A one-pot microwave irradiation synthesis of 1,2,4-triazolo[1,5-a]pyrimidines

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ABSTRACT

Synthesis of a series of triazolopyrimidines (4a-j) was achieved from different acetoacetamides, thiophene-2-carbaldehyde and 5-amino-1,2,4-triazole using microwave irradiation within 20-30 minutes with high yield. The structures of the products were supported by FTIR, PMR and mass spectral data.

Keywords: Triazolo[1,5-a]pyrimidines; Acetoacetamides; 5-Amino-1,2,4-triazole, microwave irradiation synthesis.

INTRODUCTION

The condensation of a ring of 1,2,4-triazole and another one of pyrimidine gives rise to the formation of bicyclic heterocycles known as 1,2,4-triazolopyrimidines. Among these isomeric families of compounds, 1,2,4-triazolo[1,5-a]pyrimidine derivatives are thermodynamically more stable and, thus, the most studied ones [1]. Revisions surveying the synthesis, reactivity, spectroscopic characterization and crystallographic studies of 1,2,4-triazolo[1,5-c]pyrimidines[2], 1,2,4-triazolo[4,3-a]pyrimidines[3] and 1,2,4-triazolo[4,3-c]pyrimidines[4] have also been published. Pharmacological activities, such as antitumor potency [5,6] inhibition of KDR kinase[7], antifungal effect[8,9] and macrophage activation[10]. Anticancer activity[11], Acetohydroxyacid synthase inhibitor[12], CDK-2 inhibitors[13], Anti-inflammatory[14,15], fungicidal activities[16], antimycobacterial agents[17], A2A adenosine receptor antagonists[18], latent leishmanicidal activity[19],

We have developed a new one-pot multi component synthesis of novel triazolo[1,5-a]pyridimidines (4a-j) with the advantages of short reaction time, high yield and environmental friendliness (Scheme-1).

EXPERIMENTAL SECTION

Melting points were measured in open capillaries and are uncorrected. ¹H NMR spectra were recorded on BRUKUR spectrophotometer (400MHz). Chemical shifts are expressed in units relative to TMS signal as internal reference. IR spectra were recorded on FT-IR SHIMADZU-FT-IR 8400 spectrophotometer on KBr pallets. Mass spectra were recorded on GCMS QP2010 Gas Chromatograph SHIMADZU. Thin Layer Chromatography (TLC) was performed on silica gel-G using hexane: ethylacetate solvent system.
Typical experimental procedure for the synthesis of 1,2,4 triazolopyrimidines.
A mixture of the 5-amino-1,2,4-triazole (2 mmol), acetoacetamide (1 mmol) and thiophene-2-carbaldehyde (1 mmol) in 0.4 ml of DMF was refluxed under microwave irradiation for 20-30 min. After cooling, methanol (~10 ml) was added. The reaction mixture was allowed to stand overnight and then filtered to give the solid triazolopyrimidine products (4a-j), which were crystallized from ethanol and subsequently dried in air.

Scheme-1

4,7-dihydro-N-(4-methoxyphenyl)-5-isopropyl-7-(thiophen-2-yl)-[1,2,4]triazolo[1,5-a]pyrimidine-6-carboxamide (4a)
M. p. 219 ºC; white crystals; 1H NMR (DMSO-d6) δ ppm: (δ 1.71) (s, 3H, H3), (δ 3.37) (s, 3H, H6), (δ 5.66) (s, 1H, H1), (δ 6.60-6.72) (d, 2H, Hcc'), (δ 6.75) (t, 2H, Hbc'), (δ 6.91) (d, 1H, Hbc), (δ 7.53) (t, 2H, Hbc), (δ 8.10) (s, 1H, H3), (δ 8.43) (s, 1H, H1), (δ 9.78) (s, 1H, H6), FT IR (cm−1): 3150 (N-H stretching of secondary amine), 3002 (C-H stretching of aromatic ring), 2913 (C-H asymmetrical stretching of CH3 group), 2856 (C-H asymmetrical stretching of CH2 group), 1660 (C=O stretching of amide), 1601 (C=N stretching of triazole ring), 1553 (N-H deformation of pyrimidine ring), 1515 and 1443 (C=C stretching of aromatic ring), 1421 (C-H asymmetrical deformation of CH3 group), 1350(C=S stretching), 1323(C-O-C stretching), 1021 (C-H in plane deformation of aromatic ring), 823 (C-H out of plane bending of 1,4-disubstituion), Mass: m/z 367; Anal. Calcd. for C18H17N3OS: C, 61.52; H, 4.88; N, 19.93; O, 4.55; S, 9.12. Found: C, 61.21; H, 4.23; N, 19.54; O, 4.42; S, 9.10 %.

