

Synthesis and antimicrobial studies of bis-phthiocolmonoximato Iron III complex.

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ABSTRACT

Research work incorporates methods of synthesis of the ligand (phthiocolmonoxime) its Iron (II) complexes as well as the adduct formation reactions such as halogenation, pyridination and thiocyanation. The compound Fe-1 was prepared by using the procedure reported by Previous workers. The activity of Fe-1 complex was studied by familiar bio assay type –Well method. It was observed on comparison with reference to microcidal and fungicidal activity of the complexes were found to be more effective than ligands. The antimicrobial activity of the compound increases after chelation.

KEY WORDS

phthox-phthocolmonoxime,NCS/NBS/NIS–n-halosuccinamides,Ea-Activation energy,Fe-1 to Fe-4-Fe-complex and adduct chelate compounds,NSQ-Naphthosemiquinone, SQPY-squarepyramidal.

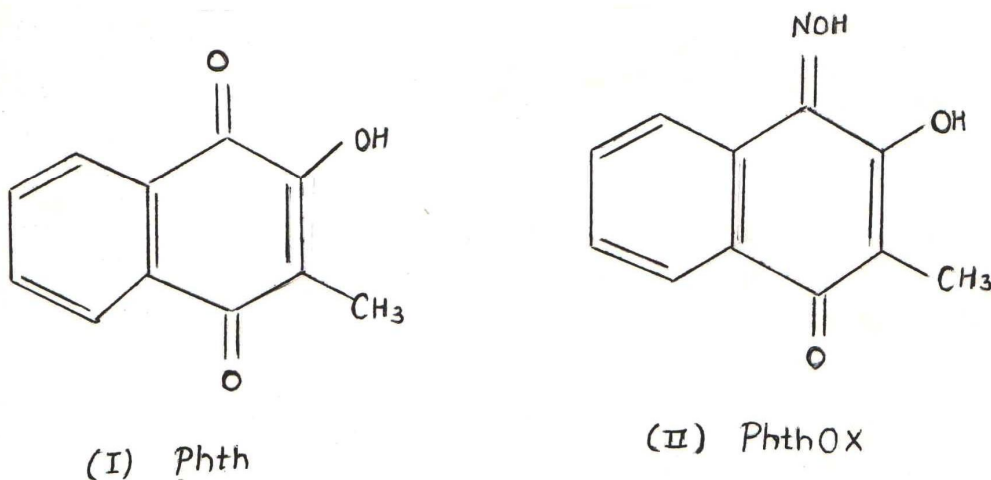
INTRODUCTION

The synthesis and antimicrobial activity of metal chelates is of great importance due to their wide applications in laser technology¹, biological Sciences^{1,2,3,4,5} and industry. There has been a growing interest in the structural study of the chelates derived from organic compounds containing nitrogen and oxygen donors with antimicrobial activity. The chelates of selective metal ions from first transition series have been found to be more active with promising results than the ligand itself against several types of bacteria⁶. The significant antimicrobial activity has shown by metal complexes with 8- hydroxyquinoline increases their importance in medical and biochemical sciences⁷.

Quinones are naturally occurring materials in soils, plants and animals. Quinones play an important role in many biological electron transfer processes particularly in respiration and photosynthesis. During these processes they are reversibly reduced while divalent metal centers are oxidized.

The derivative of quinine, Lawsonia (i.e. -hydroxyl-1, 4-naphthoquinone) is a coloring material obtained from lawsonia alba which is known as heena. It is also indicated to be useful in the treatment of various ailments, diseases and as disinfectant. Some of the derivatives of quinine show antimicrobial and antimalarial activities due to the formation of metal complexes as intermediates⁸⁻¹⁰. The hydroxyquinone derivatives of 1,4-naphthoquinone class have been of interest for sometime due to their mention as therapeutic agents in Indian system of medicine¹¹⁻¹³.

