

Research Article

Synthesis and Antimicrobial Evaluation of Some Novel Diazo Derivative of Cyclic Imides Using Diazotization Coupling Reaction

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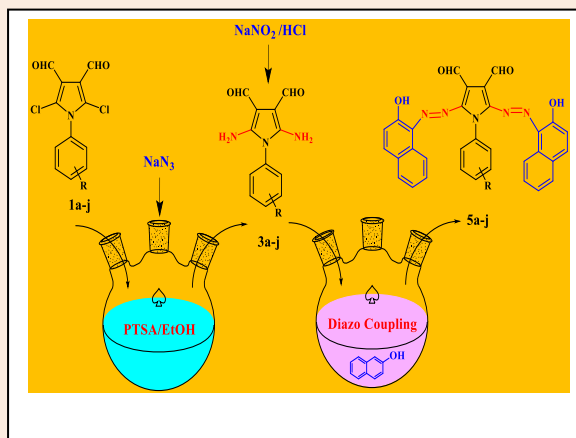
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Abstract

A series of new azo coupled derivative of N-substituted cyclic imides were prepared by diazotization-coupling reaction. All the compounds were screened for their antimicrobial and antifungal activities. Most of the synthesized compounds have shown significant antimicrobial activity. Based on our experimental findings, the new substituted azovinyl aldehyde derivatives containing cyclic imide moiety exhibiting excellent bactericidal and fungicidal potentials could be proposed for dyeing and antimicrobial finishing for silk, wool, cotton, and polyester fabrics. The structures of these compounds were confirmed by various analytical tools.

Keywords: azo coupling, diazotization, coupling reaction, cyclic imides, antimicrobial activity



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Introduction

The structural diversity and industrial as well as biological importance of nitrogen and oxygen-containing heterocycles have made them attractive targeted for synthesis over many and justify continuing efforts in the development of new synthetic strategies [1]-[3]. From last few decades, the study of the chemistry of diazo compound has been given particular impacts because of their application in pigments [4]-[9], lacks [10] and dyes [11]. It has been explored and being developed as a dyeing [12] and coloring agent [13] for the textile industries [14]. Now a day chemistry of diazo derivative synthesized using heterocyclic ring [15] exhibit a new aspect of coupling reaction [16]. Therefore, some novel diazo cyclic imides were synthesized from halo vinyl aldehyde derivative of cyclic imides. The series of reaction were carried out over halo vinyl aldehyde to synthesized diazo coupled product.

After the extensive literature search [17]-[25], it was observed that cyclic imides [26], halo vinyl aldehyde [27] and azo compounds [28] are the important pharmacophores, but till date enough efforts have not made to combine these three moieties as a single molecular scaffold. So, our objective was to synthesize and biological screening of a series of new compound incorporates these moieties.

Experimental

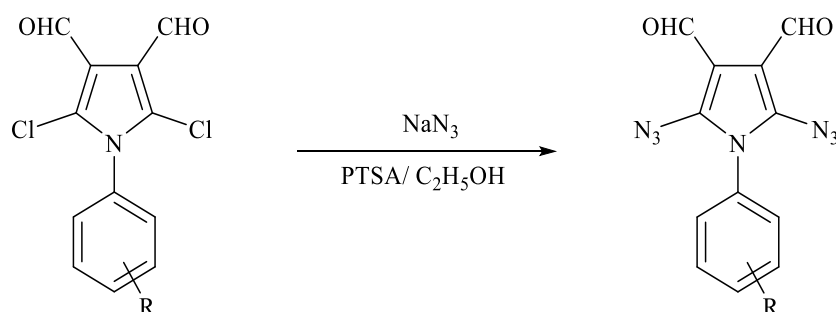
Materials and Reagents

The Melting points of all the synthesized compounds were recorded in open glass capillaries and were uncorrected. IR spectra were recorded on Lambda 7600 FTIR spectrophotometer. ¹H NMR spectra were recorded on Bruker 400 MHz in DMSO-d₆ using TMS as an internal standard. The reaction was monitored by TLC which was accomplished by using pre-coated silica gel aluminum plates with the mixture of hexane: ethyl acetate as a solvent phase. Chemical purchased were used as received.

