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Research Paper

Synthesis, Physicochemical, Morphological, and Antimicrobial Study of Schiff-Base Ligands Metal Complexes

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ABSTRACT: *The transition metal complexes were made using 2,2'-((1E,1'E)-(1,3-phenylenebis (azanylylidene)) bis (methanylylidene)) diphenol Schiff Base(SB) ligand with an equal molar ratio of metal acetate or metal salts (Metal : Ligand). There are no antibacterial findings for the SB ligand-related metal complexes SBCu, SBZn and SBCd in bacterial and fungal species. However, as compared to the chloramphenicol standard, the complexes SBZn and SBCd exhibited very strong activity against both gram-negative and gram-positive bacterial species, as well as the fungal species C. albicans. In order to obtain a typical SB ligand metal complex with a higher average particulate size and grain width than their nano range, the particle size of the metal complexes SBZn and SBCd was used in the SEM study.The particle size of the produced metal complex SBZn is 54.87 nm, which is in the nanometer range, as revealed by SEM analysis, while the particle and grain sizes of other compounds are larger.*

KEYWORDS: *Schiff Base ligands, Metal Complexes, SEM Analysis, Antimicrobial Activities*

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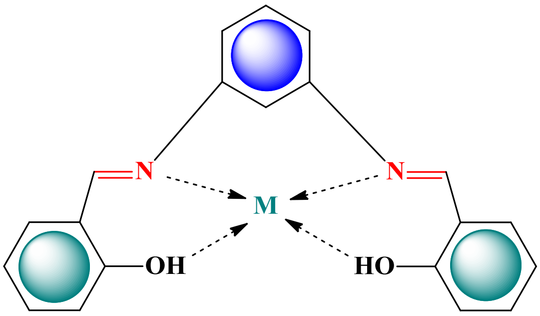
I. INTRODUCTION

Schiff bases remained important ligands even a century after their discovery in coordination chemistry. Schiff base ligands have an important role in coordination chemistry [1-5]. Even though this issue has been extensively researched [6-8] Schiff bases, it remains of major interest in inorganic chemistry to create stable complexes with different transition metal ions from aromatic/aliphatic aldehydes and amines. Because of their versatility, flexible nitrogen supporter sets produce effective chelating ligands for metal complex formation [9-10]. Schiff bases may be used as valuable intermediates in the synthesis of a broad variety of bioactive compounds. They also demonstrate a broad spectrum of biological effects, such as antibacterial, antifungal, antifertility, and anti-cancer properties [11-18]. As a consequence, the nitrogen atom reduces pathogen activity, which causes disease. Pathogens are particularly resistant to nitrogen, oxygen, and sulfur-containing compounds. The Schiff base metal complexes are active against microorganisms because the Schiff base ligands, such as azomethine or imine, contain nitrogen atoms. Transition metal complexes and metal complexes are significant chemical families because they are employed in a broad range of domains, from textile sciences to biological sciences, and so are extensively used.

Coordination chemistry is an important subject of research because it may help researchers better understand biological processes and find new medications. Transition metals are a particularly significant class of compounds because they are engaged in such a wide variety of biological functions. Metal complexes, which are well-known for speeding up drug development, typically boost a medicinal agent's potency when it is coordinated with a metal ion. To put it another way, they may be critical in the synthesis of various Schiff base ligands that are utilized as chiral auxiliaries in asymmetric synthesis, and Schiff base metal complexes have also been used in oxidation processes [19]. Because of the wide range of medicinal applications and ability to coordinate with transition metal ions that these molecules have, the synthesis and characterization of transition metal complexes containing Schiff bases has been a focus of study for many years [20-24]. Schiff base ligands in different oxidation states may coordinate and stabilize a broad spectrum of metal ions [25-27]. This paper describes the synthesis of these novel tetra dentate mononuclear Schiff base complexes.

II. EXPERIMENTAL SECTION

The researcher's full attention was concentrated on the synthesis and classification of metal complexes of Schiff bases, despite the relevance of this kind of molecule being stressed in the previous section. A substantial number of Schiff bases have been built as a result of these qualities. This flexible intermediate is expected to form complexes with metal ions such as copper (II), zinc (II), and cadmium (II) after condensation with di-substituted aromatic aldehydes and substituted aromatic diamines.



