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Synthesis, crystal structures and antibacterial studies of oxidovanadium(IV) complexes of salen-type Schiff base ligands derived from meso-1,2-diphenyl-1,2-ethylenediamine

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Abstract A series of new derivatives and previously reported Schiff base ligands and their oxidovanadium(IV) complexes were synthesized, characterized and tested as potential antibacterial agents against four human pathogenic bacteria. These N_2O_2 type Schiff base ligands were derived from the condensation of meso-1,2-diphenyl-1,2-ethylenediamine with different salicylaldehyde derivatives, and their metal complexes were obtained from the reaction of these ligands with bis(acetylacetonato)oxidovanadium(IV). Our studies showed that the metal complexes had moderate antibacterial activity, and this activity was higher than that of the free ligands against both Gram-positive and Gram-negative bacteria. Besides, it was found that the presence of more substituents on the ligands increases the antibacterial activities of both the free ligands and their complexes. The crystal structures of H_2L^4 and its corresponding complex VOL^4 were determined by X-ray crystallography.

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Introduction

Vanadium chemistry has attracted great interest for several decades [1–7]. An especially considered class of vanadium-containing compounds is oxidovanadium(IV) Schiff base complexes. This interest comes from their antibacterial [8–10], insulin mimetic [11–13], anticancer [14, 15] and catalytic properties [16, 17]. The wide variety of Schiff base ligands and their metal complexes, thanks to their easy synthesis and structural diversity as well as to the possibility of fine tuning of steric and electronic properties, has made it possible to identify several factors affecting such activities. This has led to the development of new industrially important catalysts and novel antibacterial agents relevant to global issues such as bacterial resistance [18]. In continuation of our studies on the antibacterial activity of transition metal complexes derived from Schiff base ligands, herein, we report the synthesis, characterization and results of antibacterial activity studies on a series of N_2O_2 type Schiff base ligands and their oxidovanadium(IV) complexes (Fig. 1). The Schiff base ligands were synthesized from the condensation of meso-1,2-diphenyl-1,2-ethylenediamine with salicylaldehyde derivatives. The oxidovanadium(IV) complexes were synthesized from the reaction between these ligands and bis(acetylacetonato)oxidovanadium(IV). Crystal structures of one of the ligands (H_2L^4) and the corresponding complex (VOL^4) were also determined. The in vitro antimicrobial activity of the complexes was tested against four human pathogenic bacteria, namely *Escherichia Coli* (Gram negative), *Salmonella typhi* (Gram negative), *Staphylococcus aureus* (Gram positive) and *Bacillus subtilis* (Gram positive). Our studies revealed that these complexes were moderately active against both Gram-type bacteria, although they were more active against the Gram

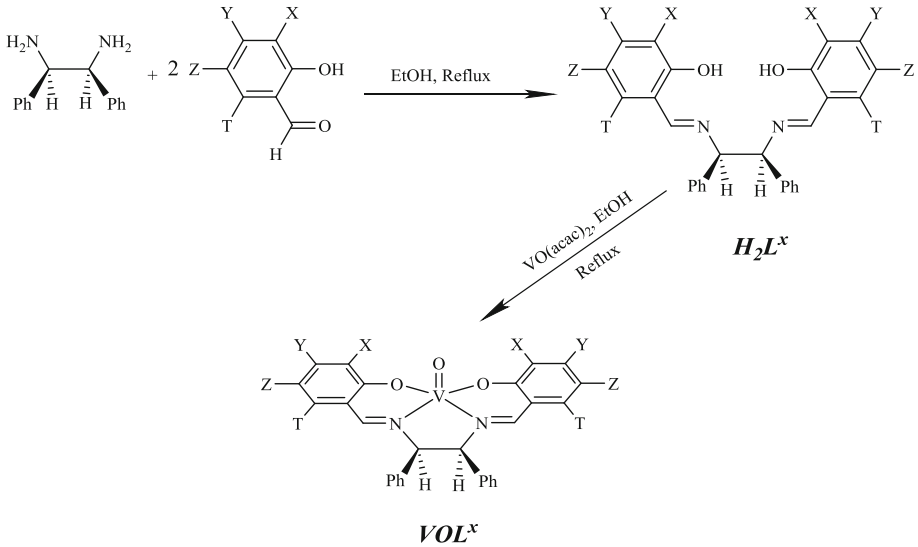

Fig. 1 Schematic representation of the synthetic procedure of the ligands (H_2L^x) and complexes VOL^x

