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Synthesis, Characterization and Anti-Microbial Screening of Iron (II) And Cobalt (II) Complexes

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Abstract
Metal complexes of Iron and Cobalt were synthesized and characterized using IR and UV Visible spectroscopy. Their decomposition temperatures and solubility in different solvents were also investigated. Both complexes were found to be soluble in Methanol, Ethanol, Chloroform, Ether, ammonia, DMSO, ethyl acetate, benzene, nitrobenzene and distilled water but insoluble in n-hexane. The decomposition temperatures of the synthesized Iron and cobalt complexes were in the range of 276^0C - 262^0C and 263^0 C- 253^0 C respectively. The IR spectra observed in all the complexes for v(C-O) and (C-O) showed a band at 1590 cm^{-1}- 1700cm^{-1} and around 1200cm^{-1} - 1700cm^{-1} respectively; (M-O) and (N-M) bands also ranged from 800cm^{-1} – 800cm^{-1} and 1000cm^{-1} -7500cm^{-1} respectively. The Uv-Vis result for the complexes showed Iron complex to have peaks at 400nm and a shoulder at 290nm; Cobalt (II) complex at 360nm and a shoulder at 400nm. Both the synthesized complexes showed the highest antimicrobial activity against E. Coli and Aspergilus at their highest concentrations. The synthesized Iron complex however, had the best anti-microbial potential than the Cobalt complex and penicillin put together when used in the highest prepared concentration: 0.20cm for the iron complex, 0.40cm and 0.3cm for the cobalt complex and penicillin respectively against E-coli and the same trend was observed for aspergillus.
Keywords: synthesis, characterization, complexes, anti-microbial, Uv-visible spectroscopy, IR spectroscopy.

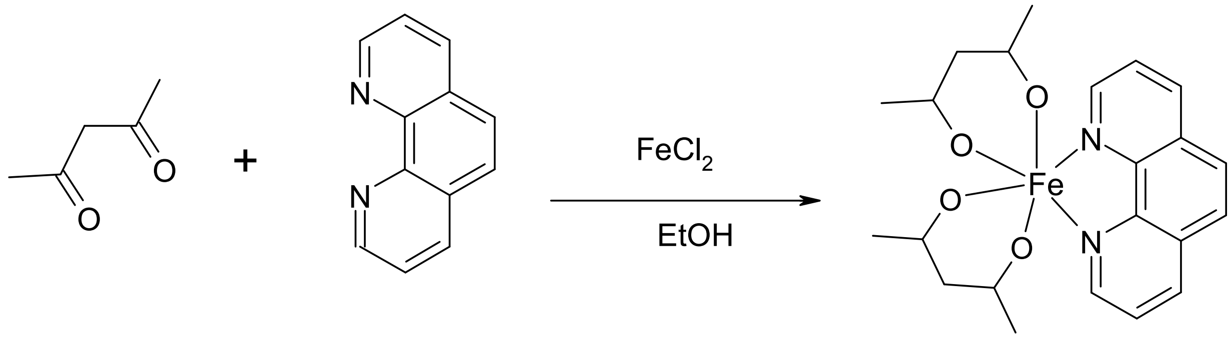
INTRODUCTION

Transition metals are known to form complexes with Schiff base ligands. Schiff bases have been used as chelating agents in the field of coordination chemistry. It is well known that N and O atoms play a key role in the coordination of metals at the active sites of numerous metallobiomolecules. Schiff base metal complexes have been widely studied because they have industrial, antifungal, antibacterial, anticancer, anti viral and herbicidal application. They serve as models for biologically important species and find application on biometric catalytic reactions (Adeola, 2009). Global scientists expand their limit of research in search for these complexes that will be of biological importance. Madelyn *et al.,* 2008 synthesized Lanthanide complexes by mixing solution of lanthanide chloride salt of Eu(III), Sm(III), and Yb(III) with the –diketonate and bpm in 2:6:1 molar proportion. Precipitates formed immediately and the resulting light yellow powders were obtained. All the three crystallized lanthanides complexes were found to give dinuclear structures by elemental analysis. The Uv-Vis absorption spectra of the complexes in $CHC1_3$ solution were dominated by the absorption of the –diketonate and bpm ligand.They also added that the spectra of all complexes are similar and have maxima around 280nm. They exhibit higher absorption peak and slightly smaller intensities. This research work is aimed at synthesizing transition metal (Fe^{2+} and Co^{2+}) complexes with 1-10 phenanthroline and acetylacetone adduct as search in the literature revealed that none of such compounds have been reported. The complexes will be tested for anti-microbial activity.

