International Journal of Scientific Research in Science and Technology
Print ISSN: 2395-6011 | Online ISSN: 2395-602X (www.ijsrst.com)
doi : https://doi.org/10.32628/IJSRST229643

Synthesis, Characterization and Antimicrobial Studies of Novel Series of 2,4,6-Trihydrazino-6- substituted-1,3,5-triazine Schiff Base Metal Complexes

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Article Info
Volume 9, Issue 6
Page Number : 283-292

Publication Issue
November-December-2022

Article History
Accepted : 10 Nov 2022
Published : 02 Dec 2022

ABSTRACT

The present work represents the synthesis, characterization, and antimicrobial studies of novel series of 2,4,6-Trihydrazino-6- substituted-1,3,5-triazine Schiff Base Metal Complexes. The characterization techniques included are thermal and antimicrobial characterization methods used to characterize newly synthesized 1,3,5 trizine derivatives and its complexes. The structural characterizations include X-ray diffraction (XRD), Fourier Transform IR Spectroscopy (FTIR), UV-visible spectroscopy, and some important properties like Thermal Properties. Scanning electron Microscopy (SEM) has been also done to study the compounds and characterization in more detail. The antibacterial activity of the ligands and their metal complexes against different bacterial pathogens were analyzed. Muller Hinton Agar was used as medium for bacterial sensitivity and testing the activity of microorganisms. For this study different organisms like *Staphalococcus aureus, Bacillus subtilis, Escherichia coli and Pseudomonas fluorescens* were used.

Keywards : Antimicrobial studies, Triazine Schiff Base Metal Complexes, Scanning electron Microscopy.

I. INTRODUCTION

Hydrazone compounds obtained by the reaction of aromatic and heterocyclic hydrazides with mono and di-aldehydes or ketones have revealed very versatile behavior in metal *"Synthesis and structural studies of some hydrazone derivatives and their vanadium complexes"* coordination [1-4].

Many researchers have synthesized a number of new hydrazones because of their ease of synthesis[5]. Hydrazones are principle compounds for drug design, as possible ligands for metal complexes, organocatalysis and also for the synthesis of heterocyclic compounds.[6-8]. The ease of preparation, increased hydrolytic stability relative to imines, and tendency toward crystallinity are the desirable characteristics of hydrazones. Due to these positive traits, hydrazones have been under study for a long time, but much of their basic chemistry remains unexplored. Hydrazones is a class of organic

compounds having the basic structure $R_1R_2C=NNH_2$. They are related to aldehydes and ketones, by the replacement of the oxygen with the $–NNH_2$ group. They are derived by the condensation of substituted hydrazides with carbonyl compounds namely aldehydes and ketones. They are formed by the action of hydrazine on ketones or aldehydes. Hydrazones are azomethines characterized by the presence of the triatomic grouping >C=N-N<. They are distinguished from other member of this class (imines, oximes etc.) by the presence of the two interlinked (–N– N–) nitrogen atoms. According to the needs of a polydentate ligand, the group functionalities are increased by condensation and substitution. Hydrazones are usually named after the carbonyl compounds from which they are obtained. They are important intermediates in heterocyclic chemistry. Hydrazone moiety plays an important key role in heterocyclic chemistry[9-15]. The most important property of hydrazones is their high physiological activity[16-21]. Hydrazones possessing an azomethine proton (–NHN=CH–) constitute an important class of compounds, contain two connected nitrogen atoms of different nature and a CN double bond that is conjugated with alone electron pair of the terminal nitrogen atom. Extensive studies have revealed that the lone pair on trigonally hybridized nitrogen atom of the azomethine group is responsible[22-24] for the chemical and biological activity.

Now a days interest is focused on the synthesis of complexes with potential medicinal applications. Similarly complexes of vanadium are very useful, due to the fact that vanadium compounds are in clinical trials as a potential treatment for non-insulin dependent diabetes mellitus[23]. Also, they are highly significant from the biological point of view[24-25]. Keeping the above facts in mind and in the present research work we report the synthesis and structure of some new hydrazone derivatives and their vanadium complexes. They are of wide interest because of their diverse biological and clinical applications. This created an interest in the present research work synthesized variety of hydrazone derivatives and its vanadium complexes also screened them for their various biological activities.

II. Experimental Technique

This part focuses mainly on the fundamentals and basic principles of the preparation techniques of 1,3,5 triazine derivatives and its complexes with transition metal ions the characterization tools, which are used in the present investigation. The characterization techniques include thermal and antimicrobial characterization methods used to characterize newly synthesized 1,3,5 trizine derivatives and its complexes. The structural characterizations include X-ray diffraction (XRD), Fourier Transform IR Spectroscopy (FTIR), UV-visible spectroscopy, and some important properties like Thermal Properties. Scanning electron Microscopy (SEM) has been also done to study the compounds and characterization in more detail.

Solvent Purification

Common solvents like chloroform, ethanol and dimethyl sulphoxide was used at various stages of this work are purified according to standard procedure described in Weissenburg series [26] andin quantitative analysis by A. I. Vogel [27].