N-(4-chlorophenyl)-4,7-dihydro-5-isopropyl-7-(thiophen-2-yl)-[1,2,4]triazolo[1,5-a]pyrimidines-6-carboxamide (4b)
M. p. 179 ºC; white crystals; 1H NMR (DMSO-d6) δ ppm: (δ 1.77) (s, 3H, H3), (δ 5.06) (s, 1H, H6), (δ 6.61-6.77) (d, 2H, Hcc'), (δ 6.79) (t, 2H, Hbc'), (δ 6.93) (d, 1H, Hbc), (δ 7.59) (t, 2H, Hbc'), (δ 8.15) (s, 1H, H3), (δ 8.49) (s, 1H, H1), (δ 9.78) (s, 1H, H6), FT IR (cm−1): 3133 (N-H stretching of secondary amine), 3010 (C-H stretching of aromatic ring), 2921 (C-H asymmetrical stretching of CH3 group), 2853 (C-H asymmetrical stretching of CH2 group), 1659 (C=O stretching of amide), 1610(C=N stretching of triazole ring), 1535 (N-H deformation of pyrimidine ring), 1510 and 1460 (C=C stretching of aromatic ring), 1440 (C-H asymmetrical deformation of CH3 group), 1401 (C-H symmetrical deformation of CH3 group), 1358 (C=S stretching), 1333 (C-N stretching), 1242 (C-O-C stretching), 1021 (C-H in plane deformation of aromatic ring), 823 (C-H out of plane bending of 1,4-disubstituion), Mass: m/z 371; Anal. Calcd. for C18H17ClN3OS: C, 58.84; H, 4.66; N, 19.06; O, 8.71; S, 8.73. Found: C, 58.61; H, 4.34; N, 19.00; O, 8.42; S, 8.53 %.

N-(4-methoxyphenyl)-4,7-dihydro-5-isopropyl-7-(thiophen-2-yl)-[1,2,4]triazolo[1,5-a]pyrimidines-6-carboxamide (4c)
M. p. 257 ºC; white crystals; 1H NMR (DMSO-d6) δ ppm: (δ 1.63) (s, 3H, H3), (δ 2.07) (s, 3H, H6), (δ 5.45) (s, 1H, H1), (δ 6.54-6.70) (d, 2H, Hcc'), (δ 6.73) (t, 2H, Hbc'), (δ 6.95) (d, 1H, Hbc), (δ 7.54) (t, 2H, Hbc'), (δ 8.23) (s, 1H, H3), (δ 8.89) (s, 1H, H1), (δ 9.70) (s, 1H, H6), FT IR (cm−1): 3144 (N-H stretching of secondary amine), 3020 (C-H asymmetrical stretching of CH3 group), 2953 (C-H asymmetrical stretching of CH2 group), 2850 (C-H asymmetrical stretching of CH3 group), 1656 (C=O stretching of amide), 1601 (C=N stretching of triazole ring), 1535 (N-H deformation of pyrimidine ring), 1515 and 1443 (C=C stretching of aromatic ring), 1421 (C-H asymmetrical deformation of CH3 group), 1400 (C-H symmetrical deformation of CH3 group), 1350(C=S stretching), 1311 (C-N stretching), 1020 (C-H in plane deformation of aromatic ring), 933 (C-H out of plane bending of 1,4-disubstituion), Mass: m/z 367; Anal. Calcd. for C18H17N3OS: C, 61.52; H, 4.88; N, 19.93; O, 4.55; S, 9.12. Found: C, 61.21; H, 4.23; N, 19.54; O, 4.42; S, 9.10 %. 

