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# SYNTHESIS, CHARACTERIZATION, BIOLOGICAL STUDY OF SCHIFF BASE METAL COMPLEXES FROM SULPHACETAMIDE SODIUM WITH GLUTARALDEHYDE

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**ABSTRACT : Schiff base ligand and its complexes were prepared and characterization using different mothed like electronic spectra (ultra-violate & Visible spectrum). FTIR magnetic moments measurements, bioactivity and molar conductivity measurements the geometry shape of the complexes is Tetrahedral geometry. The biological activity of prepared ligand and its complexes are study using *Staphylococcus aureus, Escherichia coli* and *Candida albicans* the result obtained complex were more active than ligand.**

***Key words :*** Schiff base ligand, metal complex, *Staphylococcus aureus*, *Escherichia coli*, *Candida albicans.*

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## INTRODUCTION

Sulfonamide is one of a group of chemotherapeutic agents commonly referred to as Sulfa drugs, which were discovered in the 1930's. Sulfonamides remain the most widely used as antibacterial agents in the world because of their low cost, low toxicity and excellent activity against common bacterial diseases. The clinically useful sulfonamides are derived from sulfanilamide, which is similar to Para amino benzoic acid, $NH_2$ group in sulfa drug is responsible for the activity and R group acts as modified for the activity (Sodhi *et al*, 2019 and Geersing *et al*, 2018). Most of inorganic metals play a vital role as co-factors for many important enzymatic reactions in human body. However, coordination metal complexes were gained an increasing importance in the design of respiratory, slow release and long acting drugs.

Metal ions (II) are therefore known to accelerate drug actions. The efficacies of some therapeutic agents are known to increase upon coordination, so several authors have reported the synthesis of sulfa drugs and their metal complexes which were prepared and characterized by elemental analyses, magnetic measurement and electronic absorption data. Antimicrobial activity studies on their metal chelates could have much physiological and pharmacological relevance, because the metal chelates of sulfa drugs have been found to be more bacteriostatic than the drugs themselves (Al-Mudhafar *et al*, 2016).

Inorganic compounds especially transition metals have played a significant part in the development of some cosmetic compounds and new metal-based drugs. Previously, complexes have been regarded as of interest only to the theoretical and to the inorganic chemists. However, these complexes are revealing avital role in biochemistry, polymerization processes and analytical chemistry through organic compounds preparation (Babamale *et al*, 2016).

## EXPERIMENTAL

All the chemicals and solvents used for the synthesis were obtained from Fluka company but the $CrCl_2$. $6H_2O$ $CuCl_2.2H_2O$, $NiCl_2.6H_2O$, $CoCl_2.6H_2O$ and $ZnCl_2$ salts were obtained from BDH utilized without purification. Melting point apparatus of Gallencamp MF.B-600.01 was

used. The Uv-Vis spectra for the ligand and its metal complexes were performed using Shimadzu Uv-Vis 1600 A Ultraviolet spectrophotometer in the range (190-1100) nm. IR spectra for the ligand and it's metal complexes were recorded with KBr disk utilizing FTIR spectrophotometer Shimadzu model 8400 in the range (4000-400) $cm^{-1}$. Molar Conductivity was used to measure the conductivity of the complexes at room temperature in freshly prepared 0.001 M in absolute ethanol using coring conductivity meter 220. The metal content of the complexes was measured using Perkin Elmer 5000 Atomic absorption spectrophotometer.. Magnetic susceptibility measurement was determined using magnetic susceptibility Balance of Johuson matting catalytic system division at room temperature.

**Preparation of Ligand (L)**

A solution of 1.06 gm of Sulfonamide sodium (1m mole) in 30 ml of ethanol, a solution of 1.04 gm of glutaraldehde (1m mole) in 20 ml of ethanol was added. A few drops of 10% NaOH were added to adjust the pH and the reaction mixture then refluxed with stirring for seven hours and the obtained precipitate was collected by filtration through Buchner funnel, recrystallized from methanol, and dried at room temperature (Scheme 1) (Rao *et al*, 2010).

