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New biocidal metal complexes of bisphenol-A formaldehyde polymer containing piperazine

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The polymeric ligand (BFP) was synthesized by condensation of bisphenol-A, formaldehyde, and piperazine in alkaline medium at 70–80°C. The polymer–metal complexes were synthesized by the reaction of BFP with Mn(II), Co(II), Ni(II), Cu(II), and Zn(II) acetates in 1:0.5 (ligand:metal) molar ratio. All the synthesized polymers were characterized by elemental, spectral (infrared, ¹H-NMR, and UV-Vis), magnetic moment measurements, and thermal (TGA) analysis. The ligand-field and nephelauxetic parameters have been determined from UV-Vis spectra using ligand-field theory. Elemental analyses indicate the association of water with metal for Mn(II), Co(II), and Ni(II), which is also supported by TGA. The antimicrobial activities of the synthesized polymers were studied by agar well diffusion methods against *Bacillus subtilis*, *Bacillus megaterium*, *Staphylococcus aureus*, *Escherichia coli*, *Salmonella typhi*, *Pseudomonas aeruginosa*, and *Shigella boydii*. The antimicrobial activity and thermal stability of Cu(II)–polymer were higher than the other polymer–metal complexes due to the higher stability constant of Cu(II).

Keywords: Piperazine; Condensation; Formaldehyde; Polymer–metal complexes; Antimicrobial activity; Higher stability constant

1. Introduction

The polymeric materials are ubiquitous because of their tremendous processing advantages over ceramics and metals. Incorporation of transition metals into a polymer main chain offers potential for the preparation of processible materials with properties which differ significantly from those of conventional organic polymers [1, 2]. The diverse range of coordination numbers and geometries of transition elements offer the possibility of accessing polymers with unusual conformational, mechanical, and morphological characteristics. Polymer–metal complexes might also be expected to function as convenient and processible thermal or photochemical precursors to metal-containing ceramic films, fibers, and coatings with desirable features and useful physical properties [3]. Polymer–metal complexes have widespread applications [4–7]

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in nuclear chemistry, preconcentration, recovery of trace metal ions, pollution control, hydrometallurgy, polymer drug grafts, and wastewater treatments. In addition, they are also used as mechano-chemical systems [8] and models for bioinorganic systems [9]. The development of new polymer–metal complexes is of interest owing to their potential for high thermal stability, good electrical conductivity, and catalytic activity, especially for the chemical reactions occurring in biological materials [10–12]. The incorporation of metal ion into the polymer chain could also lead to new electrical and optical characteristics, and to improved mechanical properties and heat stability. The potential applications of chelating polymer are, for example, surface coatings on metals and glasses, adhesives, high-temperature lubricants, electrical insulators, and semiconductors [13].

Several *in vivo* studies have indicated [14–17] that metal complexes possess interesting toxicological and pharmacological properties. However, a significant problem is that some lose their activity upon exposure to proteins and appear to have better affinities than the ligands/compounds for metal ions, which are deactivated when embedded in proteins.

We have previously investigated [18–21] the coordination chemistry of some antibiotics and/or antimicrobial compounds with transition metals to examine the modes of binding and to study their effect on biological activity in continuation of our ongoing research. We report herein the preparation, characterization, and antimicrobial activities of bisphenol-A formaldehyde containing piperazine (BFP) and its polymer–metal complexes to investigate the possible use of polymeric ligand and its polymer–metal complexes for the development of thermally stable antimicrobial materials. These compounds are characterized by infrared (IR) spectra, $^1\text{H-NMR}$, $^{13}\text{C-NMR}$, and electronic spectroscopy. Thermal stability of these compounds was also determined by thermogravimetric analysis (TGA). UV-Vis and magnetic moment measurements support the proposed geometry of the metal ions. In addition, the antimicrobial activities against several bacteria and fungi were examined using the agar well diffusion method. The polymeric ligand had antimicrobial and thermal properties, which were enhanced after chelation.

