by the reaction of p toluidine and salicylaldehyde and its characterization through UV, fluorescence and NMR spectroscopy.
Understanding the synthesis and characterization of Schiff bases is crucial for organic chemists and researchers working in the field. The use of spectroscopic techniques allows us to gain insight into the structure, electronic transition, and chemical environment of the synthesized compound. By exploring the process of synthesizing the Schiff base of p-toluidine and salicylaldehyde and characterizing it through UV, fluorescence, and NMR spectroscopy, we can enhance our knowledge and contribute to advancements in the field of organic chemistry. Let's delve into the details of this fascinating synthesis and characterizations.

Materials and Methods
Material: Dry ethanol, round bottom flask, glacial acetic acid, pipette, reflux tube, balloon (filled with N2 gas for inert medium), silicon oil bath, tissue paper, filter paper etc.

Procedure: A round bottom flask was taken with 20ml ethanol in it. Then calcium hydride (CaH2) of 2g was added to it. It was stirred with a magnetic bid for about 12 hours. Then the ethanol was refluxed until no more calcium hydroxide precipitate was present in the pure ethanol collected.
After ethanol was dried then the amine (p-toluidine) & aldehyde (salicylaldehyde) was taken in equimolar quantity i.e. 1:1 in round bottom flask. In that mixture specific amount (4to 5 drops) of glacial acetic acid was added to maintain the pH. This mixture was stirred in magnetic stirrer for half an hour and then it was kept in silicon oil bath, the heat was about to 70 to 800c, reaction2 was made inert with N2 gas, it was stirred for 3 hours. Schiff base ligand was formed & it was collected through filtration process.

Reaction scheme



Scheme 1: The synthetic route of Salicylidene-p-toluidine

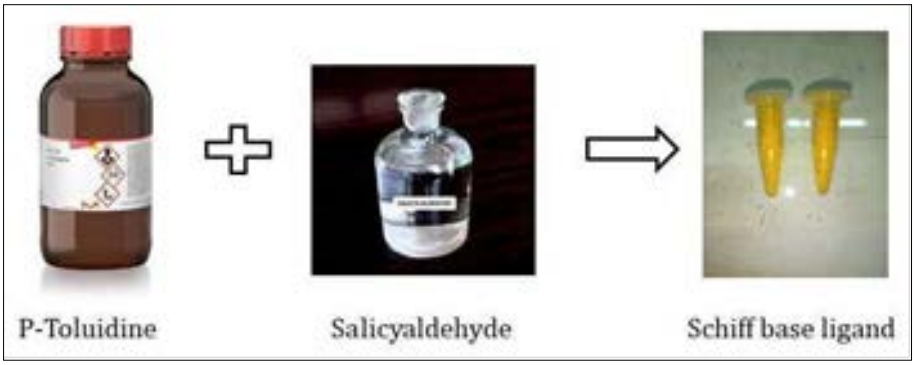


Fig 1: Flow chart of Schiff-base synthesis

Characterization

UV-Visible Spectra

The Schiff base ligand was dissolved in THF solution and it's UV-spectra was measured. Its UV data are shown and discussed in the following table. And UV plot of Schiff base ligand has been observed in Fig.2.

Table 1: Synthesis of Schiff-base ligand by taking reactants in different ratios

SL. No	Schiff base	Ratio of reactants	Time (h)	colour
1	Salicylidene-p- Toluidine	1:0.5	3:00	Pale Yellow
2	Salicylidene-p- Toluidine	1:1	3:00	Yellow
3	Salicylidene-p- Toluidine	1:2	3:00	Yellow (no change in colour)

Table 2: Characteristic UV data for Schiff base ligand

SL. No	UV band	Nature of the peak	Interpretation
1	272 nm	Broad	This is due to the imine π-π* transition
2	342 nm	Broad	This is due to the imine n- π* transition

UV-Spectra analysis of ligand binding metal (Al3+) complexes

The Schiff base ligand binding metal complex was dissolved in methanol solution and its UV-spectra was measured. And UV data are shown and discussed in the following table. And UV plot of Schiff base ligand binding metal complex has been observed in Fig.3.

Table 3: Characteristic UV data for Schiff base ligand with metal (Al^{3+}) complex

SL No	UV band	Nature of the peak	Interpretation
1	270 nm	Broad	This is due to the imine π-π* transition
2	386 nm	Broad	This is due to the imine n-π* transition

