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Synthesis, Characterisation And Antimicrobial Activities of Schiff Bases containing 1,3,4-oxadiazoleMoiety

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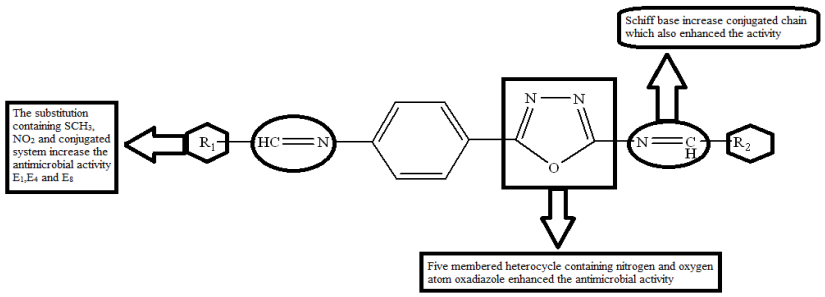
ABSTRACT

In present work PABA A reacted with substituted aldehyde B to given schiff based 4-(substitutidene amino)benzoic acid C. Which further reacted with semicarbezide and substituted aldehyde D to obtained cyclic product and schiff base as final moiety N-(4-(5-(substitutideneamino)-1,3,4-oxadiazole-2-yl) phenyl) substituedimine E. The synthesized molecules were spectrally evaluated by IR, ^{1}H NMR, ^{13}C NMR and Mass spectra. Synthetic analogues screened for their antimicrobial activities. Among the tested compounds, E_8 displayed very good antibacterial activity against E. coli and S. aureus strain. E_4, E_5 and E_8 showed very good antibacterial activity against P. aeruginosa strain. E_1 has given good antifungal activity against C. albicans species.E_7 and E_8 observed good antifungal activity against A. niger and A. clavatus species.

Graphical Abstract:



Synthesis of N-(4-(5-(substitutideneamino)-1,3,4-oxadiazole-2-yl)phenyl) substituedimine.

Keywords: Oxadiazole, schiff base, antibacterial and antifungal.

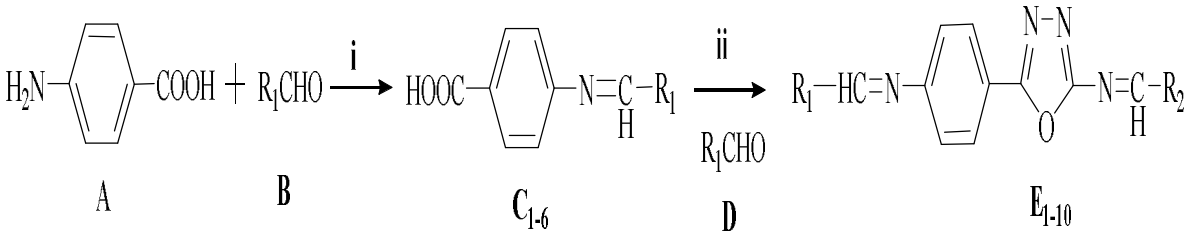
INTRODUCTION

Oxadiazole, a heterocyclic molecule containing one oxygen and two nitrogen atoms in a five-membered ring [1]. Which has attracted a wide attention for Researcher as a new therapeutic agent. Several methods have been reveals in the literature for the synthesis of 1,3,4-oxadiazoles and out of various isomers particularly 1,3,4-oxadiazole derivatives exhibit wide range of biological activities. Also various route for

the synthesis of 1,3,4-oxadiazole have been reported[2-7]. Acid hydrazides [8] have been in general use as the starting materials in some 1,3,4-oxadiazole.

Researchers have already reported that gram positive bacteria are much more susceptible to antimicrobial agents as compared to gram negative bacteria. These differences may be attributed to the fact that the cell wall in gram positive bacteria is of single layer whereas the gram-negative bacteria have multilayered cell wall. 1,3,4-oxadiazoles have significant pharmaceutical interest. It associated considerable biological activities such as antifungal [9], antibacterial [10], analgesic [11-13], antitubercular [14], cytotoxicity [15] and antioxidant agent [16,17].