2. Experimental

2.1. Materials and bacterial strains

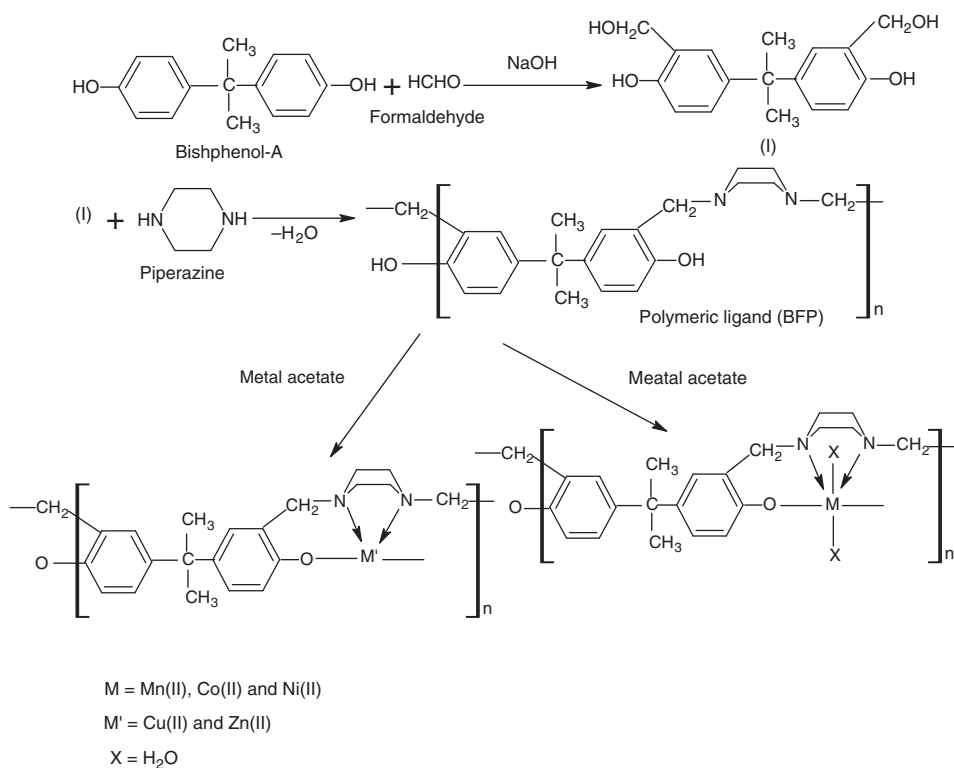
Bisphenol-A, piperazine, formaldehyde (37% aqueous solution), sodium hydroxide, manganese(II) acetate tetrahydrate, $\text{Mn}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$, cobalt(II) acetate tetrahydrate, $\text{Co}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ nickel(II) acetate tetrahydrate, $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$, copper(II) acetate monohydrate, $\text{Cu}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$, and zinc(II) acetate dihydrate, $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ (S.D. Fine Chemicals, India) were used without purification. Dimethylformamide (DMF), dimethylsulfoxide (DMSO), ethyl alcohol, methanol, and acetone (Qualigens, India) were purified by standard procedures before use. Microorganisms *Bacillus subtilis*, *Bacillus megaterium*, *Staphylococcus aureus*, *Escherichia coli*, *Salmonella typhi*, *Pseudomonas aeruginosa*, and *Shigella boydii* were collected by the Culture Collection Center, Microbiology Laboratory, School of Life Science, Jawaharlal Nehru University, New Delhi.

2.2. Synthesis of polymeric ligand (BFP)

The polymeric ligand was synthesized by condensation of bisphenol-A, formaldehyde, and piperazine in alkaline medium (scheme 1). In a 250 mL three-necked round-bottom flask, 2.28 g (0.01 mol) of bisphenol-A and 1.5 mL (0.02 mol) of formaldehyde (37% aqueous solution) were combined and the pH was adjusted to 8 with sodium hydroxide. The reaction mixture was stirred magnetically and the temperature raised to 70°C and maintained for 4 h. Then 0.86 g (0.01 mol) of piperazine was added in the reaction mixture and stirred again for 1 h at 70–80°C. The reaction was monitored by thin layer chromatography (TLC) using ethanol as eluent. The reaction mixture was cooled, precipitated into deionized water, and filtered. A light-yellow solid product was obtained, which was dried in a vacuum desiccator on calcium chloride to remove trapped solvents and water to give (BFP) polymeric ligand in 72% yield.