**Preparation of complexes**

A general method has been used for preparation new metal complexes by the reaction of 1 m mole of hydrate metal salts of chromium, copper, nickel, cobalt and zinc which dissolved in 10 ml of absolute ethanol, then mixed with 1 m mole of the ligand in the same solvent. The reaction mixture was reflexed for 3 hours at room temperature. The precipitates was filtered, washed with hot ethanol and dried at 50ºC by using an oven for 1 hour.

**Biological activity study**

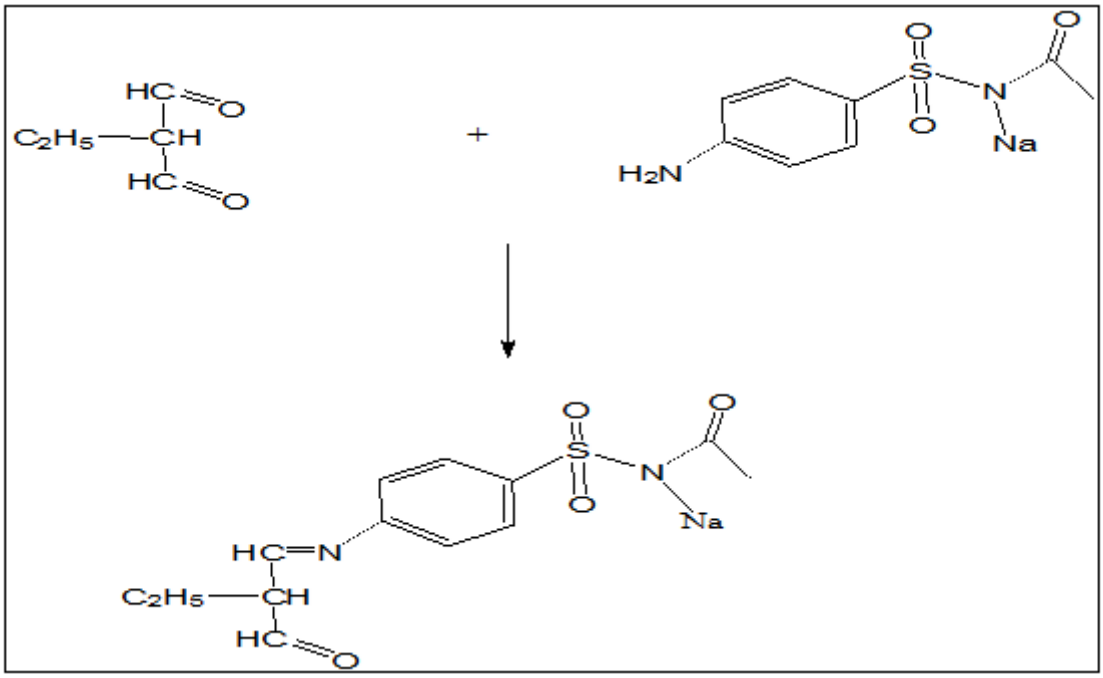
The biological screening effects of the investigation the ligand and metal complexes were tested against the bacteria *Staphylococcus aureus* and *Escherichia coli.* In addition to *Candida albicanc*, this was carried out by the disk diffusion technique, using nutrient agar as the medium (Arora *et al*, 2011). The stock solution (5 mM) of the compounds was prepared by dissolving these compounds in ethanol.

## RESULTS AND DISCUSSION

The molar ratio method was followed to measure the ratio of metal ion to ligand in complexes (M:L). Ethanol was used as a solvent. The (M: L) ratio was (1:1). Table 1 includes the physical properties; the magnetic measurements ($\mu_{eff}$ B.M) for the complexes.

