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Synthesis of novel fluorophenyl, furan tagged benzylthiopyrimidine derivatives

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A new series of 6-(4-fluorophenyl)-2-(substitutedbenzylthio)-4-(furan-2-yl)-1,6-dihydropyrimidine derivatives have been synthesized from 2-acetyl furan through a multi-step reaction sequence. The key intermediate on reacted with various substituted benzyl halides in the presence of K_2CO_3 afforded a series of title compounds. The structures of all newly synthesized compounds were characterized by IR, ^{1}H NMR, LCMS mass and C, H, N analyses.

Key words: dihydropyrimidine derivatives, fluorophenyls, furan, synthesis

INTRODUCTION

Heterocycles are ubiquitous in pharmaceutical compounds. Pyrimidine moiety is an important class of *N* containing heterocycles widely used as key building blocks for pharmaceutical agents [1–3]. Dihydropyrimidine derivatives, which can be considered as thia-analogues of the natural purine bases such as adenine and guanine, have acquired a growing importance in the field of medicinal chemistry because of their biological potential. In addition to diverse biological activities, in association with other heterocyclics, pyrimidines are known to play a crucial role in several processes of chemical and pharmacological importance as therapeutics in clinical applications. Many pyrimidine derivatives are known to exhibit antibacterial and fungicide [4–6], antimalarial [7, 8], analgesic [9], anti-HIV [10], antihypertensive [11], antitumor [12, 13], antioxidant [14–16], antimitotic [17], and anticonvulsant activities [18].

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Some dihydropyrimidines (DHPM) have emerged as integral backbones of calcium channel blockers, antihypertensive agents, adrenergic and neuropeptide antagonists. Several alkaloids containing dihydropyrimidine isolated from marine sources such as batzelladine alkaloids are reported to be potent HIV-gp-120-CD4 inhibitors [19–21] etc.

The search for new heterocyclic compounds and novel molecules for their synthesis is a major topic in contemporary organic synthesis. Therefore, versatile and widely applicable methods for their synthesis are of considerable interest. Thus the aim of the present work is to synthesize novel fluorophenyl, furan tagged benzylthiopyrimidine derivatives.

RESULTS AND DISCUSSION

An ice-cold solution of the cyclic compound 4-(4-fluorophenyl)-6-(furan-2-yl)-3,4-dihydropyrimidin-2(1H)-thione (**4**) (1 mmol) in DMF (10 ml), potassium carbonate (1.5 mmol) and substituted benzyl halides (1.3 mmol) were

taken in a 1 liter round-bottomed flask equipped with a magnetic stirrer and stirred for 1 hour. The residual portion was poured on to crushed ice, neutralized with dilute acid and the product 6-(4-fluorophenyl)-2-(substitutedbenzylthio)-4-(furan-2-yl)-1,6-dihydropyrimidine derivatives (**6 a–k**) was collected by filtration.

For the synthesis of the new materials, initially 2-acetyl furan (**1**) was treated with *p*-fluorobenzaldehyde (2) in the presence of KOH to give their corresponding 3-(4-fluorophenyl)-1-(furan-2-yl)prop-2-en-1-one (**3**). Compound **3** was reacted with thiourea in the presence of KOH toobtain 4-(4-fluorophenyl)-6-(furan-2-yl)-3,4-dihydropyrimidin-2(1*H*)-thione (**4**). Further, the cyclic compound 4-(4-fluorophenyl)-6-(furan-2-yl)-3,4-dihydropyrimidin-2(1H)-thione 4 was successfully reacted with selected substituted aldehydes in the presence of DMF solvent, to a good yields (**6a–k**) (Scheme 1).

