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RESEARCH LETTER

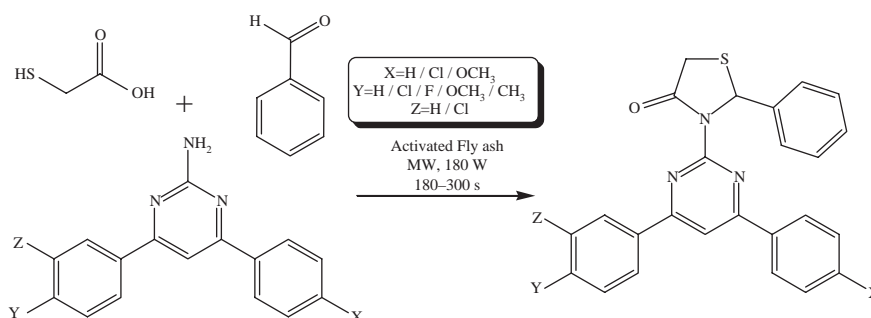
Three component one-pot synthesis of novel pyrimidino thiazolidin-4-ones catalyzed by activated fly ash

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2-Phenyl-3-(4,6-diarylpyrimidin-2-yl) thiazolidin-4-ones, 12–22, were synthesized with good yields in a short reaction time by the “one-pot” multicomponent reaction of the appropriate 2-amino-4,6-diarylpyrimidines, benzaldehyde, and thioglycolic acid under microwave irradiation in the presence of activated fly ash catalyst. The characterization of these compounds was confirmed by melting point, elemental analysis, MS, FT-IR, and one-dimensional NMR (^1H and ^{13}C) spectroscopic data.



Keywords: multicomponent reaction; 2-phenyl-3-(4,6-diarylpyrimidin-2-yl) thiazolidin-4-ones; microwave irradiation; thioglycolic acid; activated fly ash

Introduction

Nowadays the pharmaceutical industries are in need of new innovative alternate synthetic routes for synthesizing therapeutic and pharmacologically important compounds. Microwave activation as a non-conventional energy source has become a very popular and useful technology in organic chemistry. Microwave-assisted organic synthesis (MAOS) serves the need for accelerated chemical synthesis remarkably well, reducing times for the optimization and performance of reactions from hours or days to minutes. The environmental impact of organic chemical syntheses can be significantly reduced by incorporating cleaner unit processes. Solvent-free synthesis of organic compounds involving easily separable solid catalysts has attracted notable interest and offers a clean, economical and environmentally safe protocol. During the initial stage, only pozzolanic activity of fly

ash is paid attention (1,2). Many researchers devoted themselves to the research of the potential activity of fly ash and the hydration process of fly ash cement (3). Recently, microwave-assisted synthesis of “one-pot” conversion of ketones into amides, Knoevenagel condensation, Schiff bases formation, Biginelli and Hantzsch reactions were carried out using activated fly ash as catalyst (4). In addition, activated fly ash is used for the “one-pot” synthesis of 1,2,4,5-tetrazines (5) and 1,2,3-selenadiazoles (6).

Various 4-thiazolidinones have attracted considerable attention as they are endowed with wide range of pharmacological activities. Peptidoglycan is an essential component of the cell wall of both gram-positive and gram-negative bacteria. 4-thiazolidinones have been reported as novel inhibitors of the bacterial enzyme Mur B which is a precursor, acting during the biosynthesis of peptidoglycan (7). A wide

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variety of biological properties such as hypolipidaemic (8), antidegenerative (9), muscarinic receptor 1 agonist (10), antiproteolytic (11), anti-inflammatory (12), antiviral (13), antifungal (14), antibacterial (15), antitubercular (16), anticonvulsant (17), respiratory (18), and hypnotic (19) activities have been reported for 4-thiazolidinones.

Aminopyrimidine nuclei are common in marketed drugs such as anti-atherosclerotic aronixil, anti-histaminic thonzylamine, anti-anxiolytic buspirone, anti-psoriatic enazadrem, and other medicinally relevant compounds. Pyrimidines are the basic nucleus in nucleic acids and have been associated with a number of biological activities. Some notable biological activity of pyrimidine derivatives include adenosine receptor antagonists (20), kinase inhibitors (21), analgesic and anti-inflammatory (22), inhibitors of cyclin-dependent kinases 1 and 2 (23), calcium channel antagonist (24), antihistaminic (25), and antitubercular (26) activities.

In continuation of our interest in synthesizing pharmacologically important compounds in “dry media” (27,28), we planned and succeeded to synthesize a system, which comprises both 4-thiazolidinones and 2-amino-4,6-diarylpyrimidine components together to give a compact heterocyclic structure like the title 2-phenyl-3-(4,6-diarylpyrimidin-2-yl) thiazolidin-4-ones in “one-pot” catalyzed by activated fly ash under microwave irradiation.