General procedure for synthesis

2,5-diazido-1-substitutedphenyl-1H-pyrrole-3,4-dicarbaldehyde (2a-j)

In continuation of our previous work [29]-[33] a solution of 2,5-diazido-1-substitutedphenyl-1H-pyrrole-3,4-dicarbaldehyde **1a-j** (0.01 moles) in absolute ethanol (10 mL), P-toluene sulphonic acid (0.02 moles) and sodium azide (0.03 moles) were added and reaction mixture heated under reflux for time ranging between 4-6 hrs (**Scheme 1**). The refluxed mixture was added to ice cold water which precipitated compounds **2a-j**. These were filtered and recrystallized from ethanol.

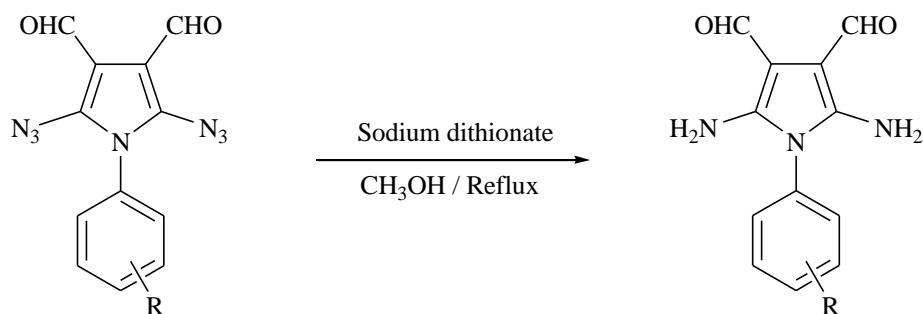


Where R a = 4H, b = 3Cl, c = 4Cl, d = 4Br, e = 2NO₂, f = 3NO₂, g = 4NO₂, h = 4OH, i = 4CH₃, j = 4OCH₃

Scheme 1 Synthesis of 2,5-diazido-1-substitutedphenyl-1H-pyrrole-3,4-dicarbaldehyde

2,5-diamino-1-substitutedphenyl-1H-pyrrole-3,4-dicarbaldehyde (3a-j)

The mixture of compounds **2a-j** (0.026 moles), sodium dithionite (0.054 moles) and methanol (12mL) was refluxed for 5 hrs (**Scheme 2**). The reaction mixture was filtered and the inorganic residues were washed with methanol. The combined methanolic solution was distilled and poured over crushed ice. The resultant solids **3a-j** was filtered washed with water dried and recrystallized using ethanol as a solvent.



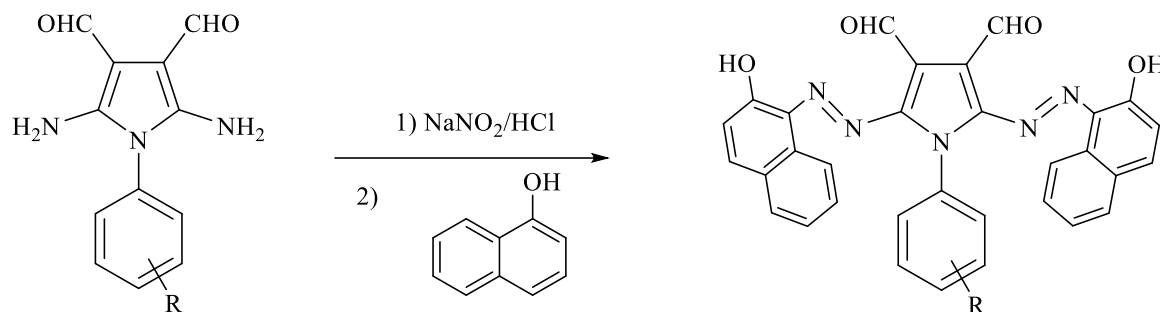
Where R a = 4H, b = 3Cl, c = 4Cl, d = 4Br, e = 2NO₂, f = 3NO₂, g = 4NO₂, h = 4OH, i = 4CH₃, j = 4OCH₃

Scheme 2 2,5-diamino-1-substitutedphenyl-1H-pyrrole-3,4-dicarbaldehyde

2,5-bis((E)-(2-hydroxynaphthalen-1-yl)diazenyl)-1-substitutedphenyl-1H-pyrrole-3,4-dicarbaldehyde (5a-j)

