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Synthesis, Characterization and Antimicrobial Activity of Transition Metal Complexes with Diethanolamine and Acetylacetone

Arshad hussain khan[1], Abdul bashar[2*], Urvashi singh[3], Mohammad maqbool dar[3] and Athar adil hashmi[3]

1. Department of Chemistry, SIGHANIA UNIVERSITY, Rajasthan-333515, **INDIA**
2. Department of Chemistry, B.P.Chaliha College, Nagarbera, Assam-781127, **INDIA**
3. Department of Chemistry, JAMIA MILLIA ISLAMIA, New Delhi-110025, **INDIA**

Email: ahk1246@gmail.com, abdulbashar01@gmail.com

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ABSTRACT

Diethanolamine and acetylacetone are versatile ligands that readily form co-ordination compounds with almost all transition metal ions. Co(II) and Fe(III) metal complexes derived from diethanolamine and acetyl-acetone have been prepared. The newly synthesized compounds have been confirmed on the basis of elemental analysis, melting points, conductivity, IR, UV-VIS spectral methods .The metal complexes have been screened for their antibacterial and antifungal activity in Mueller Hinton Media.

Keywords: synthesis, characterization, antibacterial and antifungal activity.

INTRODUCTION

Ethanolamines are a class of organic molecules containing amino and alcohol groups. The amino group may be primary (monoethanolamine, mea), secondary(diethanolamine, dea) or tertiary (triethanolamine, tea) and display reactivity of the corresponding amines. Ethanolamines are primary alcohols and therefore, reactions are typical of primary alcohols. The bifunctional nature of ethanolamines enables them to serve as a variety of commercial applications such as corrosion inhibitors, surfactants, gas purification and herbicides [1].

Ethanolamines are also versatile ligands that readily form co-ordination compounds with almost all metal ions and behave as nitrogen and oxygen donor ligands and their transition metal complexes were synthesized[2,3]. In some cases, ethanolamines lose their ethanolic hydrogen being as ethanolaminate anions, which also behave as ligands similar to ethanolamines and ethanolaminate derivatives of a number of metals have been reported recently [4-7].

Diethanolamine, dea, is an example of tridentate ligand and is a common substances used in the chemical and pharmaceutical industries as an intermediate for the production of detergents, solubilizers, cosmetics, drugs, textile finishing agents and as an absorbent for acidic gases,(Sutton,1963).

Acetylacetone is a beta-diketone (1, 3 diketone) in which two ketones are separated only by one carbon. The beta-ketone is stable as a conjugated enol rather than alpha-diketone due to the delocalization which makes the counter-ion more stable and less likely to regain the proton.The keto form[$CH_3COCH_2COCH_3$]

and enol form[$CH_3COCH=C(OH)CH_3$] co-exist in solution. This keto-enol tautomerism results in the tautomeric migration of a hydrogen atom from an adjacent carbon atom to a carbonyl group of a keto compound to produce the enol form of the compound. Although the enolate form of acetylacetone anion is most commonly found in co-ordination complexes, other possibilities are also known. Acetylacetonate is an example of a bidentate ligand.Metal acetylacetonate complexes which can be used as phase precursors in alkoxo synthesis of esters finds considerable attention[8-11]. Diketone derivatives find versatile applications in making biomolecules, agrochemicals, dyes, pigments, pharmaceuticals and stabilizers for PVC and Polyester.