Melting / Decomposition temperature

The melting/decomposition points were determined by placing a finely powdered sample in a glass capillary and heating by using digital melting point apparatus[28-29]

Preparation

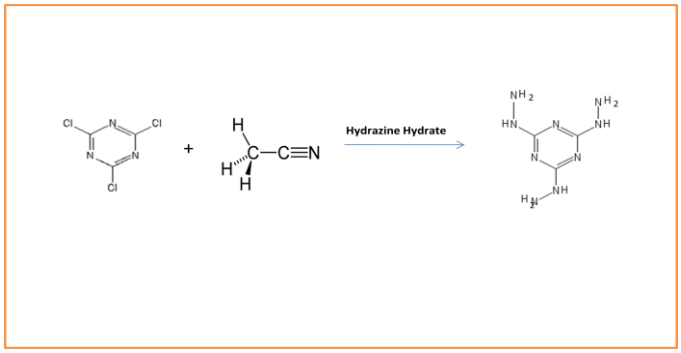
1) Synthesis of 2,4,6-Trihydrazino-6- substituted-1,3,5-triazine (TT) from Cyanuric Chloride

Method:

1.0 gram of Cyanuric chloride and 10 ml of Acetonitrile were taken in a round bottom flask. Then

03 ml Hydrazine hydrate and 10 ml Acetone were taken in a beaker and this mixture was added to the first taken which was taken in round bottom flask. This mixture was refluxed for three hours.

Reaction:



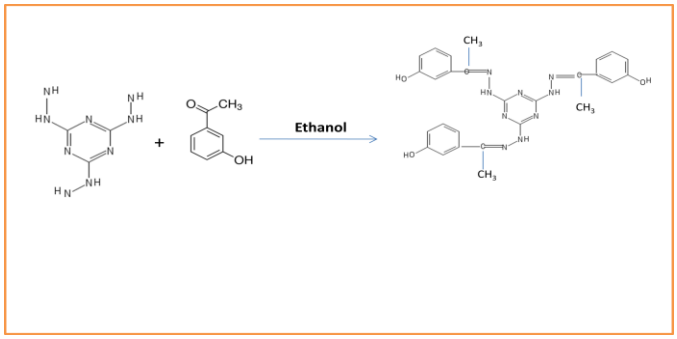
Scheme 2.1 :- Synthesis of 2,4,6-Trihydrazino-6-substituted-1,3,5-triazine

2) The Synthesis of 2,4,6-Trihydrazino-6-substituted-1,3,5-triazine Schiff Base (TTSB)

Method

1.30 gm of product A add in 0.340 gm of m-hydroxy acetophenone dissolved in a 5 ml of ethanol . Reflux for 3 hrs. The yellow powder was recrystallized from 05 ml of ethanol to afford a yellow precipitate.

Reaction:



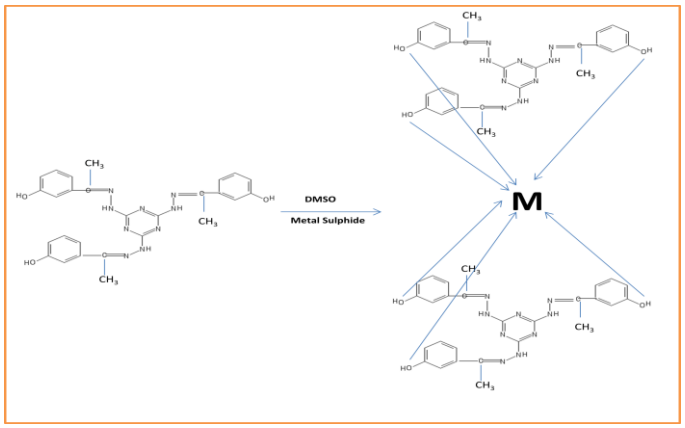
Scheme 2.1 :- Synthesis of 2,4,6-Trihydrazino-6-substituted-1,3,5-triazine Schiff Base

3) The Synthesis of 2,4,6-Trihydrazino-6- substituted-1,3,5-triazine Schiff Base Metal Complexes (TTSB Metal Complexes)

A) TTSB- Cu (II)

Method: 0.2gm of 2,4,6-Trihydrazino-6- substituted-1,3,5-triazine Schiff Base dissolved in 25 ml of dimethyl sulphoxide ,then add 0.6 gm of copper sulphate in it , mix them and reflux for 3 hr. The black powder was obtained.

Reaction



Scheme 2.1 :- Synthesis of Metal Complexes

B) TTSB- Ni (II)

Method: 0.2gm of 2,4,6-Trihydrazino-6- substituted-1,3,5-triazine Schiff Base dissolved in 25 ml of dimethyl sulphoxide ,then add 0.6 gm of nickel sulphate in it , mix them and reflux for 3 hr to get brown color precipitate.

C) TTSB- Fe (II)

Method: 0.2gm of 2,4,6-Trihydrazino-6- substituted-1,3,5-triazine Schiff Base dissolved in 25 ml of dimethyl sulphoxide ,then add 0.6 gm of iron sulphate in it , mix them and reflux for 3 hr to get black color precipitate.

