[illegible] ACTIVITY OF 1, 2 [illegible] AND ITS CHELATES WITH Fe(II), Co(II) AND Ni(II)

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Abstract

[illegible] is a very important ligand. It has a vast application in [illegible] chemistry, co-ordination chemistry and industrial chemistry. [illegible] 1, 2 naphthoquinone-1 oxime was synthesized [illegible] by using metal salts of Fe, Co and Ni. [illegible] were checked for two new bacteria [illegible] A. the result activity was found to be [illegible] was found to be resistant to Ni complex. As [illegible] 1,2 naphthaquinone-1-oxime but the metal [illegible] shows a good sensitivity against the bacteria. [illegible] (Fe, Co and Ni) was done by using 1, 2 [illegible]. Different concentrations of these metal chelates were [illegible] was recorded. The concentration taken for [illegible]. The results were recorded and compared for [illegible]. The metal chelates shows a presence of metal at truncated [illegible] the detection of metal easy using 1, 2 nahthaquinone-1-[illegible]

Keywords: 1, 2 [illegible], Organometallic, Klebsiella Pneumonia, [illegible] determination of metals, Chelates

1. Introduction

[illegible] application in the field of co-ordination chemistry, [illegible]. Naphthoquinones are widely distributed in [illegible] biological activities.(Pawar,et.al., 2011). A [illegible] 1, 2-naphthoquinones received a [illegible] activity (Shukla, et.al.2012). The chelation [illegible] the research for the past few decades.(Jadhav, [illegible] used as chelating ligands in the field [illegible] have been of great interest for many [illegible] to the results of infra-red, proton NMR and [illegible] the complexes in the solid state exists in the quinone [illegible] we have synthesised the 1, 2 NNQ 1 [illegible] were screened for two new bacteria and the [illegible] were done to find out the necessary [illegible] was recorded and analysed for further

2. Materials and Method

2.1 Preparation of 1, 2 Naphthaquinone-1-Oxime :

The ligand 1, 2-naphthoquinone-1-oxime is synthesized in laboratory as per the reported method.β-naphthol was dissolved in sodium hydroxide in 100ml distilled water and made upto 200ml. 5g of sodium nitrite dissolved in little amount of water was carefully added to the solution. The mixture was cooled by addition of ice. 10% sulfuric acid was added with help of burette with constant stirring. The temperature was maintained below 5°C. 1-nitroso-2-nahpthol (1, 2naphthaquinone-1-oxime) separates as yellow crystals. The solution was kept still for 2 hrs. filtered and washed with cold water. The product was found to be red needles. The yield was found to be almost theoretical yield. (Cumming, 1937)

2.2 Preparation of Metal Chelates of 1,2 Naphthaquinone 1 Oxime :

A stock solution of Fe (II), Co (II) and Ni(II) is prepared by using AR grade chemicals. Deionised water is used during synthesis. The chelates were prepared by mixing metal salt solution and ligand in 2: 1 proportion. (Jadhav, et.al, 2014) The mixture was constantly stirred for one hour at 1200rpm on magnetic stirrer. The pH of the solution mixture was maintained in between 5-7 by adding ammonia to the mixture. The mixture was warmed on water bath and cooled. It was filtered and dried overnight for further investigation purposes.

2.3 Antimicrobial activity testing:

Test organisms: The antimicrobial activity of ligands, metal salts and synthesized metal chelates is tested against bacteria and compared. Maintenance of culture: The cultures of bacteria were maintained on Mueller-Hinton and subcultured accordingly and preserved at 4^0 C. for 24 hours in incubator. The testing against growth of micro-organisms was carried out by using well diffusion method. (Gonewar, et.al. , 2012)

2.4 Spectroscopic determination for metals using 1,2 Naphthaquinone 1 oxime as a organic ligand:

The metal chelates were formed using the procedure discussed above. Glass vessels were cleaned by soaking in acidified solutions of potassium dichromate, followed by washing with soap water and rinsing two times with distilled water. (Kamble, et.al. 2011) The stock solution of different concentration was prepared for the detection of the metal (Chandramouleeswaran and Ramkumar, 2014). The different concentration taken into the consideration was 50ppm, 100ppm, 150ppm and 200 ppm in solvent as methanol. The UV Visible spectroscopic method was used to capture the difference in the spectroscopic values at the different concentrations. (Kamble, et.al. 2011) The instrument used for the spectra was the UV-100 Shimadzu UV Spectrophotometer.

3. Results and Discussions

The synthesis 1,2 napthquinone was characterised by using different techniques such as NMR, UV-VIS, IR ,TLC etc. The results are discussed below. The chelates were characterized by using UV–VIS, IR etc.

3.1 UV-VIS: Electronic Absorption data (λ nm) of the NQO and its metal chelate in DMSO were checked in the range of 200-600nm. The table below shows the absorption values of the compounds in nm.

Sr.No.	Name of the Compound	π – π* Transitions	π – π* Transitions	n – π* Transitions
1.	NQO	222	311	418
2.	Fe (NQO)	256	332	421
3.	Co (NQO)	260	342	522
4.	Ni(NQO)	255	340	505

The UV-VIS absorption of the organic compound and its chelates with Fe and Co increases normally but only Ni slightly decreases because of the structural deformation property of the nickel while forming a complex.

