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Synthesis, studying the spectral, thermal aspects and in vitro Antimicrobial activity of Novel Azo Co-ordination Polymers

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Abstract

4,6-Dinitroresorcinol was reduced in alcoholic alkali to give a novel azo polymer, poly [azo(1-napthol)]. These co-ordinationpolymers of polyazochelates we reprepared with cu^{+2}, Ni^{+2}, Co^{+2}, Mn^{+2} and Zn^{+2} metal ions. The novel polymer synthesized at Batch: 1 were characterized by following techniques such as elemental analysis, IR spectra & thermo gravimetric analysis. The co-ordination polymers were characterized by elemental Analysis, IR spectral and diffuse reflectance spectral studies and were evaluated for their thermalst ability by thermogravimetric Analysis. The number average molecular weight (Mn) was determined by non aqueous conductometric titration. Furthermore, the magnetic susceptibilities of these coordination polymers have been examined. Antimicrobial efficacy of all these compound also been scrutinized.

Keywords: Co-ordinationpolymers, Elemental Analysis, Infrared Spectra, Electronic Spectra, Magnetic measurements, Thermo gravimetric Analysis (TGA),Antimicrobial activities.

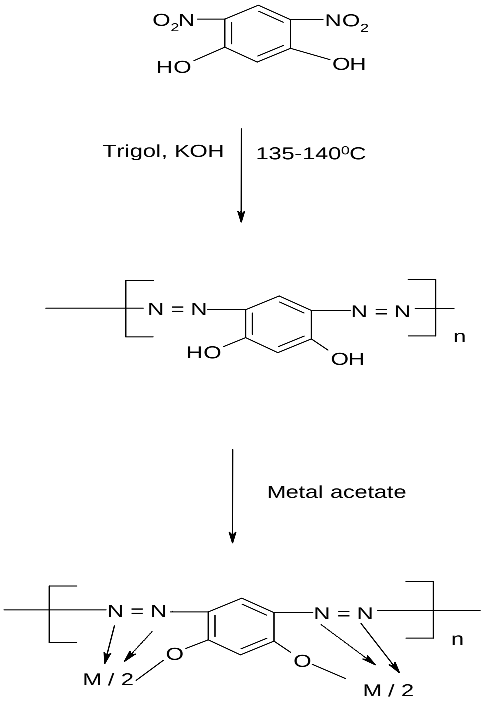
Introduction

Semi conducting catalytic properties, waste water metal recovery, protective coating materials, antibiofouling paints and antifungal activity also been studied of this polymers[1,2]. The structure shown arehaving metal ions are treated with chelating agents and these chelating agents are arrange with alternatelocations are known as the bi-chelating ligands. Several bichelating ligands were prepared as chelating agents developed such us 8-hydoroxy quinoline and salicylic Acid etc[3-6]. -SO_2, -CH_2, -O family peptides a similar group of ligands linked at the –N=N-& azo moeity[3-9].

Single crystal of coordination polymers ligated with transition metal ions in the last few years has found its applications which has undoubtedly created a widespread interest[10-16]. Co-ordination polymers are polymers composed of metal ions and di or polyfunctional ligands. Besides supramolecular chemistry of polynuclear transition metal compounds with multidentate organic ligands have also been a subject of intense research during the last few years leading to the fabrication of novel Co-ordination supramolecules and polymers[17-21].

Materials and Methods

The chemicals used were all of analytical or laboratory grade. Poly[azo (1-Napthol)] was synthesized in a double-step running information. Preparation of poly [azo(1-Napthol)] (Az) was according to a literature[22].



Where, M= Cu, Ni. CO, Zn, Mn etc.

Batch-1: Poly(azo(1-napthol)-M^{+2} polymer.
Where M=Cu^{+2}, Co^{+2}, Ni^{+2}, Mn^{+2}, Zn^{+2}

Synthesis of azoligandpoly [azo(1-napthol)](Az): Azo ligand Az was synthesised by alcoholic alkaline reduction of 4,6 dintro resorcinol in trigol and KOH by self polymerization method reported for polymerization of dinitro ary lenes[22]. Added in drop wise manor with continues stirring at room temperature the solution containing 4,6 dinitro resorcinol (17.0 gms, 0.1 mole) in trigol (150 ml) to which KOH (44.8 gm, 0.8 mole) was gradually dissolved completely. The temperature was then raised to 135-140^{0}C and the reaction was continued for 5 hr.. Then reaction was quenched by cooling to 100^{0}C, & neutralized with the ethanol- HCL (50:50 V/V) & the resulting neutralized mixture was poured into ice water (500 ml), powder was collected through filtration, washed with water & dried in oven. And finally, it was reprecipitated with water, washed and dissolved in DMF. The yield of black powder was 90%.

