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Synthesis of Some Polymer-Metal Complexes and Elucidation of their Structures

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In this study, the nine coordination polymers of Fe(III), Co(II) and Ni(II) salts have been synthesized using polyacrylamide (PAA), polt(ethylene glycol) (PEG) and poly(vinyl alcohol) (PVA) and their structures were characterized by magnetic and conductivity measurements, ultraviolet-visible (UV-VIS), FTIR spectroscopy and thermogravimetric analysis (TGA). The structures of Fe(III) complexes in the all coordination polymers were found as tetrahedral. The structures of PAA-Co(II) coordination polymer was determined as octahedral geometry whereas PEG-Co(II) and PVA-Co(II) complexes showed as tetrahedral structure. PAA-Ni(II) and PEG-Ni(II) complexes have octahedral geometry, whereas PVA-Ni(II) has a square planar structure. Besides, the stress-strain experiments of PVA-metal coordination polymers obtained rubber-like structure were carried out and the value of breaking-strain of PVA-Ni(II) complex was found to be about 17% of vulcanized natural rubber. The conductivities of the resulting polymer-metal complexes were measured by four-probe technique and were found in the range $10^{-5} - 10^{-6}\,Scm^{-1}$. Thus, it was suggested that they can be used in the field potential application of conducting polymers. TGA results revealed that among the complexes PEG-Fe(III) and PVA-Fe(III) complexes have the highest thermally stable.

Keywords conductivity, complexes, coordination polymer, polyacrylamide, poly(ethlylene glycol), poly(vinyl alcohol)

Introduction

Polymer-metal complexes have been attracting interest in many scientific and technological fields in recent years. Coordination polymers have found wideapplications in bioinorganic industry (1, 2), wastewater treatment (2), pollution control (3), hydrometallurgy (4), preconcentration (5), anionic polyelectrolyte hydrogels (6), cation-exchange resins (7), etc. The first study on the polymers including metal has been carried out by Arimoto and Haven in 1955 (8). They have been synthesized vinyl ferrocene by free radical polymerization. Recently, it has been reported that several studies and characterizations on polymer-metal complexes. Mathew et al. have prepared a series of mixture of

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polyacrylamide and dithiocarbamade and they have synthesized its complexes using Cu(II), Ni(II), Zn(II), Co(II) and Hg(II) (9). They have investigated thermal properties of the synthesized polymer-metal complexes. Moreover, it was studied catalytic effect of divinylbenzene crosslinked polyacrylamide-Cu(II) coordination complexes (10). Diab and El-Sonbati have investigated thermal properties of the polymer-metal complexes synthesized from poly (ethylene glycol) with Co(II), Ni(II), Cu(II) and Cd(II) (11). Yasuyoshi et al. (12) investigated that structural of Fe(III) and Cu(II) complex with poly(vinyl alcohol) by means of NMR techniques. Coordination polymers of poly (ethylene glycol) with Cd(II) salt have also synthesized and their structures characterized by X-Ray chromatography (13). An et al. (14) showed that the complex formation of Cr(III), Fe(III), Co(II), Ni(II), Cu(II) and Zn(II) ions with partially phosphorylated poly (vinyl alcohol) in aqueous solution is studied by means of potentiometric titration.

To the best our knowledge, there are no reports in the literature on the details, elucidation of structures, and unit form of polymer-metal complexes. In this present study, the synthesis and characterization and thermal properties of the nine coordination polymers containing donor atoms (i.e., some transition metals) are reported.

Experimental

Materials

All the reagents were purchased from Aldrich and Merck with Analytical Grade. PVA Aldrich M_w: 89 000–98 000, PEG M_w: 8 000 $d = 1.03\,g\,mL^{-1}$; PAA 50% wt solution in water M_w: 10 000. The chemicals were used as received.

Measurements

The conductivities of the coordination polymers were measured at room temperature using a four-probe technique on pressed pellets. Fourier transforms infrared (FTIR) spectra the samples were recorded on a Mattson-1000 spectrophotometer using the KBr disc technique. Thermogravimetric analysis (TGA) was performed on a DuPont 9900 Model (Thermal Analyzer) in the temperature range of 0–900°C under nitrogen atmosphere. Magnetic susceptibility measurements of the samples were performed using a Sherwood Scientific Model MKI Gouy Scale, as described previously (15). The elemental analysis (C, H, N) for polymer-metal complexes were carried out using LECO-CHNS-9320 Model elemental analyzer. The metal contents of the polymer-metal complexes were determined using a Philips Pu 9285 Model atomic absorption spectrophotometer. The UV-VIS absorption spectra were measured using Unicam UV-2 model ultraviolet-visible absorption (UV-VIS) spectrophotometer and Epson FX-870 model printer. Stress–strain measurements of the PVA-metal complexes were performed by Shimadzu AG-I 5 kN model tensile tester.