Results and discussion

The fly ash collected from Neyveli Lignite Corporation, Neyveli, Tamil Nadu, India, was utilized for catalyzing the reactions. The physical properties, such as specific gravity and specific surface area, of fly ash used were 1.9 and 127 m²/g, respectively. The chemical compositions (%) of fly ash (3) used were SiO₂, Fe₂O₃, Al₂O₃, CaO, MgO, loss of ignition, and insoluble residue in the ratio 64.03, 6.50, 15.50, 4.62, 3.00, 4.35,

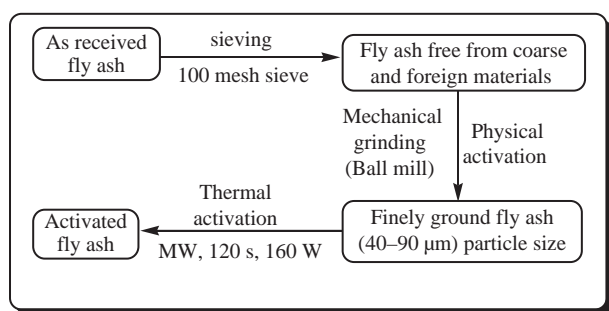
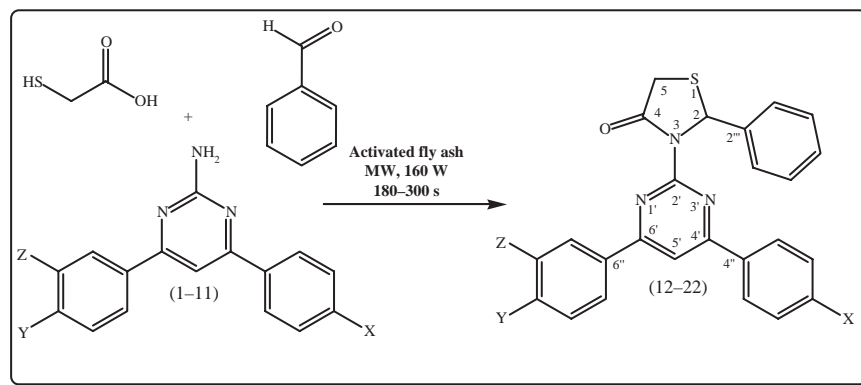


Figure 1. Flow chart for the preparation of activated fly ash.

and 2.00, respectively. The purpose of the present investigation is to activate the as-received fly ash by physical method followed by thermal method (Figure 1) and to study the influence of activated fly ash to catalyze the one-pot cyclization reaction for the formation of 2-phenyl-3-(4,6-diarylpyrimidin-2-yl) thiazolidin-4-ones.

The classical method available for the synthesis of thiazolidin-4-ones was the conversion of appropriate Schiff bases of respective amines and aldehyde by thioglycolic acid in refluxing benzene/dioxane catalyzed by *p*-toluene sulfonic acid/ZnCl₂ using a Dean-Stark apparatus for 12 h. Various problems were associated with the above synthesis such as severe reaction conditions using hazardous benzene as solvent, poor yields, difficulty in product isolation, and longer reaction times. In the present “one-pot” procedure, novel 2-phenyl-3-(4,6-diarylpyrimidin-2-yl) thiazolidin-4-ones **12–22** are synthesized by the addition of benzaldehyde and thioglycolic acid to 2-amino-4,6-diarylpyrimidines under microwave irradiation in the presence of catalytic amount of activated fly ash (50 mg) in high yields when compared with general conditions under microwave irradiation in solvent-free conditions. Initially, conversion of 2-amino-4,6-diarylpyrimidines **1–11** to 2-phenyl-3-(4,6-diarylpyrimidin-2-yl) thiazolidin-4-ones **12–22** was effected in the absence of activated fly ash. No yields were achieved. Instead, if activated fly ash was used as a dehydrating agent, the yield of the product has been improved significantly (i.e., about 95%) under microwave irradiation. The schematic representation and the analytical data of compounds **12–22** are given in Scheme 1 and Table 1, respectively. The structure of the newly synthesized compounds **12–22** is confirmed by melting point, elemental analysis, mass spectroscopy (MS), Fourier transform infrared (FT-IR), and one-dimensional nuclear magnetic resonance (NMR) (¹H and ¹³C) spectroscopic data.

