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SYNTHESIS AND CHARACTERIZATION OF FLUOROPYRIMIDINE DERIVATIVES

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ABSTRACT

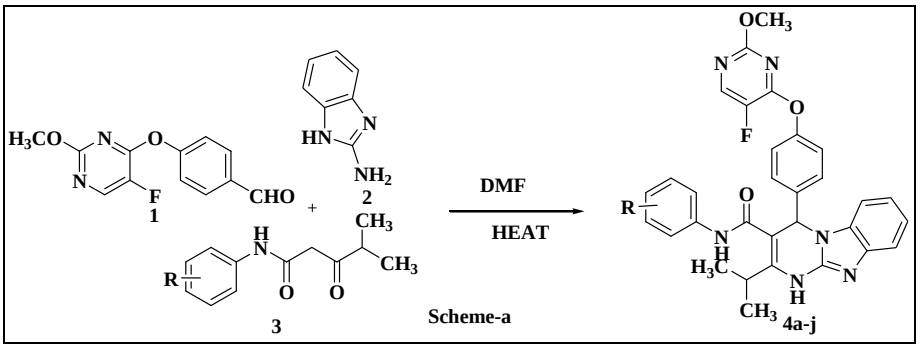
A uncomplicated and well-organized method for synthesis of pyrimidine derivatives was achieved from *N*-(substitutedphenyl)-4-methyl-3-oxopentanamide, 4-(5-fluoro-2-methoxypyrimidin-4-yloxy)benzaldehyde and 2-amino-benzimidazole refluxed with *N*,*N*'-dimehylformamide with high yield and no further purification (Colum purification) requirement for compound . FTIR, 1H NMR and mass spectral data supported the structure of all synthesized compound.

KEYWORDS: *N*-(substitutedphenyl)-4-methyl-3-oxopentanamide, 4-(5-fluoro-2-methoxypyrimidin-4-yloxy)benzaldehyde, 2-amino-benzimidazole.

INTRODUCTION

At that time pyrimidine derivatives most important research in the world like that of triazolopyrimidine is very imperative work of pyrimidine derivatives part because its biological activity like anti-microbial, anti-cancer, anti-HIV, anti-hypertensive, cardiac stimulant, anti-malarial, anti-fungal, anti-HBV, anti-microbial, anti-cancer, anti-pyretic, analgesic, anti-inflammatory, potential herbicidal, and leishmanicidal(1-21). And here new heterocyclic compounds and novel methods for their synthesis is a major topic in contemporary medicinal chemistry(22-26). Cevipabulin and its analogs represent a class of triazolo[1,5-*a*]pyrimidines and were proved to be potent anti-cancer agents with an unique mechanism of action in promoting tubulin polymerization.

We have developed a new moiety for the synthesis 4(4-(5-fluoro-2-methoxypyrimidin-4-yloxy)phenyl)-2-methyl-*N*-(sustitutedphenyl)-1,4-dihydropyrimido-[1,2-*a*]benzimidazole-3-carboxamide ***(4a-j)*** with the advantage of fine yield and environmentally lenience (**Scheme-a**).



Scheme-a

METHOD AND MATERIAL

To the mixture of *N*-(substitutedphenyl)-4-methyl-3-oxopentanamide, 4-(5-fluoro-2-methoxypyrimidin-4-yloxy)benzaldehyde and 2–amino-benzimidazole in few drops of DMF with stirring and heating at 1Hrs. After reaction complete so the add methanol/ethanol and filter solid product.

RESULTS AND DISCUSSION

N-(4-chlorophenyl)-2-isopropyl-4-(4-(5-fluoro-2-methoxypyrimidin-4-yloxy)phenyl)-1,4-dihydropyrimido-[1,2-a]benzimidazole-3-carboxamide (4a)

Yield: 60%; mp 223ºC; Anal. Calcd. for $C_{31}H_{26}ClFN_6O_3$: C, 63.64; H, 4.48; Cl, 6.06; F, 3.25; N, 14.37; O, 8.20;

