International Journal of Pharmaceutical Chemistry
ISSN: 2249-734X (Online)
Journal DOI: 10.7439/ijpc
CODEN: IJPCH3 (American Chemical Society)
Research Article

Synthesis, characterization and antimicrobial screening of some substituted 1, 3, 4 –aryl oxadiazole derivatives

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Abstract

The plethora subscribed in this research is directed towards the synthesis of 1, 3, 4-aryl oxadiazole derivatives. The acid hydrazide of Mefenamic acid and Diclofenac undergoes Schiff's reaction with substituted aromatic aldehyde to offer aryl hydrazones which upon treatment with an aromatic acid and phosphorous oxychloride offers corresponding substituted 1, 3, 4-ary-oxadiazole derivatives. The structures of the synthesized compounds were established by IR, ^{1}H-NMR, MS and elemental analysis. The synthesized compounds were screened for their anti bacterial and anti-fungal activities.
Keywords:1, 3, 4-aryl-oxadiazole, antibacterial, antifungal, anti-inflammatory activity.

1. Introduction

Mefenamic acid and Diclofenac are the non-steroidal anti-inflammatory drugs (NSAIDs) which are mostly useful for the treatment of pain and threshold. It mostly acts through the inhibition of prostaglandin synthesis by inhibition of cyclooxygenase (COX). It also exhibit bacteriostatic activity by inhibiting bacterial DNA synthesis. The long term use of this agents may lead to the development of gastrointestinal ulceration, bleeding and in some cases renal disorders. Chronic use of non-steroidal anti-inflammatory drugs may elicit the appreciable gastro-intestinal toxicity. With the aim of improving safety profile of NSAIDs chemical modification on these agents had been carried out. It has also been reported that compounds containing some substituted 1,3,4-aryl-oxadiazole moiety possess various biological activities likes antimicrobial activity[1], Anti-inflammatory activity[2], GOT, GPT AND c-GT inhibitory activity[3], Anti-cancer activity[4], Haemolytic activity [5], Antioxidant activity[6], Inhibitors of GSK-3b Kinase[7], Monoamine oxidase (MAO) inhibitors [8], Anti-tubercular activity[9], Tubulin inhibitors 10] , Lipoxygenase inhibitors[11] etc. By considering the above facts in this research we had replaced the carboxylic acid moiety of Mefenamic acid and Diclofenac by substituted 1, 3, 4- aryl Oxadiazoles.

2. Materials and Methods

2.1 Material: All the chemicals required for the synthesis were purchased from Modern science, Nashik and are of AR grade.

2.2 Methods

2.2.1 Antibacterial Activity: Anti bacterial study was carried out by using Cup-Plate Agar diffusion method. The synthesized derivatives were tested in vitro for their anti bacterial activity against *Escheria Coli (NCTC 10418)*,

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Staphylococcus aureus (NCTC 6571) and *Bacillus subtilis* which are pathogenic to human being. Leavofloxacin had been used as a standard drug.

2.2.2 Antifungal Activity

Anti fungal activity was carried out by using Cup-Plate Agar diffusion method using nutrient agar as a culture media. The synthesized compounds were tested against *Candida albicans* (ATCC 10231) and *Aspergillus Niger* (ATCC 16404). Amphotericin B had been used as a standard drug.

The synthesized compounds were dissolved in DMF and activities were carried out at a concentration of 200 µg/ml.

2.3. Experimental

Melting points were determined in open capillary method and are uncorrected. Purity of the compound was checked on Silica gel TLC plates. IR spectra were recorded on Thermo Nicolate IR 200 spectrophotometer using KBr disc method. ^{1}H NMR spectra were recorded on Bruker AMX-400, DMSO ḍ6 as solvent and TMS as internal standard. Combustion analyses were found to be within the limits of permissible errors.

2.3.1 Synthesis of Schiff's bases from acid hydrazide and aromatic aldehyde [12]

0.01 mole of an acid hydrazide was dissolved in 10 ml of water along with little ammonia and stirred continuously with drop wise addition of 0.01 moles an aromatic aldehyde until a solid mass is obtained. Filter the precipitate and recrystallized from methanol.