Solution A was prepared by mixing **3a-j** (0.01 mol) with concentrated HCl (6 mL) and water (6 mL) and cooling at a temperature below 5 °C in an ice bath. NaNO₂ (0.02 mol) was dissolved in water (20 mL) at 5 °C to obtain solution B. Then solution A was added dropwise to solution B at 5 °C with stirring. The mixture was then slowly added into the solution of 2-naphthol **4** (0.02 mol), which was dissolved in 10% NaOH (40 mL) at 5 °C. The mixture was kept chilled

in the ice bath and stirred continuously for 10 min (**Scheme 3**). The precipitate **5a-j** formed was filtered and recrystallized from glacial acetic acid, and washed with methanol and finally dried in a vacuum oven at 70 °C for 12 hours.



Where R a = 4H, b = 3Cl, c = 4Cl, d = 4Br, e = 2NO₂, f = 3NO₂, g = 4NO₂, h = 4OH, i = 4CH₃, j = 4OCH₃

Scheme 3 (2E,5E)-2,5-di(2-(naphtha-2-olen-1-yl)diazenyl)-1-substitutedphenyl-1H-pyrrole-3,4-dicarbaldehyde

2,5-bis((E)-(2-hydroxynaphthalen-1-yl)diazenyl)-1-phenyl-1H-pyrrole-3,4-dicarbaldehyde 5a:

Light brown; M. F.: C₃₂H₂₁N₅O₄; Mol. Wt.: 539.54; Percent yield: 77; Melting point (°C): 247-249; FTIR (cm⁻¹): 1695 (>C=O stretch, aldehyde), 2730 (H-C=O; C-H stretch), 3202 (C-H stretch, aromatics), 1660 (-C=C- stretch), 2902 (C-H stretch, aromatics), 1523 (C-C stretch, in ring aromatics), 3444 (O-H stretch, aromatic phenol), 1500 (-N=N- stretch); ¹HNMR (δ ppm): 9.7 (s, 2H, CHO), 5.2 (s, 2H, Ar-OH), 7.4 (s, 5H, Ar-H), 7.1 (m, 4H, Ar-H), 7.2-7.3 (m, 8H, Ar-H).

2,5-bis((E)-(2-hydroxynaphthalen-1-yl)diazenyl)-1-(3-chlorophenyl)-1H-pyrrole-3,4-dicarbaldehyde 5b:

Yellowish brown; M. F.: C₃₂H₂₀ClN₅O₄; Mol. Wt.: 573.99; Percent yield: 66; Melting point (°C): 236-238; FTIR (cm⁻¹): 1705 (>C=O stretch, aldehyde), 2827 (H-C=O; C-H stretch), 3367 (C-H stretch, aromatics), 1640 (-C=C- stretch), 3055 (C-H stretch, aromatics), 1570 (C-C stretch, in ring aromatics), 3505 (O-H stretch, aromatic phenol), 1485 (-N=N- stretch), 739 (C-Cl stretch); ¹HNMR (δ ppm): 9.3 (s, 2H, CHO), 5.3 (s, 2H, Ar-OH), 7.0 (d, 2H, Ar-H), 7.5 (d, 4H, Ar-H), 7.3 (s, 1H, Ar-H), 7.1-7.5 (m, 9H, Ar-H).

2,5-bis((E)-(2-hydroxynaphthalen-1-yl)diazenyl)-1-(4-chlorophenyl)-1H-pyrrole-3,4-dicarbaldehyde 5c:

Gray; M. F.: C₃₂H₂₀ClN₅O₄; Mol. Wt.: 573.99; Percent yield: 75; Melting point (°C): 257-259; FTIR (cm⁻¹): 1678 (>C=O stretch, aldehyde), 2717 (H-C=O; C-H stretch), 3280 (C-H stretch, aromatics), 1604 (-C=C- stretch), 3081 (C-H stretch, aromatics), 1585 (C-C stretch, in ring aromatics), 3445 (O-H stretch, aromatic phenol), 1455 (-N=N- stretch), 739 (C-Cl stretch); ¹HNMR (δ ppm): 9.9 (s, 2H, CHO), 4.9 (s, 2H, Ar-OH), 7.2 (d, 2H, Ar-H), 7.3 (d, 2H, Ar-H), 7.0 (d, 2H, Ar-H), 7.6 (m, 4H, Ar-H), 7.4-7.5 (m, 6H, Ar-H).

