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Synthesis, characterization of p-anisidine oxamic acid and their metal complexes

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Abstract:

The present investigation was carried out on Synthesis, characterization of anisidine oxamic acid and their metal complexes. A series of new mixed metal-ligand complexes is reported with p-anisidine oxamic acid as a primary ligand. The structure of these metal complexes were confirmed by various technique such as FTIR, H^{1}-NMR and UV electronic spectral studies, and their melting point, metal yield percentage were recorded. All complexes indicates a four coordination with p-anisidine oxamic acid viz, M=Mn(II), Co(II),Ni (II) and Cu(II). The study can be helpful for further molecular characterization of organic acids and their metal complexes.

Key words: p-anisidine oxamic acid, Metal ions

Introduction

Oxamic acid is the N-amine derivatives of aliphatic dibasic acids. They possess the charateristic functional group. The oxamic acid is the simplest example of such ligand which contains both carboxylic group and primary amide group.

The simplest oxamic acid is known as amino substituted glyoxylic acid (H_2N-CO-COOH,Oxm H_2). The broad chemical variety resulting from the flexibility of carbon chemistry and the structural variety of coordination compounds provides an attractive approach for

synthesizing novel materials exhibiting magnetic properties. Among others (Kahn, 1995) also reported bis (oxamato) type transaction metal complexes have been extensively used as versatile building blocks for magnaetic homo-and hetero-polymetallic systems (Kahn, 1993; Kahn,1996; Aukauloo *et al.*, 2000) for the exploration of molecular magnetic properties. A numerous studies and their metal complexes. Sharma vinod Kumar (1998) synthesized and characterized of Sc(III)-N-alpha-napthyl-oxamic acid and their complexes. The scandium complex of N-alpha-napthyl-oxamic acid had been prepared for estimation of Scandium metal. The complex formed is thermally stable upto 250°C. Ruffer *et al.*, (2007) have been worked done on synthesis, characterization and magnetic properties of new homotrinuclear Cu(II) complexes.

Keeping in view of the above facts a detailed study on Synthesis, characterization of p-anisidine oxamic acid and their metal complexes has been planned.

Experimental Methodology

1-Synthesis of P-anisidine oxamic acid (PAOA)

2.46 gm(0.02M) p-anisidine and 2.70 (0.02 M) diethyl oxalate were mixed and refluxed for two hours in round bottom flask fitted with air-condenser. The contents of the flask were allowed to cool and about 20ml of absolute alcohol was added. The oxamide formed in this reaction was then fitted out due to insoluble nature. After this a aqueous solution cabonate (0.02M) in distilled water was added to filtrate. The mixture was then steam distilled for about two hours. The sodium salt of anisidine oxamic acid was obtained. Then, it was concentrated with HCl added to this solution. The light brown crystalline product of anisidine oxamic acid was obtained. It was filtered, washed with distilled water and recrystallized from alcohol. The recrystallized product was dried over with anhydrous P_2O_5 in a vacuum desiccators.

2-Synthesis of Mn (II) , Co(II), Ni(II) and Cu(II) metal complexes of P-anisidine oxamic acid.

(a)- Synthesis of Mn(II) metal complex

0.174 gm (0.002mol) PAOA was dissolved in 10 ml methanol and mixed with 10 ml aqueous solution of 0.245 gm (0.001mol) maganese acetate tetrahydrate in round bottom flask. The

mixture was followed by pH, room temperature and refrigerated for overnight. Finally brown colored crystals were obtained.

(b)- Synthesis of Co(II) metal complex

10 ml of methanolic solution of 0.39 gm (0.002 mol) p-anisidine oxamic acid was mixed with 10 ml aqueous solution of 0.245 gm (0.001mol) cobalt acetate tetrahydrate in round bottom flask. The mixture was followed by pH, room temperature and refrigerated for overnight. Finally dark green coloured crystals was obtained.

(C)- Synthesis of Co(II) metal complex

0.20 gm (0.001) Copper acetate monohydrate dissolved in 10 ml distilled water and mixed with 10 ml aqueous solution of 0.39 gm (0.002mol) p-anisidine in round bottom flask. The mixture was followed by pH, room temperature and refrigerated for overnight. Finally reddish green coloured product was obtained.