2.3.2 Synthesis of 1, 3, 4-aryl Oxadiazoles from Schiff's bases (A_1-A_{12}) [13,14]

0.01 mole of an aromatic acid was dissolved in $POCl_3$ in a fuming cupboard with continuous stirring until a uniform solution had been formed. After which 0.01 mole a schiffs base is added and temperature of a reaction mixture raised up to 150^0C reflux continued for 2 hrs. Cooled and product is reprecipitated with addition of sodium bicarbonate and recrystallized using methanol to offer title compounds. Purity of synthesized compounds was checked using TLC. (Mobile phase: Toluene: methanol-3:1).

2.3.4 Spectral Data

2.3.4.1 Spectral Data

A_1: IR (cm^{-1}) KBr disc: 3250.15 –NH str.; 3256.23 –OH str.; 3002.58 Ar-CH str.; 2854.36 –CH_3 str.; 1684.69 –CONH str.; 1556.24 –C=N str.; 1120.36 –C-O-C str.; **^{1}H-NMR (ppm):** 6.4-7.2 15H of phenyl; 6.0 1H of 1,3,4-oxadiazole; 5.4 1H of –OH; 5.2 1H of –NH; 1.2-2.6 9H of –CH_3, **m/e** (100%): 493.

A_2: IR (cm^{-1}) KBr disc: 3260.25 –NH str.; 3185.36 –OH str.; 2986.37 Ar-CH str.; 2834.29 –CH_3 str.; 1689.28 –CONH str.; 1564.32 –C=N str.; 1089.25 –C-O-C str.; **^{1}H-NMR (ppm):** 6.2-7.8 15H of phenyl; 6.2 1H of 1,3,4-oxadiazole; 5.6 2H of –OH; 5.0 1H of –NH; 1.2-2.6 6H of –CH_3, **m/e** (100%): 479.

A_3: IR (cm^{-1}) KBr disc: 3245.25 –NH str3220.23 –OH str.; 3000.14 Ar-CH str.; 2810.37 –CH_3 str.; 1685.23 –CONH str.; 1575.24 –C=N str.; 1059.51 –C-O-C str.; **^{1}H-NMR (ppm):** 6.1-7.6 15H of phenyl; 6.1 1H of 1,3,4-oxadiazole; 5.4 2H of –OH; 4.8 1H of –NH; 0.8-1.6 6H of –CH_3, **m/e** (100%): 497.

A_4: IR (cm^{-1}) KBr disc: 3245.20 –NH str.; 3110.28 –CH=CH str.; 3025.14 Ar-CH str.; 2836.79 –CH_3 str.; 1680.24 –CONH str.; 1556.29 –C=N str.; 1060.58 –C-O-C str.; **^{1}H-NMR (ppm):** 6.4-7.8 16H of phenyl; 6.2-6.4 2H of –CH=CH; 6.0 1H of 1,3,4-oxadiazole; 5.0 1H of –NH; 2.1-3.8 9H of –CH_3, **m/e** (100%):503.

A_5: IR (cm^{-1}) KBr disc: 3240.27 –NH str.; 3220.28 –OH str.; 3125.86 –CH=CH str.; 2984.36 Ar-CH str.; 2815.34 –CH_3 str.; 1687.24 –CONH str.; 1575.69 –C=N str.; 1035.28 –C-O-C str.; **^{1}H-NMR (ppm):** 6.2-7.6 16H of phenyl; 6.2-6.4 2H of –CH=CH; 6.0 1H of 1,3,4-oxadiazole; 5.4 1H of –OH; 5.0 1H of –NH; 2.1-2.6 6H of –CH_3, **m/e** (100%): 489.

A_6: IR (cm^{-1}) KBr disc: 3250.48 –NH str.; 3184.26 –CH=CH str.; 3025.38 Ar-CH str.; 2826.38 –CH_3 str.; 1690.27 –CONH str.; 1558.34 –C=N str.; 1039.34 –C-O-C str.; 987.25 –C-Cl bend **^{1}H-NMR (ppm):** 6.4-7.8 16H of phenyl; 6.2-6.4 2H of –CH=CH; 6.2 1H of 1,3,4-oxadiazole; 5.0 1H of –NH; 2.0-2.6 6H of –CH_3, **m/e** (100%): 532.