Preparation of Coordination polymers: A solution of 0.01 mole of ligand Azo polymer (Az) in water was added drop wise to a solution of Metal (0.005 mole) in 100 ml water with vigorous stirring for 90 min. Thus, different azo ligands were in the pH ranges at 4.5 to 6 pH. The greenish blue solid that came out was as a precipitate. The polymer precipitated as suspension was digested on a boiling water bath for 2 hr. The resulting crude blue-green solid was collected by filtration, washed with a mixture of 1:1 water-ethanol-acetone. After weight adjustment of the polymer [$Az\text{-}M^{+2}$]; airdried. These polymers $Az{-}Cu^{+2}$, $Az{-}Ni^{+2}$, $Az{-}Co^{+2}$, $Az{-}Mn^{+2}$ and $Az{-}Zn^{+2}$ were obtained in nearly quantitative yield.

Antibacterial Activities: The antibacterial and antifungal activities of the Az-ligand and its coordination polymers were evaluated and tested (concentration: 50μg/ml) against gram-negative bacteria (E.coli, Salmonella thypi and Klebsiella promioe) and gram-postive bacteria (Bacillus subtilis and Staphylococcus aureus) and against plant pathogenic organisms such as Penicillium expansum, Botrydepladiathiobromine, Nigrospara Sp, and Rhizopusnigricum by the agar cup plate method. Control Revolving with Methanol Method; Area of Inhibition Zone mm 2.

Antifungal Activities: Fungicidal activity of all the compounds was checked at 1000 ppm of the tested chemicals on 7 day old culture of Rhizoctonia solani by poisoned food techniques for in-vitro. Penicillium expansum, Nigrospora Sp, Trichothesium Sp, Rhizopus nigricum different plant pathogenic organisms were prepared. The antifungal activity of ligand and its co-ordination polymers have been analyzed on each one of those media. The culturing medium PDA Potato 200g, Dextrose 20 g, Agar 20g and Water 01 litre A 5-day-old culture was used for coinfection. The tested compounds were solubilized in PDA medium at 1000 ppm concentrations and then these were autoclaved at 120^{o}C for 15 min at 15 atm pressure. The medium was poured in sterile petri plates and allowed to cool down before the organisms were inoculated. In the case of fungi, the percentage inhibition on the 5 day of mycelial growth was assessed as follows.

% Inhibition=100(x-y)/x.
X = Areaofcolony in controlplate y = Area of colony in test plate

Measurements: Elemental analysis C,H,N (C,H,N,%): a C,H,N elemental analyzer (Italy) was used for the determination of Az-ligand compound and its coordination polymers. IR Spectra were recorded form of KBr tablets for ligand and all polymer samples on Nicolet 760 D Spectrophotometer. Diffuse reflectance spectra for theCu^{+2}coordinationpolymers were recorded on a solid sample of these in powder form, using a Backman DK- 2 A Spectrophotometer equipped with a solid reflectance attachment. One of them used MgO as the references compound. Thenumber average molecular weight ofall the coordination polymers was determined by a method already published:[23] Magnetic susbceptibility Measurements of All the compunds were studied by Gouy method. $Hg\,[Co(CNS)_4]$ was used as a calibration, and Molar susceptibilities were corrected using pascal's- constants for diamagnetism of componentatoms. Then the values of effective magnetic moment μ eff was calculated.

The polymer samples were subjected to the TG test in air (slowrate) in a 'PERKIN ELMER PYRIS 1 TGA' apparatus. Electronic spectra (diffuse reflectance spectra) of the solid polymeric chelates were recorded with a Beckman DK-2A Spectrophotometer using a solid reflectance attachment with a reference compound of MgO.

Polymeric chelates were assayed for metal ions by decomposing an aliquot of chelate with mineral acid. The metal content was determined, as described in the literature. Take a sample of100 mg chelate and then add HCl — 1ml, H_2So_4 — 1ml, $HClO_4$ — 1ml and then $NaClO_4$ — 1gm; was added.