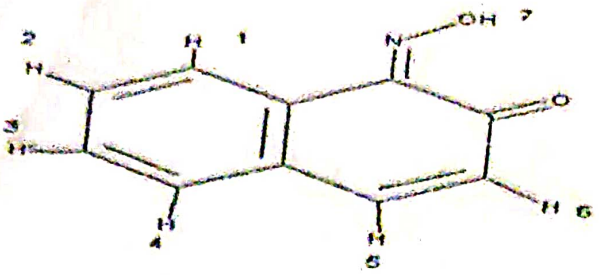
3.2 Infrared Spectral Analysis: The IR frequencies of all the compounds were recorded and found to be in the range. The observed spectral data is given in the table below

Table: The Characteristic ν IR (cm^{-1}) Bands of NQO and its metal chelates:

Sr.No.	Name of the Compound	C-H	C=N	N-O
1.	NQO	1621	1568	1136
2.	Fe (NQO)	1610	1560	1147
3.	Co (NQO)	1615	1565	1138
4.	Ni(NQO)	1615	1510	1158

The IR values of the NQO and metal chelates with Fe and Co keeps on increasing but the values of Ni chelate slightly decreases due to the structural deformating property of nickel when forming complex with the organic compounds.

3.3 NMR: The ^1H NMR was taken for synthesised organic compound 1, 2 Naphthaquinone-1-Oxime. The peaks were observed for the oxime structute as the 1, 2 naphthaquinone-1-oxime exist in tautomeric form. As oxime form is more stable than the nitroso form, the peak values of oxime are observed.



The 1H NMR values are compared with calculated and the observed

Sr. No. of Proton	Calculated δ Value	Observed δ Value
1 H	7.53	7.50
2 H	7.30	7.36
3 H	7.21	7.21
4 H	7.63	7.64
5 H	7.97	7.80
6 H	7.26	7.26
7 H	5.0	4.78

The NMR values confirm the formation of 1, 2 naphthaquinone-1-oxime from β-Naphthol. As all the values of NMR, IR, UV is in sync with the reported values.

3.4 Colour: The colour changed observed was for the metal chelates. The colour change observed was blood red for Fe (NQO), reddish brown for Co (NQO) and greenish black for Ni (NQO) complex.

3.5 The anti -microbial activity of the metal chelates: The plates were incubated at 37°C for 24 hours in incubator. The clear zone of inhibition of growth for the organism was measured.

Name of Bacteria	Sample Name	Sensitivity	Resistiyity
Klebsiella Pneumonia	NOQ	Sensitive	
	NOQ (Fe)	Sensitive	
	NOQ (Co)	Slightly Sensitive	
	NOQ (Ni)		Resistance
	Fe Salt	Sensitive	
	Co Salt	Sensitive	-
	Ni Salt	Strong Inhibitory Effect	-
Salmonella Paratyphi A	NOQ	-	Strongly Resistance
	NOQ (Fe)	Slightly Sensitive	-
	NOQ (Co)	Slightly Sensitive	-
	NOQ (Ni)	Slightly Sensitive	-
	Fe Salt	Sensitive	-
	Co Salt	Sensitive	-
	Ni Salt	Slightly Sensitive	-

3.6 Spectroscopic Determination of metal using NQO as ligand:

The various concentrations of the metal chelates were prepared such as 50ppm, 100ppm, 150ppm and 200ppm. The electronic spectra of all the samples were taken and evaluate the absorption from 200nm to 600 nm.

NQO (Fe): All the four samples of Fe were analyzed and compared with each other. The wavelength for Fe was found to be in the range of 200 to 550 nm. The Absorption was at the highest peak of 320 nm for all the samples. As the concentration decreases the absorption also starts to decrease. The absorption decreases at constant difference between two peaks.

NQO (Co): All the four samples of Co were analyzed and compared with each other. The wavelength for Co was found to be in the range of 200 to 450 nm. The Absorption was at the highest peak of 240 nm for all the samples. The absorption is at very high intensity of all the samples. The absorption of 100ppm, 150 ppm and 200 ppm does not show a substantial difference, the difference is only observed in the sample of 50 ppm.

NQO (Ni): All the four samples of Ni were analyzed and compared with each other. The wavelength for Ni was found to be in the range of 200 to 550 nm. The Absorption was at the highest peak of 230 to 360 nm for all the samples. The absorption is at very high intensity of all the samples. The absorption of 200ppm and 50 ppm show a substantial difference, the concentrations of 100 and 150 ppm show a merger at 220 to 240 nm and separates out at all 240 to 350 nm.

4. Conclusion

1,2 naphthaquinone-1-oxime can be easily prepared by using β Naphthol . The organic compound was determined by using various techniques such as NMR, IR, UV, TLC etc. All the values of the spectroscopic determinations were found to be in the range of the reported values. The only decrease in UV and IR was observed due to the specific property of the nickel metal as it gives the slight structural deformity while forming a complex with an organic compound. The metal chelates show various interesting properties such as analytical determination of metals at various concentrations, antimicrobial activity against various bacteria etc. The antimicrobial activity against the two new bacteria was determined successfully. The bacteria were Klebsiella Pneumonia and Salmonella Paratyphi A. The organic compound and its chelates shows a good antimicrobial activity against Klebsiella Pneumonia except the nickel chelate whereas the organic compound does not show the activity against the bacteria Salmonella Paratyphi A but the metal chelates of the organic compounds shows slight sensitivity against the bacteria. The spectroscopic analysis of the metal chelates was determined using different concentrations such as 50ppm, 100ppm, 150ppm, 200ppm. it shows various peaks of absorption with different metals which will make the detection of these metals possible by using 1,2 naphthaquinone-1-oxime as a ligand. The organic compound possesses a good potential of application in various fields of science and technology.

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