A_7: IR (cm^{-1}) KBr disc: 3286.84 –NH str.; 3220.54 –OH str.; 3058.48 Ar-CH str.; 2856.37 –CH_3 str.; 1694.25 –CONH str.; 1556.32 –C=N str.; 1025.39 –C-O-C str.;965.38 –C-Cl bend **^{1}H-NMR (ppm):** 6.2-7.8 15H of phenyl; 6.2 1H of 1,3,4-oxadiazole; 5.4 1H of –OH; 5.0 1H of –NH; 1.2-1.6 5H of –CH_3, **m/e** (100%): 548.

A_8: IR (cm^{-1}) KBr disc: 3265.28 –NH str.; 3226.48 –OH str.; 3012.35 Ar-CH str.; 2825.36 –CH_3 str.; 1686.26 –CONH str.; 1570.39 –C=N str.; 1070.38 –C-O-C str.;960.35 –C-Cl bend **^{1}H-NMR (ppm):** 6.4-7.8 15H of phenyl; 6.0 1H of 1,3,4-oxadiazole; 5.6 2H of –OH; 5.0 1H of –NH; 1.2-1.6 2H of –CH_2, **m/e** (100%): 534.

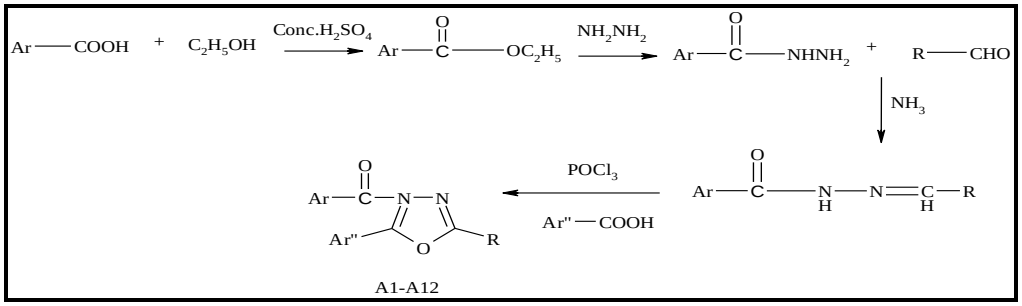
A_9: IR (cm^{-1}) KBr disc: 3265.24 –NH str.; 3225.69 –OH str.; 2986.37 Ar-CH str.; 2830.35 $–CH_3$ str.; 1684.38 –CONH str.; 1572.35 –C=N str.; 1085.34 –C-O-C str.;968.35 –C-Cl bend **^{1}H-NMR (ppm):** 6.4-7.6 15H of phenyl; 6.0 1H of 1,3,4-oxadiazole; 5.4 1H of –OH; 5.0 1H of –NH; 1.2-1.4 2H of $–CH_2$, **m/e** (100%): 552.

A_{10}: IR (cm^{-1}) KBr disc: 3245.68 –NH str.; 3226.35 –CH=CH str.; 3025.69 Ar-CH str.; 2846.38 $–CH_3$ str.; 1684.37 –CONH str.; 1576.34 –C=N str.; 1065.48 –C-O-C str.;967.28 –C-Cl bend **^{1}H-NMR (ppm):** 6.2-7.8 16H of phenyl; 6.2-6.6 2H of –CH=CH; 6.2 1H of 1,3,4-oxadiazole; 5.4 1H of –OH; 5.0 1H of –NH; 1.2-1.6 5H of $–CH_3$, **m/e** (100%): 558.

