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# POTENTIALLY BIOACTIVE SULFATHIAZOLE-BASED LIGAND AND ITS PALLADIUM (II) COMPLEX: SYNTHESIS, CHARACTERIZATION, PHYSICO-CHEMICAL AND ANTIMICROBIAL BEHAVIOR

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**ABSTRACT**

The purpose of this work is to synthesize and describe the original Schiff base and its Palladium (II) metal complex, exploring their antimicrobial properties. The base compound, Sulphathiazole was prepared, and a new Schiff base ligand was formed using salicylaldehyde. With $PdCl_2$ metal salt, the Schiff base ligand's metal complex was made. The synthesized ligand and complex structure were determined using techniques including FT-IR, elemental analysis, UV-vis, $^1$H-NMR, SEM, and magnetic susceptibility. Additionally, the antibacterial and antifungal properties of the complex and the ligand of metal were examined. An octahedral structure was found for the Pd (II) complex, and this complex compound generally demonstrated more effective antimicrobial activity compared to the ligand.

**Keywords:** Schiff Base, Sulfonamides, Sulfathiazole, Antimicrobial Activity, Transition Metals, Salicylaldehyde.

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## INTRODUCTION

A crucial component of coordination chemistry is Schiff bases because they frequently interact with ions of transition metals, which results in the formation of stable compounds.[1-2] There are several uses for Schiff bases and their metal complexes, particularly those derivatives containing thiophene, as they are of significant interest in chemistry, pharmacology, and biology. These compounds exhibit several biological functions such as antitumor, antineoplastic, antiviral, antibacterial, and antimalarial activities. The inclusion of the thiophene moiety enhances their potency in diverse biological activities.[3-5] Different aldehydes have demonstrated moderate to excellent efficacy in antibacterial, anti-urease, hemolytic, and antioxidant activities. Antibiotics are compounds or substances that hinder the growth of bacteria.[6] Research on transition metal ion coordination with various ligands has drawn more attention recently due to developments in bioinorganic chemistry and medicine.[7] Sulfathiazole, a Sulfa drug, behaves as a Schiff base ligand, which combines with the metals to form stable complexes and is bacteriostatic.[8-9] It comes under the class of sulphanilamide.[10] Sulfathiazole (TZ) are synthetic antimicrobial agents with a broad range that are effective against most gram +ve and gram –ve organisms. This work focuses on the synthesis of a ligand, its formation into a Palladium (II) metal complex, and subsequent characterization. Additionally, it examines the antimicrobial properties of the synthesized compound, comparing them to those of the Schiff base ligand.

## EXPERIMENTAL

### Material and Methods

Every chemical and solvent used was of analytical grade. Among the substances are salicylaldehyde, sulfathiazole, and $PdCl_2$. The Perkin Elmer 240 C type Elemental Analyzer at IIT Mumbai was used for elemental analysis. With KBr pellets in the 400–4000 $cm^{-1}$ range, Infrared spectra of complex and Schiff base were obtained at SAIF, Chandigarh, using an FT-IR spectrophotometer Model RZX (Perkin Elmer). A Perkin Elmer Model Synthesis Lambda 750 Ultra Violet Visible-NIR Spectrometer from Jammu


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University was used to record electronic spectra. The IISER Bhopal facility performed NMR analysis. MANIT Bhopal performed the XRD diffractometer. IIT Mumbai's Vibrating Sample Magnetometer was used for the magnetic susceptibility measurements. It was possible to ascertain the melting points and Palladium (II) complex's decomposition temperatures and the newly formed ligand using the melting point apparatus. The solution's pH was adjusted by adding the required 1M sodium hydroxide (NaOH) volume.

**Preparation of Ligand and Metal Complex**

An equimolar ethanolic solution of Sulfathiazole (0.51gm) with salicylaldehyde (0.30 gm) was mixed together and irradiated under microwave-assisted conditions for 8 min at 250W. The light yellow-colored, shiny, amorphous solid Schiff base (Sulfathiazole-Salicylaldimine) ligand was produced. For the metal complex's preparation, an equimolar TZSD Schiff base ligand (Sulfathiazole-Salicylaldimine) and metal chloride solution were refluxed at 60–70 ºC for 3 h on a heating mantle. The resulting solution was kept overnight. The brown-colored amorphous solid of the $PdCl_2$ metal complex was obtained, and filtered. It underwent many ethanol washes before being dried and weighed. The melting point was noted. Yield obtained is 60%.