2.3. Synthesis of polymer-metal complexes

The polymeric ligand (3.23 g, 0.01 mol) was dissolved in DMF (25 mL). The manganese(II) acetate tetrahydrate (1.23 g, 0.005 mol) was also dissolved in DMF (15 mL). The hot and clear solution of manganese(II) acetate tetrahydrate was added with constant stirring to the hot and clear solution of the ligand and refluxed for 2 h.



Scheme 1. Synthetic route for the preparation of polymeric ligand (BFP) and its polymer metal complexes.

Table 1. Elemental analysis of polymeric ligand (BFP) and metal complexes.

Compounds	Yield (%)	Elemental analysis, calculated (observed)			
		%C	%H	%N	%M
C ₂₁ H ₂₅ N ₂ O	72	79.57 (78.94)	9.92 (9.35)	9.35 (8.87)	– –
C ₂₁ H ₂₈ N ₂ O ₃ –Mn(II)	80	66.60 (65.81)	8.05 (7.85)	8.00 (7.74)	8.77 (8.10)
C ₂₁ H ₂₈ N ₂ O ₃ –Co(II)	82	65.12 (64.87)	8.09 (7.77)	8.95 (8.21)	9.13 (8.78)
C ₂₁ H ₂₈ N ₂ O ₃ –Ni(II)	85	65.72 (65.49)	8.56 (7.80)	8.17 (7.90)	8.49 (7.83)
C ₂₁ H ₂₄ N ₂ O–Cu(II)	70	72.10 (71.89)	6.50 (6.02)	8.73 (7.92)	9.44 (9.08)
C ₂₁ H ₂₄ N ₂ O–Zn(II)	81	72.80 (72.29)	8.48 (8.34)	6.68 (6.21)	8.80 (8.36)

A reddish-brown product of BFP–Mn(II) was precipitated by pouring it into a large excess of distilled water and then filtered. The solid was washed with distilled water and acetone and dried in a vacuum desiccator on calcium chloride, yield 80%.

The polymer–metal complexes BFP–Co(II), BFP–Ni(II), BFP–Cu(II), and BFP–Zn(II) were synthesized by mixing 1.25 g (0.005 mol), 1.24 g (0.005 mol), 0.995 g (0.005 mol), and 1.095 g (0.005 mol) of cobalt(II) acetate tetrahydrate, nickel(II) acetate tetrahydrate, copper(II) acetate monohydrate, and zinc(II) acetate dehydrate, respectively, with 3.23 g of polymeric ligand (BFP) in 1:2 ratio by similar method as mentioned above. The colored polymer–metal complexes obtained were filtered off, washed, and dried. Their yields are given in table 1.

2.4. Preparation of microbial cultures

The antimicrobial activity of polymeric ligand and its metal complexes was carried out against *B. subtilis*, *B. megaterium*, *St. aureus*, *E. coli*, *Sa. typhi*, *P. aeruginosa*, and *Sh. boydii* by agar well diffusion methods. Bacteria strains were nourished in nutrient broth (Difco) for 24 h. Bacteria were incubated on Mueller–Hinton agar. BFP and its polymer–metal complexes (50 µg mL⁻¹) were dissolved in DMSO. A circular well was made at the center of each petri dish with a sterilized steel borer. Then 0.1 mL of each test solution was added to the well and incubated at 36°C for 24 h. DMSO absorbed disks were used as controls. The resulting inhibition zones on the dish were measured in millimeters and compared with kanamycin as a standard drug for antibacterial activity.