D)) TTSB- Co (II)

Method: 0.2gm of 2,4,6-Trihydrazino-6- substituted-1,3,5-triazine Schiff Base dissolved in 25 ml of dimethyl sulphoxide ,then add 0.6 gm of copper sulphate in it , mix them and reflux for 3 hr to get dark brown color precipitate.

The coordination compounds were prepared by stirring the appropriate ligand (L_1-L_2) in ethanol under reflux with the anhydrous metal sulphates (Cu, Ni, Fe and Co). Thus, metal complexes formed are filtered and washed with the corresponding solvents and yielded metal complexes in high yield.

Thermogravimetric Analysis

The thermogravimetric analysis of all newly synthesised compounds was carried out using a Netzsch STA 409 thermal analyzer at our Institute in the temperature range 40-800°C with a heating rate 10°C/min.

X-ray diffraction (XRD)

X-ray diffraction (XRD) is a powerful technique used to uniquely identify the crystalline phases present in materials and to measure the structural properties (strain state, grain size, epitaxy, phase composition, preferred orientation and defect structure) of these phases, XRD of newly synthesized compounds have been recorded in this study. The melting points or decomposition temperature were determined by placing a finely powdered sample in a glass capillary and heating by using digital melting point Apparatus with high accuracy.

Antibacterial activity

The antibacterial activity of the ligands and their metal complexes against different bacterial pathogens were analyzed. Muller Hinton Agar was used as medium for bacterial sensitivity and testing the activity of microorganisms. For this study different organisms like *Staphalococcus aureus, Bacillus subtilis, Escherichia coli and Pseudomonas fluorescens* were used.

III. RESULTS & DISCUSSION

The Schiff bases synthesised from Cyanuric Chloride and metal(II) complexes of copper(II), nickel(II), iron (Fe) and cobalt(II) were obtained in good yield. The analytical data and physical properties of Schiff bases synthesised with transition metal(II)complexes are listed in Table 1.

S No.	Compounds	Molecular Formula	Colour	Yield obtained (in %)	Melting Point (in ^{0}C)
1.	TT	$C_3H_9N_9$	White	62 %	266 ^{0}C
2.	TTSB	$C_{27}H_{18}O_3$	Yellow	92 %	240 ^{0}C
3.	TTSB-Cu (II)	$C_{54}H_{36}O_6Cu$	Black	80 %	255 ^{0}C
4.	TTSB-Ni (II)	$C_{54}H_{36}O_6Ni$	Brown	90 %	260 ^{0}C
5.	TTSB-Fe (II)	$C_{54}H_{36}O_6Fe$	Black	85 %	290 ^{0}C
6.	TTSB-Co (II)	$C_{54}H_{36}O_6Co$	Dark Brown	75 %	285 ^{0}C

Table 1: Physical Data of Newly synthesized Compounds

Fourier Transform Infra Red Spectroscopy

FTIR characterization newly synthesised compounds and its complexes is found to be important for investigation of compounds structure that provides information about the complexation and interactions between the various constituents in the newly synthesised compounds. Each type of bond has a different natural frequency of vibration, so the identification of an absorption peak in the vibration portion of the infrared region will give a specific type of bonding [30].

The FTIR spectra for newly synthesised compounds and its complexes are shown in Figure 1

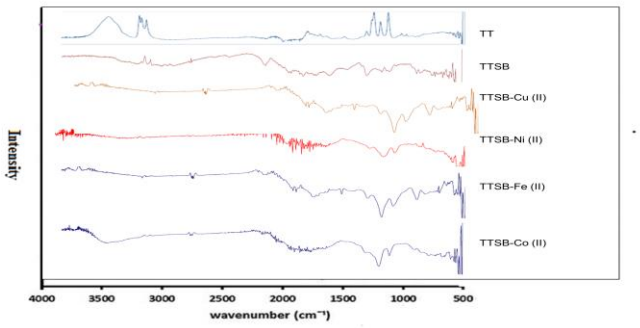


Fig.- 1 FTIR spectra of triazine derivatives

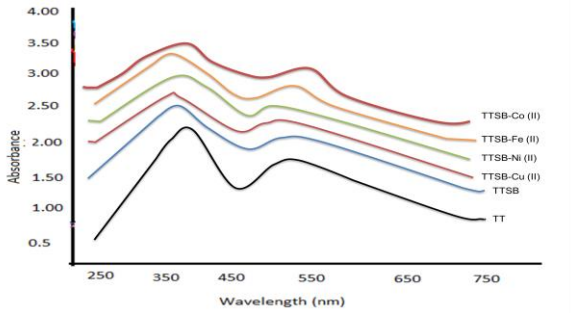


Figure – 2 : UV-Visible Spectra of TRIPOD Compounds

Figure 1 showed the absorption peaks of Schiff bases synthesised from Trihydrazino trihydrazine derivative with transition metal(II) complexes. It is good arrangement with Literature values [31-33].

