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Synthesis, spectral characterization and antimicrobial activity of Chromium (III) complex with 2-Amino phenol

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Abstract- A series of 2-aminophenols including methoxy group were synthesized using appropriate synthetic route. The structures of the compound were determined with spectroscopic techniques and analytical methods. IR spectra of the compounds including and not including hydrogen bonding were compared. The compounds 2 and 4 show the characteristic UV bands attributed to the NH-forms. On the other hand, antibacterial and antifungal activities of the compounds were investigated. Most of the compounds show selective activity toward *Staphylococcus epidermidis* and *Candida albicans*.

Key words: *Staphylococcus epidermidis*, *Candida albicans*, Chromium, UV bands

INTRODUCTION

2-Amino phenol is an organic compound having chemical formula C_6H_7NO with molar mass of 109.13g/mole. Along with its isomer 4-Amino phenol, it is an amphoteric molecule and a reducing agent.¹ It is useful reagent for the synthesis of dyes and heterocyclic compound. Reflecting its slight hydrophilic character, white powder is moderately soluble in alcohol and can be recrystallized from hot water. 2-Amino phenol becomes quite popular in coordination chemistry because of its Schiff bases, which generate a rich variety of complexes.² Recently, the oxidation of many organic compounds with ditertiary butyl chromate has become a subject of great interest and many research works has been published in this present paper, we have reported the synthesis and study of some Chromium (III) complexes of 2-Amino phenol by oxidizing it with TBC to explore the versatility of the oxidizing agent.

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MATERIALS & METHODS

Preparation of reaction mixture

Substrate/oxidant ratio 1:1-1gm CrO_3 dissolved in 10 ml of tert butyl alcohol to prepare tert butyl chromate and 1gm 2-Amino phenol dissolved in 10 ml THF.

Substrate/oxidant ratio 1:2-1gm CrO_3 dissolved in 10 ml of tert butyl alcohol to prepare tert butyl chromate and 2.4gm 2-Amino phenol dissolved in 10 ml THF.

Substrate/oxidant ratio 1:3-1gm CrO_3 dissolved in 10 ml of tert butyl alcohol to prepare tert butyl chromate and 3.16gm 2-Amino phenol dissolved in 10 ml THF.

Procedure

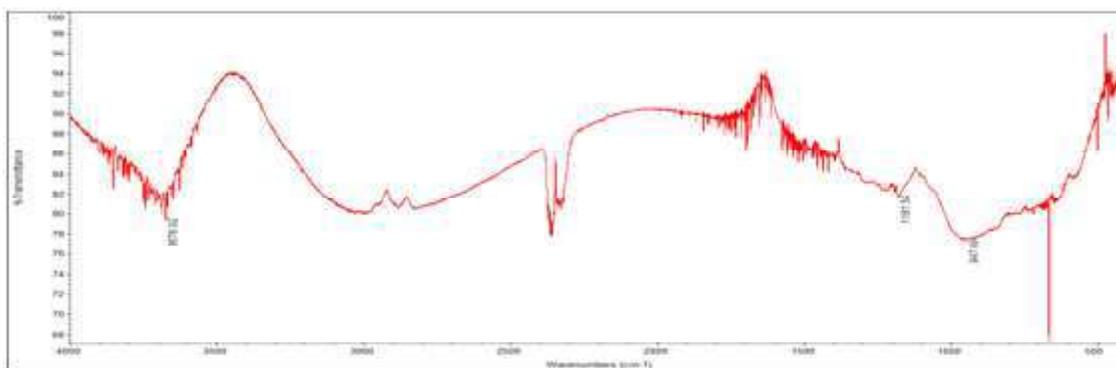
2-Amino phenol was found to be freely soluble in selected solvents namely THF and 1,4-dioxane. Oxidation of this substrate with TBC was performed in two sets, each for the two solvents.³ A solution of the oxidant TBC was prepared separately in situ from Chromium trioxide and tertiary butanol, varying the substrate/oxidant molar ratio as 1:1, 1:2 and 1:3 for each set. The two solution were mixed together to prepare a homogeneous reaction

mixture, stirred continuously for a considerable time using a magnetic stirrer and any change in consistency was noted. The mixture was heated in microwave oven.⁴ Reaction were found to be exothermic, in general, except for those in 1,4-dioxane, in which they were endothermic as indicated by a drop in temperature of the reaction mixture. The six products formed in each of these cases were washed with acetone, dried and weighed.