General structure of the Schiff base metal complexes

Figure 1

2.1Material and Methods

Melting points were recorded in uncorrected open glass capillaries. The infrared spectra (KBr Disc) were obtained using a Shimadzu FTIR-8400S spectrophotometer in the range of 4000 to 400 cm^{-1}. BRUKER AVANCE-II 400 MHz spectrophotometer was used to record ^{1}H NMR spectra. The reference values for IR and NMR prediction were obtained from reference books and the Sigma Aldrich website [28-30]. Hitachi S-4800 Type-II SEM, Japan, and ImageJ software. Thin layer chromatography was used to monitor the reaction, which was carried out on pre-coated silica gel aluminium plates in the solvent benzene. All of the metal complexes were synthesized in hours using a standard technique using commercially available materials from SD Fine and Loba company. Cupric Acetate (Monohydrate), Zinc Chloride, Cadmium Chloride are the metal acetates utilized, while all solvents and chemicals are commercially reagent grade. The complexes' ligands and metals were determined using a conventional technique.

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2.2 General Procedure for the synthesis of the metal complexes derived from Schiff Base

The transition metal complexes were synthesized in 1:1 molar ratio of metal acetate or metal salts and previously prepared 2,2'-((1E,1'E)-(1,3-phenylenebis (azanylylidene)) bis (methanylylidene)) diphenol (SB) Schiff Base ligand (Metal : Ligand). The 10 mmole Schiff Base (SB) is in use into 20 mL ethanol solution in two necked round bottom flask and kept back on the magnetic stirrer with the addition of 10 mmole metal acetate or metal salts. Then the reaction mixture is refluxed on the water bath for 4 to 5 hours with continuous stirring. After the reaction is finished, the liquid is cooled to room temperature till a coloured precipitate develops, which is then filtered and washed with ethanol several times before being dried in a desiccator under vacuum set above a white anhydrous $CaCl_2$ powder. A TLC plate and a 7:3 n-hexane/ethyl acetate solvent combination were used to evaluate the purity of each component. The following diagram depicts the fundamental structure of Schiff base metal complexes.

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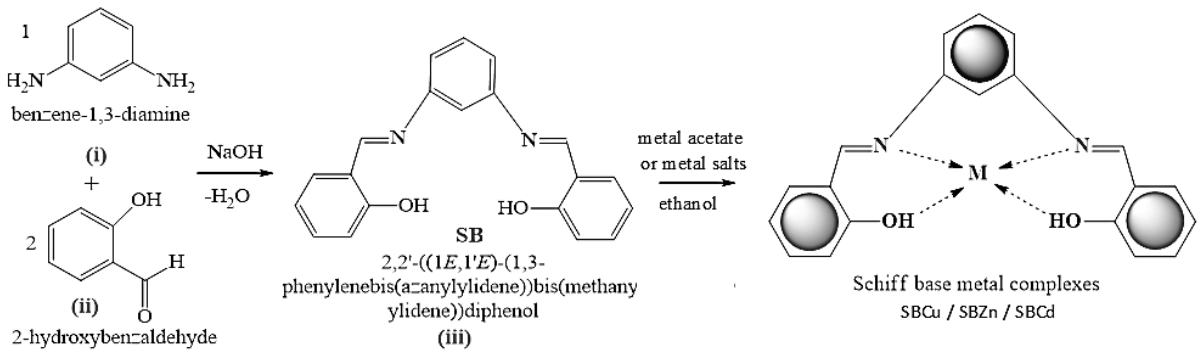

2.2.1 Copper Complex of Schiff Base (SBCu):

Chemical Formula: $C_{20}H_{16}CuN_2O_2$; Molecular Weight: 379.90; Practical yield in gram: 57.51%; Colour of the Complex Compound: Grey; Melting Point: >360 °C; Reaction time: 4-5 hrs.; Nature of Copper: Tetrahedral (immonium group formal charge +1); Elemental Analysis: Cal. C, 63.23; H, 4.25; Cu, 16.73; N, 7.37; O, 8.42 Obs. C, 64.87; H, 5.77; Cu, 17.68; N, 8.63; FTIR Analysis: tri-substituted bending for C=C: 754 cm^{-1}, C-H bending: 2011 cm^{-1}, cyclic aromatic ring C=C (3-peaks): 1384 cm^{-1}, 1442 cm^{-1} and 1510 cm^{-1}, aromatic compound C-H overtone: 1710 cm^{-1}, C-N stretching: 1260 cm^{-1}, imine C=N stretching: 1600 cm^{-1}, aldehyde C-H: 1200 cm^{-1}, Ar-OH: 3325 cm^{-1}, Metallic M-X: ~490 cm^{-1}; ^1H NMR: δ 6.78 (t, 5H, Ar-H), 7.20 (dd, 6H, Ar-H), 7.50 (s, 1H, Ar-H), 9.39 (s, 2H, -OH), 3.35 (s, 2H, -NH)