Found: C, 63.67; H, 4.40; Cl, 6.07; F, 3.21; N, 14.30; O, 8.25 %; IR (cm^{-1}): 3323 (N-H stretching of amide), 3100 (C-H stretching of aromatic ring), 2985 (C-H asymmetrical stretching of CH_3 group), 2831 (C-H symmetrical stretching of CH_3 group), 1631 (C=O stretching of amide), 1585, 1562 (C=O stretching of cyclic) 1510 (N-H deformation of pyrimidine ring), 1450 (C-H asymmetrical deformation of CH_3 group), 1381 (C-H symmetrical deformation of CH_3 group), 1290 (C-N-C stretching vibration of pyrimidine ring), 1269 (C-O-C asymmetrical stretching of OCH_3), 1076 (C-F stretching), 775 (para-substituted), 725 (C-H in out plane deformation of aromatic ring); ^{1}H NMR (DMSO-d6) δ ppm: 2.17-2.22 (s, 6H, H), 3.34 (m, 1H, H), 3.66 (s, 3H, H), 6.57 (s, 1H, H), 7.04-7.07 (dd', 4H, H), 7.23-7.30 (dd', 4H, H), 7.37-7.40 (dd', 4H, H), 7.68 (s, 2H, H), 8.57 (s, 1H, H); MS: m/z 585.

N-(4-fluorophenyl)-2-isopropyl-4-(4-(5-fluoro-2-methoxypyrimidin-4-yloxy)phenyl)-1,4-dihydropyrimido-[1,2-a]benzimidazole-3-carboxamide (4b)

Yield: 67%; mp 199ºC; Anal. Calcd. for $C_{31}H_{26}F_2N_6O_3$: C, 65.49; H, 4.61; F, 6.68; N, 14.78; O, 8.44; Found: C, 65.48; H, 4.67; F, 6.69; N, 14.70; O, 8.40%; IR (cm^{-1}): 3333 (N-H stretching of amide), 3108 (C-H stretching of aromatic ring), 2980 (C-H asymmetrical stretching of CH_3 group), 2838 (C-H symmetrical stretching of CH_3 group), 1631 (C=O stretching of amide), 1585, 1562 (C=O stretching of cyclic) 1510 (N-H deformation of pyrimidine ring), 1450 (C-H asymmetrical deformation of CH_3 group), 1381 (C-H symmetrical deformation of CH_3 group), 1290 (C-N-C stretching vibration of pyrimidine ring), 1269 (C-O-C asymmetrical stretching of OCH_3), 1076 (C-F stretching), 775 (para-substituted), 725 (C-H in out plane deformation of aromatic ring); ^{1}H NMR (DMSO-d6) δ ppm: 2.17-2.22 (s, 6H, H), 3.34 (m, 1H, H), 3.66 (s, 3H, H), 6.57 (s, 1H, H), 7.04-7.07 (dd', 4H, H), 7.23-7.30 (dd', 4H, H), 7.37-7.40 (dd', 4H, H), 7.68 (s, 2H, H), 8.57 (s, 1H, H); MS: m/z 569.

N-(4-bromophenyl)-2-isopropyl-4-(4-(5-fluoro-2-methoxypyrimidin-4-yloxy)phenyl)-1,4-dihydropyrimido-[1,2-a]benzimidazole-3-carboxamide (4c)

Yield: 57%; mp 227ºC; Anal. Calcd. for $C_{31}H_{26}BrFN_6O_3$: C, 59.15; H, 4.16; Br, 12.69; F, 3.02; N, 13.35; O, 7.63; Found: C, 59.15; H, 4.14; Br, 12.68; F, 3.07; N, 13.30; O, 7.60%; IR (cm^{-1}): 3330 (N-H stretching of amide), 3102 (C-H stretching of aromatic ring), 2984 (C-H asymmetrical stretching of CH_3 group), 2848 (C-H symmetrical stretching of CH_3 group), 1631 (C=O stretching of amide), 1588, 1566 (C=O stretching of cyclic) 1515 (N-H deformation of pyrimidine ring), 1452 (C-H asymmetrical deformation of CH_3 group), 1381 (C-H symmetrical deformation of CH_3 group), 1290 (C-N-C stretching vibration of pyrimidine ring), 1267 (C-O-C asymmetrical stretching of OCH_3), 1079 (C-F stretching), 775 (para-substituted), 725 (C-H in out plane deformation of aromatic ring); ^{1}H NMR (DMSO-d6) δ ppm: 2.17-2.22 (s, 6H, H), 3.34 (m, 1H, H), 3.66 (s, 3H, H), 6.57 (s, 1H, H), 7.04-7.07 (dd', 4H, H), 7.23-7.30 (dd', 4H, H), 7.37-7.40 (dd', 4H, H), 7.68 (s, 2H, H), 8.57 (s, 1H, H); MS: m/z 629.