Preparation of the Coordination Polymers: General Method

A solution of the PAA, PEG (4×10^{-4} mole) and PVA (2×10^{-4} mole) are dissolved in H_2O (50 mL), CH_3OH (20 mL) and H_2O (20 mL), respectively. The solution was stirred magnetically and heated at 85°C. 1.0×10^{-3} mole of metal (II) chloride dissolved in water (10 mL) was added dropwise to the solution of the polymer, it was then stirred and heated again at 85°C for an hour. The complexes were precipitated from solution using acetone and filtered, washed with acetone, and then dried in a vacuum oven. Physical characterization, microanalytical and magnetic susceptibility data of the complexes are given in Table 1.

Major IR spectra and electronic spectral data (nm) of (PAA-Me): for [PAA-Fe]; υ $(NH_2)_{(sym),(asym)}/\upsilon$ $(CH)_{(sym),(asym)}$ 3387, 3180/2926, 2857, υ $(C{=}O)_{(PAA)}/\upsilon$ $(C{=}O)_{(PAA\text{-}Fe)}$ 1662/1606, δ (NH)/υ (CN) + δ (NH) 1247/1523, υ (Fe-O)/υ (Fe-N) 658/492, λ_{max} (nm) 190, 542; for [PAA-Co]; υ $(NH_2)_{(sym),(asym)}/\upsilon$ $(CH)_{(sym),(asym)}$ 3397, 3180 / 2928, 2858, υ $(C{=}O)_{(PAA)}$ / υ $(C{=}O)_{(PAA\text{-}Co)}$ 1660/1607, δ (NH)/υ (CN) + δ (NH) 1245/1521, υ (Co–O)/υ (Co–N) 658/492 λ_{max} (nm) 190, 480; [PAA-Ni]; υ $(NH_2)_{(sym),(asym)}/\upsilon$ $(CH)_{(sym),(asym)}$ 3395, 3172/2529, 2856, υ $(C{=}O)_{(PAA)}/\upsilon$ $(C{=}O)_{(PAA-Ni)}$ 1651/1606, δ (NH)/υ (CN) + δ (NH),1127/1520, υ (Ni–O)/υ (Ni-N) 627/502, λ_{max} (nm) 190, 430, 624; Major IR spectra and electronic spectral data (nm) of (PEG–Me): for [PEG–Fe]; υ $(C{-}O{-}C)_{(sym,asym)}/\upsilon$ (CO) 1108, 1053/485, λ_m(nm) 211, 548, for [PEG–Co]; υ $(C{-}O{-}C)_{(sym,asym)}/\upsilon$ (CO) 1109, 1052/487, λ_{max} (nm) 210, 650, for [PEG–Ni]; υ $(C{-}O{-}C)_{(sym,asym)}/\upsilon$ (CO) 1108, 1052/482, λ_{max} (nm) 211, 395, 716.

Results and Discussion

The analytical data of the complexes correspond well with the main unit. Derivation from the theoretical values of polymer include metal may be due to the polymeric nature of the polymers. It is very possible that the polydispersity of the polymers are not in a narrow range. Similarly, we can say stoichiometry of polymer-metal complexes may not be uniform.

All polymer-metal complexes are soluble in water except poly(vinyl alcohol)-metal complexes are dissolved in nothing. Elemental analysis showed the best agreement with the theoretical values when we consider the ratio polymer and metal as 32 : 1, 44 : 1 and 126 : 1 for PAA-Me(II), PEG-Me(II) and PVA-Me(II), respectively.

FTIR Spectra

PVA-metal (II, III) complexes were not prepared in the form of tablets and made into pellets due to springy. The assigned absorption bands are consistent with suggested structure. For PAA-Me complexes; bands appearing at 1520, 1246 and 1227 cm^{-1} are assigned to $\upsilon_{(CN)} + \delta_{(NH)}$, $\delta_{(NH)}$ and $\upsilon_{(C-C-N-M)}$ due to out-of-plane deformation that these bands may be due to the out of phase combination of O–C–N and NH vibration mode (16, 17) (Figure 1).