2.5. Characterization

Elemental analysis (C, H, and N) of the polymeric ligand and its polymer–metal complexes was carried out on a Perkin-Elmer model 2400 elemental analyzer (Central Drug Research Institute, Lucknow). The metal percentages of the polymer–metal complexes were determined by complexometric titration against ethylenediaminetetraacetic acid (EDTA) after its decomposition with concentrated nitric acid. FTIR spectra

were recorded over the 4000–400 cm^{-1} ranges on a Perkin-Elmer IR spectrometer model 621 by using KBr pellets. A Perkin-Elmer Lambda EZ-201 was used to measure electronic spectra of the polymer–metal complexes; magnetic susceptibility measurements of the polymer–metal complexes were performed using a Gouy balance, $\text{Hg}[\text{Co}(\text{SCN})_4]$ as calibrant. $^1\text{H-NMR}$ spectra were recorded on a JEOL-GSX 300 MHz FX-1000 FTNMR spectrometer using DMSO as a solvent and tetramethylsilane (TMS) as an internal standard. The TGA was done on a TA analyzer 51 (TA Instrument).

3. Results and discussion

3.1. Composition and chemistry

The synthetic route to the polymeric ligand (BFP) is described in scheme 1, which allowed us to obtain the compounds with high purity and in good yield. Hot solutions of ligand in DMF were used during complexation, where the formations of $[\text{BFP-M}] \cdot 2\text{H}_2\text{O}$ ($\text{M} = \text{Mn}(\text{II}), \text{Co}(\text{II})$ and $\text{Ni}(\text{II})$) and $[\text{BFP-M}']$ ($\text{M}' = \text{Cu}(\text{II})$ and $\text{Zn}(\text{II})$) were rapid and gave good yields. These polymer–metal complexes were prepared by the reaction of the ligand with metal(II) acetates in 2:1 (ligand:metal) molar ratio. The polymer–metal complexes are colored, stable on prolonged exposure in air, readily soluble in DMSO and DMF, but insoluble in other common organic solvents. The elemental analytical data agreed with the proposed formulas of the ligand and polymer complexes. The structures of the ligand and polymer–metal complexes are supported by magnetic susceptibility, and FTIR, $^1\text{H-NMR}$, and elemental analysis. The microanalytical data showed BFP and metal in a 2:1 ratio in all polymer–metal complexes.

3.2. FTIR spectra

IR frequencies of all the synthesized polymeric compounds are summarized in table 2. BFP exhibited a band at 3460 cm^{-1} due to phenolic OH [22]. The presence of methylene was confirmed by strong bands at 2880 and 2740 cm^{-1} due to $\nu\text{C-H}$ asymmetric and symmetric stretching. In BFP a sharp band at 1460–1415 cm^{-1} due to δCH_2 bending and peaks between 1599 and 1503 cm^{-1} are attributed to aromatic $\text{C}=\text{C}$ stretching. All the compounds show absorptions between 1290 and 1010 cm^{-1} due to aliphatic C-N . The C-H out-of-plane bending vibrations of the aromatic ring were at 760–650 cm^{-1} [23] and the polymeric compounds show characteristic bands at 3060–3055 cm^{-1} due to aromatic $\nu\text{C-H}$. In polymer–metal complexes the phenolic–OH stretch disappears, indicating coordination with metal ions through phenolic oxygen. In all the complexes additional bands appeared at 650–610 and 550–525 cm^{-1} , supporting coordination of metal through oxygen and nitrogen [24, 25].

3.3. $^1\text{H-NMR}$ spectra

The $^1\text{H-NMR}$ spectra of BFP and its zinc complex were carried out in DMSO at room temperature using TMS as internal standard. $^1\text{H-NMR}$ spectra of polymeric ligand and its polymer–metal complexes are given in “Supplementary material”. In BFP a

Table 2. IR spectra and their assignment of BFP and its complexes.