UV-Visible spectroscopy

UV-Visible spectra of Schiff bases synthesised from Trihydrazino trihydrazine derivative with transition metal(II) complexes were recorded on Shimadzu UV-1800 spectrophotometer, all newly synthesised compounds by placing an uncoated identical conducting glass substrate in the reference beam ranging from 250 to 800 nm. A plot shown in figure - 2.

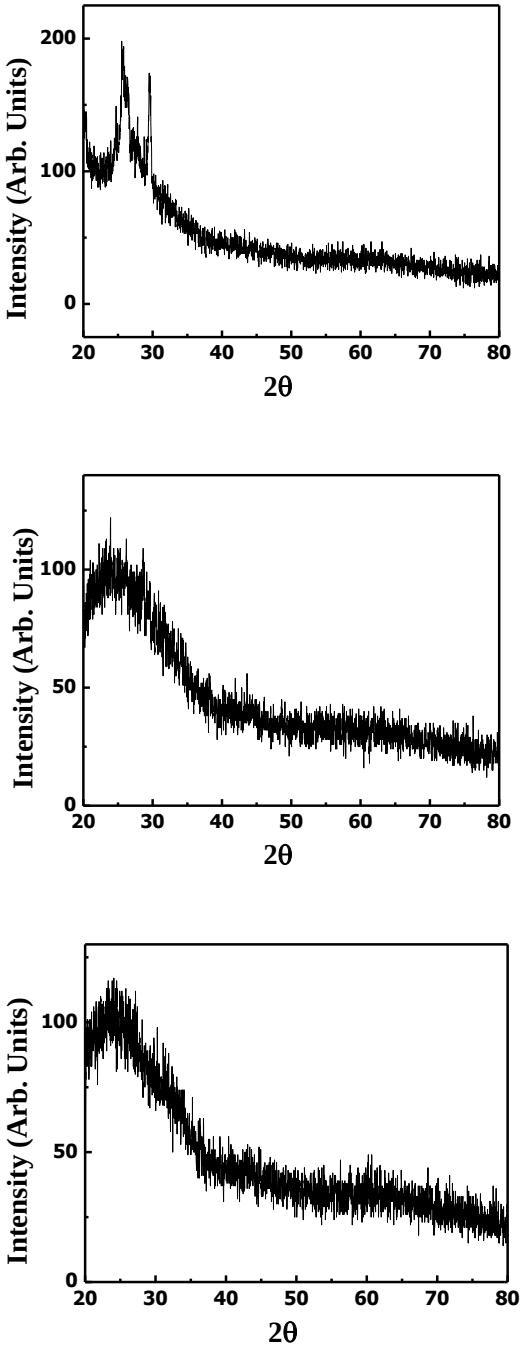
UV-Visible spectra of Schiff bases synthesized from TT with Cyanuric Chloride. ligand TTSB with transition metal (II) complexes were recorded on Shimadzu UV-1800 spectrophotometer, all newly synthesized compounds by placing an uncoated identical conducting glass substrate in the reference beam ranging from 200 to 1100 nm. Wavelength for maximum absorbance λmax and corresponding optical band gap for all samples are presented in Table 2. All compounds have a conjugate system of double bonds on their backbone, such as σ-σ*, Π-Π*, n-Π* etc. The σ-σ* transition of conjugated double bonds are related to near UV regions around 200 nm, UV-visible spectra of the spectrum of pure TT is characterized by an absorption edge at wavelength 258 nm. This absorption edge can be attributed for >C=N in backbone of compound [34--36]. Also absorbance increases Schiff bases synthesized from TTSB with substituted acetophenone and TTSB and transition metal (II) complexes.

Table – 2: UV-Visible absorption values Triazine Compound

Sr.No.	Material	λ max nm	Absorbance(au)
1	TT	259	2.697
2	TTSB	310	0.450
3	TTSB-Cu (II)	308	0.739
4	TTSB-Ni (II)	388,319,269,328,289	0.387,0.329,0.326,0.315,0.264
5	TTSB-Fe (II)	333,318,275,286	0.338,0.342,0.286,0.285
6	TTSB-Co (II)	340,3181,279,289	

Thermogram of the Schiff bases synthesized from TT with substituted aldehydes and derivation TTSB with transition metal (II) complexes indicate that they have varying degree of thermal stability and undergo

decomposition at different temperature. The percent weight loss as computed from the thermo grams of the Schiff base synthesized from TT with substituted aldehyde and derivative TTSB with transition metal (II) complexes. XRD spectra of compounds as shown bellow.



