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Synthesis of novel tripodal-benzimidazole from 2,4,6-tris(p-formylphenoxy)-1,3,5-triazine: Structural, electrochemical and antimicrobial studies

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ABSTRACT

Four new tripodal-benzimidazole derivatives were synthesized by Schiff base reaction between 2,4,6-tris(p-formylphenoxy)-1,3,5-triazine (TRIPOD) and different diamine derivatives. The structures of the obtained compounds were identified by FT-IR, ^{1}H NMR, ^{13}C NMR and UV–vis spectral data, thermal analysis and elemental analysis. Electrochemical behaviors of the compounds were studied by cyclic voltammetry in DMF including 0.1 M $[NBu_4][PF_6]$. The voltammograms showed peaks having similar characteristics except tripodal-benzimidazole including $-NO_2$ derivative. In addition, their antimicrobial activities were evaluated by using the standard disk diffusion method in dimethylformamide media. The activities were determined against 4 bacteria cultures by comparing to those of gentamycin.

1. Introduction

An important class of compounds consisting of substituted s-triazine derivatives has biological activities such as anticancer [1], antiviral [2], estrogen receptor modulators [3] and antimalarials [4–10]. In addition, many of the compounds containing imidazole structure have exhibited the antibacterial activities [11–13]. The compounds have also been used in the treatment of depression and hence gained a considerable importance [14]. Considering information above, the design and synthesis of potent antimicrobials have been an important area of immense significance for medicinal chemists [15,16]. Much afford has been devoted to the synthesis of s-triazine derivatives by different groups in the recent years due to their attractive characteristics [17–22].

The reaction of trimeric cyanuric chloride ($C_3N_3Cl_3$) with 3 equiv. of 4-hydroxybenzaldehyde in benzene yielded the desired trialdehyde in a single step, 2,4,6-tris(p-formylphenoxy)-1,3,5-triazine, coded as TRIPOD [23,24]. In this paper, we aimed to make four new benzimidazole derivatives by using TRIPOD and different diamines (o-phenylenediamine, 4-methyl-o-phenylenediamine, 4-chloro-o-phenylenediamine and 4-nitro-o-phenylenediamine). We called them as "tripodal-benzimidazole". The structure characterizations, electrochemical properties and antibacterial activities of the obtained compounds were reported.

2. Experimental

2.1. Materials and methods

All solvents, o-phenylenediamine, 4-methyl-o-phenylenediamine, 4-chloro-o-phenylenediamine and 4-nitro-o-phenylenediamine used for the synthesis were of reagent grade and used without further purification. Elemental analyses were performed on a Carlo Erba 1106 elemental analyzer. The FT-IR spectra were recorded using KBr discs (4000–440 cm^{-1}) on a Perkin Elmer 1600 series FT-IR spectrophotometer. Melting points were measured using a Buchi SMP-20 melting point apparatus. The ^{1}H and ^{13}C NMR spectra were recorded on a Bruker DPX-400 MHz in d_6–DMSO. The thermal analyses were performed on Shimadzu DTA 50 and TG 50 H models using 10 mg samples. DTA and TG curves were obtained at a heating rate of 10 °C min^{-1}. In all cases the 22–750 °C temperatures range was studied under a dry nitrogen atmosphere. UV–vis absorption spectra were obtained by using a Shimadzu UV-1800 double beam spectrophotometer.

Electrochemical measurements were performed with an EG&G Princeton Applied Research PAR 263/A2 potentiostat/galvanostat with a positive feedback. Cyclic voltammetry studies of the compounds were carried out in dimethylformamide (DMF) solution containing 0.1 M $[NBu_4][PF_6]$ as supporting electrolytes, using platinum wires of 0.2 cm diameter as a working and counter electrodes. In all cases, the reference electrode was the Ag/Ag^{+} (0.1 mol L^{-1}) in DMF with a vycor glass interfacing the working electrode compartment. Ferrocene redox couple was used as an external standard

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($E_{1/2}$ = 0.400 V, ΔE_p = 63–69 mV at 100 mV s^{-1}) under the experimental conditions [25]. Cyclic voltammograms were performed in a potential range of +1.50 to −1.80 at scan rates (ν) between 50 and 400 mV s^{-1}. All solutions were purged with nitrogen steam for 30 min before measurement and the working electrode was polished before each experiment with different alumina powders. The procedure was performed at room temperature and a nitrogen atmosphere was maintained over the solution during the measurements.