For PEG-Me complexes, new bands showed that at 485 cm^{-1}, 1108 cm^{-1} and 1050 cm^{-1} are assigned to $\upsilon_{C-O,}$ $\upsilon_{C-OC(s)}$ and $\upsilon_{C-O-C\ (as)}$, respectively. Furthermore, the new bands observed in the region of 621–658 cm^{-1} and 492–502 cm^{-1} in the far IR. These bands that is characteristic of $\upsilon_{(M-O)}$ and $\upsilon_{(M-N),}$ (18) on this basis, we can say that studied metal (II, III) ions are bound to polymer through one nitrogen with one carbonyl oxygen and glycol oxygen for PAA-metal (II) and PEG-metal (II), respectively.

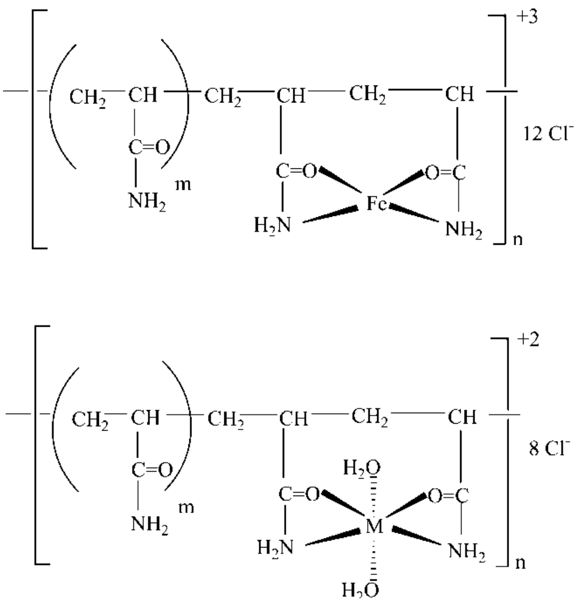
Electronic Absorption Spectra and Magnetic Susceptibility

The [PAA-Co], [PAA-Ni] and [PEG-Ni] complexes give magnetic moment values of 4.24, 3.15 and 3.21 BM, respectively, which are consistent with octahedral geometry

Table 1
Analytical and magnetic susceptibility data of the complexes

Compound		Found (calcd) %					Color and ${}^{a}\mu_{eff}$, (BM)
Abbreviation (M_w)	Suggestion structure	C	H	N	Cl	M	
[PAA-Fe]	$[FeC_{102}H_{170}O_{34}N_{34}]_4^{+3} \cdot 12Cl^-$	48.91	6.51	18.30	3.64	1.88	Yellow
10 305		(47.51)	(6.59)	(18.47)	(4.13)	(2.17)	2.29
[PAA-Co]	$[CoC_{102}H_{174}O_{36}N_{34}]_4^{+2} \cdot 8Cl^-$	48.71	6.47	18.77	2.02	1.67	Blue
10 320		(47.44)	(6.74)	(18.45)	(2.75)	(2.28)	4.24
[PAA-Ni]	$[NiC_{102}H_{174}O_{36}N_{34}]_4^{+2} \cdot 8Cl^-$	48.54	6.01	18.23	2.34	1.87	Green
10 320		(47.43)	(6.74)	(18.45)	(2.75)	(2.28)	3.10
[PEG-Fe]	$[FeC_{92}H_{184}O_{46}Cl_2]_4^{+} \cdot 4Cl^-$	53.72	8.29	—	1.32	1.98	Yellow
8 058		(54.80)	(9.13)	—	(1.76)	(2.78)	2.68
[PEG-Co]	$[CoC_{92}H_{184}O_{46}Cl_2]_4$	55.07	8.95	—	—	2.38	Blue
7 924		(55.73)	(9.29)	—	—	(2.93)	4.80
[PEG-Ni]	$[NiC_{92}H_{188}O_{48}Cl_2]_4$	50.93	7.25	—	—	2.08	Green
8 068		(54.73)	(9.12)	—	—	(2.88)	3.20
[PVA-Fe]	$[FeC_{1016}H_{2030}O_{508}]_4^{+} \cdot 4Cl^-$	49.71	8.25	—	(0.11)	(0.17)	Yellow
89 766		(54.32)	(9.05)	—	(0.16)	(0.50)	2.05
[PVA-Co]	$[CoC_{1016}H_{2030}O_{508}]_4$	52.93	8.25	—	—	(0.21)	blue
89 632		(54.40)	(9.05)	—	—	(0.26)	4.42
[PVA-Ni]	$[NiC_{1016}H_{2030}O_{508}]_4$	53.21	9.52	—	—	(0.18)	Green
89632		(54.40)	(9.05)	—	—	(0.26)	Diamagnetic

${}^{a}\mu_{eff}$: Effective magnetic moment.
BM: Bohr magneton.

