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Synthesis, characterization, crystal structures, computational studies, and antibacterial activities of two new Schiff bases derived from isophthalaldehyde

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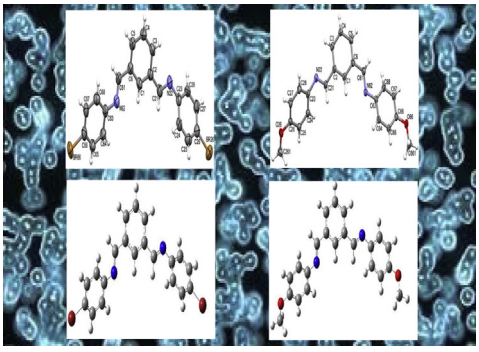
HIGHLIGHTS

- The new compounds showed moderate antibacterial activity.
- The crystal structures were determined by X-ray diffraction.
- Molecular orbital calculations were performed.
- Complexes have been characterized by the analytical and spectral methods.

GRAPHICAL ABSTRACT

Two new Schiff bases have been synthesized by the reaction between isophthalaldehyde and appropriate aniline derivatives, and characterized by various Spectroscopic techniques. The crystal structures of **1** and **2** were determined by X-ray diffraction. Moreover, structural optimization by DFT calculations have been performed and compared with the experimental data. The compounds were also screened for *in vitro* antibacterial activities against four human pathogenic bacteria and their minimum inhibitory concentrations showed moderate antibacterial activities.



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ABSTRACT

Two new Schiff bases, *N*,*N*′-(1,3-phenylenebis(methanylylidene))bis(4-bromoaniline) (**1**) and *N*,*N*′-(1,3-phenylenebis(methanylylidene))bis(4-methoxyaniline) (**2**), have been synthesized by the reaction between isophthalaldehyde and appropriate aniline derivatives, and characterized by physico-chemical and spectroscopic methods. The structures of new compounds **1** and **2** have been characterized crystallographically. Moreover, structural optimization by DFT calculations have been performed and compared with the experimental data. The compounds were also screened for *in vitro* antibacterial activities against four human pathogenic bacteria and their minimum inhibitory concentrations showed moderate antibacterial activities.

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Introduction

Schiff bases are considered as a very important class of organic compounds [1–7]. In the last years, some of these ligands have been studied extensively and have received considerable attention because of their attractive chemical and physical properties. Versatile Schiff base ligands have wide range of applications, e.g. as catalysts in hydrogenation and oxidation of olefins [8–12], in photochromic industry [13] and as fluorescent sensors for toxic metal ions [14]. Schiff base compounds also played an important role in the development of coordination chemistry [15–17]. Moreover, we can see that there in recent decade a continuing interest in study of Schiff bases has been observed, due to a variety of applications in biological, biochemical, analytical and industrial fields. Schiff bases have showed remarkable antiviral [18], antimicrobial [19], antifungal [20], antitumors [21], anticancer [22,23] and antibacterial activities [24–26]. With the growing public health awareness of the pathogenic effects, malodors and stain formations caused by microorganisms, there is an increasing need for antibacterial materials in many application areas like medical devices, health care, hygienic application, water purification systems, hospital, dental surgery equipment, textiles, food packaging, and storage [27,28]. This becomes clear, the biological activity of Schiff bases deserves further investigation. Recently we prepared a series of complexes of cobalt(III) with Schiff base ligands, and we characterized their spectroscopic, structural and cyclic voltammetric properties [29–32]. In this work, we have directed our efforts toward the synthesis, antibacterial properties and structural characterization, computational studies of two new Schiff bases (Scheme 1). The Schiff bases described here also are pincer ligands; similar compounds have already been used in the synthesis of NCN pincer complexes [33,34].

Experimental

Reagents and measurements

All solvents and chemicals were used as received and without any purification. Melting points were obtained by a Thermo Scientific 9100 apparatus. ^{1}HNMR spectra were recorded on 300 MHz Bruker Avance spectrometer using $CDCl_3$ solvent; chemical shifts (δ) are given in ppm. IR spectra were obtained as KBr plates using a Bruker FT-IR instrument and UV–Vis spectra were obtained on a Shimadzu UV-1650PC spectrophotometer.