2,5-bis((E)-(2-hydroxynaphthalen-1-yl)diazenyl)-1-(4-bromophenyl)-1H-pyrrole-3,4-dicarbaldehyde 5d:

Light gray; M. F.: C₃₂H₂₀BrN₅O₄; Mol. Wt.: 618.44 ; Percent yield: 69; Melting point (°C): 216-218; FTIR (cm⁻¹): 1690 (>C=O stretch, aldehyde), 2712 (H-C=O; C-H stretch), 3240 (C-H stretch, aromatics), 1644 (-C=C- stretch), 3076 (C-H stretch, aromatics), 1479 (C-C stretch, in ring aromatics), 3595 (O-H stretch, aromatic phenol), 1480 (-N=N- stretch), 610 (C-Br stretch); ¹HNMR (δ ppm): 9.5 (s, 2H, CHO), 5.1 (s, 2H, Ar-OH), 7.2 (d, 2H, Ar-H), 7.4 (d, 2H, Ar-H), 7.0 (d, 2H, Ar-H), 7.6 (m, 4H, Ar-H), 7.3-7.5 (m, 6H, Ar-H).

2,5-bis((E)-(2-hydroxynaphthalen-1-yl)diazenyl)-1-(2-nitrophenyl)-1H-pyrrole-3,4-dicarbaldehyde 5e:

Dark brown; M. F.: C₃₂H₂₀N₆O₆; Mol. Wt.: 584.54; Percent yield: 60; Melting point (°C): 171-173; FTIR (cm⁻¹): 1713 (>C=O stretch, aldehyde), 2802 (H-C=O; C-H stretch), 3338 (C-H stretch, aromatics), 1685 (-C=C- stretch), 2990 (C-H stretch, aromatics), 1540 (C-C stretch, in ring aromatics), 3512 (O-H stretch, aromatic phenol), 1510 (-N=N- stretch), 1280 (N-O symmetric stretch), 1525 (N-O asymmetric stretch); ¹HNMR (δ ppm): 9.5 (s, 2H, CHO), 5.1 (s, 2H, Ar-OH), 7.0 (d, 2H, Ar-H), 7.2-7.3 (m, 4H, Ar-H), 7.5-7.7 (m, 9H, Ar-H), 8.2 (m, 1H, Ar-H).

2,5-bis((E)-(2-hydroxynaphthalen-1-yl)diazenyl)-1-(3-nitrophenyl)-1H-pyrrole-3,4-dicarbaldehyde 5f:

Brown; M. F.: C₃₂H₂₀N₆O₆; Mol. Wt.: 584.54; Percent yield: 66; Melting point (°C): 210-212; FTIR (cm⁻¹): 1680 (>C=O stretch, aldehyde), 2770 (H-C=O; C-H stretch), 3308 (C-H stretch, aromatics), 1670 (-C=C- stretch), 3110 (C-H stretch, aromatics), 1464 (C-C stretch, in ring aromatics), 3235 (O-H stretch, aromatic phenol), 1455 (-N=N- stretch), 1298 (N-O symmetric stretch), 1545 (N-O asymmetric stretch); ¹HNMR (δ ppm): 9.8 (s, 2H, CHO), 5.4 (s, 2H, Ar-OH), 7.1 (d, 2H, Ar-H), 7.2-7.3 (m, 4H, Ar-H), 7.5-7.6 (m, 8H, Ar-H), 8.2 (d, 2H, Ar-H).

2,5-bis((E)-(2-hydroxynaphthalen-1-yl)diazenyl)-1-(4-nitrophenyl)-1H-pyrrole-3,4-dicarbaldehyde 5g:

Dark brown; M. F.: C₃₂H₂₀N₆O₆; Mol. Wt.: 584.54; Percent yield: 76; Melting point (°C): 224-226; FTIR (cm⁻¹): 1682 (>C=O stretch, aldehyde), 2810 (H-C=O; C-H stretch), 3290 (C-H stretch, aromatics), 1700 (-C=C- stretch), 3072 (C-H stretch, aromatics), 1502 (C-C stretch, in ring aromatics), 3279 (O-H stretch, aromatic phenol), 1440 (-N=N- stretch), 1318 (N-O symmetric stretch), 1550 (N-O asymmetric stretch); ¹HNMR (δ ppm): 10.0 (s, 2H, CHO), 5.2 (s, 2H, Ar-OH), 6.9 (d, 2H, Ar-H), 7.2-7.3 (m, 4H, Ar-H), 7.5-7.6 (m, 8H, Ar-H), 8.3 (d, 2H, Ar-H).