All the newly synthesized compounds gave moderate to high yields. Products were purified and characterized by various spectroscopic techniques. The IR spectra of compounds (**6 a–k**) showed characteristic absorption bands at 3 341–3 310 cm^{-1}, 1 621–1 568 cm^{-1}, 1 547–1 521, and 854–836 cm^{-1} corresponding to the $N–H_{str}$, $C=N_{str}$, $C=C_{str}$ and $C–F_{str}$ functions in the structures. The ^1H NMR spectra showed peaks in the range of δ 4.10–4.27 for SCH_2, δ 6.09–6.19 for =CH, δ 6.58–6.81 for the furan ring and δ 10.02–10.27 for pyrimidine NH. The mass spectrum of all the compounds showed molecular ion peak at M^+, at M+H corresponding to its molecular formula, which confirmed its chemical structure. The IR, ^1H NMR, LCMS mass spectra and elemental analysis confirmed the structure of various novel 6-(4-fluorophenyl)-2-(substitutedbenzylthio)-4-(furan-2-yl)-1,6-dihydropyrimidine derivatives (**6 a–k**).

EXPERIMENTAL

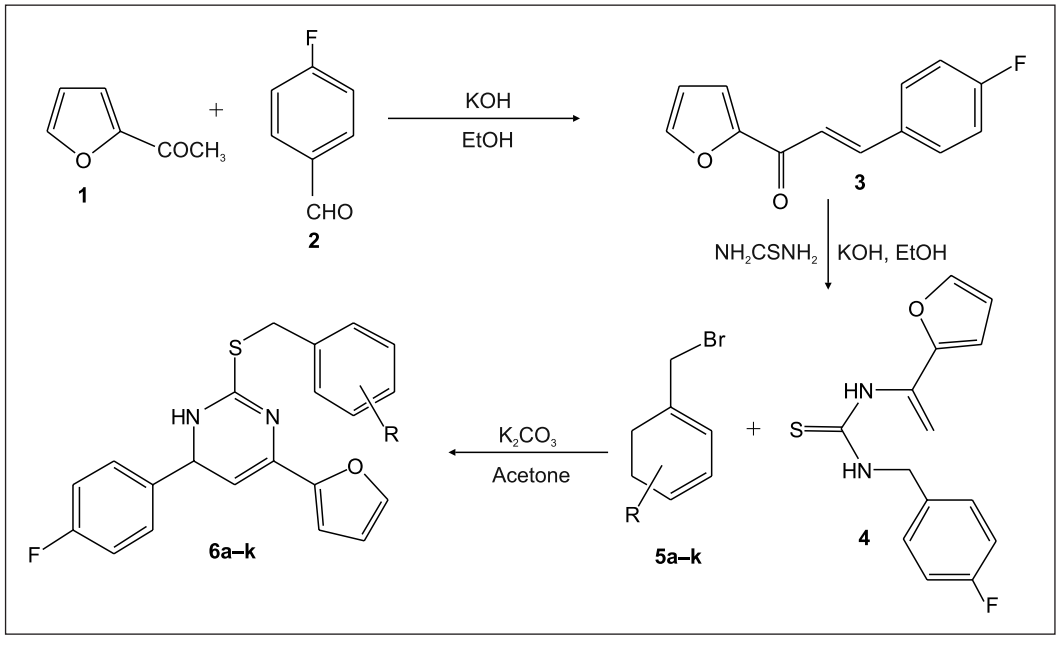
All reagents and solvents were purchased and used without further purification. Melting points were determined on a Fisher-Johns melting point apparatus and were uncorrected. Crude products were purified by column chromatography on silica gel of 60–120 mesh. IR spectra were obtained on a Perkin Elmer BX serried FT-IR 5000 spectrometer using the KBr pellet. The ^1H NMR spectra were recorded on a Varian 400 MHz spectrometer for ^1H NMR. LCMS Mass spectra were recorded on a MASPEC low resolution mass spectrometer operating at 70 eV.