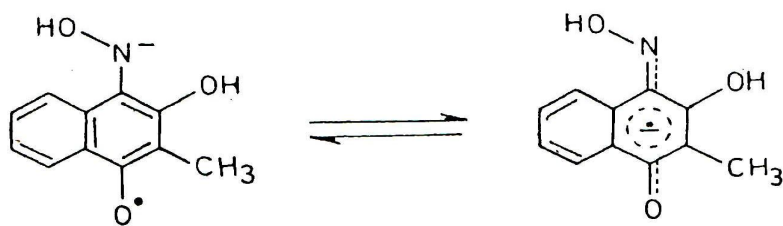
The reactivity of coordinated complex provides a reaction of great versatility when ligands as well as metal centres are prone to redox activity. Hence, it is necessary to see the intramolecular electron transfers probably in redox active ligands viz. phthiocolmonoxime (phthox) (I to IV) having biological relevance. Such electronic transmissions,



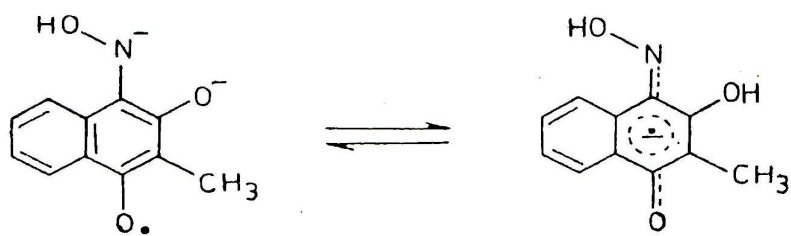
In co-ordinated ligands are well induced in "square planer" complex of nickel when it undergoes through different reactions¹⁴,



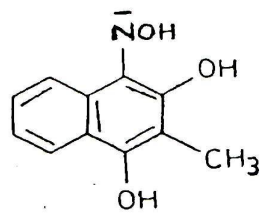
(III b) 4-oxido-1,2-naphthosemiquinoneoxime



(III c) 2-OH-1,4-naphthosemiquinoneoxime



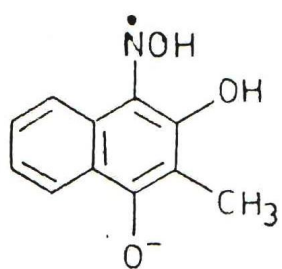
(III d) 2-oxido-1,4-naphthosemiquinoneoxime



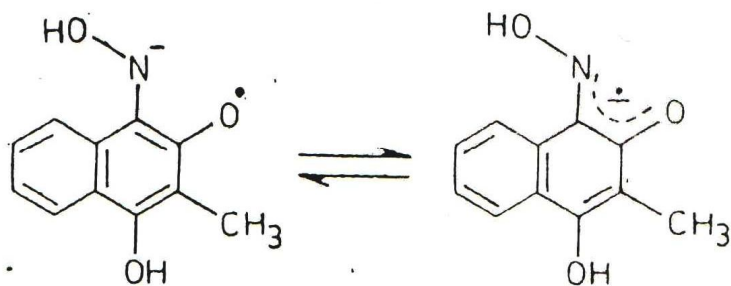
(IV) CAT



(II) NQ



(III) NSQ
iminoxy radical



(IIIa) 4-OH-1,2-naphthosemiquinoneoxime

SYNTHESIS

(i) Synthesis of Ligand :

a) Preparation of Phthiocol: - Phthiocol was prepared according to Fieser's procedure¹⁶.

b) Preparation of 1 –monoxime of Phthiocol:

1-monoxime of Phthiocol (phthiocolmonoxime) was synthesized according to the procedure to the literature¹⁵.

(ii) Synthesis of Fe -1, Bis. (Phthiocol monoxmato) Iron (II) : The compound Fe-1 was prepared by using the procedure reported by Previous workers¹⁷ as given below.