2.2.2 Zinc Complex of Schiff Base (SBZn):

Chemical Formula: $C_{20}H_{16}ZnN_2O_2$; Molecular Weight: 381.74; Practical yield in gram: 82.28%; Colour of the Complex Compound: Brown; Melting Point: 261-263 °C; Reaction time: 4-5 hrs.; Nature of Zinc: Trigonal Planar (immonium group formal charge +1); Elemental Analysis: Cal. C, 62.93; H, 4.22; N, 7.34; Zn, 17.13; O, 8.38 Obs. C, 64.95; H, 5.75; N, 8.64; Zn, 18.58; FTIR Analysis: tri-substituted bending for C=C: 750-760 cm^{-1}, C-H bending: 1620-1640 cm^{-1}, cyclic aromatic ring C=C (3-peaks): 1400 - 1510 cm^{-1}, aromatic compound C-H overtone: 1720-1750 cm^{-1}, C-N stretching: 1210-1230 cm^{-1}, imine C=N stretching: 1600-1610 cm^{-1}, aldehyde C-H: 1185-1195 cm^{-1}, Ar-OH: ~3050 cm^{-1}, Metallic M-X: ~510 cm^{-1}; ^1H NMR: δ 6.78-6.53 (t, 5H, Ar-H), 6.97-6.84 (dd, 6H, Ar-H), 7.27 (s, 1H, Ar-H), 9.26 (s, 2H, -OH), 3.57 (s, 2H, -NH)

2.2.3 Cadmium Complex of Schiff Base (SBCd):

Chemical Formula: $C_{20}H_{16}CdN_2O_2$; Molecular Weight: 428.76; Practical yield in gram: 57.44%; Colour of the Complex Compound: Light Brown; Melting Point: >360 °C; Reaction time: 4-5 hrs.; Nature of Cadmium: Trigonal Planar (immonium group formal charge +1); Elemental Analysis: Cal. C, 56.02; H, 3.76; Cd, 26.22; N, 6.53; O, 7.46 Obs. C, 57.54; H, 4.88; Cd, 26.97; N, 7.73; FTIR Analysis: tri-substituted bending for C=C: 750-760 cm^{-1}, C-H bending: 1900-2000 cm^{-1}, cyclic aromatic ring C=C (3-peaks): 1380 - 1510 cm^{-1}, aromatic compound C-H overtone: 1700 cm^{-1}, C-N stretching: 1200-1250 cm^{-1}, imine C=N stretching: 1590-1600 cm^{-1}, aldehyde C-H: 1120-1150 cm^{-1}, Ar-OH: 3390-3400 cm^{-1}, Metallic M-X: 520-530 cm^{-1}

III. SEMSTUDY OF SB METAL COMPLEXES

To better understand the morphology of metal complexes of Cu (SBCu), and Zn (SBZn) scanning electron microscopy (SEM) was used. As can be seen from the pictures, the clustered particles have a nanostructured shape and their composition reveals a tight arrangement of homogenized complexes. On closer inspection, it is clear that the particles are round and in comparable groups. The average diameter of the particle size discovered in SBCu is 2138.86 nm, whereas the average grain size is 2463.20 nm.