The antibacterial screenings were evaluated against selected two gram-positive organisms, *Bacillus subtilis* (ATCC 6633) and *Staphylococcus aureus* (ATCC 6538P), and two gram-negative organisms, *Salmonella typhimurium* (NRRL B 4420) and *E. coli* (ATCC 25922), by using "the standard disk diffusion technique" [26] by comparing with gentamycin (Genta).

2.2. Synthesis of compounds

2.2.1. Synthesis of 2,4,6-tris(p-formylphenoxy)-1,3,5-triazine (*1*)

2,4,6-Tris(p-formylphenoxy)-1,3,5-triazine (TRIPOD) was prepared by the reaction of cyanuric chloride and 4-hydroxybenzaldehyde according to previous literatures [23,24].

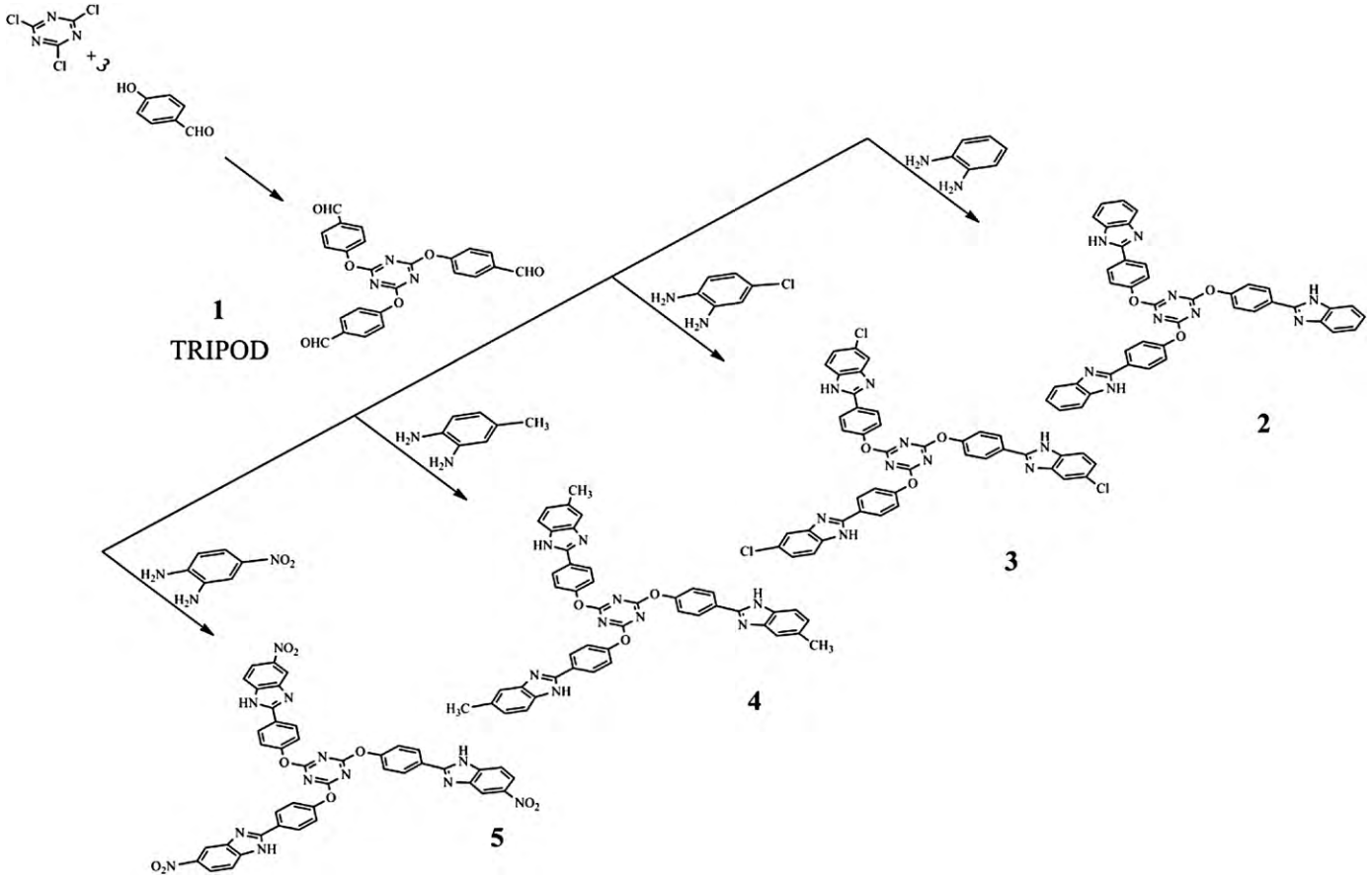
2.2.2. The synthesis of tripodal-benzimidazoles from TRIPOD (*2–5*)