The evaporated mixture was spotted in the vessel and the dried salt was dissolved in doubled distilled water made reconstituted to mark. This solution used for titrating the metal content to the standard EDTA solution. Test for antimicrobial activity against both gram positive (+) and gram negative (-), organisms was done on all sample, which was carried out as per method already reported[24,25].

Results and Discussion

Poly[azo(1-napthol)(Az)] was synthesized by reductive polymerization of 4,6 dinitro resorcinol in triethylene glycol (Trigol) and KOH. The product was a black powder. The ligand is further characterized spectroscopically and analytically by EL elemental analysis & IR spectrum as illustrated below.

Elemental Analysis: Table-1 shows the metal position and macrocyclic ligand with all their contents, C and H and N, which is also well in agreement with the hypothetical structure.

IR Analysis: Table-2 shows the Band positions observed in the recorded infrared spectra of the KBr disks of the synthesized azo ligand (Az) and its Co-ordination polymers on Nicolet FTIR-760 spectrophotometer with tentative assignments. In spectrum, significant bands due to the azo moiety phenolic hydroxyl (-OH) groups (Figure-2). The complete band appeared in ligand and all the complex at 3800- 2700 cm^{-1} is assigned to the stretching vibration of the organic-OH group. The band in which the inflections are observed in this band at 2780, 2820 $cm^{–1}$. The bands at 3200 – 3650 $cm^{–1}$ is assign to $–CH_3$ linkage & for aromatic structure IR frequency 3050, 1600, 1010 $cm^{–1}$. In the studied coordination polymers 3360 – 3400, 1420, 1280 and 1090 $cm^{–1}$ regions for the bands observed are considered the well measured bands for OH stretching, bending, rocking, wagging vibration as significant towards the presence of aqua ligand[26]. The Co-ordination polymers prepared using Az-ligand were non-soluble in regular organic solvents. Consequently, the Co-ordination polymers cannot be characterized by molecular mass by conventional techniques viz Osmometry, Viscometry, etc. The melting point of Co-ordination polymers was as high as 300^0C. The number average molecular weight (*Mn*) of Azo Polymer has been calculated by non aqueous conductometric titrations.

All the non-aqueous conductometric titrations were carried out in pyridine with standard sodium methoxide solution in pyridine as titrant base and the conductance measurements were recorded using METROHM KNODUCTOSKOP E 365 as conductance bridge. The main expected spectral changes were the development of a broadened band in the area of 3360 and 3450 cm^{-1}. The coordination chemical situation is quite free from the other but in the azo metal salts the ligand, give is a azo ligands while the other in azo ion linkages is a free ligands, The interferences of the request frequencies band was also assigned tooed Azo ligands is a chelating coordinating ligand and It through pyrazole Schiff base. No of both the complexes and in azo metal salts the oxygen of the–OH group of the ligand complex was attached to the metal ions other put frequency shifts are due to There was The frequency bands were assigned to the –N =N- group at 1406±14cm^{-1} in IR spectrum of azo ligand due to shift to the lower frequency was another noticeable band shift from the azo ligand towards the centre at the higher frequency at 1420 cm^{-1} in IR spectrum of Azo liqand corresponding to in plane –OH stretch, In the spectrum of the coordination polymer, making the M-O bond formation[27]. Additionally, 1090 & 1280 cm^{-1} C-OH group of azo moieties were bonded with a weak bond were confirmed.

Table-1: Analysis of Az-ligand and its Co-ordinationpolymers.

Compound Empirical Formula	Formula weight Mol Cal g/mol	Yield %	Elemental Analysis			
			% C	% H	% N	% M
			Calc/Found	Calc/Found	Calc/Found	Calc/Found
$C_{20}H_8N_8O_2$	392	90	43.90/43.8	2.43/2.40	31.14/34.1	-
$C_{20}H_{10}N_8O_2.Cu^{+2}.2H_2O$	493.5	74	48.63/48.60	2.43/2.40	22.69/22.65	12.86/12.80
$C_{20}H_{10}N_8O_2.Nl^{+2}.2H_2O$	489	72	49.07/49.00	2.45/2.45	22.90/22.90	12.06/12.00
$C_{20}H_{10}N_8O_2.Co^{+2}.2H_2O$	489	70	49.07/49.00	2.45/2.45	22.90/22.90	12.09/12.00
$C_{20}H_{10}N_8O_2.Mn^{+2}.2H_2O$	485	76	49.48/49.45	2.47/2.45	23.09/23.00	11.34/11.30
$C_{20}H_{10}N_8O_2.Zn^{+2}.2H_2O$	489	68	48.48/48.45	2.42/2.40	22.62/22.60	13.13/13.10

Table-2: Spectral Features and magnetic moment of metalchelates.