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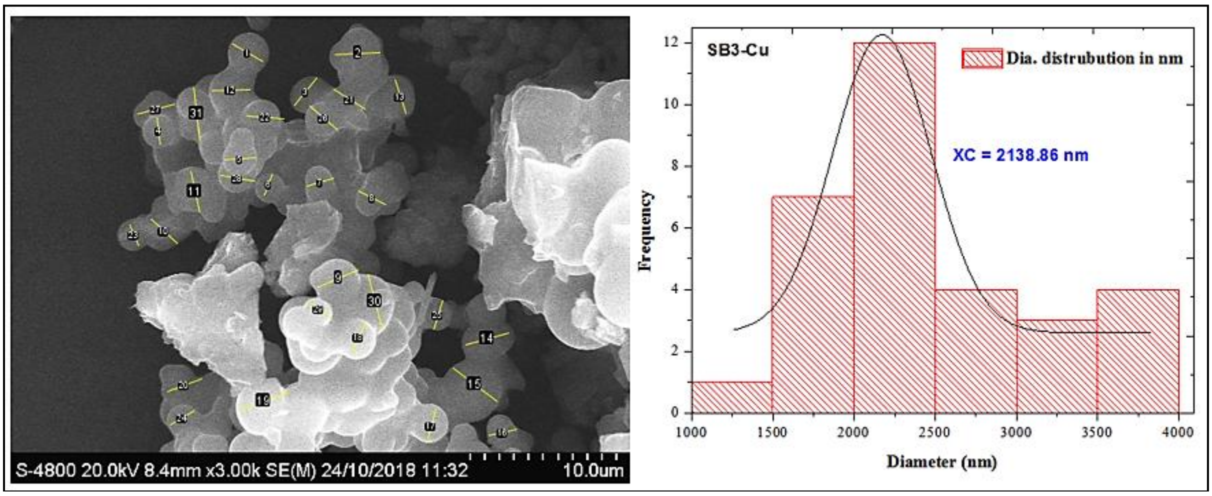
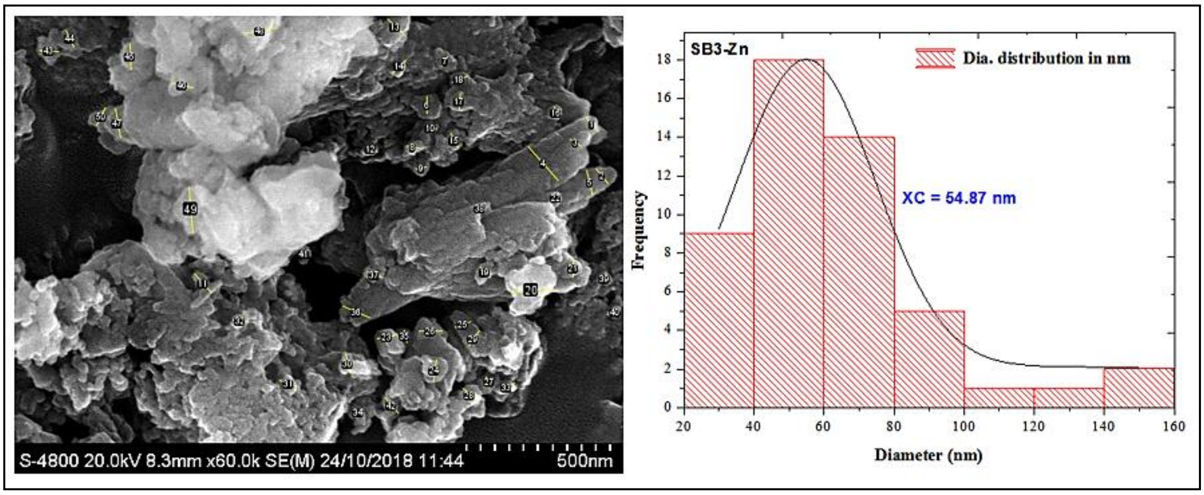


Micrograph 1.1: SEM and histogram of SBCu complex

The average diameter of the SBZn particle size was determined to be 54.87 nm, with an average grain diameter of 64.09 nm.



Micrograph 1.2: SEM and histogram of SBZn complex

The particle size of the produced metal complex SBZn is 54.87 nm, which is in the nanometer range, as revealed by SEM analysis, while the particle and grain sizes of other compounds are larger.