Schiff bases, known as Imines are compounds containing azomethine group -(HC=N)- which imports in elucidating the mechanism of transformation and racemization reaction in biological system [18] and represented by the general formula R3R2C=NR1. They are the condensed products of aldehydes or ketones and were first reported by Hugo Schiff in 1864 [19]. Originally, the classical synthetic route for synthesis of Schiff bases was reported by Schiff which involves condensation of primary amines with carbonyl compounds [20] under azeotropic distillation with the removal of water. Interests in these compounds are largely due to their relatively simple procedures of synthesis as well as significant biological activity. They are well known intermediate for the preparation of azetidinone, thiazolidinone, arylacetamide, metal complexes and many other derivatives [21]. Schiff's bases have been playing vital roles in pharmaceuticals, rubber additives, as amino protective groups in the synthetic organic chemistry and several biologically active organic compounds. They are also used as liquid crystals in analytical, medicinal, and polymer chemistry [22]. The Schiff bases possessing diversified biological applications such as antitubercular, anticancer, antibacterial, antifungal, antitumor, anthelmintic, anti-HIV, antiparasitic activities [23, 24].



Scheme 1. Synthesis of N-(4-(5-(substitutideneamino)-1,3,4-oxadiazole-2-yl) phenyl) substituedimine. Experimental conditions: (i)Methanol, 2-3 drops acetic acid, 2-4 h heating with starring; (ii) Methanol, Semicarbazide, 6-8 h reflux; $4R_1$ = $-C_8H_7$, $-C_6H_4$-4-C_3H_7, $-C_4H_3S$, $-C_6H_4$-SCH_3, $-C_6H_4$-4-NO_2, $-C_4H_3O$; $4R_2$ = $-C_5H_4N$, $-C_4H_3O$, $-C_6H_4$-SCH_3, $-C_6H_4$-4-C_3H_7, $-C_8H_7$.

MATERIALS AND METHODS

All the chemicals were used as of analytical grade. Melting points were determined on a Fisher–Johns Melting Point apparatus. A purity of the compounds was checked by TLC on silica gel plates with visualization by UV-light and iodine chamber. ^{1}H-NMR and ^{13}C-NMR spectra were recorded on Bruker Avance II 400 MHz spectrometer instruments in DMSO-d6 and chemical shifts were recorded in parts per million downfield from tetramethylsilane. Mass spectra were recorded on LC-MS Aglilent 1100 series.

General synthesis of 4-(substitutideneamino) benzoic acid (C_{1-6}): Equimolar solution of 4-amino benzoic acid (A) (0.1mol) and cinnamaldehyde aldehyde (B_1) (0.1mol) in ethanol taken in a Borosil conical flask. Then add few drops of acetic acid as catalyst. Put the reaction on electric starrer for starring with heating till precipitate obtained [25]. The progress of the reaction was monitored by TLC using n-hexane: ethyl acetate (7:1), after every 30 min. On completion of the reaction, the reaction mixture was

cooled at room temperature and was poured into ice. The product was washed by distil water. It was dried and purified by recrystallization from ethanol to obtained analytical sample (C_1).Other compounds (C_{2-6}) were prepared by same method.

Table 1. Physical data of compounds (C_{1-6})

Sr. No.	Code	Yield□	M.P.^{0}C	M.F.
1.	C_1	84	153	$C_{16}H_{13}O_2N$
2.	C_2	82	146	$C_{17}H_{17}O_2N$
3.	C_3	87	130	$C_{12}H_9O_2NS$
4.	C_4	78	142	$C_{15}H_{15}O_2NS$
5.	C_5	85	159	$C_{14}H_{12}O_4N_2$
6.	C_6	83	134	$C_{12}H_9O_3N$

General Synthesis of N-(4-(5-(substitutideneamino)-1,3,4-oxadiazole-2-yl) phenyl) substitutedimine (E_{1-10}): To a mixture of compound (C_1) (0.01mol), semicarbazide (0.012mol) and Pyridine 2-carboxaldehyde (D_1) in ethanol in a Borosil conical flask was put on starrer for starring with heating at 90^{0}C for 5-6 h [26].The TLC was checked after 1.5 h. After completion of reaction cooled the reaction mixture at room temperature and then pour over ice cold water. The precipitate obtained was wash with water followed by distilled water. Then precipitated was crystallised by ethanol to make pure product (E_1) as analytical sample. Other compounds (E_{2-6}) were prepared by same method.