Preparation of 3-(4-fluorophenyl)-1-(furan-2-yl)prop-2-en-1-one (3)

3-(4-Fluorophenyl)-1-(furan-2-yl)prop-2-en-1-one was synthesized by the mixture of KOH (0.055 mol), water (20 mL), ethanol (15 mL), 2-acetyl furan (**1**) (0.043 mol) and the *p*-fluorobenzaldehyde (**2**) (0.043 mol) stirred at 30–40 °C for 2 h and kept overnight. It was then filtered, washed with water and with ethanol, dried and refluxed with glacial acetic acid (10 mL) for 2 h. The crystals separated after cooling were filtered, washed and dried. Yield 77%, Mp 210 °C; IR (KBr) cm^{-1}: 1 733 (C=O), 1 631 (C=C), 1 030 (cyclic $C–O–C_{str}$), 823 (C-F); ^1H NMR (300 MHz, DMSO-d_6, δ ppm): 7.24 (d, 2H, Ar–H), 7.51 (d, 2H, Ar–H), 6.53–7.21 (m, 3H, –CH-furan), 6.10 (d, 2H, =CH); MS (EI) m/z 216 $[M]^+$; Anal. calcd for $C_{13}H_9FO_2$: C, 72.22; H, 4.20; Found: C, 72.23; H, 4.22.

Synthesis of 4-(4-fluorophenyl)-6-(furan-2-yl)-3,4-dihydropyrimidin-2(1H)-thione (4)

A mixture of 3-(4-fluorophenyl)-1-(furan-2-yl)prop-2-en-1-one (**3**) (1 mmol) thiourea (1 mmol) and potassium



Scheme. Synthesis path way of fluorophenyl furan dihydropyrimidine derivatives

hydroxide (2.5 g) in 95% ethanol (100 mL) was heated under reflux for 3 h. The reaction mixture was concentrated to half of its volume, diluted with water, then acidified with dilute acetic acid and kept overnight. The solid thus obtained was filtered, washed with water and recrystallized from ethanol to give 4-(4-fluorophenyl)-6-(furan-2-yl)-3,4-dihydropyrimidine-2(1*H*)-thione (**4**). Yield 72%, Mp 231 °C; IR (KBr) cm^{-1}: 3 361 (NH_{str}), 1 531 (C=C), 1 034 (cyclic C–O–C_{str}), 843 (C-F); ^1H NMR (300 MHz, DMSO-d_6, δ ppm): 7.39 (d, 2H, Ar–H), 7.41 (d, 2H, Ar–H), 6.61–6.81 (m, 3H, –CH-furan), 6.14 (d, 1H, pyrimidine H), 4.70 (d, 1H, pyrimidine H), 3.27 (s, 2H, –NH); MS (EI) m/z 274 $[M]^+$; Anal. calcd for $C_{14}H_{11}FN_2OS$: C, 61.30; H, 4.04; N, 10.21; Found: C, 61.33; H, 4.06; N, 10.24.

6-(4-Fluorophenyl)-2-(substitutedbenzylthio)-4-(furan-2-yl)-1,6-dihydropyrimidine derivatives (6a–k)

An ice-cold solution of the cyclic compound 4-(4-fluorophenyl)-6-(furan-2-yl)-3,4-dihydropyrimidin-2(1H)-thione (**4**) (1 mmol) in DMF (10 ml), potassium carbonate (1.5 mmol) and substituted benzyl halides (1.3 mmol) were taken in a 1 liter round-bottomed flask equipped with a magnetic stirrer and stirred for 1 hour. The residual portion was poured on to crushed ice, neutralized with dilute acid and the product obtained 6-(4-fluorophenyl)-2-(substitutedbenzylthio)-4-(furan-2-yl)-1,6-dihydropyrimidine derivatives (**6 a–k**) was collected by filtration.