EXPERIMENTAL

SYNTHESIS OF IRON (II) COMPLEX

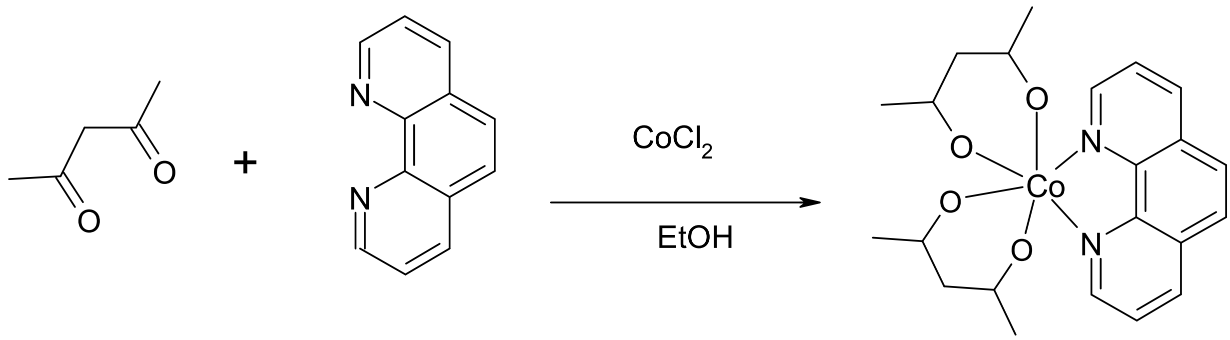
Acetylacetone (1g) was weighed into a 250ml round bottom flask. Absolute ethanol (50cm^3) was added and the mixture was stirred, which was followed by the addition of 1.97g of 1-10 phenanthroline, in ratio of 2:1 at room temperature. Iron (II) salt (1.9g) was added to the mixture, and the mixture immediately gave a dark brown color. The coloured mixture was refluxed for 2hours, allowed to cool, and the dark brown coloured powder was collected by gravity.



Scheme of the reaction

SYNTHESIS OF COBALT(II) COMPLEX

Absolute ethanol (50cm^3)was added to acetyl acetone (1g) in 250ml round bottom flask and stirred. This was followed by the addition of 1.97g of 1-10 phenanthroline, in a ratio of 2:1. Cobalt(II) salt (2.8g) was dissolved in the mixture which immediately turned to a purple color. The coloured mixture was refluxed for 2 hours, and allowed to cool. purple coloured residue was collected by means of filter paper, and the filtrate yielded 65%.



Scheme of the reaction

IN VITRO ANTI MICROBIAL ACTIVITY

SOURCE OF MICRO ORGANISM

The micro organisms used for this study were obtained from the Biology Laboratory of Umaru Musa Yar'adua University (UMYU) Katsina. The organisms used were E-coli, and Aspergillus. The preparation of the culture medium, serial dilution, sensitivity Test and all the observastion and monitoring of the antimicrobial activity were all carried out in the Biology laboratory of the same university.

PREPARATION OF THE CULTURE MEDIUM

Potato Dextrose Agar (PDA) was prepared according to the method of Harrigan W. F., 1998.

SERIAL DILUTION

Various concentrations of the synthesized complexes (1000ug/cm^3, 100ug/cm^3, 10ug/cm^3 and 1ug/cm^3), using DMSO as a solvent were prepared using serial dilution.

ANTIMICROBIAL ACTIVITY TESTING

Disk method was adopted according to the procedure prescribed by Banso, *et. al*., 1999.

CHARACTERIZATION OF THE COMPLEXES

MELTING POINT DETERMINATION

The synthesized metal complexes were analysed using the melting point aparatus to determine their melting points.

RESULTS

TABLE 1: IR spectra of the synthesized complexes showing absorption of different functional groups

Complexes	V(C-O)	(M-O)	(C-O)	(C-H)	(N-M)
[$Fe(acac)_2(phen)$]	1590.32cm^{1}	800 cm^{-1}	1200 cm^{-1}	1490 cm^{-1}	1000 cm^{-1}
[$Co(acac)_2(phen)$]	1700.67cm^{1}	800.34 cm^{-1}	1700 cm^{-1}	1200 cm^{-1}	750 cm^{-1}

TABLE 2: Results of fungal growth recorded in three (3) days for the synthesized complexes and penicilline

Concentration of the complexes (ug/cm^3)	[$Fe(acac)_2(phen)$] growth/day (cm)			[$Co(acac)_2phen$] growth/day (cm)			Penicilline growth/day (cm)		
	1	2	3	1	2	3	1	2	3
1.0	0.99	1.50	2.50	0.89	1.40	2.55	0.11	0.72	1.20
10^1	0.80	1.30	2.22	0.70	1.25	2.00	0.19	0.80	1.30
10^2	0.77	0.99	1.30	0.66	1.00	1.50	0.23	0.91	1.40
10^3	0.65	0.80	1.00	0.50	0.99	1.00	0.40	1.00	3.20