Table 2. Physical data of compounds (E_{1-10})

Sr. No.	Code	Yield□	M.P.^{0}C	M.F.
1.	E_1	62	254	$C_{23}H_{17}ON_5$
2.	E_2	68	237	$C_{23}H_{20}O_2N_4$
3.	E_3	54	224	$C_{21}H_{16}ON_4S_2$
4.	E_4	56	220	$C_{26}H_{24}ON_4$
5.	E_5	51	194	$C_{25}H_{21}O_3N_5$
6.	E_6	64	247	$C_{19}H_{13}ON_5$
7.	E_7	58	217	$C_{21}H_{15}O_2N_4$
8.	E_8	69	204	$C_{25}H_{20}ON_4$
9.	E_9	73	242	$C_{20}H_{13}O_4N_5$
10.	E_{10}	57	191	$C_{24}H_{17}O_3N_5$

RESULTS AND DISCUSSION

Synthetic steps given in a scheme in which all reaction steps completion was checked by TLC using sufficient solvents. Products obtained at different steps were purified by crystallisation using particular solvents. All intermediates were characterised by IR and ^{1}H NMR. Final products were identified by IR, ^{1}H NMR, ^{13}C NMR and Mass spectral studies. All final moieties were screened for antimicrobial activity.

Spectral characterization of compounds

C_5: IR (KBr) *v* cm^{-1}: 3058 (OH), 1710 (C=O), 1616 (C=N); ^{1}H NMR (DMSO-d_6) δ (ppm): 10.47 (s, 1H, OH), 8.73 (s, 1H, =CH), 7.65-7.15 (m, 8H, Ar-H).

C_6: IR (KBr) *v* cm^{-1}: 3062 (OH), 1696 (C=O), 1604 (C=N); ^{1}H NMR (DMSO-d_6) δ (ppm): 10.15 (s, 1H, OH), 8.56 (s, 1H, =CH), 7.75-7.17 (m, 8H, Ar-H).

E_1: IR (KBr) *v* cm-1: 2976, 2887 (CH), 1670 (C=C), 1645 (N-N), 1598 (C=N), 1092 (C-O-C); ^{1}H NMR (DMSO-d_6) δ (ppm): 8.75 (d, 1H, Ar-H pyridine), 8.53 (s, 1H, =CH), 8.06 (s, 1H, =CH), 7.87-7.66, (m, 3H, Ar-H pyridine), 7.52-7.26, (m, 9H, Ar-H), 7.18 (d, 1H, =CH), 6.65 (t, 1H, =CH).^{13}C NMR(100MHz, DMSO-d_6) δ (ppm): 163.83, 162.86 (=CH), 158.65 (C_5, Pyridine), 156.80 (C=N, Oxadiazole), 153.14 (C_2, Pyridine), 149.44 (aromatic ring), 146.71 (C_3, pyridine), 142.96 (C_1, pyridine), 139.62 (C=N, Oxadiazole),

136.25 (C=C), 128.85, 126.29, 122.69, 118.22, 114.35, 111.05, (aromatic ring), 104.52 (C=C); ESI–MS: m/z calculated 379.42, found $[M+H]^+$ 379.19.