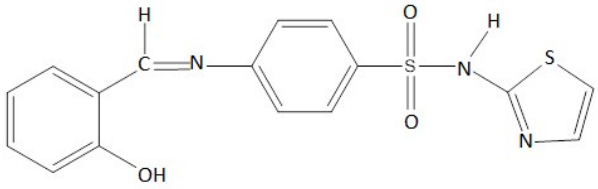


Fig.-1: Synthesized Schiff Base Ligand

## RESULTS AND DISCUSSION

Both the produced compound and the ligand are stable solids.[11] They are not soluble in pure distilled $H_2O$ but in methanol, DMSO, acetone, and ethanol. The conductometric studies suggest a 2:1 [L: M] ratio. To explain their electrolytic behavior, the complex's observed conductance value must be greater. According to the magnetic studies, the palladium complex has no magnetic moment, indicating its diamagnetic nature.[12] In accordance with the instructions in the experiments section, Schiff base was synthesized, crystalized, dried, and submitted to elemental analysis. Table-1 displays the elemental analysis findings (C, H, N, and S) employing the molecular formula.

Table-1: Physico-chemical and Analytical Information of TZSD Ligand and $[Pd(TZSD)_2]$ Complex

| Ligand/ Complex | Colour | M. Pt. (ºC) | Molr. Wt. | Elemental analysis found (calc.) % | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | C | H | N | S | Metals |
| TZSD $C_{16}H_{13}N_3O_3S_2$ | Yellow | 172 | (359.42) | 58.50 (58.47) | 3.79 (3.68) | 11.61 (11.68) | 17.76 (17.80) | --- --- |
| $[Pd(TZSD)_2]$ $C_{32}H_{24}N_6O_6S_4Pd$ | Light brown | 298 | (823.23) | 46.57 (46.69) | 2.93 (2.96) | 10.24 (10.21) | 15.53 (15.57) | 12.96 (12.92) |

**IR Spectra**

The metal ion in the middle is coordinated by the ligand via the N=CH, O-H, N, or Oxygen groups acting as a tridentate donor (Table-2).[13-14] NH stretching at 3366 $cm^{-1}$ in the ligand does not change in the palladium complex. The azomethine's linkage via its nitrogen atom ºC= N in the complex shifts the band from 1615–1618 $cm^{-1}$, which is a more significant wave number. When compared to the free ligand, the S=O vibration stretching moves to the side of a lesser wave number indicating the sulfonic acid group's involvement in coordination. The phenolic O-H band is visible in the ligand, but in the Pd(II) complex, it is absent.[17] M-N frequency may cause M-N frequency may cause the complex bands that appear in the far-infrared spectrum at 459–405 $cm^{-1}$. When comparing it to the free Schiff base, the M-O frequency corresponds to the additional bands 615–550 $cm^{-1}$. For the TZSD ligand and $[Pd(TZSD)_2]$complex, the NH stretching of the amino group emerges between 3300–3400 $cm^{-1}$.[15-17]

**$^1$H NMR Spectra**

$^1$HNMR proton spectra of the TZSD show that the peak at 12.78 ppm in the ligand and 12.75 ppm with an upfield shift of 0.03 ppm is due to the ligation of the thiazole ring's nitrogen atom to the central metal ion.[18] The ligand shows a signal in the 7.26–6.71 ppm range due to the aromatic proton (Ar-H).[19] This is because these protons are surrounded by various surroundings. The HC=N proton is visible at 8.02 ppm in the ligand, signifying azomethine-N interaction with metal ions. The O-H proton of the ligand resonates at a signal of 7.04 ppm.[20-22]

**Electronic Spectra and Magnetic Susceptibility**

There is no magnetic moment (**μ**) in the Pd(II) complex. The derived complex of Palladium exhibited bands at 35685–39458 $cm^{-1}$, 27437–25500 $cm^{-1}$ with allowed transitions $^1A_1g \rightarrow {}^1B_1g$; $^1A_1g \rightarrow {}^1A_2g$, respectively[23], illustrating the octahedral band which shows the transfer transition in the synthesized ligand.

**Scanning Electron Microscopy (S.E.M.)**

Using scanning electron micrography (SEM), both the dimension and shape of the particles of Schiff base metal complexes have been depicted. Figure-2 displays representative micrographs of the i) Ligand (TZSD); and ii) $PdCl_2$ metal complex.