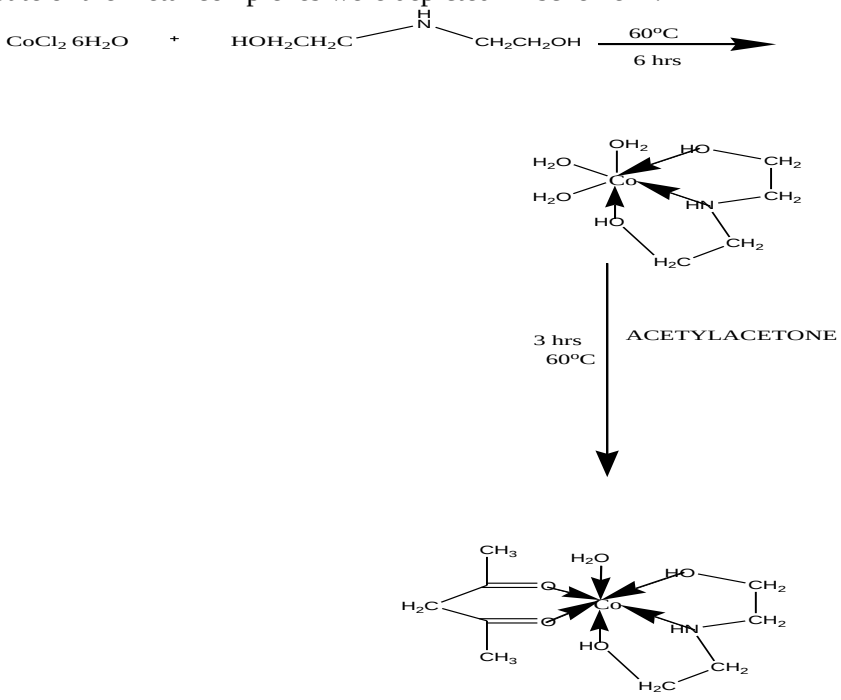
MATERIALS AND METHODS

All chemicals used were of analytical grade (AR) reagents and of highest purity available .They included ferric chloride anhydrous(fisher scientific), cobalt(II)chloride hexahydrate (RANKEM), diethanolamine (MERCK) and acetylacetone (MERCK). Elemental analysis was performed using an elemental analyser. The measurement of conductance of the complexes were measured using a conductometer at 30^0 C .The IR spectra were recorded in a spectrometer (4000-400cm-1). The UV-VIS electronic spectra (200-800nm) were performed using double beam spectrophotometer.The geometries of the metal complexes was determined using the molecular calculations.

Synthesis of the metal complexes: An ethanolic solution of MX_n(0.005 mols) M=Co and Fe, is added slowly to an to an ethanolic solution of diethanolamine (0.005mols) with constant stirring. The mixture is refluxed for 6 h at 60^0C. Then an ethanolic solution of acetylacetone (0.005 mols) is added drop wise and the mixture is refluxed for 3 h at 60^0C. The mixture is then filtered, washed with ethanol and dried the residue.

RESULTS AND DISCUSSION

The synthetic route of the metal complexes were depicted in scheme 1 .



Scheme 1: Synthetic reaction of Co-complex.

Elemental analysis and molecular conductance: The metal complex of iron is soluble in water and the complex of cobalt is soluble in hot water and ethanol. The analytical data and physical properties of the complexes are presented in table 1. The data are consistent with the calculated results from the empirical formula of each compound.

Table 1: Elemental analysis, specific conductance and melting points of the complexes.

COMPLEX	EMPERICAL	MOLE.WT g/mol	ELEMENTAL ANALYSIS FOUND(CALCULATED)%			SPECIFIC CONDUCTANCE mS/cm	MELTING POINT 0C
			C	H	N		
Co-complex	$C_9H_{21}O_5NCo$	282.2	36.38 (38.27)	7.21 (7.50)	4.761 (4.961)	O.14	315
Fe – complex	$C_9H_{21}O_5NFe$	279.1	36.88 (38.70)	7.27 (7.52)	4.82 (5.02)	O.26	186

Conductance of Fe-complex: A 1mM solution is taken for conductivity.

Conductance of Co-Complex : Co-Complex is prepared by dissolving 0.0028g. of the salt in 1mL ethanol and diluting the solution to 10 mL with double distilled water. The conductance Fe-complex=0.26 mS cm^{-1} at 30^0 C. The conductance of Co-complex=0.14 mS cm^{-1} at 30^0 C. The above conductance values indicate that the complexes are electrolytes [12].

IR spectra: The significant IR bands for the complexes are complied and presented in table2. The IR spectrum of the complexes (Fig.1), a sharp band observed at 1700 cm^{-1} is assigned to the v (C=O, carbonyl) mode. This shifts to a lower wave number suggesting the co-ordination of the carbonyl oxygen to the metal centre. A strong sharp band observed at 3400 cm^{-1} is assigned to v (>N-H) of the ligand.