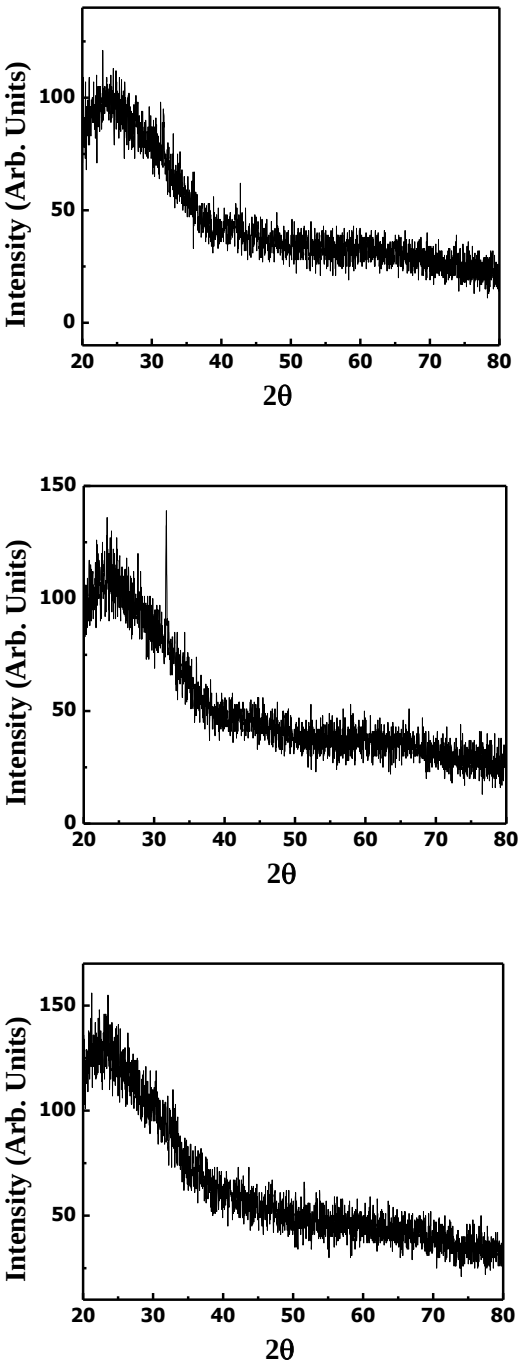
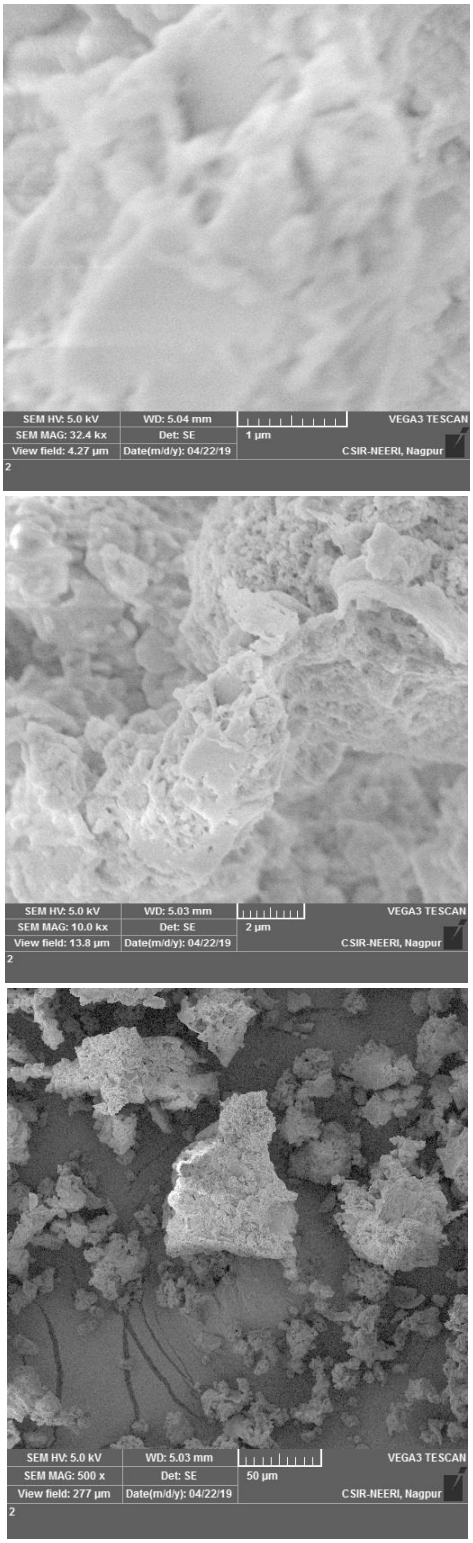


Figure 3. XRD Graph of TTSB Compounds & Complexes

Scanning Electron Microscopy (SEM)

To perform a visual analysis of a surface using scanning electron microscopy contributes to the identification of contaminates or unknown particles , the cause of failure and interactions between materials. In addition to surface evaluation, SEM

analysis was utilized for particle characterization , such as wear debris generated during mechanical wear testing. The number, size, and morphology of small particles has been analyzed by using SEM which allowed to understand the wear properties of their material.SEM analysis for all the synthesized compounds has been done (Fig. 4)