Ligands or complexes	X	Y	Z	T
H_2L^1, VOL^1	H	H	H	H
H_2L^2, VOL^2	OCH_3	H	H	H
H_2L^3, VOL^3	H	H	OCH_3	H
H_2L^4, VOL^4	H	H	Br	H
H_2L^5, VOL^5	H	H	Cl	H
H_2L^6, VOL^6	OCH_3	H	Br	H
H_2L^7, VOL^7	Br	H	Cl	H

positive ones. Besides, VOL^6 and VOL^7 complexes with more substituents on their ligands showed higher antibacterial activity.

Experimental

All chemicals were purchased from commercial sources and were used as received. Meso-1,2-diphenyl-1,2-ethylenediamine [19], the ligands [H_2L^x, $x = 1$–5] and their VO(IV) complexes [VOL^x, $x = 1$–5] were synthesized as described elsewhere [20, 21]. Melting points were obtained on a thermoscientific 9,100 apparatus. Elemental analyses were performed by using a Perkin-Elmer 2400II CHNS-O analyzer. ^{1}HNMR spectra were recorded on a 500 MHz Bruker FT-NMR spectrometer using $CDCl_3$ or DMSO-d^6 as solvent; chemical shifts (δ) are given in ppm. IR spectra were obtained as KBr plates using a Bruker FT-IR instrument. UV–Vis spectra were obtained on a Shimadzu UV-1650PC spectrophotometer. A Metrohm 757 VA instrument was employed to obtain cyclic voltammograms in DMSO solutions at room temperature (25 °C) under nitrogen atmosphere using 0.1 M tetra-*n*-butylammonium hexafluorophosphate (TBAHFP) as supporting electrolyte. A platinum working electrode, a platinum auxiliary electrode and an Ag/AgCl reference electrode were used to obtain cyclic voltammograms.

Synthesis of *N*,*N*′-bis(3-methoxy-5-bromosalicylidene)-1,2-diphenyle-1,2-ethylenediamine (H_2L^6)

This ligand was synthesized following a routine procedure [20]. In a typical experiment, to a vigorously stirred solution of meso-1,2-diphenyl-1,2-ethylenediamine (0.21 g, 1 mmol) in ethanol (25 mL) was added a solution of 3-methoxy-5-bromosalicylaldehyde (0.46 g, 2 mmol) in ethanol (40 mL). The reaction mixture was stirred and refluxed for 1 h, then the heating source was removed and the reaction mixture was left undisturbed overnight. The orange crystals of the target ligand were collected by

filtration, washed with diethyl ether and air dried. Yield: 0.60 g, 94 %. ^{1}HNMR ($CDCl_3$): 14.10 (br, 2H, OH); 7.98 (*s*, 2H, CHN); 7.38–7.25 (*m*, 10H, H_{Ar}); 7.00 (*d*, 2H, H_{Ar}); 6.86 (*d*, 2H, H_{Ar}); 4.77 (*s*, 2H, CHPh); 3.92 (*s*, 6H, OCH_3). Selected IR (cm^{-1}): 1,627 (CH=N). UV–Vis. 10^{-4} M solution in CH_2Cl_2 [λ_{max} nm, (ε M^{-1} cm^{-1})]: 279 (22,650), 347 (6,940). Anal. Calcd. for $C_{30}H_{26}Br_2N_2O_4$, C: 56.4; H: 4.1, N: 4.4. Found C: 56.5; H: 4.1, N: 4.5.