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N-(4-fluorophenyl)-4,7-dihydro-5-isopropyl-7-(thiophen-2-yl)-[1,2,4]triazolo[1,5-a]-pyrimidine-6-carboxamide (4d)

M. p. 179 °C; white crystals; 1H NMR (DMSO-d6) δ ppm: (δ 1.83) (s, 3H, H₃), (δ 5.53) (s, 1H, H₆), (δ 6.64-6.74) (d, 2H, H₄), (δ 6.79) (t, 2H, H₅), (δ 6.90) (t, 1H, H₇), (δ 7.61) (t, 2H, H₇'), (δ 8.13) (s, 1H, H₈), (δ 8.76) (s, 1H, H₉), (δ 9.88) (s, 1H, H₁₀). FT IR (cm⁻¹): 3150 (N-H stretching of secondary amine), 3021 (C-H stretching of aromatic ring), 2924 (C-H asymmetrical stretching of CH₃ group), 2851 (C-H asymmetrical stretching of CH₂ group), 1656 (C=O stretching of amide), 1609 (C=N stretching of triazole ring), 1531 (N-H deformation of pyrimidine ring), 1510 and 1455 (C=C stretching of aromatic ring), 1459 (C-H asymmetrical deformation of CH₃ group), 1424 (C-H symmetrical deformation of CH₃ group), 1353 (C=S stretching), 1323 (C-N stretching), 1032 (C-H in plane deformation of aromatic ring), 823 (C-H out of plane bending of 1,4-disubstitution), 736 (C-F stretching). Mass: m/z 355; Anal. Calcd. for C₁₇H₁₃BrN₅O₄S: C, 50.11; H, 3.12; Br, 19.11; N, 19.71; O, 4.50; S, 8.89%. Found: C, 57.35; H, 3.82; F, 5.12; N, 19.65; O, 4.21; S, 8.89%.

N-(4-bromophenyl)-4,7-dihydro-5-isopropyl-7-(thiophen-2-yl)-[1,2,4]triazolo[1,5-a]-pyrimidine-6-carboxamide (4e)

M. p. 199 °C; white crystals; 1H NMR (DMSO-d6) δ ppm: (δ 1.56) (s, 3H, H₃), (δ 5.65) (s, 1H, H₆), (δ 6.54-6.72) (d, 2H, H₄), (δ 6.78) (t, 2H, H₅), (δ 6.90) (t, 1H, H₇), (δ 7.54) (t, 2H, H₇'), (δ 8.10) (s, 1H, H₈), (δ 8.75) (s, 1H, H₉), (δ 9.75) (s, 1H, H₁₀). FT IR (cm⁻¹): 3124 (N-H stretching of secondary amine), 3011 (C-H stretching of aromatic ring), 2922 (C-H asymmetrical stretching of CH₃ group), 2853 (C-H asymmetrical stretching of CH₂ group), 1653 (C=O stretching of amide), 1603 (C=N stretching of triazole ring), 1535 (N-H deformation of pyrimidine ring), 1514 and 1456 (C=C stretching of aromatic ring), 1451 (C-H asymmetrical deformation of CH₃ group), 1421 (C-H symmetrical deformation of CH₃ group), 1352 (C=S stretching), 1328 (C-N stretching), 1045 (C-H in plane deformation of aromatic ring), 823 (C-H out of plane bending of 1,4-disubstitution), 751 (C-Br stretching). Mass: m/z 416; Anal. Calcd. for C₁₇H₁₃BrN₅OS: C, 57.45; H, 3.97; Br, 19.12; N, 16.68; O, 3.64; S, 7.65%.