Under inert atmosphere the deaerated solution of ferrous sulphate heptahydrate 0.005 Mole in anhydrous methanol and 5ml of TEOP was added to the deaerated warm methanolic solution to Phthioncolmonoxime 0.01 moles with constant stirring. The reaction mixture was continuously stirred for few hours under nitrogen atmosphere at about 70-80⁰ C. The precipitated chelates was then filtered , Washed with cold water then with distilled anhydrous methanol and finally With pet -ether. The compound was dried under vacuum room temperature.

RESULTS AND DISCUSSION

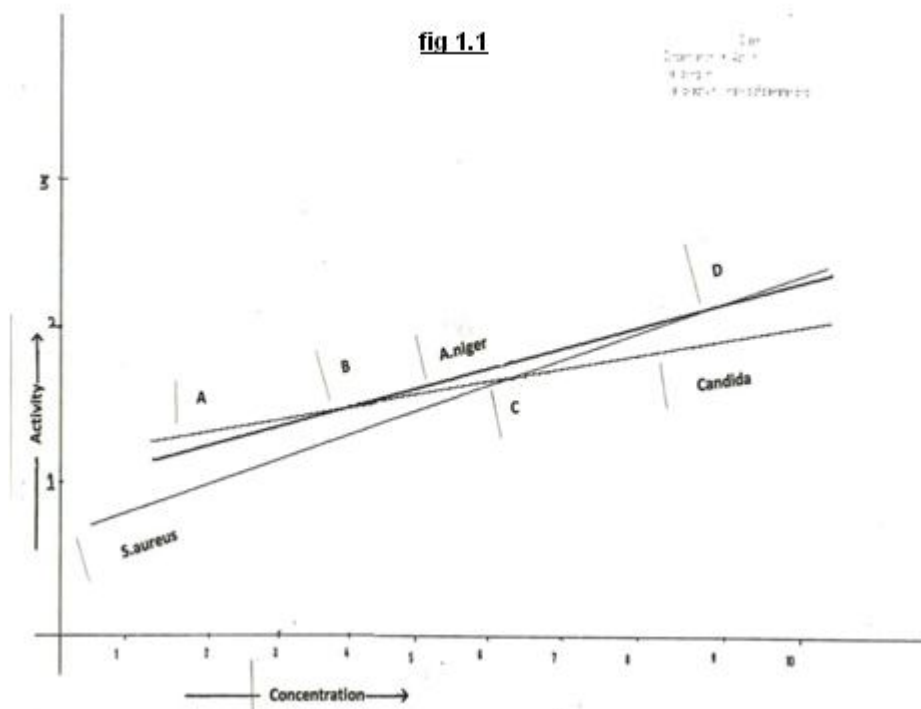
Bactericidal and fungicidal activity of Fe complex:

The activity of Fe-1 complex was studied by familiar bio assay type –Well method.

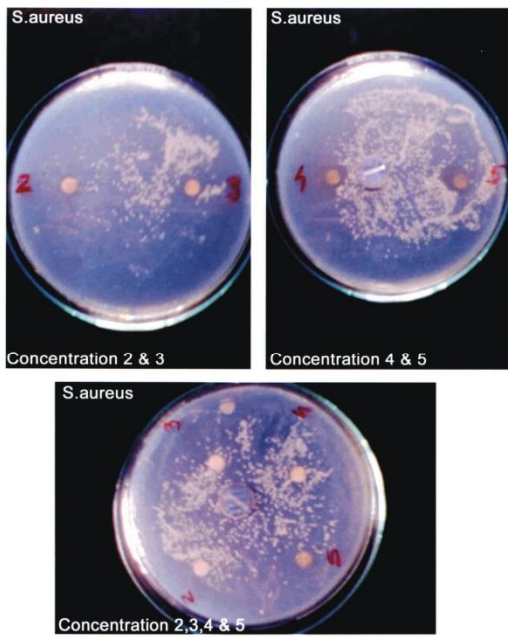
The general nutrient agar (Himedia) was used. The sets of petri-dishes are filled with nutrient agar and sterilized . On cooling these are inoculated with cultures of micro – organisms viz. A.niger,S. aureus Candida. The wells prepared on media and are filled with Fe-complex solution with various concentrations. .After incubation zone of inhibition were observed which are shown in the table given below,