Table 2. IR spectral data for the metal complexes

Complex	V(C=O)	V(M-N)	V(C-N)	V(>N-H)	V(C-O)	V(M-O)
Co-complex	1700	457-464	1600	3400	1100	537-555
Fe-complex	1700	457-464	1600	3400	1100	537-555

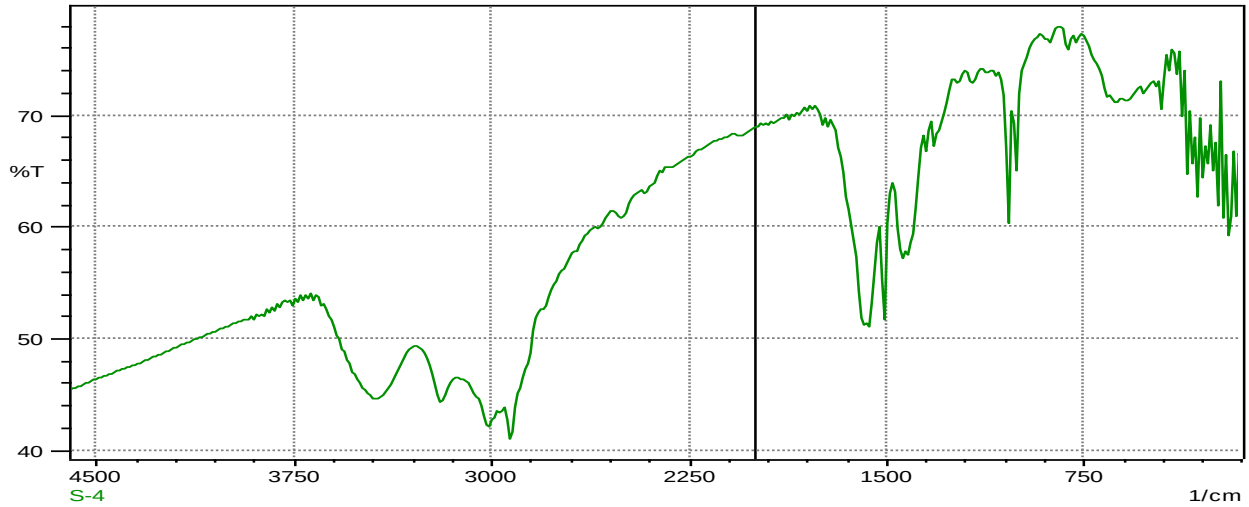


Fig 1. IR Spectrum of Fe –complex.

UV-VIS-Electronic spectra: The UV-VIS spectral data of the complexes are presented in table 3. The electronic absorption spectra of the Co- complex is made in ethanol and then diluting with double distilled water while the Fe- complex is soluble in double distilled water in the range 200-700 nm. For the Co-complex, absorption peak was found at 270.2 nm, 213.8 nm and 209.2 nm (Fig. 2) and for the Fe-complex, absorption peak was found at 378.0 nm, 330.0 nm, 281.5 nm, 442.5 nm, 356.0 nm, 300.5nm and 239.5 nm (Fig.3).

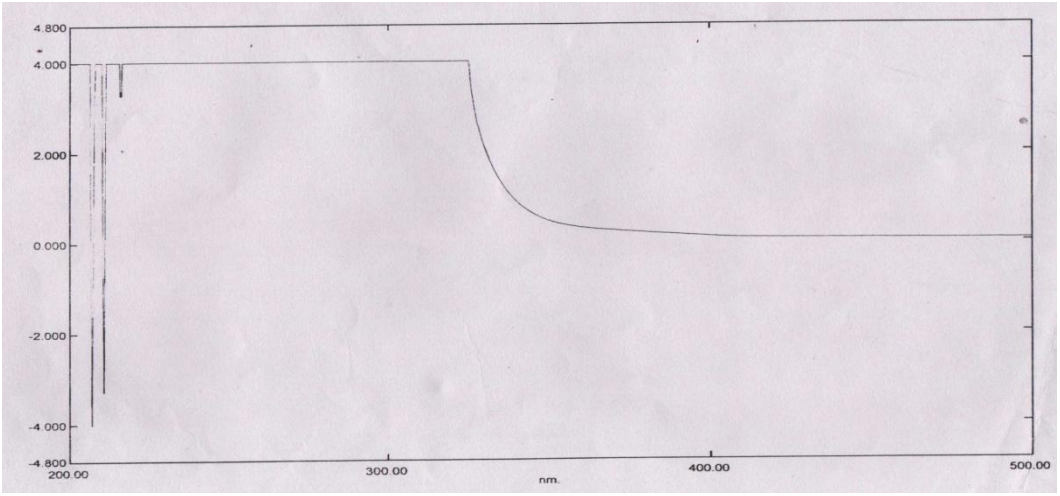


Fig 2: UV-VIS-Spectrum of Co-complex.