2-(Benzylthio)-6-(4-fluorophenyl)-4-(furan-2-yl)-1,6-dihydropyrimidine (6a)

Yield: 78%; mp 251–253 °C; IR (υ cm^{-1}, KBr): 3 316 (NH), 1 577 (C=N), 1 523 (C=C), 842 (C–F) cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ 10.02 (s, 1H, NH-pyrimidine), 7.48 (d, 2H, Ar–H), 7.21–7.32 (m, 3H, Ar–H), 7.16 (d, 2H, Ar–H), 7.09 (d, 2H, Ar–H), 6.61–6.80 (m, 3H, furan), 6.12 (d, 1H, =CH), 5.10 (d, 1H, CH), 4.10 (s, 2H, SCH_2); LCMS: (*m/z*) 524 (M+H, 100%). Anal. calcd. for $C_{27}H_{17}FN_2OS$: C, 69.21; H, 4.70; N, 7.69. Found: C, 69.18; H, 4.73; N, 7.65.

2-(3-Fluorobenzylthio)-6-(4-fluorophenyl)-4-(furan-2-yl)-1,6-dihydropyrimidine (6b)

Yield: 86%; mp 204–205 °C; IR (υ cm^{-1}, KBr): 3 328 (NH), 1 594 (C=N), 1 545 (C=C), 848 (C–F) cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ 10.17 (s, 1H, NH-pyrimidine), 7.27 (t, 1H, Ar–H), 7.14 (d, 2H, Ar–H), 7.05 (d, 4H, Ar–H), 6.62–6.77 (m, 3H, furan), 6.16 (d, 1H, =CH), 5.16 (d, 1H, CH), 4.19 (s, 2H, SCH_2); LCMS: (*m/z*) 558 (M+H, 100%). Anal. calcd. for $C_{21}H_{16}F_2N_2OS$: C, 65.95; H, 4.22; N, 7.33. Found: C, 66.02; H, 4.18; N, 7.29.

2-(4-Fluorobenzylthio)-6-(4-fluorophenyl)-4-(furan-2-yl)-1,6-dihydropyrimidine (6c)

Yield: 72%; mp 195–197 °C; IR (υ cm^{-1}, KBr): 3 339 (NH), 1 576 (C=N), 1 537 (C=C), 838 (C–F) cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ 10.15 (s, 1H, NH-pyrimidine), 7.42 (d, 2H, Ar–H), 7.15 (d, 2H, Ar–H), 7.07 (d, 2H, Ar–H), 6.61–6.78 (m, 3H, furan), 6.15 (d, 1H, =CH), 5.14 (d, 1H, CH), 4.18 (s, 2H, SCH_2); LCMS: (*m/z*) 558 (M+H, 100%). Anal. calcd. for $C_{21}H_{16}F_2N_2OS$: C, 65.95; H, 4.22; N, 7.33. Found: C, 66.02; H, 4.18; N, 7.29.

2-(4-Fluoro-2-(trifluoromethyl)benzylthio)-6-(4-fluorophenyl)-4-(furan-2-yl)-1,6-dihydropyrimidine (6d)

Yield: 88%; mp 232 °C; IR (υ cm^{-1}, KBr): 3 320 (NH), 1 568 (C=N), 1 526 (C=C), 840 (C–F) cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ 10.27 (s, 1H, NH-pyrimidine), 7.14 (d, 2H, Ar–H), 7.06 (m, 4H, Ar–H), 6.99 (s, 1H, Ar–H), 6.58–6.71 (m, 3H, furan), 6.18 (d, 1H, =CH), 5.22 (d, 1H, CH), 4.27 (s, 2H, SCH_2); LCMS: (*m/z*) 538 (M+H, 100%). Anal. calcd. for $C_{22}H_{15}F_5N_2OS$: C, 58.66; H, 3.36; N, 6.22. Found: C, 58.74; H, 3.42; N, 6.29.