E_2: IR (KBr) *v* cm-1: 3157, 3034 (CH_3), 2863 (CH_2), 1648 (N-N), 1595 (C=N), 1093 (C-O-C);); ^{1}H NMR (DMSO-d_6) δ (ppm): 8.53 (s, 1H, =CH), 8.06 (s, 1H, =CH), 7.83-7.18 (m, 8H, Ar-H), 6.87-6.53 (m, 3H, Ar-H furfural),2.87 (t, 2H, CH_2), 1.64 (m, 2H, CH_2), 1.18 (d, 3H, CH_3); ^{13}C NMR(100MHz, DMSO-d_6) δ (ppm): 158.65 (=CH), 156.80 (C=N, Oxadiazole), 153.13 (=CH), 149.44 (aromatic ring), 146.71 (C_4, furfural), 142.96 (aromatic ring), 139.62 (C=N, Oxadiazole), 136.25, 128.85, 122.69(C_2, furfural), 118.23(C_3, furfural), 114.35, 111.05, 100.46 (aromatic ring), 40.95 (CH_2), 25.54 (CH_2), 13.49 (CH_3); ESI–MS: m/z calculated 384.43, found $[M+H]^+$ 384.56.

E_3: IR (KBr) *v* cm-1: 3065 (CH_3), 1646 (N-N), 1602 (C=N), 1139 (C-S), 1092 (C-O-C), 814 (C-S-C); ^{1}H NMR (DMSO-d_6) δ (ppm): 8.80 (s, 1H, =CH), 8.36 (s, 1H, =CH), 7.71-7.21 (m, 8H, Ar-H), 7.17-6.84 (m, 3H, Ar-H thiophene), 2.38 (s, 3H, CH_3).^{13}C NMR(100MHz, DMSO-d_6) δ (ppm): 160.00 (=CH), 157.23 (C=N, Oxadiazole), 152.86 (=CH), 148.24 (aromatic ring), 144.34 (C_1, thiophene), 142.96 (aromatic ring), 138.56(C=N, Oxadiazole), 132.25, 130.14(C_2, furfural), 129.41, 128.85, 128.50(C_4, furfural), 127.23(C_3, furfural), 126.92, 126.29, 124.82, 122.37, 114.35, 111.05, (aromatic ring), 14.87 (SCH_3); ESI–MS: m/z calculated 372.45, found $[M+H]^+$ 372.78.

E_4: IR (KBr) *v* cm-1: 3183, 3068 (CH_3), 2870 (CH_2), 1646 (N-N), 1603 (C=N), 1139 (C-S), 1093 (C-O-C); ^{1}H NMR (DMSO-d_6) δ (ppm): 8.63 (s, 1H, =CH), 8.14 (s, 1H, =CH), 7.62-7.18 (m, 12H, Ar-H), 2.78 (t, 2H, CH_2),2.27 (s, 3H, CH_3), 1.61 (m, 2H, CH_2), 1.14 (d, 3H, CH_3); ^{13}C NMR(100MHz, DMSO-d_6) δ (ppm): 158.46 (=CH), 156.67 (C=N, Oxadiazole), 152.93 (=CH), 149.21 (aromatic ring),143.26, 142.07 (aromatic ring), 139.51 (C=N, Oxadiazole), 136.25, 132.8,129.4, 128.85, 126.9 114.35, 111.05, (aromatic ring), 41.15 (CH_2), 25.72 (CH_2), 14.64 (SCH_3), 13.49 (CH_3); ESI–MS: m/z calculated 408.49, found $[M+H]^+$ 408.06.

E_5: IR (KBr) *v* cm-1: 3173, 3053 (CH_3), 2856 (CH_2), 1644 (N-N), 1608 (C=N), 1535, 1353 (NO_2), 1139 (C-S), 1091 (C-O-C); ^{1}H NMR (DMSO-d_6) δ (ppm): 8.59 (s, 1H, =CH), 8.10 (s, 1H, =CH), 7.68-7.18 (m, 12H, Ar-H),2.75 (t, 2H, CH_2), 1.66 (m, 2H, CH_2), 1.14 (d, 3H, CH_3); ^{13}C NMR(100MHz, DMSO-d_6) δ (ppm): 158.52 (=CH), 156.59 (C=N, Oxadiazole), 152.93 (=CH), 149.25 (aromatic ring),142.86, 141.74 (aromatic ring), 139.51 (C=N, Oxadiazole), 136.25, 132.78,129.36, 128.85, 126.9 114.35, 111.15, (aromatic ring), 41.26 (CH_2), 25.64 (CH_2), 13.49 (CH_3); ESI–MS: m/z calculated 439.49, found $[M+H]^+$ 439.78.