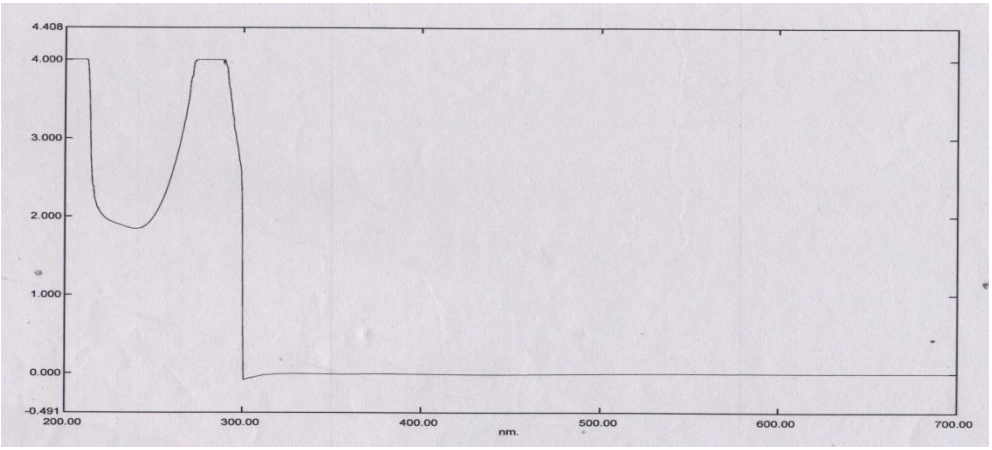


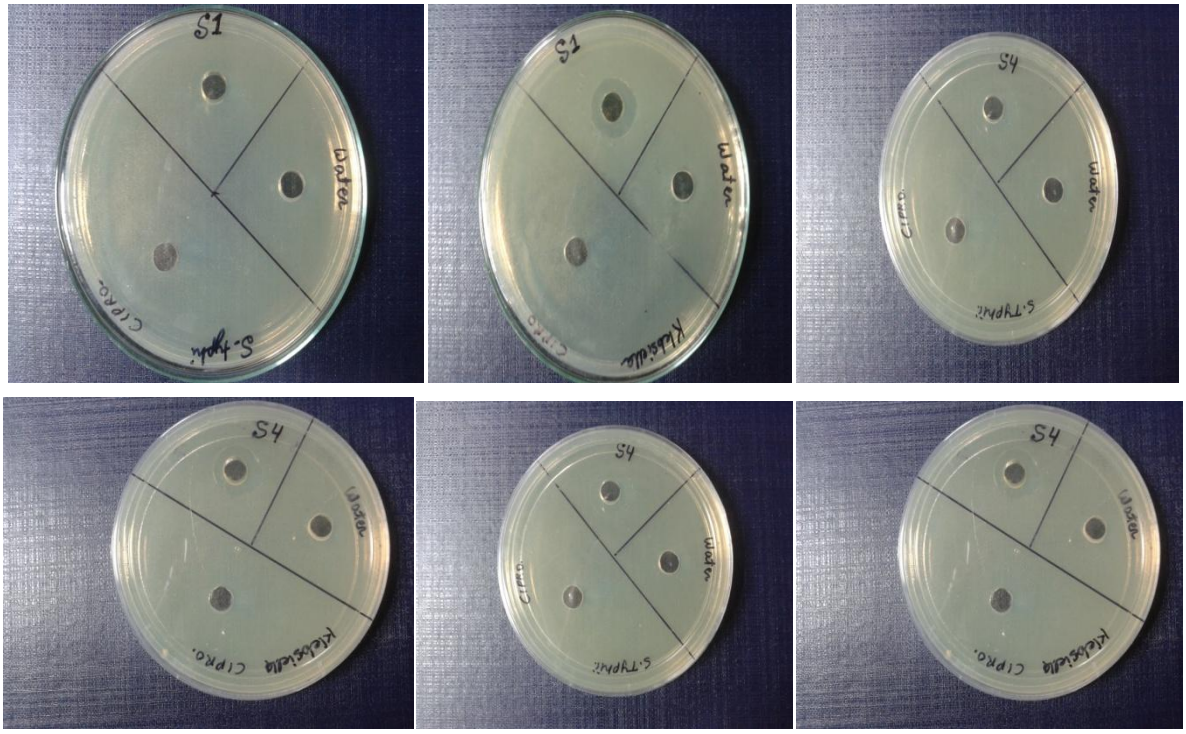
Fig 3: UV-VIS-Spectrum of Fe-Complex.