Co-ordination polymers (Metal Chelates)	μ_{eff} BM	Electronic Spectraldatacm^{-1}	Transition	IR spectral features common For all cm^{-1}
Az–Cu^{+2}	5.88	23370	Change Transfer	3800–2700
$C_{20}H_{10}N_8O_2.Cu^{+2}.2H_2O$		15650	$^2B1g \rightarrow ^2A1g$	1420–OH Azomoiety
Az–Ni^{+2}	3.1	22500	$^3A_{2g}(F) \rightarrow ^3T_{1g}(F)$	2780– CH_2
$C_{20}H_{10}N_8O_2.Nl^{+2}.2H_2O$		15290	$^3A_{2g}(F) \rightarrow ^3T_{1g}(F)$	2820
Az–Co^{+2}	5.15	24660	$^4T_{1g}(F) \rightarrow ^6T_{2g}(V^1)$	3200–3650-CH_3
$C_{20}H_{10}N_8O_2.Co^{+2}.2H_2O$		19990	$^4T_{2g}(F) \rightarrow ^4A_{2g}(V^2)$	3050

		8792	$^4T_{1g}(F)\rightarrow{}^4T_{1g}(P)$	1600 Aromatic
				1010
Az–Mn^{+2}	3.35	24040	$^6A1g\rightarrow{}^4T2g(^4G)$	1406±14–N=N–
($C_{20}H_{10}N_8O_2.Mn^{+2}.2H_2O$)		18650	$^6A1g\rightarrow{}^6A1g(^4Eg)$	1110C-O-M
		16225	$^6A1g\rightarrow{}^4A1g(^4G)$	1280&O-M
				1090Bands
Az–Zn^{+2}	D*	-	-	-
($C_{20}H_{10}N_8O_2.Zn^{+2}.2H_2O$)				

Table-3: Number Average Mol. Wt (*Mn*) of poly [azo(1-napthol)] estimated by non- aqueous conductometric Titrations.

Azo Polymer (Azo- ligand)	Milli equivalents of base required for neutralization of one OH Group (First Break) (Y)	Milli equivalents of base required for Complete neutralization (X)	Degree of polymerization D_p=X/Y	Number average molecular weight (*Mn*)
Az-ligand	80	835	10.5	1250

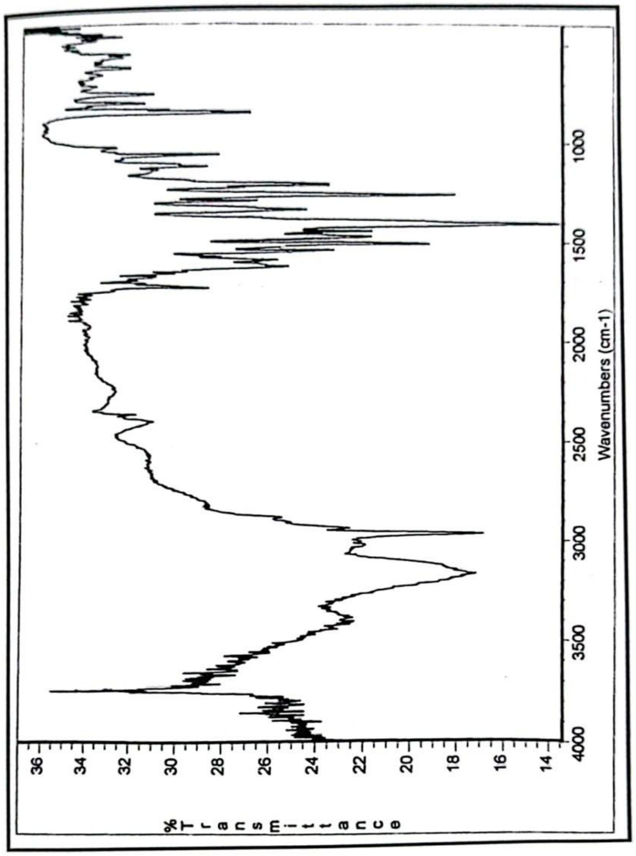


Figure-2: IR Spectrum of $C_{20}H_{08}N_8O_2$ poly[azo(1-Napthol)]/[Azo-ligand].