(D)- Synthesis of Ni(II) metal complex

0.30 gm (0.001) Nikil acetate monohydrate dissolved in 10 ml distilled water and mixed with 10 ml aqueous solution of 0.39 gm (0.002mol) p-anisidine in round bottom flask. The mixture was followed by pH, room temperature and refrigerated for over night. Finally reddish green coloured product was obtained.

2. Characterization

Thin Layer chromatography (TLC) :- Authenticity and purity of the synthesized compounds were checked by running a single spot on TLC and elemental analysis was analyzed for C,H and N by elementar vario EL III carlo Erba 1108, Elemental analyzer at IIT, New Delhi.

FTIR (Fourier Transform Infra-red) spectrum:- the synthesized compounds were characterized by FTIR,H-NMR and electronic spectral studies to elucidate the probable structure of P-anaisidine oxamic acids and its metal complexes. Shimadzu UV-vis 1600 A ultraviolet spectrophotometer in the range from 190-1100nm has finished the ultraviolet. The ligand visible spectrum with its metal complexes. The molar conductivity was used by the coring conductivity meter 220 to measure the conductivity of fully complexes at room

temperature in a newly prepared $1x10^{-3}$ M in aqueous solvent using the KBr pellet using FTIR spectrophotometer shimadzu Analytical Instrument Facility at IIT, Delhi in the range (4000-400) cm^{-1}, IR spectra for the ligand and its metal complexes were recognized.

Results and Discussion

It is clear from Table-1 that the solubility of p-anisidine oxamic acid shows variability in different chemical compounds. The present author revealed that the NMR and FTIR spectral data of p-anisidine oxamic acids show singlet and δ 11.191 ppm respectively which may be attributed due to three protons of $–OCH_3$ group oxamic acids exhibits a singlet at δ4.2784 due to one proton of ›N-H group. PAOA (p-anisidine oxamic acids) exhibits δ 6.9145 ppm to δ 7.6975 ppm due to unsymmetrical pattern of four protons of benzene ring. PAOA exhibits singlet δ 10.6494 due to one proton of –COOH group. Electronic spectral data of synthesized metal complexes are summarized in following heads:

A-**Study of Mn(II) complex**

The electronic spectra of Mn(II) complex show three bands in the range of 17500-18000, 21500-23000 and 24500-27000 cm^{-1} corresponding to the transition $^4A_{1g}$-

^6A_1g, $^4E_1g(G)$ -^6A_1g and $^4Eg(D)$-^6A_1g respectively. It is clear that the synthesized Mn(II) metal complex have on octahedral geometry.

B-**Study of Co(II) complex**

The electronic spectra of Co(II) complex show three bands in the range of 11000-12000, 14000-17000 and 17500-20000 cm^{-1} corresponding to the transition $^4T_{1g}$-

^4T_2g (F), $^4T_1g(G)$ -$^4A_1g(F)$ and ^4T_1g-$^4T_1g(P)$ respectively. It is clear from table-2 that the synthesized Co(II) metal complex have on octahedral geometry.

C-**Study of Ni(II) complex**

The electronic spectra of Ni(II) complex show three bands in the range of 12000-14000, 18500-20000 and 25500-28500 cm^{-1} corresponding to the transition $^3A_{2g}$-^2T_2g, ^3A_2g -$^3A_1g(F)$ and

^3A_2g-^3T_1g(P) respectively. It is clear from table-3 that the synthesized Ni (II) metal complexes have on octahedral geometry.

D-**Study of Cu(II) complex**

The electronic spectra of Cu (II) complex performed three bands in the range of 11500-15500, 16000-18500 and 25500-26500 cm-^{1} corresponding to the transition $^2B_{1g}$-^2A_1g, ^2B_1g -^2B_2g(F) and ^2B_1g-^2E_1g respectively. It is clear that the synthesized Ni(II) metal complex have on octahedral geometry.