The compounds **2–5** were firstly synthesized by using the procedure described in the literature [27]. To a stirring solution of TRIPOD (441 mg, 1 mmol) in ethanol (25 mL), $NaHSO_3$ (381 mg, 3 mmol) in ethanol (25 mL) was added at room temperature. The reaction mixture was treated with o-phenylenediamine (324 mg, 3 mmol), 4-chloro-o-phenylenediamine (426 mg, 3 mmol), 4-methyl-o-phenylenediamine (366 mg, 3 mmol), and 4-nitro-o-phenylenediamine (459 mg, 3 mmol) in dimethylformamide (20 mL) and boiled under reflux. After 3 h the contents were poured into iced-water (60 mL) and filtered. The precipitate was crystallized from ethanol.

Data for (**2**). Yield: 74%; m.p.: 220 °C; Elemental analysis (Found: C, 71.42; H, 3.83; N, 17.78%). Calc. for $C_{42}H_{27}N_9O_3$: C, 71.48; H, 3.86; N, 17.86%. IR (KBr) ν_{max}/cm^{-1}: 3420 (N–H), 2927 (C–H_{Ar}), 1571 (C=N), 1561 (C=$N_{triazine}$), 1213 (C–N), 1367 (C_{Ar}–O–C). ^1H NMR (400 MHz, DMSO–d_6, 25 °C) (δ: ppm): 7.72 (d, 6H, Ar–H, *J* 8.7 Hz), 7.33 (d, 6H, Ar–H, *J* 7.2 Hz), 7.28 (d, 6H, Ar–H, *J* 8.7 Hz), 6.80 (d, 6H, Ar–H, *J* 7.2 Hz), 12.15 (s, 3H, N–H). ^{13}C NMR (100 MHz, DMSO–d_6, 25 °C) (δ: ppm): 181.84, 154.35, 141.58, 137.93, 132.42, 128.07, 122.93, 121.66, 115.41.

Data for (**3**). Yield: 69%; m.p.: 235 °C; Elemental analysis (Found: C, 62.32; H, 2.96; N, 15.52%). Calc. for $C_{42}H_{24}Cl_3N_9O_3$: C, 62.35; H, 2.99; N, 15.58%. IR (KBr) ν_{max}/cm^{-1}: 3421 (N–H), 2922 (C–H_{Ar}), 1573 (C=N), 1564 (C=$N_{triazine}$), 1214 (C–N), 1364 (C_{Ar}–O–C), 840 (C–Cl). ^1H NMR (400 MHz, DMSO–d_6, 25 °C) (δ: ppm): 7.73 (s, 3H, Ar–H), 7.67 (d, 3H, Ar–H, *J* 8.5 Hz), 7.33 (d, 6H, Ar–H, *J* 7.4 Hz), 7.28 (d, 3H, Ar–H, *J* 8.5 Hz), 6.78 (d, 6H, Ar–H, *J* 7.4 Hz), 12.33 (s, 3H, N–H). ^{13}C NMR (100 MHz, DMSO–d_6, 25 °C) (δ: ppm): 181.87, 154.41, 141.62, 139.39, 136.05, 132.47, 128.24, 123.36, 121.69, 116.82, 115.83.