2,5-bis((E)-(2-hydroxynaphthalen-1-yl)diazenyl)-1-(4-hydroxyphenyl)-1H-pyrrole-3,4-dicarbaldehyde 5h:

Bright Red; M. F.: C₃₂H₂₁N₅O₅; Mol. Wt.: 555.54; Percent yield: 71; Melting point (°C): 261-263; FTIR (cm⁻¹): 1708 (>C=O stretch, aldehyde), 2790 (H-C=O; C-H stretch), 3346 (C-H stretch, aromatics), 1637 (-C=C- stretch), 2995 (C-H stretch, aromatics), 1603 (C-C stretch, in ring aromatics), 3500, 3600 (O-H stretch, aromatic phenol), 1475 (-N=N- stretch); ¹HNMR (δ ppm): 9.8 (s, 2H, CHO), 5.0 (s, 1H, Ar-H), 5.3 (s, 2H, Ar-OH), 6.7 (d, 2H, Ar-H), 7.0 (d, 2H, Ar-H), 7.1 (d, 2H, Ar-H), 7.2-7.3 (m, 4H, Ar-H), 7.5-7.6 (m, 6H, Ar-H).

2,5-bis((E)-(2-hydroxynaphthalen-1-yl)diazenyl)-1-(p-tolyl)-1H-pyrrole-3,4-dicarbaldehyde 5i:

Reddish brown; M. F.: C₃₃H₂₃N₅O₄; Mol. Wt.: 553.57; Percent yield: 70; Melting point (°C): 266-268; FTIR (cm⁻¹): 1692 (>C=O stretch, aldehyde), 2823 (H-C=O; C-H stretch), 3261 (C-H stretch, aromatics), 1672 (-C=C- stretch), 3040 (C-H stretch, aromatics), 1490 (C-C stretch, in ring aromatics), 3550 (O-H stretch, aromatic phenol), 1500 (-N=N- stretch), 1450, 1355 (C-H bend and rock, aromatic alkyl); ¹HNMR (δ ppm): 9.7 (s, 2H, CHO), 5.0 (s, 2H, Ar-OH), 7.5 (d, 2H, Ar-H), 7.6 (d, 4H, Ar-H), 7.0-7.3 (m, 10H, Ar-H), 2.4 (s, 3H, Ar-CH₃).

2,5-bis((E)-(2-hydroxynaphthalen-1-yl)diazenyl)-1-(4-methoxyphenyl)-1H-pyrrole-3,4-dicarbaldehyde 5j:

Violet; M. F.: C₃₃H₂₃N₅O₅; Mol. Wt.: 569.57; Percent yield: 66; Melting point (°C): 230-232; FTIR (cm⁻¹): 1720 (>C=O stretch, aldehyde), 2698 (H-C=O; C-H stretch), 3390 (C-H stretch, aromatics), 1690 (-C=C- stretch), 3121 (C-H stretch, aromatics), 1585 (C-C stretch, in ring aromatics), 3400 (O-H stretch, aromatic phenol), 1482 (-N=N- stretch), 1469, 1370 (C-H bend and rock, alkyl); ¹HNMR (δ ppm): 9.8 (s, 2H, CHO), 4.9 (s, 2H, Ar-OH), 6.8 (d, 2H, Ar-H), 7.0 (d, 2H, Ar-H), 7.2-7.3 (m, 6H, Ar-H), 7.5 (d, 2H, Ar-H), 7.6 (d, 4H, Ar-H), 3.8 (s, 3H, Ar-OCH₃).

Results and Discussion

Chemistry:

The starting compounds of azo vinyl aldehyde **5a-j** were prepared by the reaction of 2,5-diazo-1-substitutedphenyl-1H-pyrrole-3,4-dicarbaldehyde **2a-j** using sodium dithionite. Diazo coupling reaction were carried out over 2,5-diamino-1-substitutedphenyl-1H-pyrrole-3,4-dicarbaldehyde **3a-j** gives the diazonium salt, followed by the coupling reaction using 2-naphthol. The series of 2,5-bis((E)-(2-hydroxynaphthalen-1-yl)diazenyl)-1-(substituted phenyl)-1H-pyrrole-3,4-dicarbaldehyde **5a-j** were synthesized in reasonable yields. The structure of azo vinyl was confirmed by FT-IR and ¹HNMR analysis.