N-(4-methylphenyl)-2-isopropyl-4-(4-(5-fluoro-2-methoxypyrimidin-4-yloxy)phenyl)-1,4-dihydropyrimido-[1,2-a]benzimidazole-3-carboxamide (4d)

Yield: 60; mp 221ºC; Anal. Calcd. for $C_{32}H_{29}FN_6O_3$: C, 68.07; H, 5.18; F, 3.36; N, 14.88; O, 8.50; Found: C, 68.03; H, 5.14; F, 3.34; N, 14.84; O, 8.54%; IR (cm^{-1}): 3326 (N-H stretching of amide), 3107 (C-H stretching of aromatic ring), 2984 (C-H asymmetrical stretching of CH_3 group), 2845 (C-H symmetrical stretching of CH_3 group), 1635 (C=O stretching of amide), 1582, 1569 (C=O stretching of cyclic) 1518 (N-H deformation of pyrimidine ring), 1454 (C-H asymmetrical deformation of CH_3 group), 1386 (C-H symmetrical deformation of CH_3 group), 1290 (C-N-C stretching vibration of pyrimidine ring), 1266 (C-O-C asymmetrical stretching of OCH_3), 1079 (C-F stretching), 775 (para-substituted), 725 (C-H in out plane deformation of aromatic ring); ^{1}H NMR (DMSO-d6) δ ppm: 2.17-2.22 (s, 6H, H), 3.34 (m, 1H, H), 3.66 (s, 3H, H), 6.57 (s, 1H, H), 7.04-7.07 (dd', 4H, H), 7.23-7.30 (dd', 4H, H), 7.37-7.40 (dd', 4H, H), 7.68 (s, 2H, H), 8.57 (s, 1H, H), 2.30 (s, 3H, H); MS: m/z 565.

N-(4-nitrophenyl)-2-isopropyl-4-(4-(5-fluoro-2-methoxypyrimidin-4-yloxy)phenyl)-1,4-dihydropyrimido-[1,2-a]benzimidazole-3-carboxamide (4e)

Yield: 50; mp 220ºC; Anal. Calcd. for $C_{31}H_{26}FN_7O_5$: C, 62.52; H, 4.40; F, 3.19; N, 16.46; O, 13.43; Found C, 62.52; H, 4.40; F, 3.19; N, 16.46; O, 13.43%; IR (cm^{-1}): 3320 (N-H stretching of amide), 3096 (C-H stretching of aromatic ring), 2980 (C-H asymmetrical stretching of CH_3 group), 2827 (C-H symmetrical stretching of CH_3 group), 1621 (C=O stretching of amide), 1583, 1558 (C=O stretching of cyclic) 1505 (N-H deformation of pyrimidine ring), 1442 (C-H asymmetrical deformation of CH_3 group), 1378 (C-H symmetrical deformation of CH_3 group), 1289 (C-N-C stretching vibration of pyrimidine ring), 1261 (C-O-C asymmetrical stretching of OCH_3), 1071 (C-F stretching), 775 (para-substituted), 725 (C-H in out plane deformation of aromatic ring); ^{1}H NMR (DMSO-d6) δ ppm: 2.17-2.22 (s, 6H, H), 3.34 (m, 1H, H), 3.66 (s, 3H, H), 6.57 (s, 1H, H), 7.04-7.07 (dd', 4H, H), 7.23-7.30 (dd', 4H, H), 7.37-7.40 (dd', 4H, H), 7.68 (s, 2H, H), 8.57 (s, 1H, H); MS: m/z 596.