Data for (**4**). Yield: 72%; m.p.: 240 °C; Elemental analysis (Found: C, 72.23; H, 4.39; N, 16.79%). Calc. for $C_{45}H_{33}N_9O_3$: C, 72.28; H, 4.45; N, 16.86%. IR (KBr) ν_{max}/cm^{-1}: 3430 (N–H), 2962 and 2926 (C–H), 1572 (C=N), 1490 (C=$N_{triazine}$), 1215 (C–N), 1364 (C_{Ar}–O–C). ^1H NMR (400 MHz, DMSO–d_6, 25 °C) (δ: ppm): 7.59 (d, 3H, Ar–H, *J* 8.3 Hz), 7.51 (s, 3H, Ar–H), 7.32 (d, 6H, Ar–H, *J* 7.3 Hz), 7.07 (d, 3H, Ar–H, *J* 8.3 Hz), 6.77 (d, 6H, Ar–H, *J* 7.3 Hz), 11.93 (s, 3H, N–H). ^{13}C NMR (100 MHz, DMSO–d_6, 25 °C) (δ: ppm): 181.83, 154.34, 141.52,



Scheme 1. The synthesis of the tripodal-benzimidazoles.

Table 1
Decomposition steps with the temperature range and weight loss for tripodal-benzimidazoles.

Compound	Temperature range (°C)	Weight loss found (Calcd.) (%)	Fragment
$C_{42}H_{27}N_9O_3$ (**2**)	250–345 355–675	49.66 (49.78) 39.05 (39.15)	H_2, N_2, C_6H_6, CO
$C_{42}H_{24}Cl_3N_9O_3$ (**3**)	240–343 350–680	56.08 (56.13) 24.14 (24.20)	Cl_2, H_2, N_2, C_6H_6, CO
$C_{45}H_{33}N_9O_3$ (**4**)	245–367 375–695	52.55 (52.61) 36.85 (36.94)	CH_4, N_2, C_6H_6, CO
$C_{42}H_{24}N_{12}O_9$ (**5**)	220–375 375–685	57.80 (57.85) 32.78 (32.85)	NO_2, H_2, N_2, C_6H_6, CO

137.86, 134.95, 132.12, 128.02, 123.24, 121.61, 116.12, 115.34, 20.86.

Data for (**5**). Yield: 63%; m.p.: 195 °C; Elemental analysis (Found: C, 60.11; H, 2.97; N, 19.88%). Calc. for $C_{42}H_{24}N_{12}O_9$: C, 60.00; H, 2.88; N, 19.99%. IR (KBr) ν_{max}/cm^{-1}: 3419 (N–H), 2926 (C–H_{Ar}), 1569 (C=N), 1501 (C=$N_{triazine}$), 1211 (C–N), 1367 (C_{Ar}–O–C), 1558 (NO_2). ^{1}H NMR (400 MHz, DMSO–d_6, 25 °C) (δ: ppm): 7.78 (s, 3H, Ar–H), 7.71 (d, 3H, Ar–H, *J* 8.6 Hz), 7.38 (d, 6H, Ar–H, *J* 7.4 Hz), 7.32 (d, 3H, Ar–H, *J* 8.6 Hz), 6.82 (d, 6H, Ar–H, *J* 7.4 Hz), 12.42 (s, 3H, N–H). ^{13}C NMR (100 MHz, DMSO–d_6, 25 °C) (δ: ppm): 181.92, 154.52, 141.73, 139.45, 136.12, 132.54, 128.33, 123.41, 121.76, 116.91, 115.88.