N-(3-chloro-4-fluorophenyl)-4,7-dihydro-5-isopropyl-7-(thiophen-2-yl)-[1,2,4]triazolo[1,5-a]-pyrimidine-6-carboxamide (4f)

M. p. 222 °C; white crystals; 1H NMR (DMSO-d6) δ ppm: (δ 1.74) (s, 3H, H₃), (δ 5.64) (s, 1H, H₆), (δ 6.54-6.72) (d, 2H, H₄), (δ 6.89) (t, 1H, H₅), (δ 6.95) (d, 1H, H₆), (δ 7.54-7.59) (t, 2H, H₇), (δ 8.21) (s, 1H, H₈), (δ 8.79) (s, 1H, H₉), (δ 9.81) (s, 1H, H₁₀). FT IR (cm⁻¹): 3164 (N-H stretching of secondary amine), 3068 (C-H stretching of aromatic ring), 2964 (C-H asymmetrical stretching of CH₃ group), 2861 (C-H asymmetrical stretching of CH₂ group), 1660 (C=O stretching of amide), 1608 (C=N stretching of triazole ring), 1553 (N-H deformation of pyrimidine ring), 1514 and 1454 (C=C stretching of aromatic ring), 1451 (C-H asymmetrical deformation of CH₃ group), 1428 (C-H symmetrical deformation of CH₃ group), 1354 (C=S stretching), 1328 (C-N stretching), 1042 (C-H in plane deformation of aromatic ring), 832 (C-H out of plane bending of 1,4-disubstitution), 751 (C-Br stretching), 659 (C-F stretching). Mass: m/z 390; Anal. Calcd. for C₁₇H₁₃ClF₅N₅OS: C, 52.38; H, 3.36; Cl, 9.09; F, 4.87; N, 17.96; O, 4.10; S, 8.23. Found: C, 52.12; H, 3.24; Cl, 9.01; F, 4.56; N, 17.84; O, 4.01; S, 8.12%.

N-(3,4-dichlorophenyl)-4,7-dihydro-5-isopropyl-7-(thiophen-2-yl)-[1,2,4]triazolo[1,5-a]-pyrimidine-6-carboxamide (4g)

M. p. 227 °C; white crystals; 1H NMR (DMSO-d6) δ ppm: (δ 1.64) (s, 3H, H₃), (δ 5.64) (s, 1H, H₆), (δ 6.60-6.72) (d, 2H, H₄), (δ 6.89) (t, 1H, H₅), (δ 6.92) (d, 1H, H₆), (δ 7.56-7.60) (t, 2H, H₇), (δ 8.24) (s, 1H, H₈), (δ 8.82) (s, 1H, H₉), (δ 9.86) (s, 1H, H₁₀). FT IR (cm⁻¹): 3156 (N-H stretching of secondary amine), 3065 (C-H stretching of aromatic ring), 2966 (C-H asymmetrical stretching of CH₃ group), 2856 (C-H asymmetrical stretching of CH₂ group), 1656 (C=O stretching of amide), 1645 (C=N stretching of triazole ring), 1565 (N-H deformation of pyrimidine ring), 1512 and 1456 (C=C stretching of aromatic ring), 1456 (C-H asymmetrical deformation of CH₃ group), 1421 (C-H symmetrical deformation of CH₃ group), 1353 (C=S stretching), 1328 (C-N stretching), 1042 (C-H in plane deformation of aromatic ring), 833 (C-H out of plane bending of 1,4-disubstitution), 751 (C-Cl stretching). Mass: m/z 406; Anal. Calcd. for C₁₇H₁₃Cl₂N₅OS: C, 50.26; H, 3.23; Cl, 17.45; N, 17.24; O, 3.94; S, 7.89. Found: C, 50.11; H, 3.12; Cl, 17.42; N, 17.13; O, 3.64; S, 7.43%.

N-(3-chlorophenyl)-4,7-dihydro-5-isopropyl-7-(thiophen-2-yl)-[1,2,4]triazolo[1,5-a]-pyrimidine-6-carboxamide (4h)