TABLE 3: Size of inhibition(cm) for the synthesized complexes against E. Coli and Aspergillus recorded on Day 4

compounds	Orgnism	1.0 ($\mu g/cm^3$)	10^1 ($\mu g/cm^3$)	10^2 ($\mu g/cm^3$)	10^3 ($\mu g/cm^3$)
$Fe(acac)_2phen$	E-coli	0.40	0.30	0.25	0.20
	Aspergillus	0.60	0.52	0.45	0.30
$Co(acac)_2phen$	E-coli	0.60	0.58	0.53	0.40
	Aspergillus	0.95	0.62	0.75	0.70
Penicilline (control)	E-coli	0.45	0.41	0.38	0.30
	Aspergillus	0.80	0.72	0.65	0.61

DISCUSSION

The transition metal complexes synthesized are crystalline solids, which have relatively high melting temperatures.The Iron complex formed brown crystalline solid with a melting point of 276^{0}C - 262^{0}C. The Cobalt complex gave a purple crystalline solid with melting point of 263^0 C- 253^0 C. The high melting temperatures of the synthesized complexes showed that they are stable comlpexes. The intense colour of complexes is due to the (MLCT) Metal Ligand Charge Tranfer (Anders, L., 2011)

The IR spectra observed in all the complexes indicated v(C-O) band for all the complexes as; 1590.32cm^{-1}- 1700.67cm^{-1} and (C-O) bands around 1200cm^{-1} - 1700cm^{-1} . (C-H) bands ranged from 1490cm^{-1} -1200cm^{-1} . (M-O) and (N-M) bands ranged from 800cm^{-1} – 800cm^{-1} and 1000cm^{-1} -7500cm^{-1} respectively (table 1) which all conformed to the Infrared spectral interpretation by Brian, 1999.

The Uv-Vis result for the complexes showed Iron complex to have peaks around 400nm and a shoulder around 290nm, Cobalt (II) complex showed a peak around 360nm and a shoulder around 400nm (Table 2). These findings were also in agreement with the research findings of Bell *et al.,* 2002.

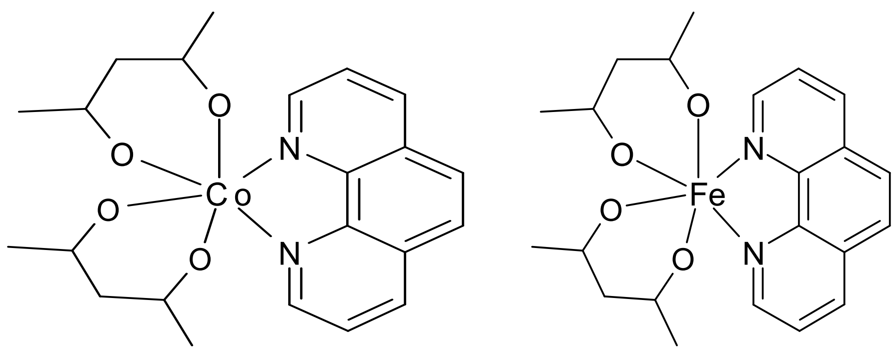


Figure 1: The proposed structure of cobalt and iron complexes

The synthesized metal complexes of Iron and Cobalt were screened for anti microbial activity potential in comparison with the synthetic antibiotic penicillin. The size of the bacterial growth was observed to spread throughout the discs containing the media when the complexes were applied in varying concentrations. This indicated that the sythesized complexes had a significant effect on the growth of the bacteria when the growth inhibition was measured (Table 3). The complex of Iron showed the highest microbial activity against E-coli at its lowest concentration compared to the cobalt complex and penicillin. This showed that the Iron complex is more active than the penicillin and the complex of Cobalt relative to *E.coli* because the size of inhibition (the distance between the compounds concentration on the disc and that of the fungal growth) were measured, and the highest activity shown by the iron complex was 0.40cm activity followed by the penicillin which had the highest concentration 0.45cm and then lastly the cobalt complex with microbial activity of 0.6cm all at 10ug concentration (Table 3).

Three days observation of the colony growth by the fungi revealed that the higher the concentration of the metal complexes in the media, the lower the colony growth. This trend was however reversed in the case of penicilline which showed an increase in fungal growth as the penicilline concentration was increased and hence had the least inhibition. This showed that penicilline complex may not be effective as an anti- fungal treatment (Table 2). Generally speaking and based on the findings of this research iron complex proved to be the best anti-microbial agent against E-coli, Aspergillus and fungi than the Cobalt complex and Penicilline.

CONCLUSION

Based on the findings of this research, synthesized Iron complex proved to be the most effective complex as an antimicrobial agent against E- coli and Aspergillus than the synthesized cobalt complex and also synthetic penicillin.

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