Synthesis of *N*,*N*′-bis(3-bromo-5-chlorosalicylidene)-1,2-diphenyle-1,2-ethylenediamine (H_2L^7)

This ligand was also synthesized following a procedure similar to that of H_2L^6 except 3-bromo-5-chlorosalicylaldehyde was used instead of 3-methoxy-5-bromosalicylaldehyde. Yield 0.62 g, 96 %. ^{1}HNMR (DMSO-d^6): 7.91 (*s*, 2H, CHN); 7.51 (*d*, 2H, H_{Ar}); 7.33–7.23 (*m*, 10H, H_{Ar}); 7.02 (*d*, 2H, H_{Ar}); 4.76 (*s*, 2H, CHPh). Selected IR (cm^{-1}): 1,627 (CH=N). UV–Vis. 10^{-4} M solution in CH_2Cl_2 [λ_{max} nm, (ε M^{-1} cm^{-1})]: 276 (18,980), 341 (12,150). Anal. Calcd. for $C_{28}H_{20}Br_2Cl_2N_2O_2$, C: 52.0; H: 3.1, N: 4.3. Found C: 52. 2; H: 3.2, N: 4.5.

Synthesis of {*N*,*N*′-bis(3-methoxy-5-bromosalicylaldiminato)-1,2-diphenyle-1,2-ethylenediamine}oxidovanadium(IV) (VOL^6)

To a vigorously stirred solution of H_2L^6 (0.32 g, 0.5 mmol) in boiling ethanol (50 mL) was added a solution of bis(acetylacetonato)oxidovanadium(IV) (0.14 g, 0.5 mmol) in ethanol (25 mL). The reaction mixture was refluxed for 1 h, and then the green solution was left undisturbed overnight until the green microcrystalline target compound precipitated. The crystals were collected by filtration, washed with cold ethanol (20 mL) and air dried. Yield: 0.31 g, 88 %. Selected IR (cm^{-1}): 1,612 (CH=N), 995 (V=O). UV–Vis. 10^{-4} M solution in CH_2Cl_2 [λ_{max} nm, (ε M^{-1} cm^{-1})]: 283 (27,230), 299.5 (27,090), 401 (7,660), 609 (180). Anal. Calcd. for $C_{30}H_{24}Br_2N_2O_5V$, C: 51.1; H: 3.4, N: 4.0. Found: C: 51.3; H: 3.7, N: 4.1.

Synthesis of {*N*,*N*′-bis(3-Bromo-5-chlorosalicylaldiminato)-1,2-diphenyle-1,2-ethylenediamine}oxidovanadium(IV) (VOL^7)

This compound was synthesized following a procedure similar to that of VOL^6 except H_2L^7 was used instead of H_2L^6. Yield: 0.31 g, 86 %. Selected IR (cm^{-1}): 1,604 (CH=N), 995 (V=O). UV–Vis. 10^{-4} M solution in CH_2Cl_2[λ_{max} nm, (ε M^{-1} cm^{-1})]: 277 (19,790), 290.5 (19,950), 390.5(9,330), 606 (163). Anal. Calcd. for $C_{28}H_{18}Br_2Cl_2N_2O_3V$, C: 47.2; H: 2.5, N: 3.9.Found, C: 47.5; H: 2.7, N: 3.7.

X-ray crystallography

Diffraction data were collected at room temperature by the ω-scan technique on an Oxford Diffraction Xcalibur four-circle diffractometer with Eos CCD detector, equipped with graphite-monochromatized MoK_α radiation source ($\lambda = 0.71{,}073$ Å). The data were corrected for Lorentz-polarization as well as for absorption effects [22]. Precise unit-cell parameters were determined by a least-squares fit of 1,430 (**1**) and 2,641 (**2**) reflections of the highest intensity, chosen from the whole experiment. The structures were solved with SIR92 [23] and refined with the full-matrix least-squares procedure on F^2 by SHELXL97 [24]. The scattering factors incorporated in SHELXL97 were used. The function $\sum w(|F_o|^2 - |F_c|^2)^2$ was minimized, with $w^{-1} = [\sigma^2(F_o)^2 + (A\cdot P)^2 + B\cdot P]$ ($P = [\text{Max}\ (F_o^2, 0) + 2F_c^2]/3$). All non-hydrogen atoms were refined anisotropically. All hydrogen atoms—except OH H12 atom in 1, which was freely refined—were inserted in idealized positions and refined riding with the atoms to which they were bonded.