2-(4-Trifluoromethyoxy)benzylthio-6-(4-fluorophenyl)-4-(furan-2-yl)-1,6-dihydropyrimidine (6e)

Yield: 82%; mp 205–207 °C; IR (υ cm^{-1}, KBr): 3 341 (NH), 1 575 (C=N), 1 540 (C=C), 851 (C–F) cm^{-1}; ^1H NMR (400 MHz, DMSO–d_6): δ 10.23 (s, 1H, NH–pyrimidine), 7.18 (d, 2H, Ar–H), 7.09 (d, 2H, Ar–H), 6.95–7.01 (m, 4H, Ar–H), 6.60–6.75 (m, 3H, furan), 6.17 (d, 1H, =CH), 5.20 (d, 1H, CH), 4.25 (s, 2H, SCH_2); LCMS: (*m/z*) 553 (M^+, 100%). Anal. calcd. for $C_{22}H_{16}F_4N_2O_2S$: C, 58.92; H, 3.60; N, 6.25. Found: C, 58.85; H, 3.51; N, 6.31.

2-(4-Methylbenzylthio)-6-(4-fluorophenyl)-4-(furan-2-yl)-1,6-dihydropyrimidine (6f)

Yield: 92%; mp 189–190 °C; IR (υ cm^{-1}, KBr): 3 310 (NH), 1 608 (C=N), 1 547 (C=C), 836 (C–F) cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ 10.11 (s, 1H, NH-pyrimidine), 7.20 (d, 2H, Ar–H), 7.11 (d, 2H, Ar–H), 7.01–7.06 (d, 4H, Ar–H), 6.63–6.81 (m, 3H, furan), 6.10 (d, 1H, =CH), 5.12 (d, 1H, CH), 4.11 (s, 2H, SCH_2), 2.21 (s, 3H, CH_3); LCMS: (*m/z*) 542 (M+H, 100%). Anal. calcd. for $C_{22}H_{19}FN_2OS$: C, 69.82; H, 5.06; N, 7.40. Found: C, 69.87; H, 5.12; N, 7.34.

2-(4-Methoxybenzylthio)-6-(4-fluorophenyl)-4-(furan-2-yl)-1,6-dihydropyrimidine (6g)

Yield: 80%; mp 244–246 °C; IR (υ cm^{-1}, KBr): 3 324 (NH), 1 573 (C=N), 1 521 (C=C), 854 (C–F) cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ 10.13 (s, 1H, NH-pyrimidine), 7.18 (d, 2H, Ar–H), 7.08 (d, 2H, Ar–H), 6.98–7.03 (m, 4H, Ar–H), 6.62–6.80 (m, 3H, furan), 6.15 (d, 1H, =CH), 5.14 (d, 1H, CH), 4.16 (s, 2H, SCH_2), 3.71 (s, 3H, OCH_3); LCMS: (*m/z*) 569 (M+H, 100%). Anal. calcd. for $C_{22}H_{19}FN_2O_2S$: C, 66.99; H, 4.85; N, 7.10. Found: C, 67.07; H, 4.89; N, 7.08.

2-(4-Nitrobenzylthio)-6-(4-fluorophenyl)-4-(furan-2-yl)-1,6-dihydropyrimidine (6h)

Yield: 84%; mp 195–197 °C; IR (υ cm^{-1}, KBr): 3 340 (NH), 1 582 (C=N), 1 530 (C=C), 850 (C–F) cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ 10.19 (s, 1H, NH-pyrimidine), 7.81–7.92

(m, 4H, Ar–H), 7.14 (d, 2H, Ar–H), 7.06 (d, 2H, Ar–H), 6.59–6.77 (m, 3H, furan), 6.16 (d, 1H, =CH), 5.18 (d, 1H, CH), 4.21 (s, 2H, SCH_2); LCMS: (*m/z*) 551 (M^+, 100%). Anal. calcd. for $C_{21}H_{16}FN_3O_3S$: C, 61.60; H, 3.94; N, 10.26. Found: C, 61.65; H, 3.89; N, 10.31.