Spectral character

Absence of characteristic C-H stretching frequency for an aldehyde group suggests that the amino phenol aldehyde group has been oxidized to a carboxylic group. This is further confirmed by the appearance of relevant peaks near 761.02 cm^{-1} for (C-O) stretching vibrations of a carboxylic group. The characteristic peaks for aromatic (C-H) stretching, aromatic (C-C) and (C=C) in-ring stretching. The presence of Cr-O and Cr=O bonds in these complexes is indicated by the (Cr-O) stretching frequencies ranging from $478.65\text{--}454.10\text{ cm}^{-1}$ and (Cr=O) peaks near $761.02, 667.55\text{ cm}^{-1}$. The broad and strong absorption bands appearing from $1161.69\text{--}947.69\text{ cm}^{-1}$ shows the presence of the H-Bonded (O-H) group, intensity and peak broadening varying greatly with variation in substrate/ oxidant ratio. This indicates the difference in the extent of coordination of the (O-H) oxygen atom with the Cr atom.⁵ The lowest (O-H)

stretching frequency is centred at 3341.10 cm^{-1} in the compound labelled AP-101, while the maximum value recorded is for AP-111 at 3676 cm^{-1} . A strong (C-O) carbonyl absorption is visible near 1369.46 cm^{-1} . However, the vibrations near $1574.87\text{--}1556.97\text{ cm}^{-1}$, $1488.19\text{--}1393\text{ cm}^{-1}$ and $1283.43\text{--}1203.11\text{ cm}^{-1}$, respectively, indicate that the aromatic ring in amino phenol is intact and has not undergone any decomposition/ cleavage during the course of oxidation. The presence of (C-O) stretching vibrations for the methoxy group near 667.56 cm^{-1} in the IR spectrum of these complexes is indicative of the oxygen atom of the other group of Amino phenol being uncoordinated. This is supported by the weak alkyl (C-H) stretching vibrations appearing near 1147.07 cm^{-1} for each of these compounds. Other weak absorption bands for bending and rocking vibrations of (C-H) group were recorded near 686 cm^{-1} and $478.65\text{--}454.10\text{ cm}^{-1}$, respectively.⁶ A comparative spectral analysis for the six oxidation products of Aminophenol shows a considerable shift in the relative intensities and peak positions of these vibrational frequencies, which may be attributed to a difference in the percentage composition of Cr metal and variable extent of its coordination with the ligand. The strong peaks at 3341.22 cm^{-1} and 2373 cm^{-1} are probably due to the impurities present in the KBr. A very weak peak near 3676.92 cm^{-1} is characteristic of CO, absorption.



Antimicrobial Activity

This section will describe the antibacterial activities of Cr (III) complexes as well as their ligands.^{1-3,7} It has also been proposed that chelation decreased the polarity of the metal ion mainly due to partial sharing of its positive charge with a donor group throughout the entire chelate ring system. This chelation process, therefore, enhanced the lipophilic nature of the central metal ion, which in turn facilitated its permeation through the lipid layer of

the membrane, thereby allowing the metal complex to more easily cross the bacterial membrane, increasing the complexes' activity. In addition, many other factors, such as solubility, dipole moment, conductivity that influenced by a metal ion, could contribute to the antibacterial activity of the complex. On chelation, the delocalisation of pi-electrons over the whole chelate ring increases the liposolubility of the complexes. The hydrocarbon tail

serves as a lipophilic group to move the compound through the cell's semi-permeable membrane and block metal-binding sites within microorganism enzymes. The latter was attributed to the fact that complexes may deactivate different cellular enzymes, which play a vital role in the microorganism metabolic pathways. It was also suggested that the toxicant's ultimate action was the denaturation of one or more cell proteins, impairing normal cellular processes as a consequence. Other factors that also increased the antimicrobial activity were the solubility, conductivity and length of bond between metal and ligand.

A comparative study of Schiff base and its complex indicate that the metal complexes exhibit higher antibacterial activity than the free ligands.^{4,8} This is probably due to the greater lipophilic nature of the complex. Such increased activity of the metal chelets can be explained on the basis of overtones concept and chelation theory. According to overtones concept of cell permeability the lipid membrane that surrounds the cell favours the passage of only lipid soluble materials due to which liposolubility is an important factor which controls the antimicrobial activity. On chelation the polarity of the metal ion will be reduced to a greater extent due to the overlap of the ligand orbital and partial sharing of positive charge of metal ion with donor groups.

RESULT & DISCUSSION

The expected broad peak for O-H stretching (H-bonded) at 3294.94 cm^{-1} , bidentate carboxylic acid functioning as ligand at 1489.05 cm^{-1} , C-O stretching at 1230.58 cm^{-1} , O-C stretching at 1369.46 cm^{-1} and M-O stretching at 680.66 cm^{-1} are present in the curve. Similarly, the loss pattern is just what we expect for the formulation as shown. We observe that the FTIR curves (table -3) of sample R3 contain almost all the peaks which are expected for its formulation. We observe that the FTIR curves contain almost all the peaks which are expected for its Formulation $\text{Cr}_2\text{O}_3 \cdot \text{CH}_3\text{COOH} \cdot \text{CH}_3\text{COCOOH} \cdot 2\text{H}_2\text{O}$. It is supported by the peaks at 3298.28 cm^{-1} (O-H stretching), 964.41 cm^{-1} (C-O-H stretching) 1489.05 cm^{-1} (bidentate carboxylate ligand), 844.82 cm^{-1} (-COOH co-ordinated), 964.41 cm^{-1} (C-O stretching) etc. Also the loss pattern in TGA-DTA curve supports the proposed formulation. The first stage loss between the temperature ranges 31.98-140°C corresponds to the fragmentation of CH_3COOH .

The second stage loss of lattice water. The substrate might have been co-ordinated through two COOH groups, as the loss is taking at higher temperature.

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