Antibacterial activity studies

Bacterial strains

The metal complexes were tested against a panel of microorganisms, namely *Salmonella typhi* (ATCC 19430, Gram negative); *Escherichia Coli* (ATCC 25922, Gram negative); *Staphylococcus aureus* (ATCC 25923, Gram positive); and *Bacillus subtilis* (ATCC No. 6633, Gram positive).

Determination of minimum inhibitory concentrations (MIC) and minimal bactericidal concentrations (MBC)

Minimum inhibitory concentrations (MICs, mg/mL) were determined by the broth microdilution method following the procedures recommended by the National Committee for Clinical Laboratory Standards [25, 26]. MICs were defined as the lowest concentrations of compounds which inhibit the growth of microorganisms. All tests were performed in triplicate. Minimal bactericidal concentrations (MBCs) were also measured following a standard procedure [27]. Then, 100 µL volumes of all clear (no growth) tubes from a dilution MIC test were spread onto separate agar plates and incubated at 37 °C for 24 h. The MBC (mg/mL) was defined as the lowest concentration of the complex at which no growth occurred.

Results and discussions

Synthesis

The reaction between meso-1,2-diphenyl-1,2-ethylenediamine and different salicylaldehyde derivatives yielded the target Schiff base ligands in good yield. Typically, a color change to yellow or orange showed the proceeding of the reaction, and the completion of the reaction was determined by TLC. The reaction between the tetradentate Schiff base ligands and bis(acetylacetonato)oxidovanadium(IV) in ethanol gave the oxidovanadium(IV) Schiff base complexes as green microcrystalline compounds. The green color of the complexes is indicative of their monomeric form [28]. The elemental analyses also confirmed the synthesis of the complexes.

Spectroscopic characterization of the ligands and complexes

In the ^{1}HNMR spectra of the free Schiff base ligands, a broad (br) band at around 14 ppm was assigned to the phenolic OH protons. This signal was not observed for H_2L^7 which is expected and is due to the broadening of the band by rapid exchange processes. The sharp singlet (*s*) signals at around 8 ppm were assigned to the azomethine protons. The aromatic protons of the phenyl rings of the meso-diamine appeared in the aromatic region at around 7.2–7.4 ppm as multiplets (*m*). The aromatic signals of the salicylaldehyde part of the Schiff base ligands were also observed in the aromatic region of the spectra as doublets (*d*).

In the IR spectra of the ligands, the presence of an intense band at 1,627 cm^{-1} was indicative of the formation and presence of iminic groups (CH=N). This band was shifted to lower wave numbers upon coordination to the metal center which meant that the iminic nitrogen atoms of the Schiff base ligands were participating in coordination. The vanadyl (V=O) stretching vibration at 995 cm^{-1} was suggestive of the absence of any intermolecular (…V=O…V=O…) interactions. This indicated the monomeric form of the complexes, while the polymeric forms usually give this band at around 850–880 cm^{-1} [28]. The UV–Vis spectra of the free ligands were similar to those of previously reported spectra for similar ligands. The intense bands at around 270 and 341 nm were assignable to the $\pi \rightarrow \pi^*$ transitions in the Schiff base ligands. The second band that was blue shifted by about 40 nm in VOL^x complexes was assigned to the $\pi \rightarrow \pi^*$ transitions of azomethine groups. This blue shift was due to the coordination to the VO(IV) metal center. The bands at around 400 and 600 nm are also assigned to the LMCT and $d \rightarrow d$ transitions, respectively [29].

Table 1 Redox potential data for 10^{-3} mol L^{-1} solution of VOL^x ($x = 4, 6, 7$) in DMSO containing 0.1 mol L^{-1} TBAHFP and scan rate 50 mV/s. Potentials are versus Ag/AgCl

Complex	E^0 (mV)	Ep_c (mV)	Ep_a (mV)	ΔE (mV)
VOL^6	515	470	560	90
VOL^4	560	506	614	108
VOL^7	620	590	650	60