Micro.org.	Fe-complex Cocn. (mg)	Zone of inhibition Diameter (cms).
Aspergillus niger	2	1,4
	4	1.7
	6	1.8
	8	
S. aureus	3	1.1
	4	1.2
	5	1.5
Candida	2	1.2
	4	1.4
	6	1.6
	8	1.8

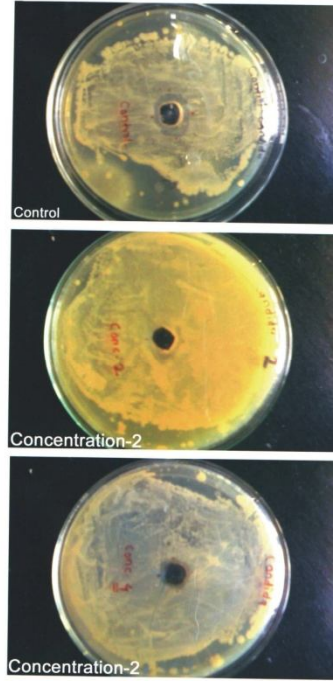
The above results indicate that in general the zone of inhibition increases with the increase in concentration of Fe-1 complex.



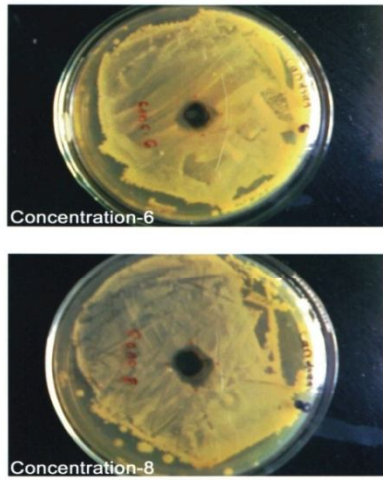
Antibacterial Activity of Fe-Complex



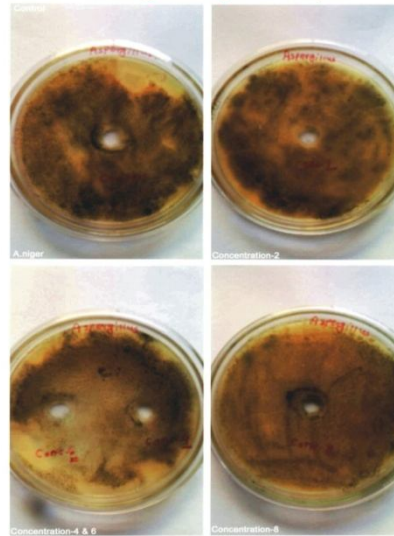
Activity of Fe-Complex Against Yeast



Activity of Fe-Complex against Yeast



Antifungal Activity of Fe-Complex



At the initial conc. at point 'A' reveals that the Fe-complex was more active on candida (yeast), least effective on S-aureus and effect on A. niger is near to the effect on candida. Fe-complex concentration at point B the effect on A. niger and candida was same and effect on s-aureus was increasing but less as compared to A- niger and candida Fe- complex concentration at point C the fungicidal activity was increasing for A-niger as compared to candida Fe-complex concentration at point D shows that the activity on S-aureus was more as compared to A-niger and candida.

The above results shows that the Fe metal complex was found to be more active on s-aureus than A –niger and candida at higher conc. While at lower concentration the metal complex activity was more on A-niger and candida while least on S-aureus.

In was observed the on comparison with reference to microtidal and fungicidal activity of the complexes were found to be more effective than ligands. The antimicrobial activity of the compound increases after chelation. Other factors such as solubility, molar conductivity and magnetic susceptibility are also responsible for increasing antimicrobial activity of the complex^{18,19}

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