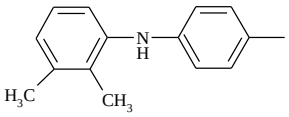
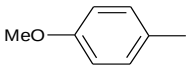
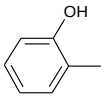
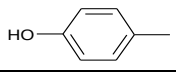
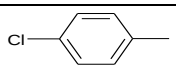
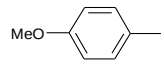
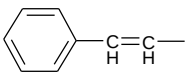
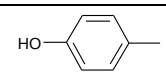
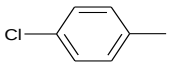
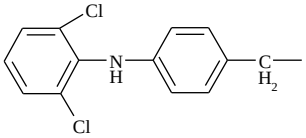
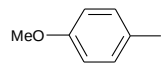
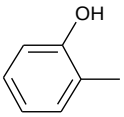
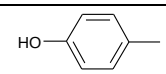
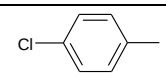
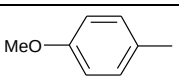
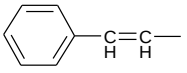
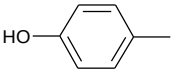
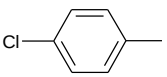
A_{11}: IR (cm^{-1}) KBr disc: 3246.85 –NH str.; 3235.36 –CH=CH str.; 3226.34 –OH str.; 3025.61 Ar-CH str.; 2856.30 $–CH_3$ str.; 1685.64 –CONH str.; 1565.32 –C=N str.; 1075.30 –C-O-C str.; 986.25 –C-Cl bend **^{1}H-NMR (ppm):** 6.4-7.8 16 H of phenyl; 6.4-6.6 2H of –CH=CH; 6.2 1H of 1,3,4-oxadiazole; 5.4 1H of –OH; 5.0 1H of –NH; 1.2-1.4 2H of $–CH_2$, **m/e** (100%): 544.

A_{12}: IR (cm^{-1}) KBr disc: 3255.68 –NH str.; 3247.68 –CH=CH str.; 3025.64 Ar-CH str.; 2810.34 $–CH_3$ str.; 1687.32 –CONH str.; 1576.24 –C=N str.; 1085.25 –C-O-C str.; 976.38 –C-Cl bend **^{1}H-NMR (ppm):** 6.2-7.6 16 H of phenyl; 6.4-6.6 2H of –CH=CH; 6.2 1H of 1,3,4-oxadiazole; 5.0 1H of –NH; 1.2-1.4 2H of $–CH_2$, **m/e** (100%): 562.

Scheme



A1-A12

Comp. Code	Ar	R	Ar'
A_1			
A_2			
A_3			
A_4			
A_5			
A_6			
A_7			
A_8			
A_9			
A_{10}			
A_{11}			
A_{12}			

3. Results and Discussion

Thestructures of the synthesized derivatives of 1, 3, 4-aryl-oxadiazoles (A_1-A_{12}) were established by IR, ^{1}H-NMR, Mass spectra and elemental analysis. The purity of synthesized compounds had been checked on TLC plates using Toluene: Methanol (3:1) as a mobile phase. The IR, ^{1}H-NMR and Mass data reported in manuscript under section of spectral data. The IR spectra shows absorption bands like 3250-3280cm-1 (-NH str.), 3220-3250 cm-1 (-OH str.), 2980-3050 cm-1 (Aromatic –CH str.), 2840-2880 cm-1 (aliphatic –CH str.), 1685-1695 cm-1 (-CONH str.), 1550-1585 cm-1 (-C=N str.), 1030-1080 (-C-O-C str.) which are characteristic feature of 1,3,4-aryl-oxadiazoles. ^{1}H-NMR shows the peaks in 6.4-7.8 (Aromatic H), 6.0-6.2 (H of 1,3,4-oxadiazole), 5.4-5.6 (H of –OH group), 5.0 (H of –NH), 1.2-2.6 (H of $–CH_3$ substituent of phenyl ring).

The synthesized compounds were subjected for anti bacterial activity. Out of twelve compounds the compounds **A_1, A_4, A_8** and **A_{10}** had shown significant antibacterial activity. The structural features of the compounds like presence of electron donating group likes $–CH_3$, $-OCH_3$ along with hydroxyl (-OH) group was thought to increase the biological activity. While other compounds which possess electron withdrawing substituents like –Cl, -OH might be responsible for decrease in activity. In case of antifungal activity compounds **A_5, A_6 and A_{11}** shows significant activity as they posses higher percentage of electron donating groups like $–CH_3$, $-OCH_3$ which might increases the antifungal activity of these derivatives beside this these derivatives also contains a –CH=CH linkage this impartation of unsaturation character in the compounds also responsible for increase in biological activities. Mostly Diclofenac derivatives shows significant antimicrobial activity as it brings inhibition of DNA synthesis.