M. p. 199 °C; white crystals; 1H NMR (DMSO-d6) δ ppm: (δ 1.45) (s, 3H, H₃), (δ 5.58) (s, 1H, H₆), (δ 6.56-6.70) (d, 2H, H₄), (δ 6.80) (t, 1H, H₅), (δ 6.92-7.08) (d, 2H, H₇), (δ 7.52) (d, 1H, H₇'), (δ 7.52) (d, 1H, H₈), (δ 8.33) (s, 1H, H₉)
1H, H₂), (δ 8.86) (s, 1H, H₃), (δ 9.78) (s, 1H, H₄). FT IR (cm⁻¹): 3165 (N-H stretching of secondary amine), 3055 (C-H stretching of aromatic ring), 2975 (C-H asymmetrical stretching of CH₃ group), 2853 (C-H asymmetrical stretching of CH₃ group), 1642 (C=O stretching of amide), 1564 (C=N stretching of triazole ring), 1561 (N-H deformation of pyrimidine ring), 1508 and 1451 (C=O stretching of aromatic ring), 1445 (C-H asymmetrical deformation of CH₃ group), 1412 (C-H symmetrical deformation of CH₃ group), 1356 (C=O stretching), 1324 (C-N stretching), 1041 (C-H in plane deformation of aromatic ring), 843 (C-H out of plane bending of 1,4-disubstitution), 752 (C-Cl stretching). Mass: m/z 388; Anal. Calcd. for C₉H₉BrN₆O₃S: C, 55.74; H, 4.68; Cl, 9.14; N, 18.06; O, 4.12; S, 8.27. Found: C, 55.65; H, 4.46; Cl, 9.08; N, 18.00; O, 4.04; S, 8.11%.

N-(3-bromophenyl)-4,7-dihydro-5-isopropyl-7-(thiophen-2-yl)-[1,2,4]triazolo[1,5-a]-pyrimidine-6-carboxamide (4i)
M. p. 189 °C; white crystals; 1H NMR (DMSO-d6) δ ppm: (δ 1.24) (s, 3H, H₂), (δ 5.49) (s, 1H, H₃), (δ 6.43-6.64) (d, 2H, H₆'), (δ 6.72) (t, 1H, H₄), (δ 6.86-7.00) (d, 2H, H₅), (δ 7.59) (d, 1H, H₇), (δ 7.65) (d, 1H, H₈), (δ 8.21) (s, 1H, H₉), (δ 8.98) (s, 1H, H₄), (δ 9.89) (s, 1H, H₉). FT IR (cm⁻¹): 3213 (N-H stretching of secondary amine), 3032 (C-H symmetrical stretching of CH₃ group), 2959 (C-H asymmetrical stretching of CH₃ group), 2835 (C-H asymmetrical stretching of CH₃ group), 1624 (C=O stretching of amide), 1635 (C=N stretching of triazole ring), 1556 (N-H deformation of pyrimidine ring), 1511 and 1449 (C=C stretching of aromatic ring), 1442 (C-H asymmetrical stretching of CH₃ group), 1346 (C=O stretching), 1314 (C=O stretching of aromatic ring), 1297 (C=O in plane deformation of aromatic ring), 846 (C-H out of plane bending of 1,4-disubstitution), 689 (C-Br stretching). Mass: m/z 432; Anal. Calcd. for C₁₀H₈BrN₆O₃: C, 50.01; H, 4.20; Br, 18.48; N, 16.20; O, 3.70; S, 7.42. Found: C, 49.46; H, 4.04; Br, 18.34; N, 16.10; O, 3.63; S, 7.33%.

N-(3-methoxyphenyl)-4,7-dihydro-5-isopropyl-7-(thiophen-2-yl)-[1,2,4]triazolo[1,5-a]-pyrimidine-6-carboxamide (4j)
M. p. 168 °C; white crystals; 1H NMR (DMSO-d6) δ ppm: (δ 1.17) (s, 6H, H₂), (δ 2.24) (m, 1H, H₃), (δ 5.24) (s, 1H, H₉), (δ 6.43-6.66) (d, 2H, H₆'), (δ 6.78) (t, 1H, H₄), (δ 6.87-7.12) (d, 2H, H₅), (δ 7.64) (d, 1H, H₈), (δ 7.87) (d, 1H, H₆), (δ 8.33) (s, 1H, H₇), (δ 8.43) (s, 1H, H₈), (δ 9.46) (s, 1H, H₉). FT IR (cm⁻¹): 3245 (N-H stretching of secondary amine), 3023 (C-H symmetrical deformation of CH₃ group), 2954 (C-H asymmetrical stretching of CH₃ group), 2831 (C-H asymmetrical stretching of CH₃ group), 1626 (C=O stretching of amide), 1612 (C=N stretching of triazole ring), 1564 (N-H deformation of pyrimidine ring), 1524 and 1487 (C=O stretching of aromatic ring), 1453 (C-H asymmetrical deformation of CH₃ group), 1419 (C-H symmetrical deformation of CH₃ group), 1346 (C=O stretching), 1314 (C=O stretching of aromatic ring), 1164 (C=O in plane deformation of aromatic ring), 846 (C-H out of plane bending of 1,4-disubstitution), 689 (C-Br stretching). Mass: m/z 383; Anal. Calcd. for C₁₀H₈BrN₆O₃: C, 59.51; H, 5.52; N, 18.26; O, 8.34; S, 8.36. Found: C, 59.23; H, 5.13; N, 18.15; O, 8.23; S, 8.31%.