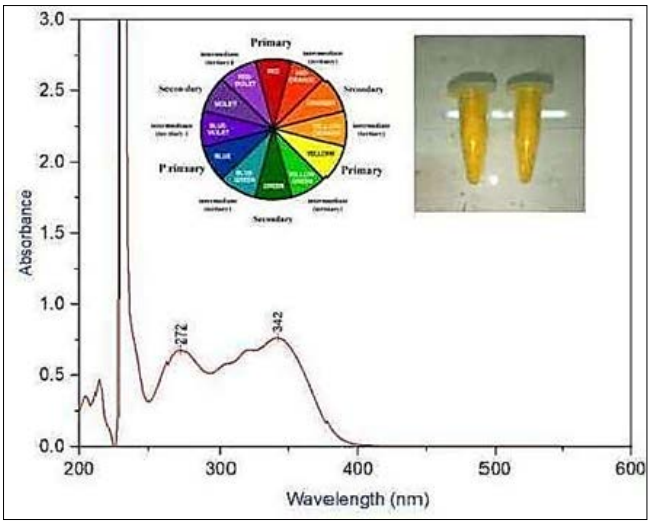


Fig 2: UV-Visible spectra for Schiff base ligand

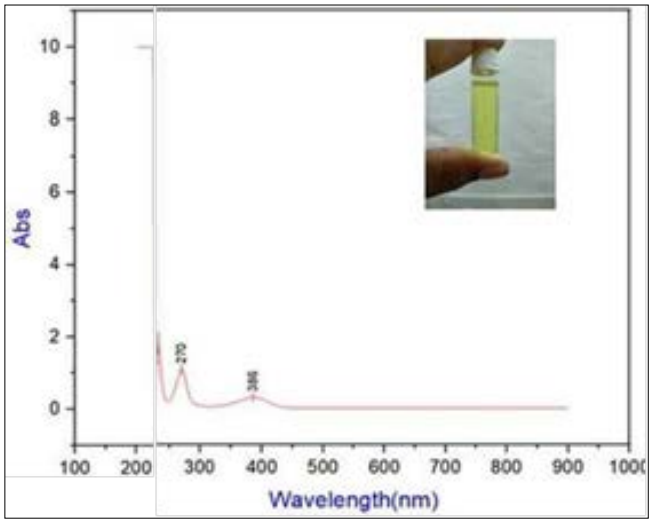


Fig 3: UV-Visible spectra for Schiff base ligand with metal complex (Al3+)

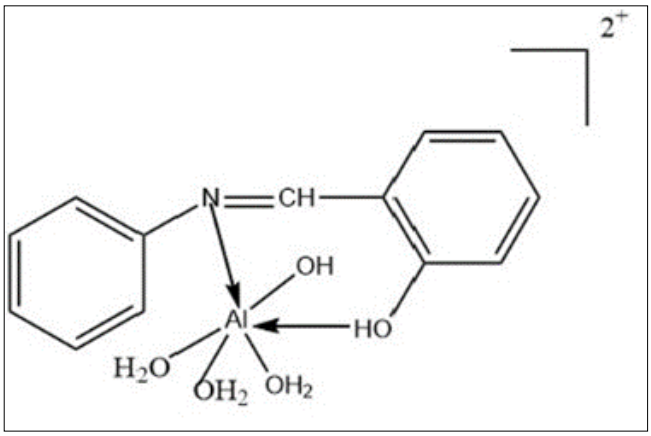


Fig 4: Probable structure of Schiff base ligand binding with Al ion

Fluorescence

The fluorescence spectra of Schiff base ligand and its metal complex were studied at room temperature (298K) which are shown in Fig.5 and Fig.6 respectively. The metal complex of Schiff- base ligand exhibited blue photoluminescence with a maximum emission around 343 nm upon excitation at 270 nm. Whereas free ligand emission was around 390 nm. The increase in the intensity of fluorescence may be cause by the bridge connection of the ligand to the metal atom, which increased its conformational rigidity and decreased the non-radiative energy loss. The emission observed in Schiff-base metal complex is assigned to the ($\pi \rightarrow \pi^*$) intraligand fluorescence.

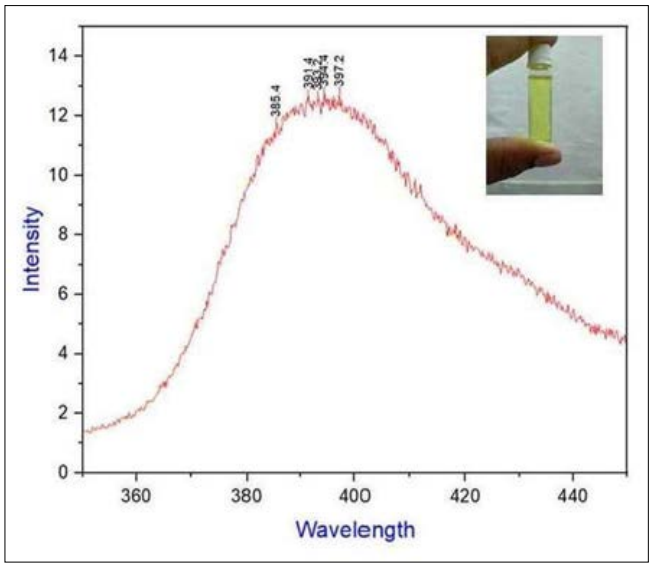


Fig 5: Fluorescence spectra of Schiff base ligand (λex=270nm &λemission≈397nm)