Compounds	Assignments							
	Phenolic-OH	ν ArC-H	AliC-N	ArC=C	C-H (asym. and sym.)	δ CH ₂	ν M-O	M \leftarrow N
BFP	3460	3050	1280	1580	2880-2740	1460	-	-
BFP-Mn(II)	-	3054	1015	1515	2880-2740	1429	650	552
BFP-Co(II)	-	3050	1025	1500	2880-2740	1435	610	550
BFP-Ni(II)	-	3055	1015	1540	2880-2740	1427	650	525
BFP-Cu(II)	-	3050	1040	1500	2880-2740	1430	653	550
BFP-Zn(II)	-	3052	1015	1565	2880-2740	1427	650	553

Table 3. Electronic spectra, magnetic moments, and ligand field parameters of polymer-metal complexes.

Abbreviation	Magnetic moment (BM)	Electronic spectral data					
		Electronic Trans. (cm ⁻¹)	Assignment	10Dq	B	β	β^0
BFP-Mn(II)	5.92	24,210 20,995 16,667	${}^4E_{1g}(G) \leftarrow {}^6A_{1g}$ ${}^4T_{2g}(G) \leftarrow {}^6A_{1g}(F)$ ${}^4T_{1g}(G) \leftarrow {}^6A_{1g}(F)$	7650	695	0.72	28
BFP-Co(II)	4.72	21,250 12,628 9804	${}^4T_{1g}(P) \leftarrow {}^4T_{1g}(F)$ ${}^4A_{2g}(F) \leftarrow {}^4T_{1g}(F)$ ${}^4T_{2g}(F) \leftarrow {}^4T_{1g}(F)$	11,440	817	0.84	16
BFP-Ni(II)	2.97	23,200 16,430 9750	${}^3T_{1g}(P) \leftarrow {}^3A_{2g}(F)$ ${}^3T_{1g}(F) \leftarrow {}^3A_{2g}(F)$ ${}^3T_{1g}(F) \leftarrow {}^3A_{2g}(F)$	9550	865	0.80	20
BFP-Cu(II)	1.83	15,310 23,945	${}^2A_{1g} \leftarrow {}^2B_{1g}$ Charge transfer	-	-	-	-
BFP-Zn(II)	Diamagnetic	-	No transitions	-	-	-	-

multiplet at 7.3 ppm was assigned to Ar-OH and resonances of aromatic protons appeared between 6.9 and 6.2 ppm [26, 27]. The OH attached to CH₂ appeared at 3.7-3.0 ppm and the methyl protons of bisphenol-A at 1.9 ppm. The resonances of Ar-CH₂-N and N-CH₂-CH₂-N appeared at 3.2-3.0 and 2.7-2.5 ppm, respectively. In the ¹H-NMR spectrum of BFP-Zn(II), Ar-OH was not observed, confirming the Ar-O-M bond [28], which is also supported by FTIR spectra due to the appearance of M-O bond. Other peaks in the spectrum of polymer-metal complexes did not show any shift compared with the spectrum of polymeric ligand.

3.4. Electronic spectra

The electronic spectra of the polymer-metal complexes were recorded in DMSO. Electronic spectral bands and their magnetic moments are depicted in table 3. BFP-Mn(II) exhibited three bands at 16,667, 20,995, and 24,210 cm⁻¹, which may correspond to ${}^4T_{1g}(G) \leftarrow {}^6A_{1g}(F)$, ${}^4T_{2g}(G) \leftarrow {}^6A_{1g}(F)$, and ${}^4E_{1g}(G) \leftarrow {}^6A_{1g}(F)$ transitions, respectively. These data were used to calculate 10Dq, B, and β as 7650, 695,

Table 4. Thermal properties of BFP and its complexes.