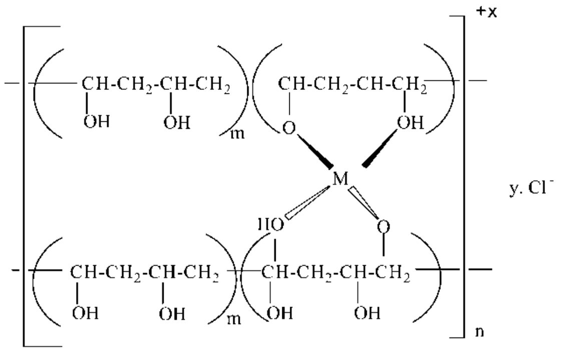


M: Co(II) and Ni(II)
m: 32, n: 4

Figure 1. Suggested structure of PAA-metal complexes.

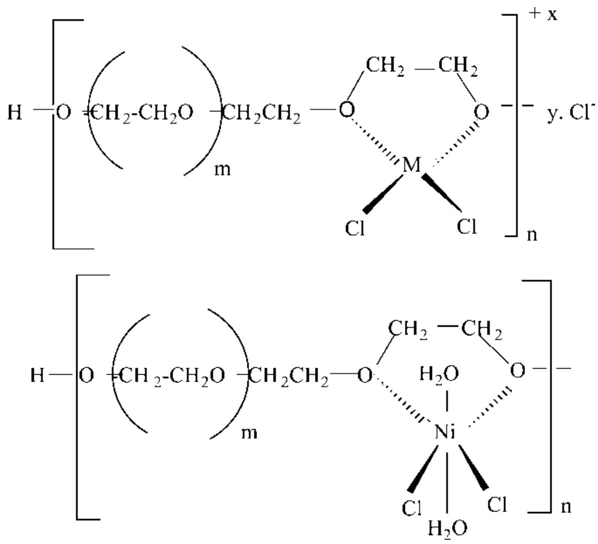
(Scheme 1) (19). [PAA-Fe], [PVA-Co], [PVA-Fe], [PEG-Co] and [PEG-Fe] complexes give magnetic moment values of 2.29, 4.42, 2.05, 4.80 and 2.68 BM, respectively, which are typical for tetrahedral Me(II) chelates (Figure 2).

[PVA-Ni] complex is diamagnetic as aspect for the square-planer structures (Figure 3). Important bands of the PAA-Me and PEG-Me are observed at 191 nm and 210 nm. It may be attributed to σ-σ^* type transitions (19). Electronic bands were not recorded due to no dissolved for PVA-Me complexes. The spectra of the PAA-Fe(III)



M: Fe(III), Co(II), Ni(II) m: 126, n: 4
x : 1 , - , -
y : 4 , - , -

Figure 2. Suggested structure of PVA-metal complexes.



M: Co(II), Fe(II); m: 44, n: 4
x: - , 1
y: - , 1

Figure 3. Suggested structure of PEG-metal complexes.

and PEG-Fe(III) complexes shows bands in the range of 541–548 nm. Hence, tetrahedral geometry may be assigned to these complexes (20).

Electronic spectrum of PAA-Co(II) complex shows a medium intense band at 480 nm. The position band together with its magnetic moment value (4.24 BM) indicates that this complex has an octahedral stereochemistry (20). More intense band in the d-d electronic spectra and high magnetic moment values support a tetrahedral configuration for the PEG-Co(II) complex. All of the nickel(II) complexes show two d-d bands between 624–716 nm and 395–430 nm indicating an octahedral structure.

Thermogravimetric (TGA) Analysis

Thermal data of the polymer-metal complexes are given in Table 2. As seen from Table 2, decomposition temperatures (T_i, T_m, T_f) of the polymer-metal complexes are different from each other. The PAA-metal and PVA-metal complexes decompose with three steps, whereas the PEG-metal complexes decompose with one step. The I-VI complexes are thermally stable up to 60, 53, 71, 62, 45 and 43°C, respectively. In the decomposition process of the I-VI complexes, the mass losses corresponded to absorption H_2O or CH_3OH leaving in the first stages of the decomposition. The second and third steps occur within the temperature range 185–393°C. The second step corresponds to polymer-metal decomposition and third step leads to the decomposition of polymer-metal to metal salts.

PEG-metal complexes are thermally decomposed in a step. According to initial decomposition temperature (T_i), PEG-Fe(III) has the highest thermally stability with 304°C. This decomposition occurs within the temperature range, 290–452°C. All of the synthesized polymer-metal complexes exhibit adequate thermal stability.