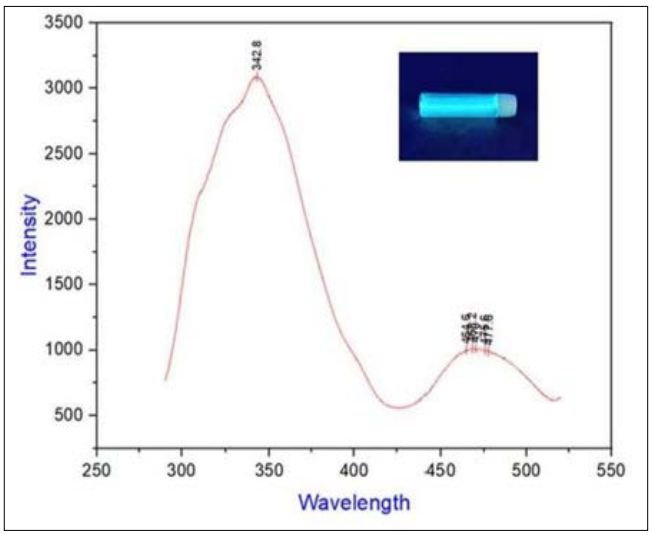


Fig 6: Fluorescence of Schiff base ligand with metal complex (Al3+) (λex=270nm & λemission≈343 nm)

^{1}H-NMR spectra

The NMR spectrum of Schiff-base ligand is shown below. CDCl3 was used as a solvent and TMS was used as internal standard. The NMR spectrum shows an azomethine proton as a singlet at chemical shift of 8.777 ppm (H). The double of doublets at 7.76 ppm (J = 7.9, 1.4, 0.4 Hz) corresponds to H1. The double of doublets at 7.40 ppm (J = 8.0, 7.6, 1.4 Hz) corresponds to H2. The double of doublets at 7.20 ppm (J = 8.0, 1.5,0.5 Hz), corresponds to H3. The double of doublets at 7.13 ppm (J =7.9, 7.6, 1.1 Hz) corresponds to H4. The double of doublets at 7.02-7.26 (J = 8.0, 1.6, 0.5 Hz) corresponds to H5. The double of doublets at 6.91 ppm (J = 8.0, 1.1, 0.4 Hz) corresponds to H6. The singlets at 2.21 ppm correspond to three H of methyl.

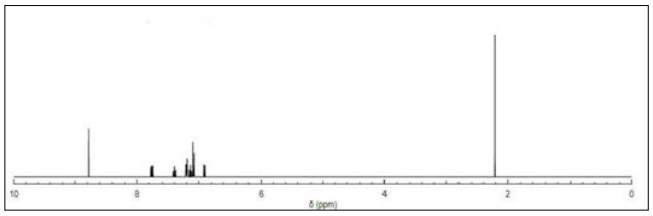


Fig 7: 1H-NMR spectrum of Schiff base ligand (Salicylidene-p-toluidine)

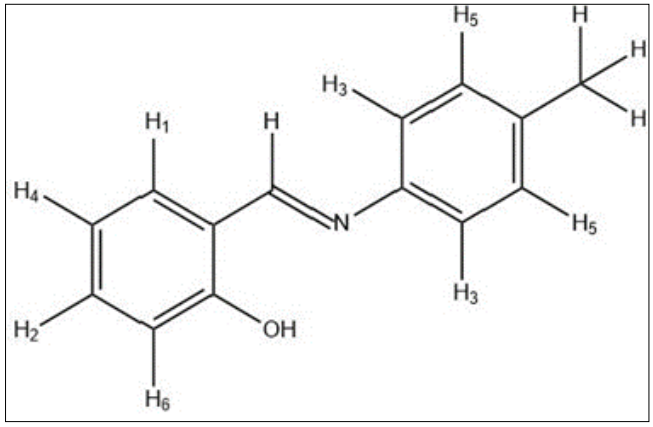


Fig 8: Atom numbering of Salicylidene-p-toluidine

Conclusion

The synthesis of p-toluidine and salicylaldehyde Schiff base was successfully conducted and characterized using various spectroscopic techniques such as UV, fluorescence, and NMR spectroscopy. The reaction between p- toluidine and salicylaldehyde yielded the desired Schiff base compound, which was confirmed through the UV and fluorescence spectra, showing the presence of conjugated double bonds. Additionally, NMR spectroscopy provided further evidence of the molecule's structure and confirmed the formation of the expected Schiff base. The data collected from these spectroscopic analyses allowed for the characterization of the synthesized compound, proving the success of the reaction and the formation of the desired product. Overall, this study highlights the significance of Schiff bases in organic synthesis and their potential applications in various fields. Further research can be conducted to explore the properties and potential uses of this Schiff base compound.

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