**Infra-red spectroscopy**

The FTIR spectra of the ligand and its metal complexes illustrated in Table 2 and Fig. 1, 2. The band c=n and c=o are shifting to the lower wave length because the bond with the metal is through there the coordination through nitrogen and oxygen atoms of this group are further supported by appearing a new weak band (565-532)$cm^{-1}$ and (450-420)$cm^{-1}$ in the spectra of the mentioned complexes which may be refer to m-n




**Scheme 1 :** Preparation route of Ligand (Schiff Base).

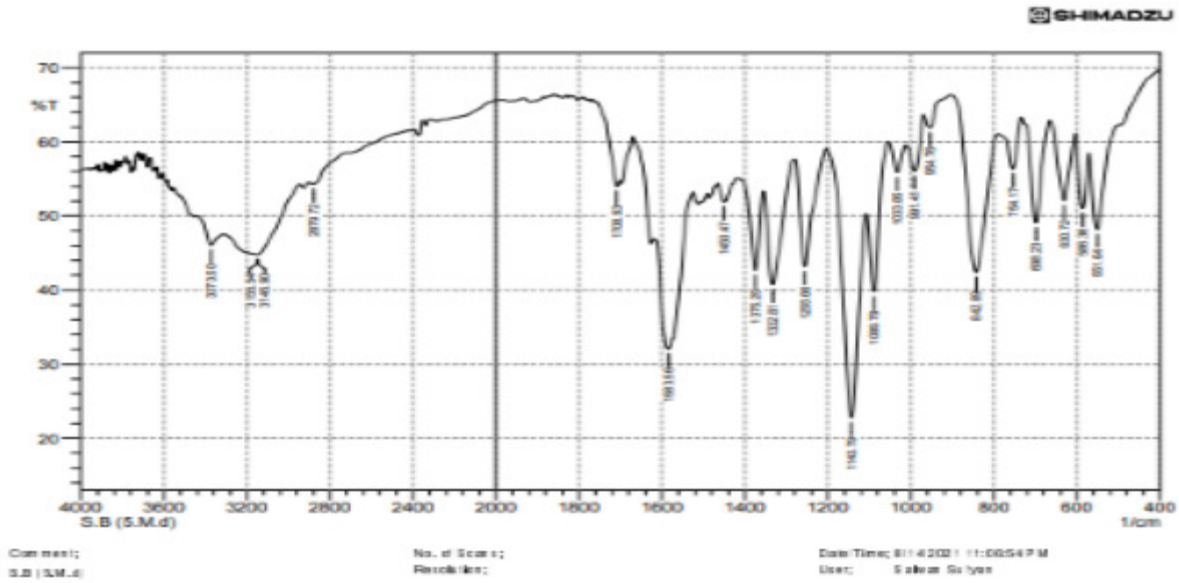



**Fig. 1 :** Ligand (Schiff Base) Infrared Spectrum.

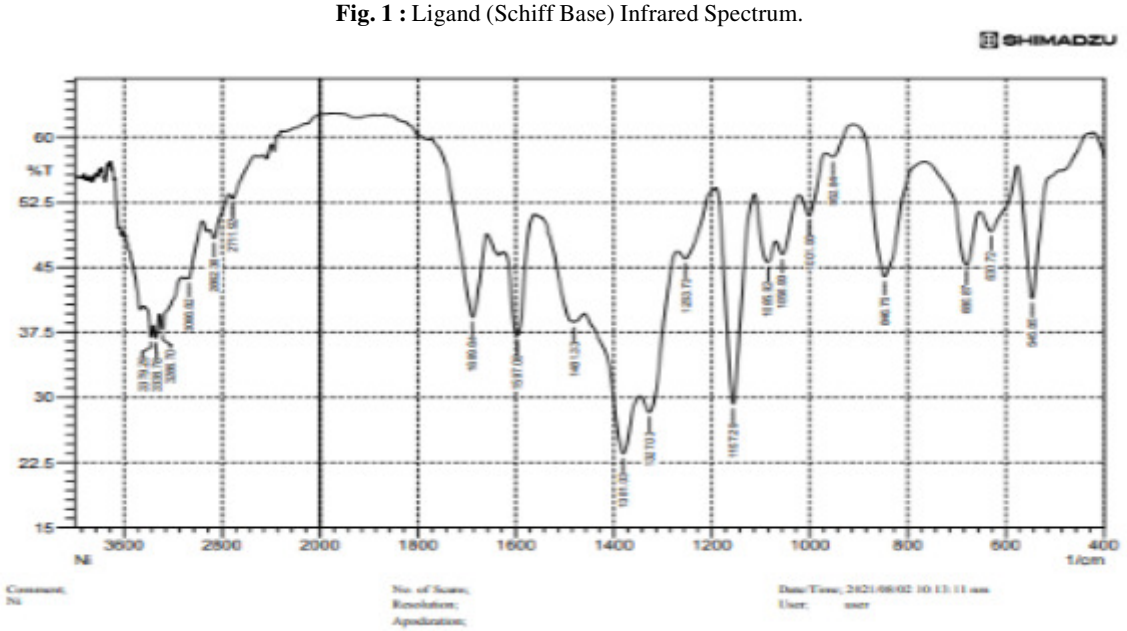



**Fig. 2 :** The Ni Complex Infrared Spectrum.

**Table 1 :** The results of microanalysis and some physical properties of ligands and their complexes.

| Compounds | Color | M.P. °C | ì $_{eff}$ B.M | Yield % | Elemental and Metal analysis Found. (Cal.) % | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | *C* | *H* | *N* | *S* | *M* |