3. Results and discussions

The synthesis of TRIPOD and compounds **2**–**5** were given in Scheme 1. In the ^{1}H NMR spectra of compounds **2**–**5**, the signals were detected at about 12.15, 12.33, 11.93 and 12.42 ppm, respectively. All signals appeared as broad singlets and were attributed to the N–H in the tripodal-benzimidazole [28]. A broad correlation can be observed between the donor or acceptor properties of the group attached to 5-position of the tripodal-benzimidazole nucleus and the chemical shift of the nitrogen proton of the tripodal-benzimidazole ring in compounds **3** and **5**. The presence of N–H was also identified by FT-IR spectroscopy as a sharp band at about 3362–3435 cm^{-1} [28]. Also, the vibrations of the triazine C=N of compounds **2**–**5** were observed at 1567–1560 cm^{-1} range [16,17].

Thermal stabilities of compounds **2**–**5** were investigated and their plausible degradation [29] schemes were presented in Table 1. Also, the decomposition change for compound **2** was given in Fig. 1. Thermal decomposition of the anhydrous compounds starts in the range of 220–495 °C and completes in the range 550–650 °C. The observed weight losses for all compounds are in good agreement with the calculated values.

It can be evaluated a communication between the electron donating and acceptor termini by comparing the maximum absorbance (λ_{max}) values [28]. The UV-vis absorption spectra of compounds **2**–**5** were recorded in DMF at room temperature and showed in Fig. 2. The λ_{max} values of compounds **2**–**5** were obtained as 310, 313, 314 and 341 nm, respectively. As can be seen, while the differences of λ_{max} for compounds **2**–**4** are nearly negligible, that of compound **5** is noteworthy. The presence of nitro group in the structure leads to the bathochromic shift in the electronic spectra of compound **5**. The shift was attributed to more extensive electron delocalization [30].

The cyclic voltammograms (CVs) of compounds **2**–**5** were obtained in DMF solution. The CVs of compounds **2**, **3** and **4** are more or less similar with minor variation in the position and appearance, whereas that of compound **5** strongly shows cathodic wave having different characteristic in the negative range. For this reason, the electrochemical behaviors of compound **2** and **5** are shown in Fig. 3a and b, respectively.

As can be seen in Fig. 3a, the CV of compound **2** has a reduction peak at −1.64 V, without any cathodic peak at reverse scan, indicating that the peak is electron transfer processes having irreversible nature. The cathodic peak can be attributed to electrochemical cleavage of π-bonding in triazine ring [31]. On the other hand, at the anodic side, the CV showed very weak wave near the limit of anodic range, but the nature of the peak could not be satisfactorily evaluated by analyzing the cyclic voltammetric data.

In Fig. 3b, in addition to these peaks, we have obtained very strongly reduction peak for compound **5** at the negative potential

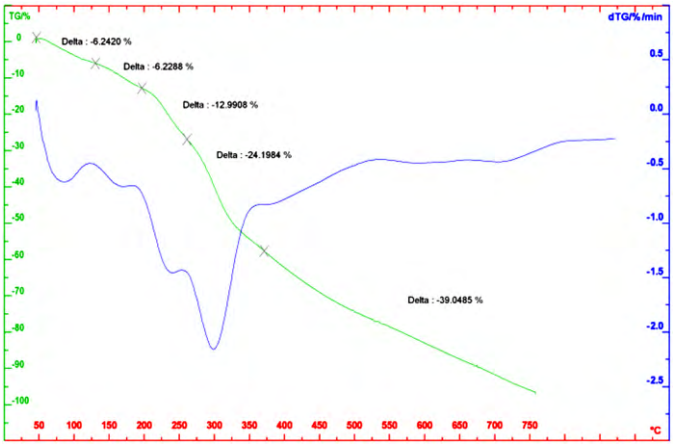


Fig. 1. The thermal decomposition for compound **2**.