APPLICATIONS

The disc diffusion method was used for the screening of antimicrobial activity. Antibacterial activity of N-(4-(5-(substitutideneamino)-1,3,4-oxadiazole-2-yl) phenyl) substituedimine (E_{1-10}) was screened against two species *E. coli* and *P. Aeruginosa* Gram-positive bacteria *i.e.* two species of *S. aureus* and *S. pyogenes* as Gram-negative bacteria. The antifungal activity of the compounds was tested against two fungal organisms namely *C. albicans*, *A. niger* and *A. clavatus*. Antibacterial studies were compared against chloramphenicol, ciprofloxacin, norfloxacin and ampicillin as standard drugs. Nystatin and Griseofulvin were used for antifungal activity studies as reference compounds. From screened Compounds E_8 was found to be excellent active and E_1 good active against *E. coli*. Molecules E_4, E_5, and E_8 exhibited excellent activity and E_7 good activity against *P. aeruginosa*. Compounds showed excellent potency was E_8 and E_2, E_4, E_5, E_7 were good potency against *S. aureus*. Moieties E_1, E_4, E_5, E_6, E_7 and E_8 exhibited considerable activity against *S. pyogenes*, when compared with Standard drugs. Derivatives found to be potent were E_1, E_4, E_5, E_9 and E_{10} against *C. albicans*. Compound E_7 and E_8 possessed moderate activity against *A. niger* and molecule E_7 and E_8 demonstrated moderate activity against *A. clavatus*, when compared with reference drugs.

Table 3. Antibacterial activity compounds (E_{1-10})

MINIMAL BACTERICIDAL CONCENTRATION(µgmL^{-1})				
Compound No.	***E.COLI***	***P.AERUGINOSA***	***S.AUREUS***	***S.PYOGENUS***
	MTCC 443	**MTCC 1688**	**MTCC 96**	**MTCC 442**
E_1	62.5	500	125	100
E_2	125	125	100	125
E_3	250	250	250	200
E_4	250	50	100	100
E_5	125	25	62.5	100
E_6	100	125	125	100
E_7	125	100	100	62.5
E_8	25	25	50	100
E_9	250	250	500	500
E_{10}	250	250	500	125
Drug	Micromolar (µgmL^{-1})			
AMPICILLIN	100	-	250	100
CHLORAMPHENICOL	50	50	50	50
CIPROFLOXACIN	25	25	50	50
NORFLOXACIN	10	10	10	10

Table 4. Antifungal activity compounds (E_{1-10})

MINIMAL FUNGICIDAL CONCENTRATION(µgmL^{-1})			
Compound No.	***C.ALBICANS***	***A.NIGER***	***A.CLAVATUS***
	MTCC 227	**MTCC228**	**MTCC1323**
E_1	250	>1000	>1000
E_2	1000	>1000	>1000
E_3	1000	>1000	>1000
E_4	500	>1000	>1000
E_5	500	500	1000
E_6	1000	1000	1000
E_7	1000	250	250
E_8	1000	250	250
E_9	500	1000	1000
E_{10}	500	1000	1000
Drug	Micromolar (µgmL^{-1})		
NYSTATIN	100	100	100
GRESEOFULVIN	500	100	100

CONCLUSIONS

The present work was synthesis of N-(4-(5-(substitutideneamino)-1,3,4-oxadiazole-2-yl) phenyl) substituedimine. Synthesised molecule identified by spectral data IR, ^{1}H NMR, ^{13}C NMR and Mass. From synthesised molecules perfomed antifungal, antibacterial and antituberculer biological studies. Some compounds give excellent biological activities and some gives sufficient activities.

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