| $C_{13}H_{16}N_2O_4S$ | Orange | 156 | — | 72 | 51.22(52.65) | 6.00(6.28) | 7.77(8.9) | 5.88(5.40) | — |
| $CrC_{13}H_{16}N_2O_4SCl_2$ | Green | 120 | 2.78 | 82 | 31.07(30.04) | 5.89(6.02) | 4.66(4.56) | 5.63(5.46) | 5.25(5.37) |
| $CuC_{13}H_{16}N_2O_4SCl_2$ | Deep green | 118 | 1.76 | 78 | 33.98(34.43) | 5.63(5.42) | 4.79(4.55) | 4.98(4.62) | 4.58(5.04) |
| $NiC_{13}H_{16}N_2O_4SCl_2$ | Brown | 122 | 3.29 | 88 | 35.08(36.49) | 6.65(7.12) | 4.52(4.33) | 5.01(4.88) | 5.53(5.72) |
| $CoC_{13}H_{16}N_2O_4SCl_2$ | Brown | 100-115d | 4.33 | 79 | 32.99(33.09) | 5.99(6.12) | 4.98(4.62) | 5.12(5.02) | 5.33(5.20) |
| $ZnC_{13}H_{16}N_2O_4SCl_2$ | Orang | 88 | di | 65 | 36.22(36.07) | 6.02(6.33) | 4.97(4.57) | 5.78(5.66) | 5.77(5.62) |

d = decomposition degree.

and m-o bonds (Nakamato, 2009).

## Electronic spectra

The electronic spectrum of the ligand exhibits two bands 37455cm$^{-1}$ to the (π-π*) transition which located on the aromatic group and 25899 cm$^{-1}$ to the (n→π*), may be refers to co, cn and nh group (Iman *et al*, 2011).