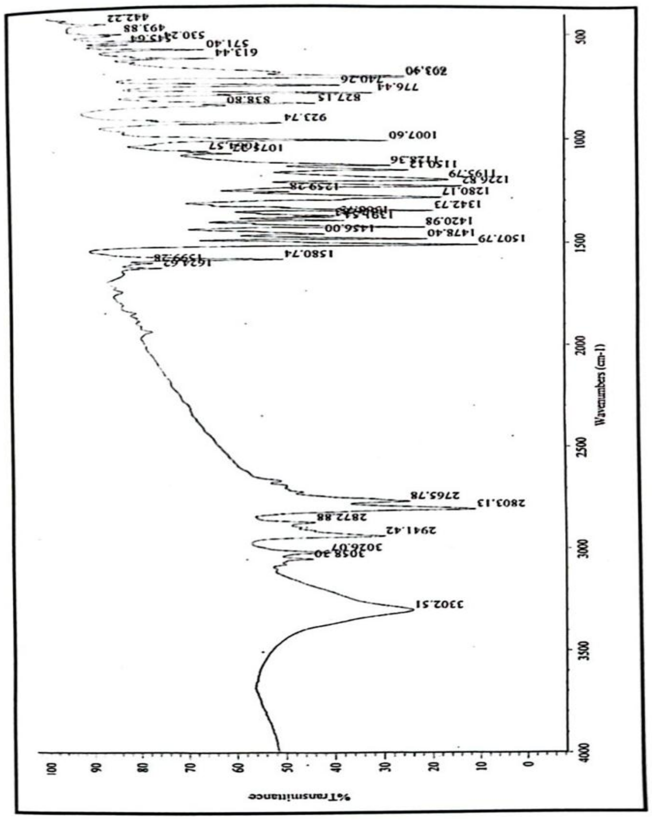


Figure-3: IR Spectrum of $C_{20}H_{10}N_8O_2$. $Cu^{+2}.2H_2O$ Co-ordination Polymer/Polyazochelate.

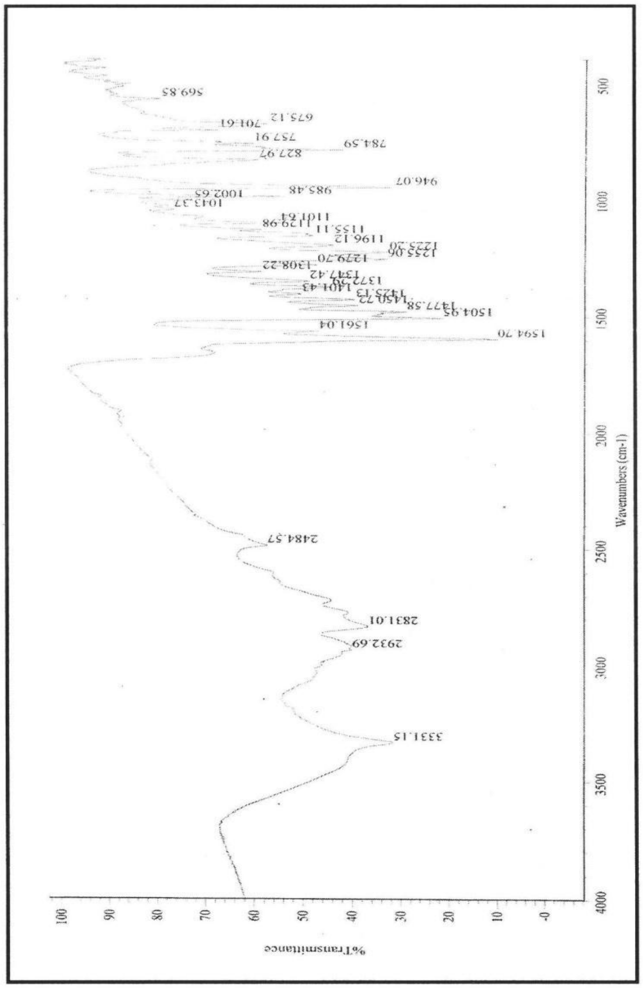


Figure-4: IR Spectrum of $C_{20}H_{10}N_8O_2$. Nl^{+2}.$2H_2O$ Co-ordination Polymer/Polyazochelate.