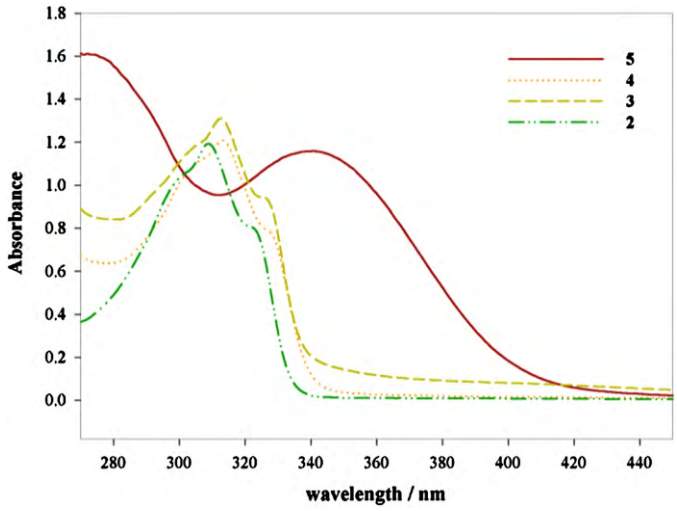


Fig. 2. UV–vis absorption spectra of compounds **2**–**5** in DMF.

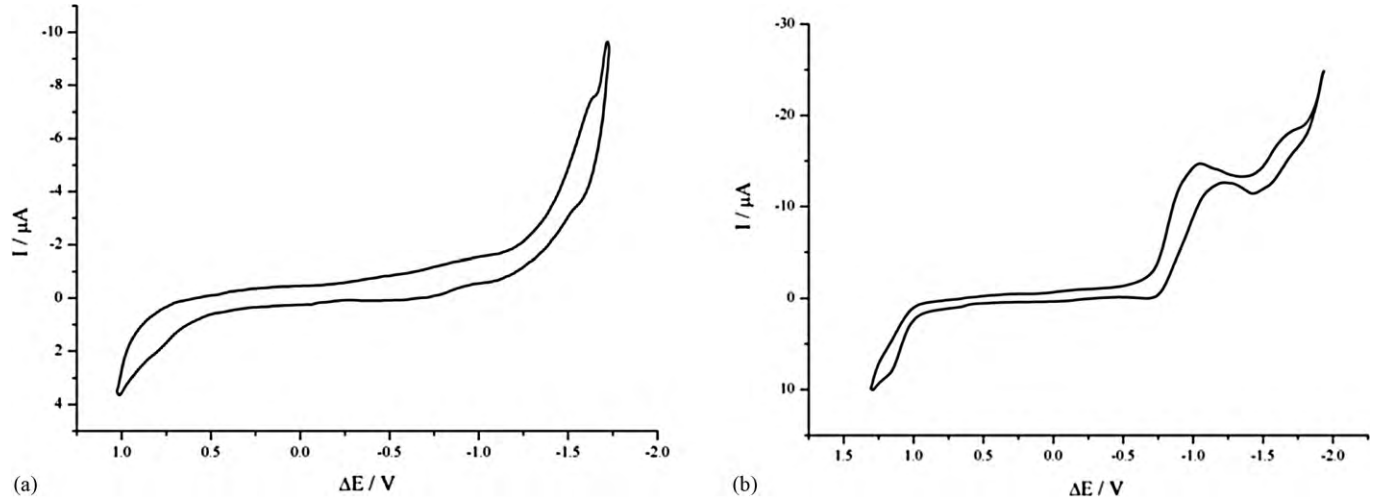


Fig. 3. Cyclic voltammograms (CVs) of compound **2** (a) and **5** (b) at the experimental conditions; 1 mM compound in 0.1 M [NBu_4] [PF_6]–DMF solution; $\nu = 50\,mV\,s^{-1}$.

side ($E_{pc} = -1.05$ V), in agreement with data obtained in the literature for the reduction of the nitro groups present in the structure [32,33]. As shown in Eq. (1), this peak is attributed to an electron transfer leading to the formation of a nitro radical anion generated by the peripheral p-nitrophenyl groups [33].