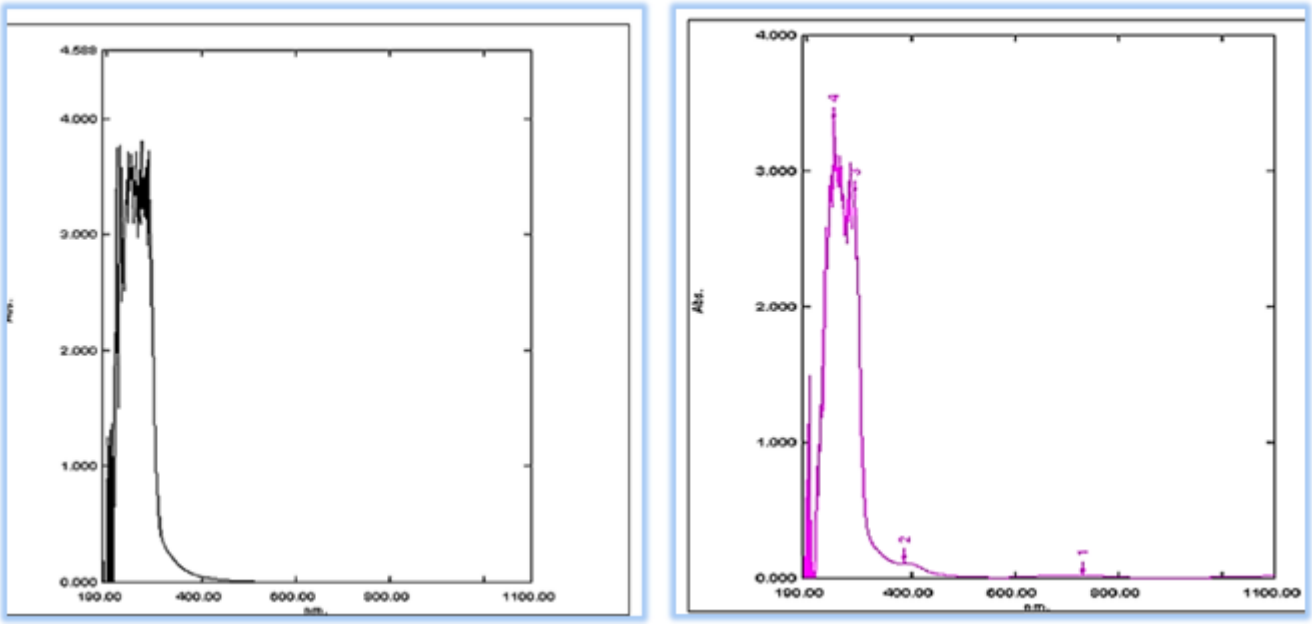
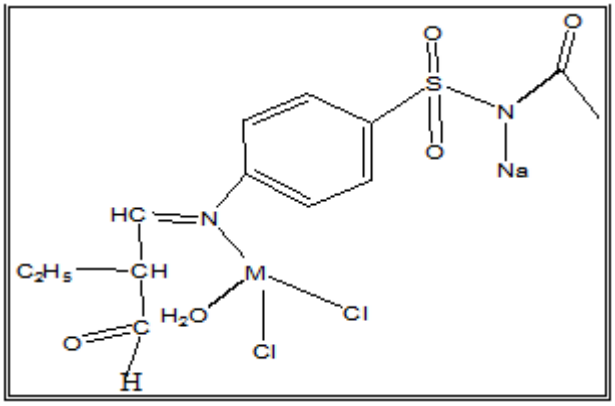



**Fig. 3 :** Electronic spectrum of ligand & Ni complex.




M=Cr, Cu, Ni. Co, Zn

**Fig. 4 :** The proposed structural formula of the complexes.

$\rightarrow {}^{2}e_{(d)}$ and charge transfer respectively, as shown in Fig. 3 and Table 3, the broadness of the band is due to the ligand field and the jahn-teller effect. the magnetic moment is 1,76 bm which confirmed the tetrahedral geometry (Sahar *et al*, 2021; Mahdi, 2019 and Figgis *et al*, 2000).

Spectrum of Cr, Ni, Co complexes shown in Fig. 3, the geometry of these complexes were tetrahedral (Fig. 4).

Finally, spectrum of complexes does not show any d-d electronic transitions in the visible region, but shows absorption band at 332 (30120 $cm^{-1}$) the magnetic moment

**Table 2 :** Infrared spectrum bands of the ligand (Schiff Base) and its metal complexes.

| Compounds | í C=N | íNH | í C=O | í $SO_2$ Sym Asym | í CH arom.alph | í $H_2O$ | í M-N | í M-O |
|---|---|---|---|---|---|---|---|---|
| $C_{13}H_{16}N_2O_4S$ | 1635 | 3237 | 1705 | 1145<br>1252 | 3060<br>2965 | 3445 | — | — |
| $CrC_{13}H_{16}N_2O_4SCl_2$ | 1622 | 3230 | 1681 | 1142<br>1251 | 3062<br>2960 | 3406 | 538 | 428 |
| $CuC_{13}H_{16}N_2O_4SCl_2$ | 1602 | 3233 | 1677 | 1144<br>1252 | 3060<br>2972 | 3417 | 565 | 424 |
| $NiC_{13}H_{16}N_2O_4SCl_2$ | 1600 | 3220 | 1680 | 1141<br>1255 | 3066<br>2958 | 3410 | 532 | 426 |
| $CoC_{13}H_{16}N_2O_4SCl_2$ | 1606 | 3220 | 1688 | 1139<br>1256 | 3062<br>2962 | 3413 | 540 | 433 |
| $ZnC_{13}H_{16}N_2O_4SCl_2$ | 1620 | 3213 | 1685 | 1142<br>1249 | 3060<br>2961 | 3412 | 539 | 423 |