Magnetic Measurements: Table-2 shows Magnetic moments of metal complexes. Two bands at about 15650 and 23370 cm^{-1} in the electronic spectrum of complex 5 shows in Figure-5 demonstrate that complex 5 is a Cu^{+2} complex. The First band most probably represents $^2B_{1g}\rightarrow{}^2A_{1g}$ transition and the second band is CT. Moreover the distorted octahedral structures with ct [28,29] value is recorded as the first dual band in case of the metal ion of Cu^{+2}. The spectrum of the metal complex of Nl^{+2} shows two well separated bands at 22500 and 15290 cm^{-1} are designated as $^3A_{2g}(F)\rightarrow{}^3T_{1g}(F)$ and $^3A_{2g}(F)\rightarrow{}^3T_{1g}$ (F) transition, respectively indicating the octahedral environment for Ni^{+2}ions. Observed μ_{eff} range 3.0-3.20 BM for above moiety.

The Co^{+2} metal complex exhibit three absorption bands at 24660, 19990 & 8792 cm^{-1} (Figure-6) which were allocated as $^4T_{1g}$ (F) → $^6T_{2g}$ (V1), $^4T_{1g}$ (F) → $^4A_{2g}$ (V2) and $^4T_{1g}$ (F) → $^4T_{1g}$ (P)(V3) transitions respectively. Here V2, V3 transitions are considered. These absorption bands and the value of μ_{eff} confirm octahedral configuration of metal complex of Co^{+2} [30,31]. The derived values of observed μ in the range of 4.4 – 5.

There were two bands in the spectrum of Mn^{+2} polymeric complex around 18650 cm^{-1} and 24040 cm^{-1}. The former bands could be assigned to $^6A_{1g}\rightarrow{}^4T_{2g}$ (4G) and $^6A_{1g}\rightarrow{}^6A_{1g}$ (4Eg) and $^6A_{1g}\rightarrow{}^4A_{1g}$ (4G) transitions. For this compound absorption band have give two band: the first band at wave number 18650 cm^{-1} correspond $^6A_{1g}\rightarrow{}^4T_{1g}$ (4G) and second band at wave number 24040 cm^{-1} correspond of $^6A_{1g}\rightarrow{}^4T_{2g}$ (4G). On analyzing these data, it can be interpreted as the octahedrally coordinated structure of the compound kept in an Mn^{+2} environment.

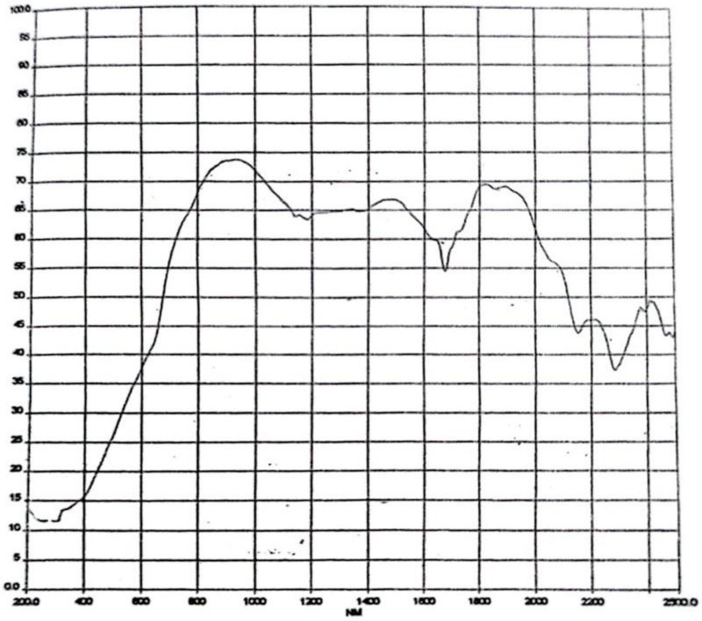


Figure-5: Reflectance spectrum of $C_{20}H_{10}N_8O_2$.Cu^{+2}.$2H_2O$ Co-ordination Polymer.