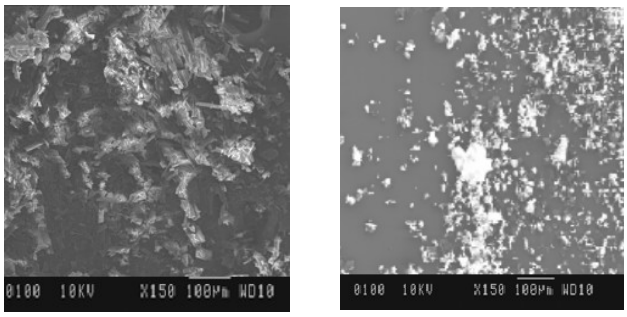


Fig.-2: S.E.M of Schiff Base and its Complex

**Antibacterial Assay**

The technique utilized to determine the antibacterial activity was disc diffusion. The compounds were evaluated against Gram +ve and –ve organisms (Table-3). The synthesized complex revealed potential antibacterial activity compared with the ligands, which was lower than the standard. Based on the inhibition zone created against the tested microbe or bacteria, it is observed that the complex of TZSD exhibited enhanced antibacterial properties comparable to its ligand.[24-25]

Table-2: IR Data of the TZSD with its Palladium Complex

| Ligand/Complex | ν(C-H) | ν(HC=N) | ν(Phenolic)OH | ν(N-H) | ν(C-S) | ν(S=O ) | ν M-N | ν M-O |
|---|---|---|---|---|---|---|---|---|
| TZSD | 2976 | 1615 | 3456 | 3366 | 756 | 1032 | … | … |
| [Pd(TZSD)$_2$] | 2982 | 1618 | - | 3365 | 759 | 1020 | 412 | 605 |

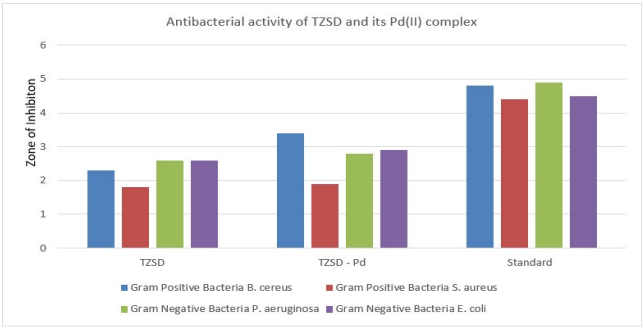


Fig.-3: Antibacterial Assay of Ligand and its Metal Complex

Table-3: Zone of Inhibition for TZSD and its Derived Metal Complex

| Ligand / Complex | Gram + ve Bacteria | | Gram - ve Bacteria | |
|---|---|---|---|---|
| | *B. cereus* | *S. aureus* | *P. aeruginosa* | *E. coli* |
| TZSD | 2.3 | 1.8 | 2.6 | 2.6 |
| TZSD - Pd | 3.4 | 1.9 | 2.8 | 2.9 |
| Standard | 4.8 | 4.4 | 4.9 | 4.5 |

**Antifungal Assay**

A varied antifungal activity was shown by the complex of the synthesized ligand TZSD against *Aspergillus niger* and *Candida albicans* ranging between 2.5–4.0 mm (Table-4). The Pd (II) complex of the Schiff base TZSD was found to be potential. Thus, complexation increases the biological activity.[26-27]

Table-4: Zone of inhibition for TZSD and its derived metal complex (mm)

| Compounds | *Candida albicans* | *Aspergillus niger* |
|---|---|---|
| TZSD | 2.6 | 2.8 |
| $[Pd(TZSD)_2]$ | 3.3 | 3.9 |
| Standard | 4.3 | 4.8 |

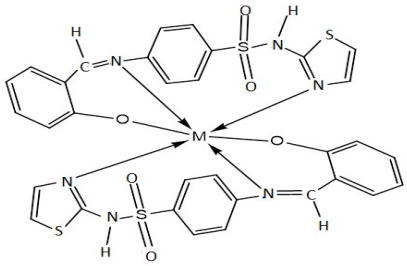

Fig.-4: The Complex's Suggested Structure Including Pd (II) Metal

## CONCLUSION

The newly synthesized Schiff base showed a tridentate nature via N, O, and N towards the central metal ion. Spectroscopic studies show that the metal complex exhibits octahedral geometry (structure). Scanning Electron Microscopy analysis observes the well-defined crystalline properties of synthesized metal complexes. The synthesized complex exhibits a higher biological activity. These findings indicate that synthesized Schiff base and complex have a sizable potential for use as medicines in the biological area.

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## CONFLICT OF INTERESTS

The authors affirm that they have no competing interests.

## AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing it, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID IDs, given below:

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## REFERENCES

1. I.P. Ejidike, P.A. Ajibade, *Molecules,* **20(6)**, 9788(2015), https://doi.org/10.3390/molecules20069788
2. P. Das, W. Linert, *Coordination Chemistry Rev*iews, **11(3)**, 1(2016), https://doi.org/10.1016/j.ccr.2015.11.010
3. C.M. da Silva, D.L. da Silva, L.V. Modolo, R.B. Alves, M.A. de Resende, C. Martins and Â. de Fátima, *Journal of Advanced research*, **2(1)**, 1(2011), https://doi.org/10.1016/j.jare.2010.05.004
4. A.M. Abu-Dief and I.M.A. Mohamed, *Beni-suef University Journal of Basic and Applied Sciences*, **4(2)**, 119(2015), https://doi.org/10.1016/j.bjbas.2015.05.004
5. L.H. Abdel-Rahman, A.M. Abu-Dief, M.S.S. Adam and S.K. Hamdan, *Catalysis Letters*, **146(8)**, 1373(2016), https://doi.org/10.1007/s10562-016-1755-0
6. K.A. Maher, S.R. Mohammed, *International Journal of Current Research and Review*, **7(2)**, 6(2015), https://doi.org/10.31782/2231-2196
7. E. Guler, F. Sevgi, B. Ozkalp, *Journal of Molecular Structure*, **1048(18)**, 476(2013), https://doi.org/10.1016/j.molstruc.2013.06.017
8. Joseyphus, R. Selwin, and M. Sivasankaran Nair, *Mycobiology*, **36(2)**, 93(2008), https://doi.org/10.4489/MYCO.2008.36.2.093
9. S. E.Turner and R. C. Anderson, *Journal of the American Chemical Society*, **71(3)**, 912(1949).
10. G. Kaur, H. Kaur, S. Lata and D.D. Bhandari, *Systematic Reviews in Pharmacy*, 14(7), 474(2023), https://doi.org/10.31858/0975-8453.14.7.474-478
11. M. Alias, H. Kassum, C. Shakir, *Journal of the Association of Arab Universities for Basic and Applied Sciences*, **15(1)**, 28(2014), https://doi.org/10.1016/j.jaubas.2013.03.001
12. Ifeanyi. E. Otuokere, J.C. AnyanwN, K.K. Igwe, *Communication in Physical Sciences*, **5(4)**, 45(2020).
13. C.D. Sheela, A. Gomathi, S. Ravichandran, P. Tharmaraj. *Journal of Coordination Chemistry*, **63(5)**, 884(2010), https://doi.org/10.1080/00958971003660416
14. K. Nakamotto, Infrared Spectra of Inorganic and Coordination Compounds, John Wiley, New York, 1976.
15. M. L. H. Nair and L. Sharma, *Journal of the Indian Chemical Society,* **86(2)**, 133(2009).
16. I. Vogel, Longman Green and Co., London, 455(1954).
17. L. J. Bellamy, Chemical application of spectroscopy, International Scientific Publication, New York, p. 551,552(1956).
18. C. N. R. Rao, Chemical Application of Infrared Spectroscopy, Academic press, New York, 260(1963).
19. A. Reiss, T. Caproiu, N. Stanica, *Asian Journal of Chemistry*, **21(7)**, 5305(2009).
20. M. R. Silverstein, X. F. Webster, Spectroscopic Identification of Organic Compounds, 6th Edition, Wiley India, 165(2009).
21. Z. Demircioğlu, F. A. Özdemir, O. Dayan, Z. Şerbetçi, and N. Özdemir, *Journal of Molecular Structure,* **II61**, 122(2018), https://doi.org/10.1016/j.molstruc.2018.02.063
22. A. Mobinikhaledi, N. Forughifar, M. Kalhor, *Turkish Journal of Chemistry* **34(3)**, 367(2010), https://doi.org/10.3906/kim-0906-49
23. M. S. Refat, I. M. El-Deen, H. K. Ibrahim, S. El-Ghool, *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*, **65(5)**, 1208(2006), https://doi.org/10.1016/j.saa.2006.01.049
24. Dar, Bilal Ahmad, Sheeraz Ahmad Teli, Amar Sohail Mirza, and Bharti Nema, *Journal of Pharmaceutical Negative Results,* **32(2)**, 736(2023), https://doi.org/10.47750/pnr.2023.14.S01.101
25. C. H. Collins and Lyne, Microbiological Methods, 4th Edition Butter-worth, London, 235(1976).
26. G. G. Mohamed, M. M. Omar, A. M. M. Hindy, *Spectrochimica Acta,* **62(4-5),** 1140(2005), https://doi.org/10.1016/j.saa.2005.03.031
27. K. Shanthalakshmi, M. Bhat, S. L. Belagali, *Journal of Chemical and Pharmaceutical Research*, **8(10)**, 240(2016).

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