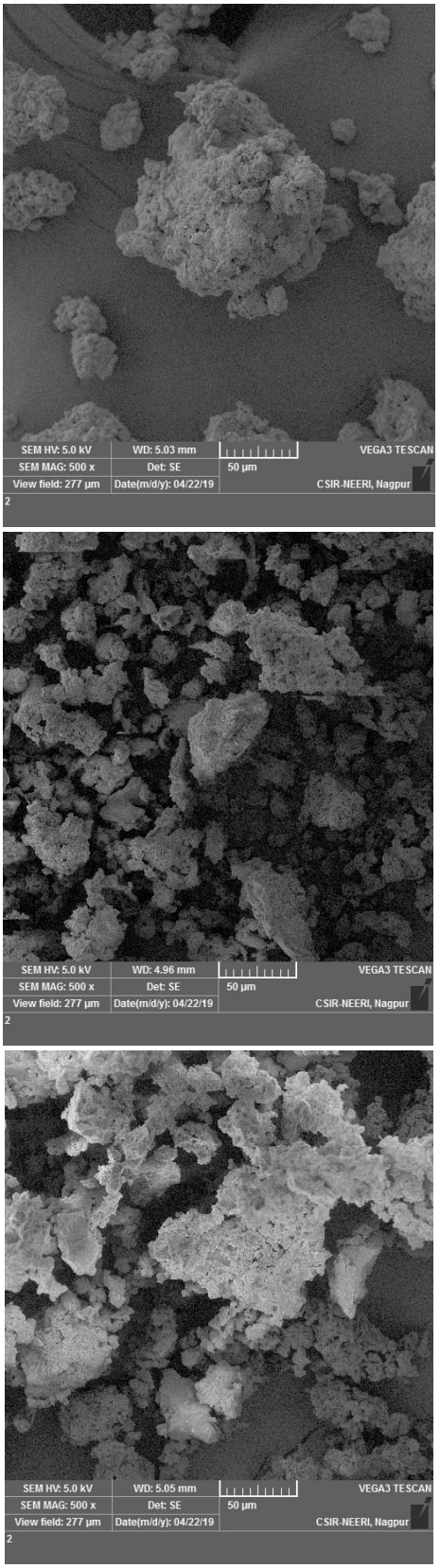


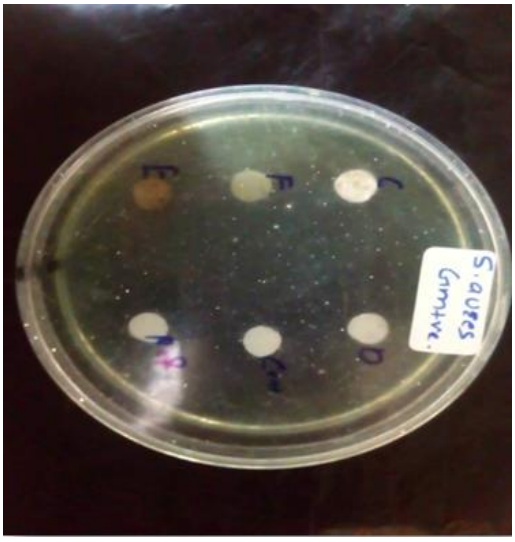
Figure 4. SEM Diagram TTSB Compounds & Complexes

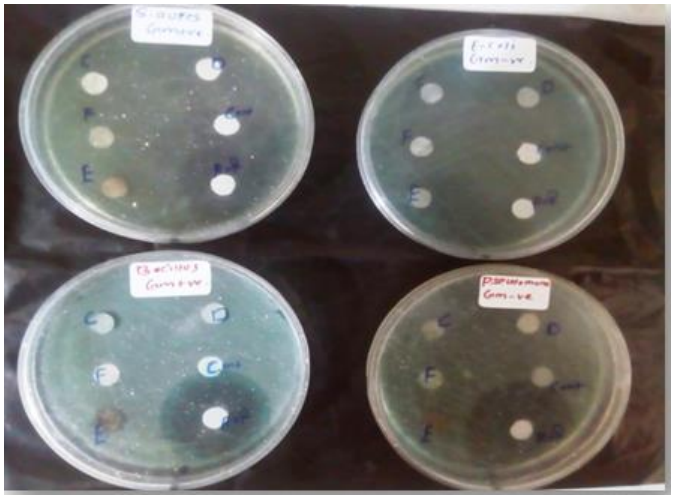
Antibacterial properties of Schiff base complexes

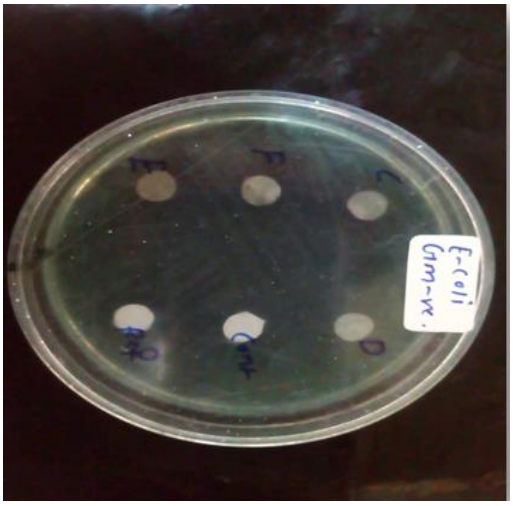
Table 3 : Showing Antimicrobial Sensitivity Test (After 24 Hours at 37 ^{0}C)