Electrochemistry

Cyclic voltammetry studies were performed in DMSO solutions at room temperature (25 °C) using 0.1 M tetra-*n*-butylammonium hexafluorophosphate (TBAHFP) as supporting electrolyte. A platinum working electrode, a platinum auxiliary electrode and an Ag/AgCl reference electrode were used to obtain cyclic voltammograms. The electrochemical properties of $VOL^{1–5}$ complexes are previously reported [20, 21]. The redox data for VOL^6 and VOL^7 complexes are collected in Table 1. The electrochemical data for VOL^4 in DMSO are also shown in this table for comparison. Cyclic voltammetry studies of oxidovanadium(IV) Schiff base complexes usually exhibit a reversible or quasi-reversible redox wave for V^{IV}/V^V process. It has been shown that the presence of electronegative substituents on the Schiff base ligands shifts the E^0 of the occurred V^{IV}/V^V process to more positive values [20, 21]. The same results were observed in this study.

X-ray crystallography

Description of the crystal structures

Crystallographic data and final structure refinement parameters for both structures are collected in Table 2. Selected bond lengths and angles for VOL^4 are listed in Table 3. Figure 2 shows a perspective view of the ligand H_2L^4. The molecule is C_i-symmetrical, and the middle point of the C1–C1 (−x, 1 − y, 1 − z) bond lies on the center of inversion. There are classical intramolecular N–H···O hydrogen bonds between the hydrogen atoms of the OH groups and nitrogen atoms in meso-1,2-diphenyl-1,2-ethylenediamine (Table 4). The dihedral angle between the planes defined by the rings of the salicylidene fragment and the phenyl substituent is 30.41(6)°, compared to just 23.8° in H_2L^1 and 63.76° in the (−)-(1S,2S) isomer [30, 31]. An ORTEP representation of VOL^4 is shown in Fig. 3. The asymmetric unit of VOL^4 consists of two crystallographically independent, but chemically identical and geometrically very similar complexes that, however, show some differences in their environment. The V(IV) atom is five-coordinated in a distorted square-pyramidal environment.

Table 2 Crystal data, data collection and structure refinement parameters for H_2L^4 and VOL^4

Compound	H_2L^4	VOL^4
Empirical formula	$C_{28}H_{22}Br_2N_2O_2$	$C_{28}H_{20}{\cdot}Br_2N_2O_3V$
Formula weight	578.30	643.22
Crystal system	Triclinic	Triclinic
Space group	P-1	P-1
Unit-cell dimensions (Å, °)		
a	6.5028 (9)	11.9421 (6)
b	9.5765 (13)	13.1778 (8)
c	11.1719 (16)	16.9847 (7)
α	103.090 (12)	91.345 (4)
β	104.410 (12)	96.935 (4)
γ	105.616 (12)	100.877 (5)
Volume (Å^3), Z	616.00 (17), 1	2602.7 (2), 4
Calculated density (g cm^{-3})	1.56	1.64
Absorption coefficient (mm^{-1})	3.32	3.48
F(000)	290	1,276
θ Range for data collection (°)	3.36–28.04	2.82–28.44
Limiting indices	$-7 \le h \le 7$	$-15 \le h \le 13$
	$-12 \le k \le 12$	$-17 \le k \le 17$
	$-13 \le l \le 14$	$-22 \le l \le 22$
Reflections		
Collected	4,070	18,090
Unique (Rint)	2,485 (0.030)	10,853 (0.040)
With $I > 2\sigma(I)$	2,001	5,923
Number of parameters/ restrains	158/0	649/0
Goodness of fit on F^2	1.032	1.03
Final R index [$I > 2\sigma(I)$]	R1 = 0.0392, wR2 = 0.0794	R1 = 0.0651, wR2 = 0.0935
R index [all data]	R1 = 0.0540, wR2 = 0.0874	R1 = 0.1391, wR2 = 0.1162
Largest difference peak and hole (e Å^{-3})	0.34/−0.45	1.055/−1.166

Table 3 Selected geometrical parameters for VOL^4

Bond lengths (Å)			
O1A-V1A-O12A	109.42 (17)	O12A-V1A-N8A	86.23 (14)
O1A-V1A-O28A	108.33 (16)	O12A-V1A-N24A	146.14 (15)
O1A-V1A-N8A	106.54 (17)	O28A-V1A-N8A	144.85 (15)
O1A-V1A-N24A	103.95 (16)	O28A-V1A-N24A	88.48 (15)
O12A-V1A-O28A	86.43 (14)		
Bond angles (°)			
V1A-O1A	1.593 (3)	V28A-O28A	1.928 (3)
V1A-O12A	1.919 (3)	V1A-N8A	2.049 (4)
V1A-N24A	2.043 (4)		