Antimicrobial susceptibility test (5a-j):

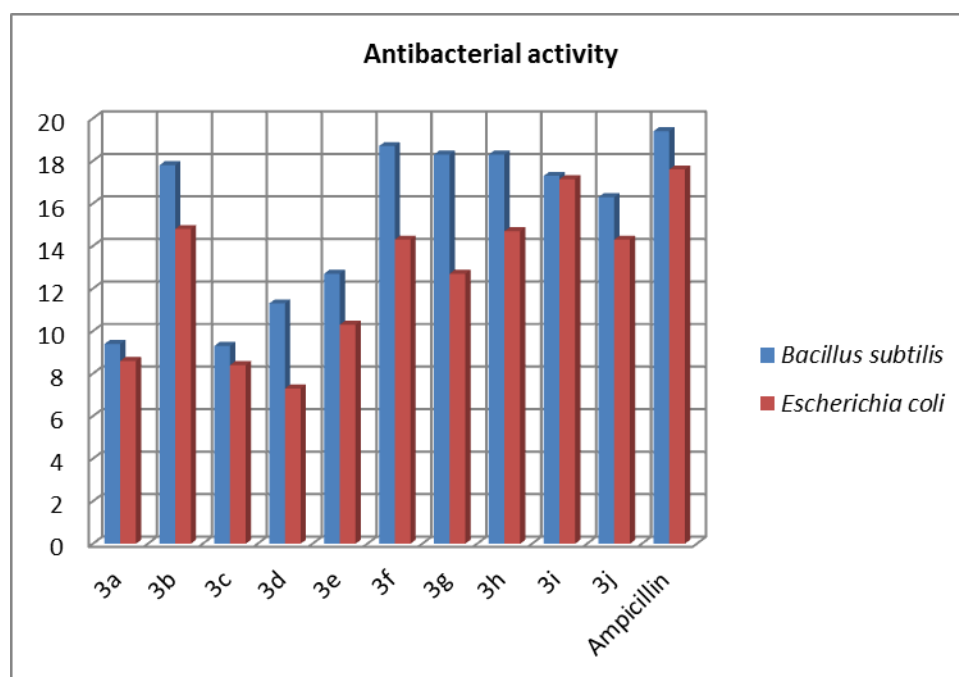
The disc diffusion method was used to screen the antimicrobial activity. In vitro antimicrobial activity was screened by using Mueller Hinton Agar (MHA) obtained from Hi-media. The MHA plates were prepared by pouring 15 mL of molten media into sterile Petri plates. The plates were allowed to solidify for 5 minutes and 0.1 % inoculum suspension was swabbed uniformly and the inoculum was allowed to dry for 5 minutes. The fix concentrations were loaded on 6 mm sterile disc. The loaded disc was placed on the surface of the medium and the compound was allowed to diffuse for 5 minutes and the plates were kept for incubation at 37°C for 24 hrs. At the end of incubation, inhibition

zones formed around the disc were measured with the transparent ruler in millimeter. All the synthesized compounds **5a-j** were screened for their antibacterial activity against gram- positive bacteria *Bacillus subtilis* (MCMB-310) and gram negative bacteria *Escherichia coli* (MCMB-301) using DMF solvent. Ampicillin was used as standard and results were shown in the **graph 1**. The same procedure was followed for the fungus using Potato Dextrose Agar (PDA) as a nutrient medium. The antifungal activities against *Candida albicans* (NCIM-3471) and *Aspergillus niger* (NCIM- 545) strains using DMSO solvent in using Amphotericin-B as a standard revealed in the **graph 2**. All the results of the synthesized compounds were carried out by the triplicate format N=3 with Mean \pm SD. The calculated data were tabulated in **Table 1**;