2-(3-Methyl-4-nitrobenzylthio)-6-(4-fluorophenyl)-4-(furan-2-yl)-1,6-dihydropyrimidine (6i)

Yield: 76%; mp 232–233 °C; IR (υ cm^{-1}, KBr): 3 335 (NH), 1 621 (C=N), 1 529 (C=C), 841 (C–F) cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ 10.18 (s, 1H, NH-pyrimidine), 7.68–7.87 (d, 2H, Ar–H), 7.41 (s, 1H, Ar–H), 7.19 (d, 2H, Ar–H), 7.07 (d, 2H, Ar–H), 6.59–6.78 (m, 3H, furan), 6.14 (d, 1H, =CH), 5.16 (d, 1H, CH), 4.15 (s, 2H, SCH_2), 2.39 (s, 3H, CH_3); LCMS: (*m/z*) 539 (M+H, 100%). Anal. calcd. for $C_{22}H_{18}FN_3O_3S$: C, 62.40; H, 4.28; N, 9.92. Found: C, 62.37; H, 4.25; N, 9.86.

2-(4-Fluoro-2-nitrobenzylthio)-6-(4-fluorophenyl)-4-(furan-2-yl)-1,6-dihydropyrimidine (6j)

Yield: 71%; mp 217–219 °C; IR (υ cm^{-1}, KBr): 3 318 (NH), 1 560 (C=N), 1 543 (C=C), 852 (C–F) cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ 10.25 (s, 1H, NH-pyrimidine), 7.51–7.64 (m, 3H, Ar–H), 7.14 (d, 2H, Ar–H), 7.05 (d, 2H, Ar–H), 6.58–6.72 (m, 3H, furan), 6.19 (d, 1H, =CH), 5.20 (d, 1H, CH), 4.22 (s, 2H, SCH_2); LCMS: (*m/z*) 552 (M+H, 100%). Anal. calcd. for $C_{21}H_{15}F_2N_3O_3S$: C, 59.01; H, 3.54; N, 9.83. Found: C, 59.07; H, 3.51; N, 9.87.

2-(2,4-Dimethylbenzylthio)-6-(4-fluorophenyl)-4-(furan-2-yl)-1,6-dihydropyrimidine (6k)

Yield: 79%; mp 226–228 °C; IR (υ cm^{-1}, KBr): 3 321 (NH), 1 572 (C=N), 1 524 (C=C), 838 (C–F) cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ 10.06 (s, 1H, NH-pyrimidine), 7.27 (d, 2H, Ar–H), 7.15 (d, 2H, Ar–H), 7.01–7.05 (m, 3H, Ar–H), 6.64–6.81 (m, 3H, furan), 6.09 (d, 1H, =CH), 5.08 (d, 1H, CH), 4.12 (s, 2H, SCH_2), 2.19 (s, 6H, $2CH_3$); LCMS: (*m/z*) 565 ($(M)^+$, 100%). Anal. calcd. for $C_{23}H_{21}FN_2OS$: C, 70.38; H, 5.39; N, 7.14. Found: C, 70.43; H, 5.35; N, 7.19.

CONCLUSIONS

In conclusion, we have described a simple and efficient protocol for the synthesis of novel fluorophenyl, furan tagged benzylthiodihydropyrimidine derivatives (**6 a–k**) with good yields. These novel classes of new dihydropyrimidine derivatives reported have a probability to emerge as a valuable lead series with great potential to be used as agents, and as promising candidates for further efficacy evaluation.

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NAUJŲ FLUOROFENIL BENZILTIOPIRIMIDINO DARINIŲ SU PRIJUNGTĄJA FURANO ŽYMA SINTEZĖ

S a n t r a u k a

Nauji 6-(4-fluorofenil)-2-(pakeistų benziltio)-4-(furan-2-il)-1,6-dihidropirimidino dariniai per kelias stadijas susintetinti iš 2-acetilfurano.