Table.1. Solubility of P-anisidine oxamic acid

S.No.	**Compounds**	**Solubility**
1.	$CHCl_3$	Insoluble
2.	$(CH_3)_2Co$	soluble
3.	CCl_4	Insluble
4.	C_2H_5OH	Insluble
5.	DMF	Soluble
6.	DMSO	Soluble
7	C_6H_6	Partial soluble
8	CS_2	Insoluble
9	$C_6H_5CH_3$	Insoluble
10	H_2O	Soluble

Table.2. Physical and elemental properties of synthesized oxamic acids (PAOA) and their metal complexes.

s.no	Compound	Molecular formula	Colour	% elemental analyses						m.p/decomp-osition temperature (2°C)
				Cabon		Hydrogen		Nitrogen		
				C	F	C	F	C	F	
1.	PAOA	$C_9H_9O_4N$	light brown	55.38	55.07	4.79	4.65	7.18	7.96	215 °C

Table.3.H-NMR spectral data of synthesized oxamic acids

S.no	PAOA	Probable assignments
1	3.7395 (3H,s)	Singlet due to three protons of $–OCH_3$ group
2	4.2784(1H,s)	Singlet due to one proton of N-H group
3	6.9145-7.6975 (4H,m)	Unsymmetrical pattern of four protons of benzene ring
4	10.6494(1H,s)	Singlet due to three protons of –COOH group

Abbreviations- s=singlet, t=triplet, m=multiplet

Similar results have been obtianed by number of workers such as Nzikayel *et al.*,(2017) worked done on synthesis, FTIR and electronic spectral studies of metal (II) complexes of pyrazine 2-carboxylic acid derivatives. Egbele *et al.*,(2014) have synthesized and characterized of mixed 1-10 phenanthroline and vancomycin ligands-Meta(II) Complexes. Hossain (2012) reported mixed ligand complexes of Co(II) and Fe (II) with maleic acid and heterocyclic amines using FTIR and ^{1}H-NMR spectroscopy. The findings of present author were supported by Kamruddin and Roy (2001). They synthesized and characterized eleven new metal complexes like Cr(II),Mn(II), Fe(II), Co(II), Ni(II) and Cu(II) of pyridyl thioacetic acid and 2-pyrimidyl thioacetic acid. These compounds are also exhibited biocidal activities.

Synthesis and characterization are novel features for the confirmation of organic compounds. Malik and Ahmed (2022) also synthesized and characterized Di and triorganotin (iv) complex with n-Butylhydrogen phthalate.

Conclusion

On the basis of present findings, the present author can be concluded that method of functional group transformation was achieved from FTIR, ^{1}H-NMR spectroscopy. These analyses indicate coordination of all the metals to the p-anisidine oxamic acid. In present day, there is a need of more advanced technologies for nomenclature of unknown organic acids with coordination of metal complexes.

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References:

[1]- Sharma,Vinod Kumar 1998. Synthesis and Characterization of Sc(III)-N-α-napthyl-oxamic acid complex.Asian Journal of Chemistry. Vol.1(3):472-475.

[2]-Kamruddin, S.K. and A.Roy 2001. Synthesis and Characterization of Cr (III), Mn(II), Fe(III), Co (III) and Cu(II) complexes of 4-pyridylthioacetic acid and 2-pyrimidyl thioacetic acid. Vol.40,p:211-212.

[3]- Malik, Abdul and Ejaz Ahmed 2022. Synthesis and characterization and biocidal studies of the newly synthesized Di and triorganotin (iv) complex with n-Butylhydrogen phthalate.Research Gate link.

[4]-Ruffer, Tobias,; Bjorn Brauer,; Annie K. Powell,;Ian Hewitt,; Georgeta Salvan 2007. Synthesis, characterization and Magnetic properties of new homotrinuclear copper (II) Complex. Inorganica Chimica Acta 360**:**3475-3483.

[5]-O.Kahn.1993. Molecular magnetism,first ed., VCH,Weinheim.

[6]-O.Kahn1996.Adv.Inorg.chem.43:179

[7]-Nzikayel S.;Akpan I.J.and Adams EC. 2017. Synthesis, FTIR and electronic spectral studies of metal (II) complexes of pyrazine 2-carboxylic acid derivatives. Med. Chem.7-11.

[8]-Egbele R.O., Osakwe S.A.;Nzikayel S.2014. Synthesis and characterization of mixed 1-10 Phenanthroline and vancomycin ligands-Metal (II) complexes.World Journal of Applied Science and technology. 6:171-176.

[9]- Hossain,BM.;Islam RF.; Salam AM.;Yousuf AM 2012. synthesis and characterization of mixed ligand complexes of Co(II) and Fe(III) with maleic acid and heterocyclic amines. Journal of Bangladesh chemical society 25:139-145.