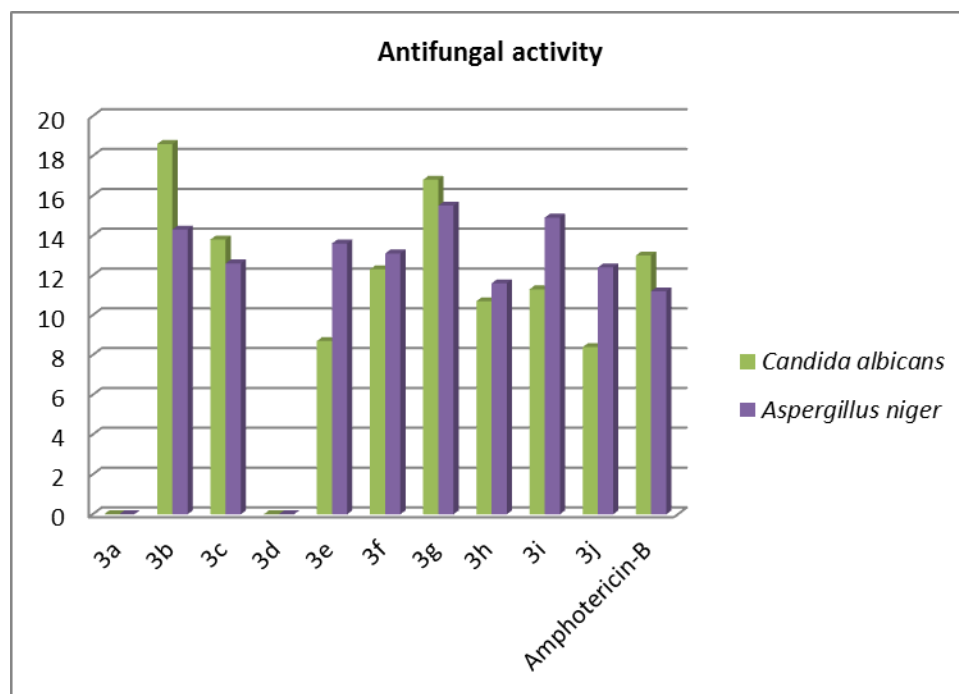
Table 1 Antimicrobial activity of synthesized diazo compound

Entry	Zone diameter in mm (Mean \pm S.D.)			
	<i>Bacillus subtilis</i>	<i>Escherichia coli</i>	<i>Candida albicans</i>	<i>Aspergillus niger</i>
3a	9.38 \pm 0.24	8.55 \pm 0.28	--	--
3b	17.76 \pm 1.25	14.84 \pm 0.61	18.57 \pm 0.27	14.27 \pm 0.49
3c	9.33 \pm 1.22	8.43 \pm 0.57	13.83 \pm 0.22	12.56 \pm 0.78
3d	11.33 \pm 0.13	7.26 \pm 1.15	--	--
3e	12.66 \pm 0.33	10.32 \pm 0.52	8.69 \pm 0.39	13.63 \pm 0.39
3f	18.66 \pm 0.5	14.33 \pm 0.53	12.34 \pm 0.34	13.15 \pm 3.76
3g	18.33 \pm 0.55	12.66 \pm 0.23	16.77 \pm 0.40	15.45 \pm 0.39
3h	18.33 \pm 0.54	14.66 \pm 1.04	10.68 \pm 0.22	11.65 \pm 1.13
3i	17.33 \pm 0.37	17.11 \pm 0.03	11.30 \pm 0.32	14.95 \pm 3.20
3j	16.33 \pm 0.57	14.33 \pm 0.57	8.40 \pm 0.04	12.37 \pm 0.64
Ampicillin	19.36 \pm 0.04	17.63 \pm 0.06	--	--
Amphotericin-B	--	--	12.98 \pm 0.44	11.38 \pm 0.54

Keynote: Zone of inhibition measured in mm (Mean \pm S.D.) (N=3) ('--' means no zone of inhibition)



Graph 1 Antibacterial activities of 5a-j



Graph 2: Antifungal activities of 5a-j

Conclusions

An entire new series of 2,5-bis((E)-(2-hydroxynaphthalen-1-yl)diazenyl)-1-(substituted phenyl)-1H-pyrrole-3,4-dicarbaldehyde **5a-j** have been synthesized in facile manner from 2,5-diazido-1-substitutedphenyl-1H-pyrrole-3,4-dicarbaldehyde **1a-j** in good yield. Based on our experimental findings, the new substituted azovinyl aldehyde derivatives containing cyclic imide moiety **5a-j** exhibiting excellent bactericidal and fungicidal potentials could be proposed for dyeing and antimicrobial finishing for silk, wool, cotton, and polyester fabrics.

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