IV. ANTIMICROBIAL POTENCIES OF SB METAL COMPLEXES

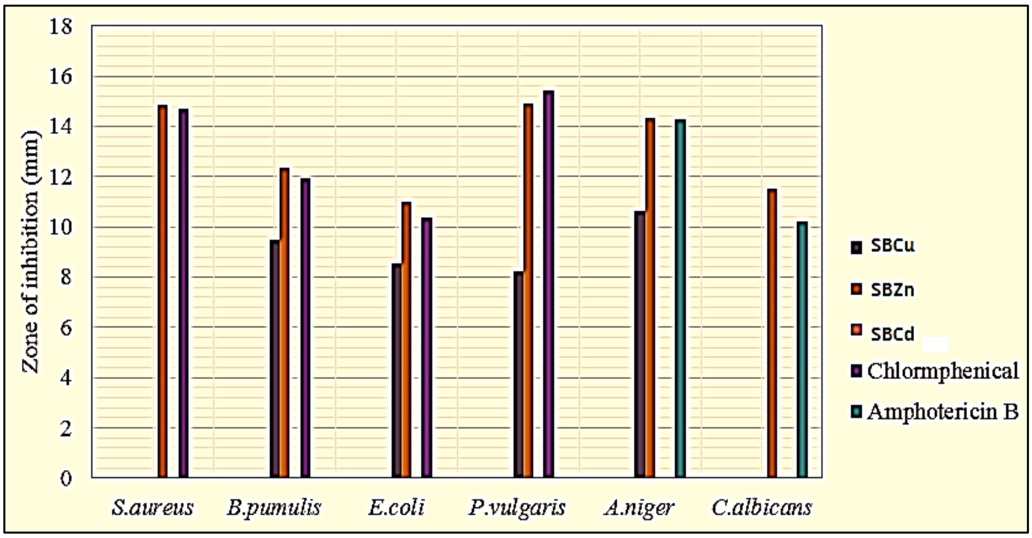
Antimicrobial studies have been performed on almost all of the metallic compounds created in order to identify their antibacterial and antifungal properties. The results of the tests are presented in detail, with comparisons made between them and the results of conventional medications that are also available.

Table 1.1: Antimicrobial activities of the synthesized SB complexes

Compound Code	Zone of inhibition is measure in 'mm'					
	E. coli	*P. vulgaris*	*S. aureus*	*B. pumulis*	*C. albican*	*A. niger*
SBCu	8.54	8.23	--	9.47	--	10.61
SBZn	11.02	14.92	14.86	12.35	11.53	14.31
SBCd	10.42	15.86	13.99	12.11	11.78	15.50
Ctrl	0.0	0.0	0.0	0.0	0.0	0.0
Std-I	10.39	15.43	14.72	11.92	NA	NA
Std-II	NA	NA	NA	NA	10.22	14.27

'- -' means no growth

The researcher has been invested metal complexes containing Schiff base centered ligands. All anti-microbial medicines are of a moderate strength when a cationic or anionic state ligand is discovered. Nearly all of the complex compounds produced in this chapter were evaluated in vitro for antibacterial activity against bacterial strains that were categorized as gram positive or gram negative in the discussion. As a solvent, DMSO was utilized with nutritional agar as the culture medium, and doses of 100g/mL per disc were employed with *Bacillus pumulis*, *Staphylococcus aureus*, and *Escherichia coli* and *Proteus vulgaris*. *Aspergillus niger* and *Candida albicans* antifungal activities were also studied and verified. The inhibitory zones were measured in millimeters (mm) and the data was put into table 1.1.



Graph 1.1: Antimicrobial activities of the synthesized SB complexes

V. RESULT AND DISCUSSION

The main purpose of this chapter is to evaluate the produced metal complexes' physicochemical, structural, and antibacterial characteristics. The goal of the study was to collect and analyses data on complex structure, core metal ion coordination structure, ligand atomic description, and antibacterial inhibitor efficacy assessment. The SB ligand related metal complexes antimicrobial activities of SBCu, SBZn and SBCd have no results amongst the bacterial and fungal species and showed very potent activities against both gram negative and positive bacterial as compared with the Chloramphenicol standard and also very potent against *C. albicans* fungal species. The particle size of the metal complexes synthesized SBCu and SBZn have been utilized in the SEM research as the typical SB ligand metal complexes that had a larger average particulate size diameter and grain diameters compared to their nano range.

VI. CONCLUSION

Schiff bases' coordination to transition metals, main group metals, and main group organometallics has gotten a lot of attention recently. Based on the coordinating potential of the ligand under consideration, the aforementioned metallic derivatives of Schiff bases have displayed a broad variety of structural motifs. Due to their wide range of uses as ligands in the synthesis of metal complexes in the elemental, spectral, nanomaterial, biological, and pharmaceutical application fields, Schiff bases and their derivative products have gotten a lot of interest.The particle size of the produced metal complex SBZn is 54.87 nm, which is in the nanometer range, as revealed by SEM analysis, while the particle and grain sizes of other compounds are larger.The antimicrobial activities of the Schiff Base ligand-related metal complexes SBCu, SBZn, and SBCd are not seen in bacteria or fungi. However, as compared to the Chloramphenicol standard, the complexes SBZn and SBCd shown very strong activity against both gram negative and positive bacteria, as well as against *Candida albicans* fungal species. The antibacterial effect with zone of inhibition of all the synthesized complexes along with the gram-negative bacteria *P. vulgaris* and *E. coli* as presented in the research work.

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