Table 2
Thermal analysis data for studied polymer-metal complexes

Compound no	[a]Thermally decomposed			Weight loss (%)	Residue at 900°C (wt%)	Conductivity × 10^5 (σ, Scm^{-1})
	T_i	T_{max}	T_f			
PAA-Fe(III) (I)	60	91	112	6	24	0.36
	247	280	320	20		
	391	372	466	51		
PAA-Co(II) (II)	53	87	110	22	19	0.39
	231	260	290	12		
	374	380	489	47		
PAA-Ni(II) (III)	71	87	113	18	17	1.74
	185	214	252	19		
	360	440	490	46		
PVA-Fe(III) (IV)	62	87	125	4	22	1.58
	251	291	328	31		
	376	421	495	43		
PVA-Co(II) (V)	45	82	104	3	28	0.85
	289	303	330	34		
	390	434	517	35		
PVA-Ni(II) (VI)	43	75	103	3	10	0.85
	278	304	330	52		
	393	430	460	35		
PEG-Fe(III) (VII)	304	356	430	83	17	1.26
PEG-Co(II) (VIII)	295	362	452	88	12	2.92
PEG-Ni(II) (IX)	290	353	420	86	14	3.12

T_i: Inital degradation temperature.
T_{max}: Maximum degradation temperature.
T_f: Finally degradation temperature.

Conductivity Measurements

The results of the measurements are summarized in Table 2. As seen in the Table, PEG-Ni(II) has the highest conductivity with value of 3.13 Scm^{-1}, whereas PAA-Fe(III) has the lowest conductivity with value of 0.36 Scm^{-1}. Polymer-metal complexes are found to be semi-conducting, although polymers do not conduct. As can be seen in Table 2, the conductivities of the PEG-metal complexes, which have coordinated with two oxygen atoms in the structure, are higher than the other. This fact can be attributed to delocalization of LUMO electrons in the oxygen molecule showing inductive effect. Oxygen molecule have sp^3, sp^3 and sp^2 hybrid for PVA-metal, PEG-metal and PAA-metal complexes, respectively. Electron donation due to the hybridized type increases in the order:

Table 3

Compound	Breaking-strain (%)
PVA-Fe(III)	51.7
PVA-Co(II)	53.4
PVA-Ni(II)	83.3
[a]Polyizopren	550
[a]Poly(butadien-co-izopren)	400
[a]Poly(izobuten-co-izopren)	650
[a]Polychloropren	500

Breaking-strain results of PVA-metal complexes.
[a]Literature values(23).

PAA-Me(II/III) < PVA-Me(II/III) < PEG-Me(II/III). This is an expected result because sp^2-hybrid has more electron donating property than sp^3-hybrid; hence, conjugation increases chelation ability in the polymer-metal complexes.

Increasing conjugation in polymer-metal complexes may be attributed to high conductivity (21). Moreover, metals improve interchain charge transfer, e.g, high molecular weight complexes show high conductivities due to intermolecular charge transfer (22).

Stress-Strain of PVA-Metal Complexes

Amazingly, among the polymer-metal complexes, PVA-metal complexes exhibit a rubber-like structure. For that reason, stress-strain experiments of the PVA-metal complexes were performed.

Table 3 shows that among the PVA-metal complexes, PVA-Ni(II) complex had the highest breaking-strain with a value of 83%. Besides, as seen from Table 4, the breaking-strain of PVA-Ni(II) complex demonstrated about 17% of vulcanized natural rubber (23).

The stress-strain may be attributed to metal complexes of PVA due −OH group. The electron-donating effect of the −OH group is higher than oxygen atom or $-NH_2$ group in the studied polymers. The polarity of $-OH^-$ group may be reduced to a great delocalization due to electron donating (24). In this delocalization, the strong intramolecular hydrogen bond may be reveals (Figures 2, 4) (25).

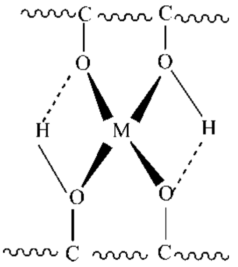


Figure 4. Hydrogen bond in the Suggested Structure of PEG-metal complexes.

Conclusions

In summary, polymer-metal complexes were synthesized by using the template method. Chemical structures were investigated and solid state conductivities were in comparison. While studied polymers have not conducted, their metal complexes have conductivity. This may be attributed to delocalization of *d*-electrons in the metal which has a hybrid orbital. The conjugation length in complexes may lead to higher conductivity. So this study may be useful for various applications.

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