Spectrum of cu complex electronic spectrum of deep green complex displayed two bands at (470 and 300) nm, (21277 and 33333) cmî¹ which assigned to the $^{2}t_{2(d)}$ value is diamagnetic which was attributed to the metal to ligand charge transfer (Sahar *et al*, 2020).

**Table 3 :** The electronic spectrum of the ligand and its metal complexes.

| Compound | $ë_{max}$ nm | Wave number $cm^{-1}$ | Assignment |
|---|---|---|---|
| $C_{13}H_{16}N_2O_4S$ | 390<br>260 | 25641<br>38462 | n-ð*<br>ð-ð* |
| $CrC_{13}H_{16}N_2O_4SCl_2$ | 350<br>551<br>802 | 28571<br>181488<br>12469 | $^4A_2 \rightarrow {}^4T_2$<br>$^4A_2 \rightarrow {}^4T_1$,<br>$^4A_2 \rightarrow {}^4T_{1\ (p}$ |
| $CuC_{13}H_{16}N_2O_4SCl_2$ | 470<br>300 | 21277<br>33333 | $^2T_2(D) \rightarrow {}^2E(D)$<br>charge transfer |
| $NiC_{13}H_{16}N_2O_4SCl_2$ | 451<br>753<br>819 | 22173<br>13280<br>12210 | $^3T_{1(F)} \rightarrow {}^3A_{2(F)}$<br>$^3T_{1(F)} \rightarrow {}^3T_{1(P)}$<br>$^3T_{1(F)} \rightarrow {}^3T_{2(F)}$ |
| $CoC_{13}H_{16}N_2O_4SCl_2$ | 350<br>512<br>670 | 28571<br>19531<br>14925 | $^4A_{2(F)} \rightarrow {}^4T_{1(P)}$<br>$^4A_{2(F)} \rightarrow {}^4T_{1(F)}$<br>$^4A_{2(F)} \rightarrow {}^4T_{2(F)}$ |
| $ZnC_{13}H_{16}N_2O_4SCl_2$ | 325 | 30769 | charge transfer |

**Table 4 :** shows the effect of ligand and it's metal complex toward *Staphylococcus aureus*, *Escherichia coli* and *Candida albicans.*

| Compounds | *Staphylococcus aureus* | *Escherichia coli* | *Candida albicans* |
|---|---|---|---|
| EtOH | - | - | - |
| $C_{13}H_{16}N_2O_4S$ | + | + | + |
| $CrC_{13}H_{16}N_2O_4SCl_2$ | ++ | + | + |
| $CuC_{13}H_{16}N_2O_4SCl_2$ | - | - | - |
| $NiC_{13}H_{16}N_2O_4SCl_2$ | + | + | ++ |
| $CoC_{13}H_{16}N_2O_4SCl_2$ | ++ | + | + |
| $ZnC_{13}H_{16}N_2O_4SCl_2$ | + | ++ | + |

(-) no signification.
(+) slight significant zone of which (5-10 mm).
(++)moderated significant zone of which (11-20 mm).

## Antibacterial and antifungal activities

The results obtained for biological activity for the preparation ligand and its metal complexes are shown in Table 4. The free ligand showed a slightly *antimicrobial* and *antifungal* activities against both kinds of bacteria. the cobalt nickel, chromium, zinc complexes shows the moderated inhibition against both organisms and *antifungal* effect compared with free ligand in both concentration. The copper complex showed a non-activity respectively against all organism used in this study.

## CONCLUSION

The new schiff base ligand and its metal complexes were successfully synthesized and characterized. The mode of bonding and overall structure of the complexes was determined through physio-chemical and spectroscopic methods. The free ligand (l) and its metal complexes show significant antimicrobial activity.the all complexes are found more effective than the free ligand.

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