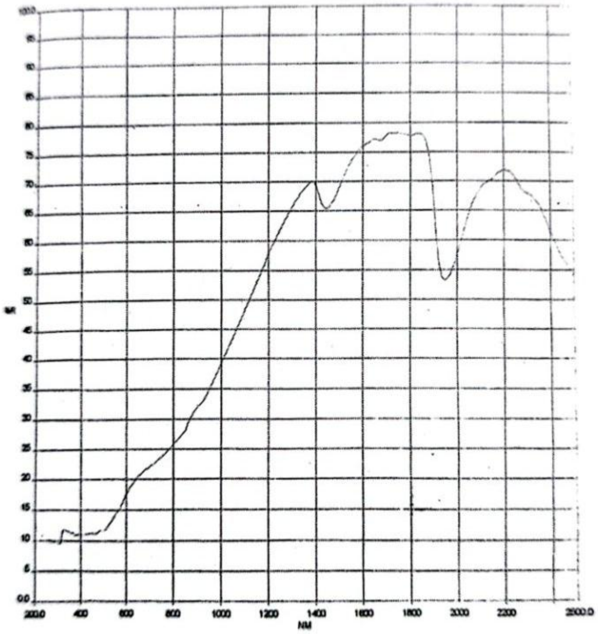


Figure-6: Reflectance spectrum of $C_{20}H_{10}N_8O_2$.Co^{+2}.$2H_2O$ Co-ordination Polymer.

Thermal Studies: The TGA data for Co-ordination polymers sample at different temperatures shows that even after 300^0C. The degradation of the co-ordination polymers is very much healthy. At a temperature ranging between $350\text{-}400^0$C, the degradation rate reaches a maximum. Metal oxides, which are produced "in situ", can account for this acceleration. For me each of the polymers lost about 80% of its mass when heated to 450^oC. All coordinated polymer samples showed considerable weight loss in the region of 200-to 350^oC, as confirmed by thermograms Figure-6. This is perhaps because of the existence of a bound water molecule. Nikolaev[32] has also suggested.

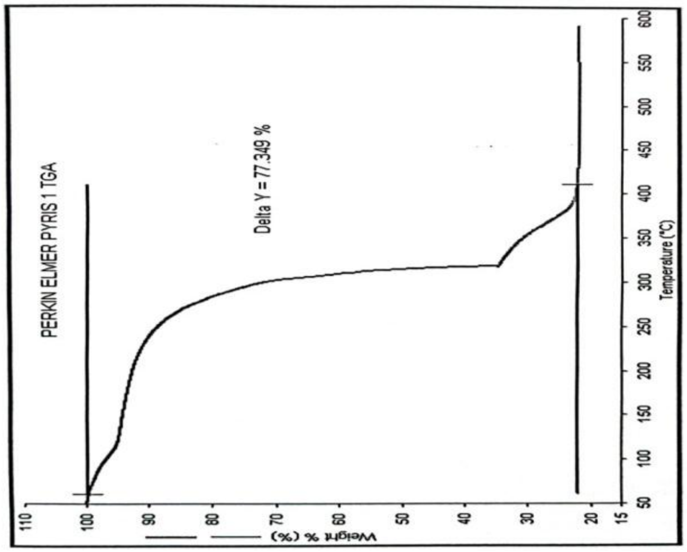


Figure-7: TGA Thermogram of Azopolychelate–($C_{20}H_{10}N_8O_2.Cu^{+2}.2H_2O$) (Co-ordination Polymer).

Table-4: TGA data for Az-ligand containg Co-ordination Polymers.

Co-ordination Polymers (Metal Chelates)	%Wt loss at temperature			
	250	300	350	400
Az–Cu^{+2} $C_{20}H_{10}N_8O_2.Cu^{+2}.2H_2O$	6.2	7.8	16	70
Az–Ni^{+2} $C_{20}H_{10}N_8O_2.Nl^{+2}.2H_2O$	7.5	24.8	66	70
Az–Co^{+2} $C_{20}H_{10}N_8O_2.Co^{+2}.2H_2O$	2.8	5.3	24	67
Az–Mn^{+2} $C_{20}H_{10}N_8O_2.Mn^{+2}.2H_2O$	6.6	14.8	24	68
Az–Zn^{+2} $C_{20}H_{10}N_8O_2.Zn^{+2}.2H_2O$	7.0	20.0	60	75

Antibacterial Activities: Table-5 shows Antibacterial data (calculated from) Table-6 shows Antifungal data (calculated from Antimicrobial). The augmentation in the antimicrobial activity can be elucidated from the principle of Overtone[33] and chelation theory of Tweedy[34].