N-(3-chlorophenyl)-2-isopropyl-4-(4-(5-fluoro-2-methoxypyrimidin-4-yloxy)phenyl)-1,4-dihydropyrimido-[1,2-a]benzimidazole-3-carboxamide (4f)

Yield: 61%; mp 201ºC; Anal. Calcd. for $C_{31}H_{26}ClFN_6O_3$: C, 63.64; H, 4.48; Cl, 6.06; F, 3.25; N, 14.37; O, 8.20; Found: C, 63.64; H, 4.44; Cl, 6.07; F, 3.24; N, 14.34; O, 8.24 %; IR (cm^{-1}): 3327 (N-H stretching of amide), 3104

(C-H stretching of aromatic ring), 2983 (C-H asymmetrical stretching of CH_3 group), 2835 (C-H symmetrical stretching of CH_3 group), 1628 (C=O stretching of amide), 1576, 1560 (C=O stretching of cyclic) 1509 (N-H deformation of pyrimidine ring), 1449 (C-H asymmetrical deformation of CH_3 group), 1377 (C-H symmetrical deformation of CH_3 group), 1294 (C-N-C stretching vibration of pyrimidine ring), 1265 (C-O-C asymmetrical stretching of OCH_3), 1077 (C-F stretching), 775 (para-substituted), 725 (C-H in out plane deformation of aromatic ring); ^{1}H NMR (DMSO-d6) δ ppm: 2.17-2.22 (s, 6H, H), 3.34 (m, 1H, H), 3.66 (s, 3H, H), 6.57 (s, 1H, H), 7.04-7.07 (dd', 4H, H), 7.23-7.30 (dd', 4H, H), 7.37-7.40 (dd', 4H, H), 7.68 (s, 2H, H), 8.57 (s, 1H, H); MS: m/z 585.

N-(2-chlorophenyl)-2-isopropyl-4-(4-(5-fluoro-2-methoxypyrimidin-4-yloxy)phenyl)-1,4-dihydropyrimido-[1,2-a]benzimidazole-3-carboxamide (4g)

Yield: 51%; mp 187ºC; Anal. Calcd. for $C_{31}H_{26}ClFN_6O_3$: C, 63.64; H, 4.48; Cl, 6.06; F, 3.25; N, 14.37; O, 8.20; Found: C, 63.61; H, 4.45; Cl, 6.01; F, 3.24; N, 14.35; O, 8.28 %; IR (cm^{-1}): 3331 (N-H stretching of amide), 3092 (C-H stretching of aromatic ring), 2988 (C-H asymmetrical stretching of CH_3 group), 2837 (C-H symmetrical stretching of CH_3 group), 1637 (C=O stretching of amide), 1589, 1568 (C=O stretching of cyclic) 1518 (N-H deformation of pyrimidine ring), 1458 (C-H asymmetrical deformation of CH_3 group), 1391 (C-H symmetrical deformation of CH_3 group), 1298 (C-N-C stretching vibration of pyrimidine ring), 1276 (C-O-C asymmetrical stretching of OCH_3), 1089 (C-F stretching), 775 (para-substituted), 725 (C-H in out plane deformation of aromatic ring); ^{1}H NMR (DMSO-d6) δ ppm: 2.17-2.22 (s, 6H, H), 3.34 (m, 1H, H), 3.66 (s, 3H, H), 6.57 (s, 1H, H), 7.04-7.07 (dd', 4H, H), 7.23-7.30 (dd', 4H, H), 7.37-7.40 (dd', 4H, H), 7.68 (s, 2H, H), 8.57 (s, 1H, H); MS: m/z 585.

N-(3-fluorophenyl)-2-isopropyl-4-(4-(5-fluoro-2-methoxypyrimidin-4-yloxy)phenyl)-1,4-dihydropyrimido-[1,2-a]benzimidazole-3-carboxamide (4h)

Yield: 60%; mp 193ºC; Anal. Calcd. for $C_{31}H_{26}F_2N_6O_3$: C, 65.49; H, 4.61; F, 6.68; N, 14.78; O, 8.44; Found: C, 65.40; H, 4.64; F, 6.64; N, 14.74; O, 8.44%; IR (cm^{-1}): 3318 (N-H stretching of amide), 3098 (C-H stretching of aromatic ring), 2994 (C-H asymmetrical stretching of CH_3 group), 2840 (C-H symmetrical stretching of CH_3 group), 1621 (C=O stretching of amide), 1579, 1573 (C=O stretching of cyclic) 1517 (N-H deformation of pyrimidine ring), 1459 (C-H asymmetrical deformation of CH_3 group), 1388 (C-H symmetrical deformation of CH_3 group), 1294 (C-N-C stretching vibration of pyrimidine ring), 1279 (C-O-C asymmetrical stretching of OCH_3), 1065 (C-F stretching), 775 (para-substituted), 725 (C-H in out plane deformation of aromatic ring); ^{1}H NMR (DMSO-d6) δ ppm: 2.17-2.22 (s, 6H, H), 3.34 (m, 1H, H), 3.66 (s, 3H, H), 6.57 (s, 1H, H), 7.04-7.07 (dd', 4H, H), 7.23-7.30 (dd', 4H, H), 7.37-7.40 (dd', 4H, H), 7.68 (s, 2H, H), 8.57 (s, 1H, H); MS: m/z 569.