$$R\text{–}NO_2 + e^- \rightarrow R\text{–}NO_2^-. \quad (1)$$

Scan rate dependency experiments showed that the peak current is linearly dependent on the square root of the scan rate. Also, the peaks shifted to more negative values with increasing scan rate. Moreover, it was observed that the cathodic peak is changed with the concentration of compound **5**. These results indicate that this cathodic peak (E_{pc}) has electrochemically irreversible nature [34]. For the irreversible electron transfer, these results can be also evaluated according to the following equation [35]:

$$\alpha = \frac{1.857RT}{nF(E_{pc} - E_{pc/2})} \quad (2)$$

where $E_{pc/2}$ is the half cathodic peak potential and n is the total number of electrons involved in the reaction. The value of α was found to be 0.29. Thus, the value indicates the electron transfer process has irreversible nature. The data of UV–vis absorption and cyclic voltammetry were tabulated in Table 2.

The antibacterial activities of compounds **2**–**5** to be tested were dissolved in DMF to final concentration of 0.1%. The results of antibacterial studies are given in Fig. 4. Compound **3** and **5** exhibited antibacterial activity against *S. aureus* and *B. subtilis* higher than compound **2** and **4**. In addition, all compounds exhibited no activities against *S. typhimurium* in the DMF medium.

Table 2
Electronic absorption and cyclic voltammetric data for the compounds **2**–**5**.

Compound	λ_{max} [a] (nm)	Reduction potentials[b] (V)	
		E_{pc1} [c]	E_{pc2} [c]
2	310	–	−1.64
3	313	–	−1.65
4	314	–	−1.62
5	341	−1.05	−1.69

[a] Conditions: 5.0×10^{-5} M in DMF solution.
[b] Conditions: 1 mM compounds in DMF/0.1 M [NBu_4] [PF_6], Pt-disk electrode *vs.* Ag/Ag^+ reference electrode; $\nu = 50\,mV\,s^{-1}$ (see Section 2 for further details).
[c] Peak potentials for chemically irreversible process.

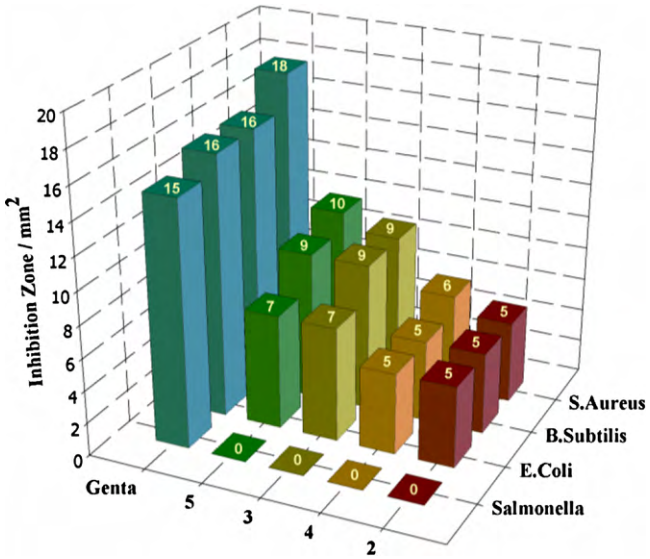


Fig. 4. Antimicrobial activities of **2**–**5** in DMF media.

4. Conclusions

In this study, compound **1** was synthesized by the reaction of cyanuric chloride and 4-hydroxybenzaldehyde according to the literature. Compounds **2**–**5** were originally synthesized using the methods described in the literature by condensation of **1** with o-phenylenediamine, 4-methyl-o-phenylenediamine, 4-chloro-o-phenylenediamine and 4-nitro-o-phenylenediamine. The structures of the obtained octupolar compounds were identified by FT-IR, ^1H NMR, ^{13}C NMR and UV–vis spectral data, thermal analysis and elemental analysis. Also, the electrochemical behaviors of compounds **2**–**5** were obtained by using cyclic voltammetry and compared with previous studies in the literature. As a consequence, the antibacterial activities of compounds **2**–**5** were observed in vitro against selected two gram-positive organisms and two gram-negative organisms.

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