Antimicrobial activity
The in vitro antibacterial activity was performed against Gram-positive bacteria including Staphylococcus aureus (MTCC 96), Streptococcus pyogenes (MTCC 442) and Gram negative bacteria including Escherichia coli (MTCC 443), Pseudomonas aeruginosa (MTCC 424). Yeast including Candida albicans (MTCC 227) and fungi Aspergillus clavatus (MTCC 1323) were used to test antifungal activity. Known antibiotics like Ampicillin and Chloramphenicol (the reference anti bacterial drugs) and Fluconazole (the reference antifungal drug) were used for comparison. The antimicrobial activities are summarized in Table A.

RESULTS AND DISCUSSION

The different 4,7-dihydro-N-(substitutedphenyl)-5-isopropyl-7-(thiophen-2-yl)-[1,2,4]triazolo [1,5-a]pyrimidine-6-carboxamide were synthesised by the cyclocondensation of 5-amino-1,2,4-triazole (2 mmol), substituted acetooacetamidine (1 mmol) and thiophene-2-carbaldehyde (1 mmol) in 0.4 ml of DMF was refluxed under microwave irradiation. The M.P. of the synthesized compounds was checked by the given literatures. The purity of the compounds was analyzed by TLC. The structures of the synthesized compounds 3a-j were confirmed on the basis of spectral and elemental analysis. The IR spectrum of these compounds exhibited bands due to 3245 (N-H stretching of secondary amine), 3022 (C-H stretching of aromatic ring), 2954 (C-H asymmetrical stretching of CH₃ group), 2831 (C-H asymmetrical stretching of CH₃ group), 1626 (C=O stretching of amide), 1612 (C=N stretching of triazole ring), 1564 (N-H deformation of pyrimidine ring), 1524 and 1487 (C=O stretching of aromatic ring), 1453 (C-H asymmetrical deformation of CH₃ group), 1413 (C-H symmetrical deformation of CH₃ group), 1335 (C=S stretching), 1313 (C=N stretching), 1023 (C-H in plane deformation of aromatic ring). Further in their 1H NMR
(DMSO-d6) δ ppm: (δ 1.17) (s, 6H, 2CH3), (δ 2.24) (m, 1H, isopropyl proton), (δ 5.24) (s, 1H, -CH), (δ 6.41-6.90) (d, 3H, thiol ring.), (δ 6.91-8.33) (m, 4H, Haloginated Aromatic ring), (δ 8.42) (s, 1H, triazol ring), (δ 8.56) (s, 1H, Amide), (δ 9.46) (s, 1H, pyrimidine ring) peaks confirms the formation of title compounds.

Table-1: Antimicrobial activity of compounds 3a-j

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<th>Compound</th>
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<th>Antifungal activity</th>
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AmpliCil
Chloramphenicol
Fluconazole

CONCLUSION

The newly synthesized compounds 4,7-dihydro-N-(substitutedphenyl)-5-isopropyl-7-(thiophen-2-yl)-[1,2,4]triazolo[1,5-alpyrimidine-6-carboxamide, in the study of antibacterial activity compound 3d, 3j were found to be active. In the study of antifungal activity compounds 3a, 3c, 3i, were active. It can be concluded that this class of compounds certainly holds great promise towards the pursuit to discover novel classes of antimicrobial agents.

REFERENCES