Test Compound	GM +VE BACTERIA		GM -VE BACTERIA	
	Staphalococcus aureus	*Bacillus subtilis*	*Escherichia coli*	*Pseudomonas fluorescens*
C (Complex Cu)	-----	-----	-----	10 mm
D (Complex Ni)	11 mm	-----	-----	11 mm
E (Complex Fe)	-----	11 mm	-----	-----
F (Complex Co)	-----	-----	12 mm	-----
Reference Antibiotic	32 mm (OFLOXACIN)	35 mm (OFLOXACIN)	20 mm (OFLOXACIN)	45 mm (OFLOXACIN)
Control Disc	-----	-----	-----	-----

On the basis of result/ antimicrobial testing, it is noted that the given Compound C found to be Antimicrobial against Pseudomonas fluorescens (11 mm). Compound D having antimicrobial against Staphalococcus aureus (11 mm) and Pseudomonas fluorescens (11 mm). Compound E having antimicrobial against Bacillus subtilis (11 mm). Compound F having antimicrobial against Bacillus subtilis (12 mm) (Table:3).







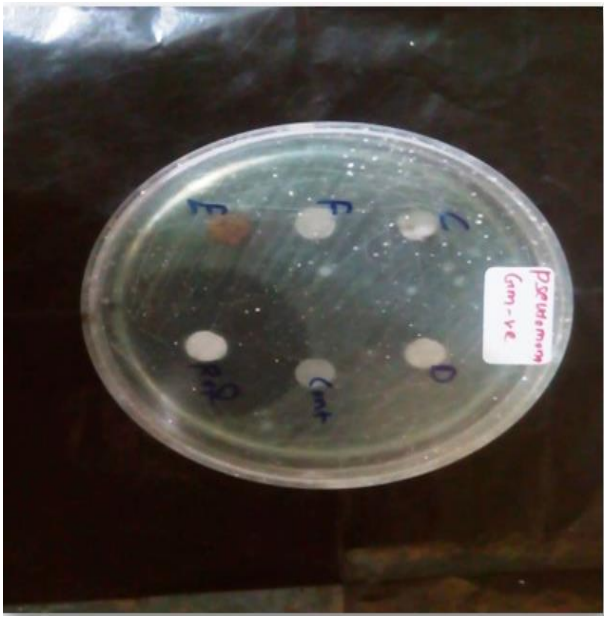


Figure 5 : Showing Antimicrobial Sensitivity Test (After 24 Hours at 37 ^{0}C

IV. CONCLUSION

In the present investigation, synthesis and characterization of Schiff bases synthesized from TTD with substituted aldehyde and derivative TTSB with transition metal (II) complexes have been attempted due to their wide range of applications in various fields of science. The present investigation is summarized in the form of the following conclusions. In modification and complexion process positive change is occur in TT backbone and this change is most important for further characterization. The spectral data shows the remarkable and positive change occur in TT after reaction with substituted aldehydes. Also spectral data shows the remarkable and positive change occur in TT compounds after complexion of TTSB.

Thermogram of the Schiff bases synthesized from TT with substituted aldehyde and derivation TTSB with transition metal (II) complexes indicate that they have varying degree of thermal stability and undergo decomposition at different temperature. The percent weight loss as computed from the thermo grams of the Schiff base synthesized from TT with substituted m-Hydroxy acetophenone and derivative TTSB with transition metal (II) complexes.

The antimicrobial activities of all the synthesized compounds are given in solvent DMSO. Different Gram positive and Gram negative strains are used for antimicrobial study. All compound shows different zone of inhibition.

The experimental studies in the present investigation shows that the uses of TT and TTSB brings some change in structure and enhances thermal properties of the TT compounds.

V. Acknowledgement

The authors gratefully acknowledge the Government Vidarbha Institute of Science and Humanities.

VI. REFERENCES

[1]. Rodriguez-Aguelles M.C., Ferrari M.B., Bisceglie F., Pelizzi C., Pelosi G., Pinelli S. and Sassi M., J. Inorganic Biochemistry, Vol. 98 (2), 313-321,2004.

[2]. Uehara K., Morimoto K. and Shijo Y., Analyst, Vol. 117 (6), 977-979, 1992.

[3]. Barbazan P., Carballo R., Covelo B., Lodeiro C., Lima J.C., and Vazquez-Lopez E.M., Eur. J. Inorg. Chem., Vol. 17, 2713-272, 2008.

[4]. Banerjee S., Mondal S., Chakraborty W., Sen S., Gachhui R., Butcher R.J., Slawin AMZ. , Mandal C and Mitra S., Polyhedron, 28, 2785, 2009.

[5]. Ghavtadze N., Frohlich R. and Wurthwein EU., Eur. J. Org. Chem., 3656, 2008.