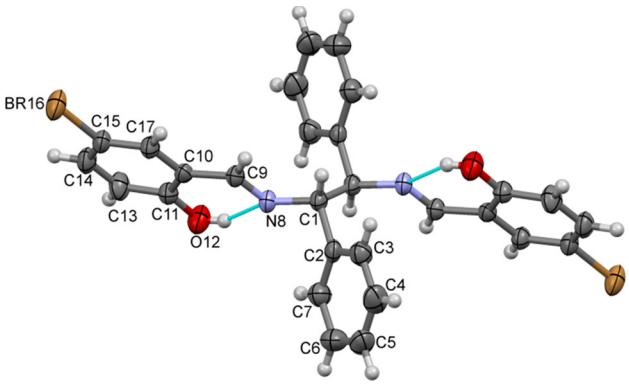

Fig. 2 Perspective view of the H_2L^4; displacement ellipsoids are drawn at 50 % probability level, hydrogen atoms are shown as spheres of arbitrary radii. Intramolecular hydrogen bonds are shown as *blue lines*. The non-labeled part is related by the symmetry operation −x, 1 − y, 1 − z to the labeled one. (Color figure online)

Table 4 Hydrogen bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	DHA
H_2L^4				
O12-H12···N8	0.72 (5)	2.02 (5)	2.640 (4)	145 (5)
VOL^4				
C1A -H1A···O1B^{i}	0.98	2.30	3.255 (6)	166
C9A-H9B···O28B^{i}	0.93	2.58	3.474 (6)	161
C17B-H17B···O1A^{ii}	0.98	2.44	3.390 (6)	162
C22A –H22A···O1B^{iii}	0.93	2.44	3.147 (9)	133
C25B –H25B···O12A^{ii}	0.93	2.34	3.260 (6)	170

Symmetry codes: ^{i} *x*,1 + *y*, *z*; ^{ii} −1+*x*,*y*,*z*; ^{iii} −*x*, − *y*, − *z*

The basal square plane is constituted by the two iminic nitrogen atoms and two phenoxide oxygen atoms of the Schiff base ligand, H_2L^4, which acts as a dianionic tetradentate ligand. The apical position is occupied with the oxo ligand. The bond angles in the basal plane of the complex are all smaller than the ideal angle of 90°, whereas the vertex angles are larger than 90° as a result of the central vanadium atom deviating from the basal plane toward the axial ligand. The average V–O_{ax} and V–O_{eq} as well as V–N_{eq} bond lengths for this complex are quite similar to the ones observed similar structures [28]. In the crystal structure, VOL^4 is connected with infinite chains along the c axis by weak intermolecular hydrogen bonds (Table 4).

Antibacterial studies

The synthesized oxidovanadium(IV) Schiff base complexes were screened for in vitro antibacterial activity

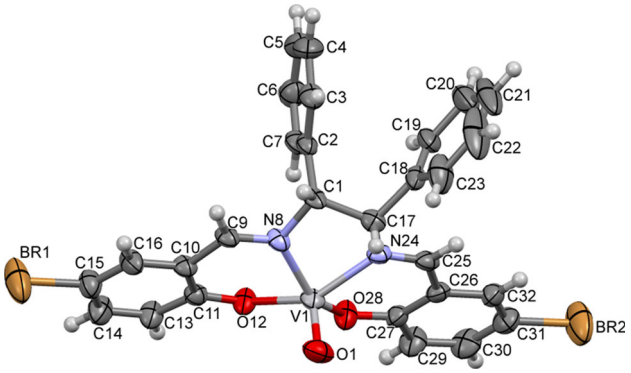

Fig. 3 Perspective view of one (A) of the symmetry-independent complexes of VOL^4 with labeling scheme. Displacement ellipsoids are drawn at 50 % probability level; hydrogen atoms are shown as spheres of arbitrary radii