Synthesis of the Schiff bases

*Synthesis of the N,N′-(1,3-phenylenebis(methanylylidene))bis (4-bromoaniline) (**1**)*

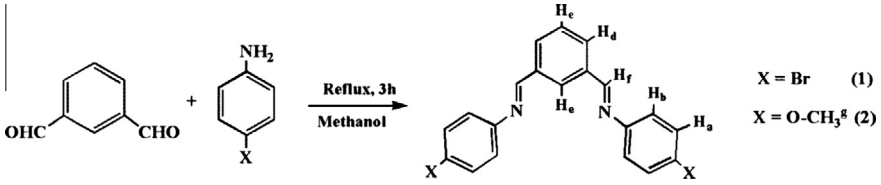
The Schiff base ligand, **1**, was prepared following literature [34]: to the stirred solution of isophthalaldehyde (0.001 mol) in absolute ethanol (5 mL) was added dropwise a solution of 4-bromoaniline (0.002 mol) in absolute ethanol (5 mL) and the resulting colorless solution refluxed for 3 h. The product separated on evaporation of the solvent was filtered, washed with ethanol. The single crystals suitable for X-ray data collection were obtained by slow evaporation of the methanol solution after 3 days. The crystals were filtered off, washed with a small amount of cold methanol and dried under vacuum. Yield: 75%. M.p. 121 °C. FT-IR (KBr, cm^{-1}): ν_{max} 1620 (s, C═N), 1481, 1573 (C═C). UV–Vis: λ_{max} (nm) (ε, $Lmol^{-1}\,cm^{-1}$) (CH_3CN): 194 (135,800), 229.5 (50,300), 266 (71,800), 319 (39,700). ^{1}H NMR ($CDCl_3$, δ (ppm)): 7.12 (m, $4H_a$, phenyl), 7.53 (m, $4H_b$, phenyl), 7.6 (t, $1H_c$, phenyl), 8.04 (d–d, $2H_d$ phenyl), 8.41 (s, $1H_e$, phenyl), 8.52 (s, $2H_f$, H—C═N—).

Table 1
Crystal data collection and structure refinement.

Compound	**1**	**2**
Formula	$C_{20}H_{14}Br_2N_2$	$C_{22}H_{20}N_2O_2$
Formula weight	442.13	344.40
Crystal system	Monoclinic	Orthorhombic
Space group	P2/n	Aba2
a (Å)	7.5573(8)	14.3347(3)
b (Å)	6.2803(7)	38.4760(7)
c (Å)	36.716(4)	6.4275(10)
β (°)	94.228(9)	90
V (Å^3)	1737.9(3)	3545.04(11)
Z	4	8
D_x (g cm^{-3})	1.690	1.29
$F(000)$	872	1456
μ (mm^{-1})	4.67	0.665
Θ range (°)	3.07–27.12	2.3–75.43
hkl range	$-8 \leqslant h \leqslant 9$	$-17 \leqslant h \leqslant 18$
	$-7 \leqslant k \leqslant 8$	$-48 \leqslant k \leqslant 38$
	$-46 \leqslant l \leqslant 46$	$-7 \leqslant l \leqslant 7$
Reflections		
Collected	16,097	7882
Unique (R_{int})	3587 (0.094)	3351 (0.020)
With $I > 2\sigma(I)$	2252	3264
Number of parameters	218	235
Weighting scheme		
A	0.075	0.0368
B	0.0000	0.4174
Final R index [$I > 2\sigma(I)$]	R_1 = 0.0731	R_1 = 0.0274
	wR_2 = 0.1596	wR_2 = 0.0683
R index [all data]	R_1 = 0.1220	R_1 = 0.0283
	wR_2 = 0.1889	wR_2 = 0.0690
Goodness-of-fit on F^2	1.08	1.04
Max/min $\Delta\rho$ (e Å^{-3})	0.63/−0.77	0.13/−0.14

*Synthesis of the N,N′-(1,3-phenylenebis(methanylylidene))bis (4-methoxyaniline) (**2**)*

The Schiff base ligand, **2**, was prepared following the same procedure as **1** except 4-methoxyaniline was used instead of 4-bromoaniline. Yield: 96%. M.p.: 137 °C. FT-IR (KBr, cm^{-1}): 1620 (m, C═N). UV–Vis: λ_{max} (nm) (ε, $Lmol^{-1}\,cm^{-1}$) (CH_3CN): 192 (73,300), 230 (37,900), 270 (38,700), 339 (35,900). Yield: 76%. M.p. 137 °C. FT-IR (KBr, cm^{-1}): ν_{max} 1620 (s, C═N), 1483, 1574 (C═C). UV–Vis: λ_{max} (nm) (ε, $Lmol^{-1}\,cm^{-1}$) (CH_3CN): 192.5 (73,300), 229.5 (37,900), 270 (38,700), 339 (35,900). ^{1}H NMR ($CDCl_3$, δ (ppm)): 3.85 (s, $6H_g$—O—CH_3), 6.96 (m, $4H_a$, phenyl), 7.28 (m, $4H_b$, phenyl), 7.57 (t, $1H_c$, phenyl), 8.01 (d–d, $2H_d$ phenyl), 8.38 (s, $1H_e$, phenyl), 8.57 (s, $2H_f$, H—C═N—).



Scheme 1. The preparation of the Schiff bases.