[6]. Turan-Zitouni G., Blache Y. and Guven Boll K., Chim.Farm., Vol. 140 (6), 397- 400, 2001. 7. Peng G. & Yunyang W., Heterocyclic Communications, Vol. 19 (2), 113-119, 2013.

[7]. Samudranil P., Balavardhana R., Journal of Organometallic Chemistry, Vol. 731, 67-72, 2013.

[8]. Junbiao C., Gang H., Hongxu L., Wenquan Y., Yueteng Z., Junbiao C., Lihong D., Yujiang L. and Xuejun Y., Journal of Organic Chemistry, Vol.78, 10337- 103431, 2013.

[9]. Frederic L., Martin C., Jean M. and Pierrick N., Tetrahedron, Vol. 67, 8187- 8194, 2013.

[10]. Ke C., Yang H., Qing-Shan L., Xiang L., Ru Y. and Hai-Liang Z., Bioorg. Med. Chem., Vol. 20, 903- 909, 2012.

[11]. Jacobsen, Eric Tan N. and Kian L., Angewandte Chemie - International Edition, Vol. 46, 1315-13171, 2007.

[12]. French F.A. & Blanz E.J., J Med. Chem., Vol. 9, 585, 1966.

[13]. Idem, Cancer Res., Vol. 26, 1638, 1966.

[14]. Idem, Ibid, Vol. 28, 2419, 1968.

[15]. Agrawal Kroshna C., Booth Barbara and Sartorelli Alan C., J Med. Chem., Vol.11(4), 700-703, 1968.

[16]. Mohan M., Gupta N.S., Gupta M.P., Kumar A., Kumar M. and Jha N.K., Inorg. Chimica Acta., Vol. 152(1), 25-36, 1988.

[17]. Mohan M., Kurnar A., Kurnar M. and Jha N.K., Ibid, Vol. 136, 65, 1987.

[18]. Ma T.S. & Tien T. M., Antobiotics. Chemotherapy (Washington), Vol. 3, 49l, 1953.

[19]. Sessler J. L, Tomat E and Vincent M L "Chem. Commun.", Vol. 10, 4486. 2006.

[20]. Haratake M, Fukunaga M and Nakayama M "J Biol Inorg Chem." Vol.10, 250-258, 2005. 22. Kianfar A.H and Mohebbib S., "J. Iran. Chem. Soc". Vol. 4, 215, 2007.

[21]. MohamadArshad, Taki Ahmed Khan and Alam Khan; International Journal of Pharma Sciences and Research, Vol. 5, No. 04, 2014.

[22]. Supun Katugampala, Inoka C. Perera, Chandrika Nanaya Kara and Theshini Perera, Bioinorganic Chemistry and Applications, Vol. 20188, No, 07, 2018.

[23]. Sonmez, M. and M. Sekerci, Polish J. Chem, Vol. 76, No. 907-914 2002.

[24]. Keypour, H. S. Salehzadeh and R.V. Parish, J. Molecules, Vol. 7, N0. 140- 144, 2002.

[25]. Reddy, V.N. Patil and S.D. Angadi, E-J. Chem., Vol. 5(3), No. 577-583, 2008.

[26]. Hanna W.G. and M.M. Moaead, Transition Metal Chemistry, Vol. 26, No.644, 2001.

[27]. Pandeya, S.N., G. Nath and E. Clercq De, PharmaActaHelv, Vol.74, No. 11, 1999.

[28]. Ravanasiddappa M.T. Suresh K. Syed, S.C. Radhavendray C. Basavaraj and S.D. Angadi, E-J.Chem., Vol. 5(2), No. 395-403,2008.

[29]. Del Paggio, A.A., D.R. McMillin, Inorg Chem., Vol. 22, No. 691, 1983.

[30]. Canpolat, E. and M. Kaya, J. Coord. Chem., 123, 2004.

[31]. R. Shanmugakala, P. Thamaraj, C.D. Sheela and C. Anitha, International Journal of Inorganic Chem., Vol. 2012, No.7, 2012.

[32]. S.S. Djebbor, B.O. Benali, J.P. Deloume, Polyhedron, Vol. 16, 2175, 1997.

[33]. P. Bhattacharyya, J. Parr, A.T. Ross, J. Chem. Soc. Datton. Trans, 3149 1996.

[34]. World Health Organization Expert Committee on Biolgical Standardization, Technical report series 822.W.H.O, Geneva, 1992.

Cite this article as :

S. S. Ingale, M. P. Wadekar, G. H. Murhekar, V. G. Thakare, "Synthesis, Characterization and Antimicrobial Studies of Novel Series of 2,4,6-Trihydrazino-6- substituted-1,3,5-triazine Schiff Base Metal Complexes", International Journal of Scientific Research in Science and Technology (IJSRST), Online ISSN : 2395-602X, Print ISSN : 2395-6011, Volume 9 Issue 6, pp. 283-292, November-December 2022. Available at doi :
https://doi.org/10.32628/IJSRST229643
Journal URL : https://ijsrst.com/IJSRST229643