Table -3 Electronic spectral data for the metal complexes

COMPLEX	Absorbance (nm)	Assignment	Geometry
Co-complex	270.2	Octahedral	Octahedral
-do-	213.8	Pi-pi*	-do-
-do-	209.2	Pi-pi*	-do-
Fe-complex	378.0	Octahedral	Octahedral
-do-	330.0	n-pi*	-do-
-do-	281.5	Pi-pi*	-do-

APPLICATIONS

Antimicrobial activity: An antimicrobial is a substance that kills or inhibits the growth of micro-organism such as bacteria, fungi or protozoans. The discovery of antimicrobials like penicillin's by Alexender Fleming and tetracyclines paved the way for better health for millions around the world. The in-vitro biological screening effects of investigated compounds were tested against the bacteria Salmonella typhi and Klebsiella pneumonie and fungi. Stock solution of 2mg/ml concentration were prepared by dissolving the compounds in hot water and serial dilution of the compounds were prepared in sterile water to determine the minimum inhibition concentration (MIC). The nutrient Mueller Hinton media (MH Media) was poured into petriplates. Different dilution of the stock solution was applied on the 10 mm diameter sterile disc. The discs were placed in incubator for 3 days. The antibacterial and antifungal potential of the complexes were assessed in terms of zone of inhibition (12) of bacterial and fungal growth in figures 4. The minimum inhibitory concentration(MIC) were calculated as the highest dilution showing complete inhibition of the tested bacterial and fungal strain and are reported in table 4-5.



S-1 = Co –complex, S-4 = Fe –complex.

Fig 5: Disk diffusion assay showing zones of inhibition in the presence of compounds.

Table 4: Determination of MIC for antibacterial and antifungal activity of the Co-complex.

Micro-organism	2.0 m g/ml	1.7m g/ml	1.5 m g/ml	1.2 m g/ml	1.0 m g/ml	0.7m g/ml	0.5m g/ml	0.1m g/ml
S. typhi	-	-	-	+	+	+	+	+
K. pneumo-nie	-	-	-	-	+	+	+	+
Fungi	-	-	-	-	+	+	+	+

Table-5: Determination of MIC values for antibacterial and antifungal activity of the Fe-complex.

Micro-organism	2.0mg /ml	1.7mg /ml	1.5mg /ml	1.2mg /ml	1.0mg /ml	0.7mg /ml	0.5mg /ml	0.1mg /ml
S. typhi	-	-	-	+	+	+	+	+
K. pneu- monie	-	-	-	-	+	+	+	+
Fungi	-	-	-	-	+	+	+	+

The Co-complex is effective against both bacteria and fungi. It is evident from the table-4, that the MIC-value for Co-complex against bacteria S. typhi is 1.5mg mL^{-1} while for bacteria K. pneumonie MIC value is 1.2mg m L^{-1}.

The Co-complex is antifungal at MIC- value of 1.2mg m L^{-1}.The Fe-complex is effective against both bacteria and fungi. It is evident from the table-5, that the MIC –value for Fe-complex against bacteria S. typhi is 1.5mg m L^{-1} while for bacteria K. pneumonie MIC-value is 1.2 mg m L^{-1}. The complex is antifungal at MIC-value of 1.2mg m L^{-1}.

CONCLUSIONS

In this paper, we have reported the synthesis of Co(II) and Fe(III) metal complexes with diethanolamine and acetylacetone. The complexes were charactrrized by spectral methods and analytical data. Based on these an octahedral geometry has been assigned for both the complexes. The antimicrobial studies carried out with the complexes confirms that they are antibacterial and antifungal agents with their MIC values.

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