N-(3-bromophenyl)-2-isopropyl-4-(4-(5-fluoro-2-methoxypyrimidin-4-yloxy)phenyl)-1,4-dihydropyrimido-[1,2-a]benzimidazole-3-carboxamide(4i)

Yield: 53%; mp 201ºC; Anal. Calcd. for $C_{31}H_{26}BrFN_6O_3$: C, 59.15; H, 4.16; Br, 12.69; F, 3.02; N, 13.35; O, 7.63; Found: C, 59.10; H, 4.10; Br, 12.69; F, 3.09; N, 13.38; O, 7.64%; IR (cm^{-1}): 3328 (N-H stretching of amide), 3106 (C-H stretching of aromatic ring), 2984 (C-H asymmetrical stretching of CH_3 group), 2848 (C-H symmetrical stretching of CH_3 group), 1631 (C=O stretching of amide), 1588, 1566 (C=O stretching of cyclic) 1515 (N-H deformation of pyrimidine ring), 1452 (C-H asymmetrical deformation of CH_3 group), 1381 (C-H symmetrical deformation of CH_3 group), 1290 (C-N-C stretching vibration of pyrimidine ring), 1267 (C-O-C asymmetrical stretching of OCH_3), 1079 (C-F stretching), 775 (para-substituted), 725 (C-H in out plane deformation of aromatic ring); ^{1}H NMR (DMSO-d6) δ ppm: 2.17-2.22 (s, 6H, H), 3.34 (m, 1H, H), 3.66 (s, 3H, H), 6.57 (s, 1H, H), 7.04-7.07 (dd', 4H, H), 7.23-7.30 (dd', 4H, H), 7.37-7.40 (dd', 4H, H), 7.68 (s, 2H, H), 8.57 (s, 1H, H); MS: m/z 629.

N-(2-fluorophenyl)-2-isopropyl-4-(4-(5-fluoro-2-methoxypyrimidin-4-yloxy)phenyl)-1,4-dihydropyrimido-[1,2-a]benzimidazole-3-carboxamide(4j)

Yield: 55%; mp 179ºC; Anal. Calcd. for $C_{31}H_{26}F_2N_6O_3$: C, 65.49; H, 4.61; F, 6.68; N, 14.78; O, 8.44; Found: C, 65.47; H, 4.64; F, 6.69; N, 14.74; O, 8.40%; IR (cm^{-1}): 3325 (N-H stretching of amide), 3098 (C-H stretching of aromatic ring), 2989 (C-H asymmetrical stretching of CH_3 group), 2839 (C-H symmetrical stretching of CH_3 group), 1645 (C=O stretching of amide), 1593, 1576 (C=O stretching of cyclic) 1500 (N-H deformation of pyrimidine ring), 1443 (C-H asymmetrical deformation of CH_3 group), 1377 (C-H symmetrical deformation of CH_3 group), 1296 (C-N-C stretching vibration of pyrimidine ring), 1275 (C-O-C asymmetrical stretching of OCH_3), 1084 (C-F stretching), 775 (para-substituted), 725 (C-H in out plane deformation of aromatic ring); ^{1}H NMR (DMSO-d6) δ ppm: 2.17-2.22 (s, 6H, H), 3.34 (m, 1H, H), 3.66 (s, 3H, H), 6.57 (s, 1H, H), 7.04-7.07 (dd', 4H, H), 7.23-7.30 (dd', 4H, H), 7.37-7.40 (dd', 4H, H), 7.68 (s, 2H, H), 8.57 (s, 1H, H); MS: m/z 569.

CONCLUSION

In loftiness, we include syntheses of narrative pyrimidine derivatives using appropriate and trouble-free method. This method produces these products in supreme yields and difficulty-free workup. The isolated products are unadulterated and no need of further purification.

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