Table 1: Analytical data of synthesized compounds (A_1-A_{12})

Compound code	Molecular formula	Mole. Wt.	M.P. (^{0}C)	Elemental analysis Found (Cald.)			Rf Value	% Yield
				C	H	N		
A_1	$C_{30}H_{27}N_3O_4$	493.57	271-273	73.01 (72.89)	5.51 (5.21)	7.51 (7.23)	0.48	54
A_2	$C_{29}H_{25}N_3O_4$	479.54	236-241	72.64 (72.38)	5.25 (4.96)	8.76 (8.39)	0.61	51
A_3	$C_{29}H_{24}ClN_3O_3$	497.99	238-242	69.95 (69.68)	4.86 (4.39)	8.44 (8.25)	0.64	62
A_4	$C_{32}H_{29}N_3O_3$	503.61	268-273	76.32 (76.03)	5.80 (5.58)	8.34 (8.13)	0.49	64
A_5	$C_{31}H_{27}N_3O_3$	489.58	301-305	76.05 (75.89)	5.56 (5.28)	8.58 (8.31)	0.58	68
A_6	$C_{32}H_{28}ClN_3O_2$	532.02	318-323	73.62 (73.26)	5.41 (5.12)	8.05 (7.86)	0.61	64
A_7	$C_{29}H_{23}C_{l2}N_3O_4$	548.43	308-313	63.51 (63.21)	4.23 (3.98)	7.66 (7.38)	0.60	78
A_8	$C_{28}H_{21}Cl_2N_3O4$	534.40	287-293	62.93 (62.59)	3.96 (3.68)	7.86 (7.64)	0.54	58
A_9	$C_{28}H_{20}Cl_3N_3O_3$	552.85	309-315	60.83 (60.59)	3.65 (3.29)	7.60 (7.38)	0.57	53
A_{10}	$C_{31}H_{25}Cl_2N_3O_3$	558.47	278-283	66.67 (66.31)	4.51 (4.25)	7.52 (7.21)	0.48	57
A_{11}	$C_{30}H_{23}Cl_2N_3O_3$	544.44	272-277	66.18 (65.98)	4.26 (3.98)	7.72 (7.28)	0.57	59
A_{12}	$C_{30}H_{22}Cl_3N_3O_2$	562.89	269-273	64.02 (63.85)	3.94 (3.58)	7.47 (7.14)	0.53	61

Table 02: Antibacterial and antifungal activity of synthesized compounds (A_1-A_{12})

Compd.	Zone of inhibition at 200µcg/mL (in mm.)				
	E. coli	***B. Subtilis***	***S. aureus***	***A. niger***	***C. albicans***
A_1	24	**25**	**26**	18	**22**
A_2	20	23	22	16	21
A_3	20	24	**25**	19	**22**
A_4	**25**	**26**	23	20	21
A_5	24	23	**26**	**21**	**22**
A_6	20	22	24	18	**23**
A_7	21	23	22	20	21
A_8	22	24	**25**	20	**22**
A_9	23	22	20	18	**22**
A_{10}	24	**26**	23	19	21
A_{11}	**25**	23	24	**21**	**23**
A_{12}	**26**	22	24	20	**22**
Levofloxacin	**26**	**25**	**26**	-	-
Amphotericin B	-	-	-	**22**	**23**

4. Conclusion

Various 1, 3, 4-aryl-oxadiazole derivatives of Mefenamic acid and Diclofenac were prepared by using water phase reaction. The synthesis of acid hydrazones was carried out using stirring in presence of water and alkali. The synthesized derivatives of 1,3,4-aryl-oxadiazoles were subjected for antibacterial and antifungal activity. The synthesized compounds were compared with Leavofloxacin and Amphotericin B. The compounds show activities at a concentration of 200µg/ml.

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