The anti-lipidic assays more frequently quoted have not directly investigated the properties of the test compounds but made indirect judgment on the potency of test compounds in the penetration of inducing of clearing a photographic emulsion capable of reversible reacting with the compounds, claiming an apparent correlation to compound activity in some of this case but failing in others. This decreases the polarity of the metal ion D symbonding orbital and charge sharing is only partial, and arises because not all of the positive charge is lost on donation to the donor groups. It also enhances the delocalisation of of π-electrons over the entire chelate ring and thereby imparts lipophilicity to the complexes. This increased lipophilicty of the host makes the Coordination polymer able to penetrate into the lipid membranes enhanced, and finally coordinate the metal binding sites in the enzymes of the microorganisms. They also poison the cells own respiration process, which further reduces and ultimately blocks protein synthesis (necessary for their rapid growth).

With reference to Table-5 and Table-6, the metal complexes displayed marked biocidal effect as compared to free ligands, which shows a sharp increase in activity of the coordination polymer expresses the marked higher biocidal activity taken the noted data confirmed among that complexes of the metal respective metal Cu^{+2} was proved to be most active. Reported that the Cu^{+2} complex have much toxicity. In hindsight it is not surprising, since the copper salts are commonly used as a fungicide. The Co-ordination polymers have shown more antimicrobial activity as compared to free ligand, due to chelation of metal ions.

Table-5: Antibacterial activity of coordination polymers.

Compound	Zone of inhibition at 1000 ppm (%)				
	Gram+ve		Gram–ve		
	Bacillus Subtilis	Staphylococcus Aureus	Klebsiella Promioe	Samonella Typhi	E. Coil
Az-Ligand	08	11	08	07	06
$C_{20}H_{10}N_8O_2$. Cu^{+2}. $2H_2O$	26	25	24	27	23
$C_{20}H_{10}N_8O_2$. Nl^{+2}. $2H_2O$	19	24	20	22	21
$C_{20}H_{10}N_8O_2$. Co^{+2}. $2H_2O$	22	19	17	18	18
$C_{20}H_{10}N_8O_2$. Mn^{+2}. $2H_2O$	20	23	22	25	20
$C_{20}H_{10}N_8O_2$. $Zn^{+2}.2H_2O$	18	20	15	17	17

Table-6: Antifungal Activity of Coordination polymers.

Zone of Inhibition at 1000 ppm(%)					
Compounds	Penicillium Expansum	Botrydepladia thibromine	Nigrospara Sp.	Tichothesium Sp.	Rhizopus nigricum
Az-Ligand	11	08	10	06	09
$C_{20}H_{10}N_8O_2$. $Cu^{+2}.2H_2O$	26	27	24	23	24
$C_{20}H_{10}N_8O_2$. $Nl^{+2}.2H_2O$	21	21	19	21	20
$C_{20}H_{10}N_8O_2$. $Co^{+2}.2H_2O$	22	18	18	18	22
$C_{20}H_{10}N_8O_2$. $Mn^{+2}.2H_2O$	16	20	22	20	19
$C_{20}H_{10}N_8O_2$. $Zn^{+2}.2H_2O$	12	22	20	16	18

Conclusions

The results of present investigation make the following conclusions. The design & synthesis of azo-ligand has been successfully shown by IR & Spectral Studies, in this report we summerise the properties of some novel coordination polymers of azo-ligand derivatives with transition metal cations, which have been synthesized in lab. The bioassay of all the synthesized compounds was carried out. The complexe sex habited bioactivity against Gram-negative and Gram-positive bacteria and plant pathogenic microorganisms, Further, the ligand complexes were found to be more active than the free ligands and were able to kill some bacterial strams providing new metal-based bactericidal agents. Electronic and magnetic moment values of the complexes in turn were used to glean information about the geometry of the more complexes. Their magnetic moment values suggested an octahedral geometry. The stability of ligand and its metal complexes (true form of ions) is good. As a result of the insertion of metal ions, the ligand and its metal complexes also show good microbicidal activity, however, this is greater than that observed with the ligand.

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