Compounds	Weight loss (%) at the indicated temperature (°C)			Characteristic weight left (%) at 800°C
	200	400	600	
BFP	10	100	100	0
BFP-Mn(II)	12	39	70	8.5
BFP-Co(II)	11	38	67	7
BFP-Ni(II)	10	40	68	8
BFP-Cu(II)	11	39	69	9
BFP-Zn(II)	13	42	68	7.8

and 0.72 cm^{-1} , respectively. BFP-Mn(II) has a magnetic moment of 5.92 BM, close to the calculated value of high spin metal ion. On the basis of μ_{eff} value and electronic spectral bands, an octahedral geometry is proposed for BFP-Mn(II) [29]. BFP-Co(II) has a magnetic moment value 4.72 BM due to four unpaired electrons and showed bands at 9804, 12,628, and $21,250 \text{ cm}^{-1}$, which were assigned to ${}^4\text{T}_{2g}(\text{F}) \leftarrow {}^4\text{T}_{1g}(\text{F})$, ${}^4\text{A}_{2g}(\text{F}) \leftarrow {}^4\text{T}_{1g}(\text{F})$, and ${}^4\text{T}_{1g}(\text{P}) \leftarrow {}^4\text{T}_{1g}(\text{F})$ transitions, respectively, suggesting octahedral environment around Co(II) [30]. The calculated values of 10Dq , B , and β are 11,440, 817, and 0.84 cm^{-1} , respectively. Three bands observed for BFP-Ni(II) at 9750, 16,430, and $23,200 \text{ cm}^{-1}$ are assigned to ${}^3\text{T}_{2g}(\text{F}) \leftarrow {}^3\text{A}_{2g}(\text{F})$, ${}^3\text{T}_{1g}(\text{F}) \leftarrow {}^3\text{A}_{2g}(\text{F})$, and ${}^3\text{T}_{1g}(\text{P}) \leftarrow {}^3\text{A}_{2g}(\text{F})$ transitions, respectively. The calculated values of 10Dq , B , and β are 9550, 865, and 0.80 cm^{-1} , respectively. The observed magnetic moment for this compound is 2.97 BM and suggested octahedral structure [31].

The above discussion strongly indicates an octahedral geometry around Mn(II), Co(II), and Ni(II) in the polymer-metal complexes, indicating occupation of two coordinating sites by H_2O out of six in making the octahedral environment. The electronic absorption spectrum of BFP-Cu(II) exhibited broad bands at 15,310 and $23,945 \text{ cm}^{-1}$ assigned to ${}^2\text{A}_{1g} \leftarrow {}^2\text{B}_{1g}$ and charge transfer, respectively, indicating square-planar geometry around copper. The magnetic moment of BFP-Cu(II) is 1.83 BM, in accord with square-planar geometry [32].

3.5. Thermogravimetric analysis

Thermal analyses of the polymeric ligand and its metal complexes were carried out to find the thermal stability from 100°C to 800°C (table 4 and figure 1). Up to 100°C the ligand loses only 2% weight, due to absorbed water [33]. The 24% weight loss from 190°C to 300°C corresponded to loss of piperazine, in two steps, 14% and 10% weight loss due to hydrocarbons and nitrogen, respectively. About 68% rapid weight loss occurs due to bisphenol-A. The ligand decomposed completely into volatile product to 350°C . All the polymer-metal complexes were more stable than the ligand. The initial decomposition temperature of Mn(II), Co(II), and Ni(II) coordination polymers were between 120°C and 140°C with 3–7% weight loss due to two coordinated waters [34]. The piperazine was not dissociated from the polymer chain, due to coordination with metal. The methylene (uncoordinated part) of piperazine was completely decomposed by 300°C ; bisphenol molecules were not easily decomposed due to the formation of

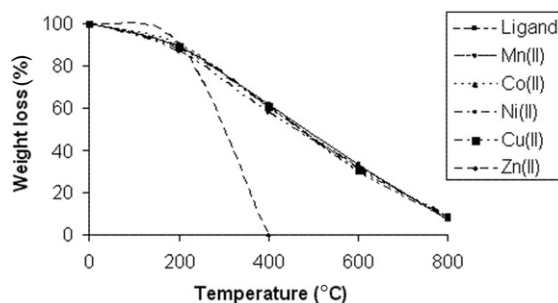


Figure 1. TGA of polymeric ligand and its polymer-metal complexes.

Table 5. Antibacterial activities of BFP and its complexes.