against two Gram-negative (*E. coli* and *S. typhi*) and two Gram-positive (*S. aureus* and *B. Subtilis*) bacterial strains according to the literature protocol [25–27], and the results are collected in Table 5. According to this data, the complexes showed higher antibacterial activities than the free ligands. This increased activity results from coordination and could be explained by Overtone's concept [32] and Tweedy's theory [33]. According to Overtone's concept of cell permeability, lipo-solubility is an important factor that governs the antimicrobial activity of antibacterial agents. Chelation reduces the polarity of the metal ions to a greater extent due to the overlap of the ligand orbitals and partial sharing of positive charge of metal atom with donor groups. Besides, it increases the delocalization of π-electrons over the whole chelate ring and increases the lipophilicity of the complexes. This increased lipophilicity enhances the penetration of the complexes into the lipid membrane and increases their antibacterial activities. These complexes showed broad spectrum antibacterial activity against both Gram-positive and Gram-negative bacteria. Comparison of the MIC values of these complexes with those of similar ones in the literature shows that the studied complexes are moderate antibacterial agents [34]. It has been previously suggested that the presence of more electronegative or electron-donating groups on the ligands results in increased antibacterial activity of the ligands themselves and their metal complexes [35]. Our studies also confirmed this suggestion. H_2L^1 with no substituents on the salicylaldehyde moiety has the least antibacterial activity (Table 5). The presence of other substituents on the ligands increased their antibacterial activity and H_2L^6 and H_2L^7 with two substituents on the salicylaldehyde moiety showed the highest activities. The complexes also showed similar features, and VOL^6 and VOL^7 showed highest antibacterial activities. This increased activity could also be described by the penetration theory. The presence of more substituents, especially halogen substituents, increases the lipophilicity of the ligands and their complexes and hence increases the membrane penetration and antibacterial activities. The electrochemistry of the complexes could also affect the antibacterial properties of the complexes. As it is previously stated, the presence of more electronegative substituents shifts the E^0 value of V^{IV}/V^V redox process to more positive values. This is itself due to the electron deficiency of the central metal atom. This electron deficiency may increase the interaction of the complexes with the bacteria and increase their antibacterial properties. However, more

Table 5 MIC and MBC values for the ligands and complexes against the studied bacteria (mgL^{-1})

	Escherichia coli		*Salmonella Typhi*		*Staphylococcus aureus*		*Bacillus subtilis*	
	MIC	MBC	MIC	MBC	MIC	MBC	MIC	MBC
L^1	4.75	5.00	4.25	4.75	2.50	2.75	2.75	3.00
L^2	4.50	4.75	4.00	4.25	1.50	1.75	2.00	2.25
L^3	4.00	4.25	3.50	4.00	1.25	1.00	1.75	2.00
L^4	3.75	4.00	3.25	3.50	1.25	1.75	1.50	1.75
L^5	2.50	3.00	2.00	2.50	0.50	0.75	0.50	0.75
L^6	2.00	2.50	1.25	1.50	0.22	0.25	0.40	0.65
L^7	2.25	2.75	1.25	1.50	0.25	0.38	0.50	0.75
VOL^1	3.50	4.00	3.25	3.75	1.50	1.75	1.50	1.75
VOL^2	3.75	4.00	3.25	3.50	0.75	1.00	1.00	1.25
VOL^3	3.25	3.75	2.75	3.25	0.62	0.75	0.75	1.00
VOL^4	3.00	3.50	1.75	2.00	0.25	0.50	0.50	0.75
VOL^5	3.50	4.00	2.75	3.00	0.62	0.70	0.75	1.25
VOL^6	2.00	2.25	1.25	1.50	0.02	0.05	0.08	0.10
VOL^7	2.75	3.00	2.50	2.75	0.08	0.12	0.12	0.25

work is required to find clear structural, electronic and electrochemical correlation with antibacterial properties of such antibacterial agents. It might be also mentioned that the complexes we have studied showed better activity against Gram-positive bacteria.

Supplementary material

CCDC 894215 and 894216 contains the supplementary crystallographic data for H_2L^4 and [VOL^4]. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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