Abbreviation	Zone of inhibition (mm) 50 µg per disk						
	<i>E. coli</i>	<i>B. subtilis</i>	<i>B. megaterium</i>	<i>St. aureus</i>	<i>Sa. typhi</i>	<i>P. aeruginosa</i>	<i>Sh. boydii</i>
BFP	14	15	14	16	18	19	18
BFP-Mn(II)	16	17	15	16	19	18	18
BFP-Co(II)	18	17	19	18	20	20	18
BFP-Ni(II)	16	16	17	18	21	21	18
BFP-Cu(II)	18	17	18	19	22	22	19
BFP-Zn(II)	17	19	17	18	20	21	17
Kanamycin ^a	15	16	18	17	20	20	18
DMSO ^b	–	–	–	–	–	–	–

^aStandard drug (positive control).

^bSolvent (negative control) antifungal activity.

covalent bond between polymeric ligand and metal ion. The thermal stability of the polymer-metal complexes were BFP-Cu(II) > BFP-Zn(II) > BFP-Mn(II) > BFP-Co(II) > BFP-Ni(II) > BFP. All are completely decomposed to the corresponding metal oxides as the residual weight at 800°C [35] (table 4).

3.6. Antimicrobial activity

The antimicrobial activities of BFP and its metal complexes were screened by agar well diffusion methods, given in table 5 and shown in figure 2. All the polymers show promising antibacterial activity against all the bacteria. The polymeric ligand shows highest zone of inhibition of 19 mm against *P. aeruginosa*. The polymer-metal complex BFP-Cu(II) shows zones of inhibition of 19, 22, 22, and 19 mm against *St. aureus*, *Sa. typhi*, *P. aeruginosa*, and *Sh. boydii*. BFP-Ni(II) shows zones of inhibition of 18, 21, 21, and 18 mm against these bacteria. The maximum zones of inhibition of 20 and 21 mm for BFP-Zn(II) were when it was treated against *Sa. typhi* and *P. aeruginosa*. BFP-Mn(II) shows lowest antibacterial activity of the polymer-metal complexes, while BFP-Cu(II) shows highest zone of inhibition. The antimicrobial activities revealed that the presence of N and O donors inhibited the enzyme production because enzymes that require free hydroxyl for their activity appear to be especially susceptible to

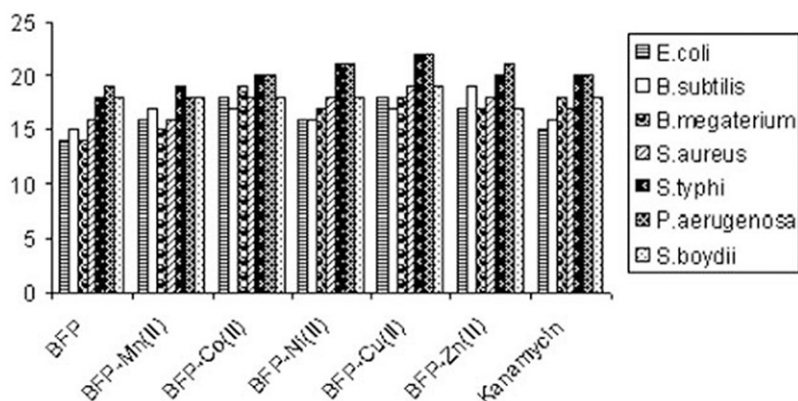


Figure 2. Antimicrobial activity of polymeric ligand and its metal complexes.

deactivation by polymer–metal complexes. The activity of the polymers was increased by the coordination of the metal ion [36–39].

4. Conclusion

BFP and its metal complexes were prepared in alkaline medium. The polymer–metal complexes were not soluble in most organic solvents. TGA data of polymer–metal complexes showed higher thermal stability than ligand. The antimicrobial activities revealed that all the polymer–metal complexes show better antibacterial activity than their corresponding ligand with Cu(II) more active and more thermally stable than other polymer–metal complexes due to higher stability constant of Cu(II). Since these polymers are good antibacterial agents, they can be used for medical and biomaterial applications.

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