The conversion of 2-phenyl-3-(4,6-diarylpyrimidin-2-yl) thiazolidin-4-ones **12–22** from 2-amino-4,6-diarylpyrimidines **1–11** by the present procedure was believed to be followed *via* 2-mercapto-N-(4,6-diarylpyrimidin-2-yl) acetamides and rapidly rearranged to give in the second step. The attempt to isolate the respective 2-mercapto-N-(4,6-diarylpyrimidin-2-yl) acetamides from the reaction mixture was unsuccessful. A plausible reaction mechanism (Scheme 2) has been proposed for the conversion of 2-amino-4,6-diarylpyrimidines to the 2-phenyl-3-(4,6-diarylpyrimidin-2-yl) thiazolidin-4-ones catalyzed by activated fly ash under microwave irradiation.



Scheme 1. Multicomponent reaction for the synthesis of novel 2-phenyl-3-(4,6-diarylpyrimidin-2-yl) thiazolidin-4-ones in “dry media” under microwave irradiation.

Experimental

Performing TLC assessed the reactions and the purity of the products. All the reported melting points were taken in open capillaries and were uncorrected. Infra-red (IR) spectra were recorded in KBr (pellet forms) on a Nicolet-Avatar-330 FT-IR spectrophotometer and noteworthy absorption values (cm^{-1}) alone are listed. ^1H and ^{13}C NMR spectra were recorded at 400 and 100 MHz, respectively on Bruker AMX 400 NMR spectrometer using $\text{DMSO-}d_6$ as solvent. The ESI +ve MS spectra were recorded on a Bruker Daltonics LC-MS spectrometer. Satisfactory microanalysis was obtained on Carlo Erba 1106 CHN analyzer. BIOTAGE Initiator microwave synthesizer, a Swedish scientific microwave oven, was used for the irradiation. By adopting the literature precedent 2-amino-4,6-diarylpyrimidines **1–11** (29), was synthesized.

Experimental method for the synthesis of 2-phenyl-3-(4,6-diphenylpyrimidin-2-yl) thiazolidin-4-one **12**

A mixture containing 0.01 mole of 2-amino-4,6-diphenylpyrimidine **1**, 0.01 mole of thioglycolic acid, 0.01 mole of benzaldehyde, and activated fly ash (50 mg) was added in an alumina bath and mixed properly with the aid of a glass rod (10 s) and then irradiated in a microwave oven for 180 s at 160 W (monitored by thin layer chromatography, TLC). After completion of the reaction, the reaction mixture was extracted with ethyl acetate (3×5 mL). The catalyst and other solid wastes were removed by filtration. The combined organic layer was washed with 10% sodium bicarbonate solution followed by water three times and then dried over anhydrous MgSO_4 . The organic layer was concentrated *in vacuo* to furnish the products, which were purified by column chromatography using silica gel (100–200 mesh), with ethyl acetate – petroleum ether (bp 40–60) in the ratio (2:8) as eluent. IR (KBr) (cm^{-1}):

3125, 3033, 2927, 2851, 1716, 1627, 1576, 1350, 710, 698, 649; ^1H NMR (δ ppm): 3.21 (d, 1H, CH_{2a} at H_{5a} , $J=15.37$ Hz), 3.38 (d, 1H, CH_{2e} at H_{5e} , $J=15.37$ Hz), 5.25 (s, 1H, CH at H_2), 7.19–8.37 (m, 16H, H_{arom}). A singlet for CH proton at position 5' of pyrimidine moiety is merged with aromatic protons. ^{13}C NMR (δ ppm): 34.0 C-5, 62.5 C-2, 108.1 C-5', 131.4 C-2'', 125.9–128.8 $-\text{C}_{\text{arom}}$, 139.1 C-4'', 139.1 C-6'', 161.3 C-4', 161.3 C-6', 163.8 C-2', 170.6 C-4.

The compounds **13–22** were synthesized correspondingly

3-(4'-(4''-chlorophenyl)-6'-phenylpyrimidin-2'-yl)-2-phenylthiazolidin-4-one

13 IR (KBr) (cm^{-1}): 3120, 3033, 2927, 2851, 1696, 1627, 1575, 1310, 894, 710, 650, 647; ^1H NMR (δ ppm): 3.22 (d, 1H, CH_{2a} at H_{5a} , $J=15.37$ Hz), 3.39 (d, 1H, CH_{2e} at H_{5e} , $J=15.34$ Hz), 5.27 (s, 1H, CH at H_2), 7.31–8.44 (m, 15H, H_{arom}). A singlet for CH proton at position 5' of pyrimidine moiety is merged with aromatic protons. ^{13}C NMR (δ ppm): 33.9 C-5, 62.6 C-2, 108.8 C-5', 127.5–133.1 $-\text{C}_{\text{arom}}$, 131.4 C-2'', 135.9 *ipso* C, 139.1 C-4'', 139.7 C-6'', 164.9 C-4', 165.0 C-6', 162.9 C-2', 170.6 C-4.

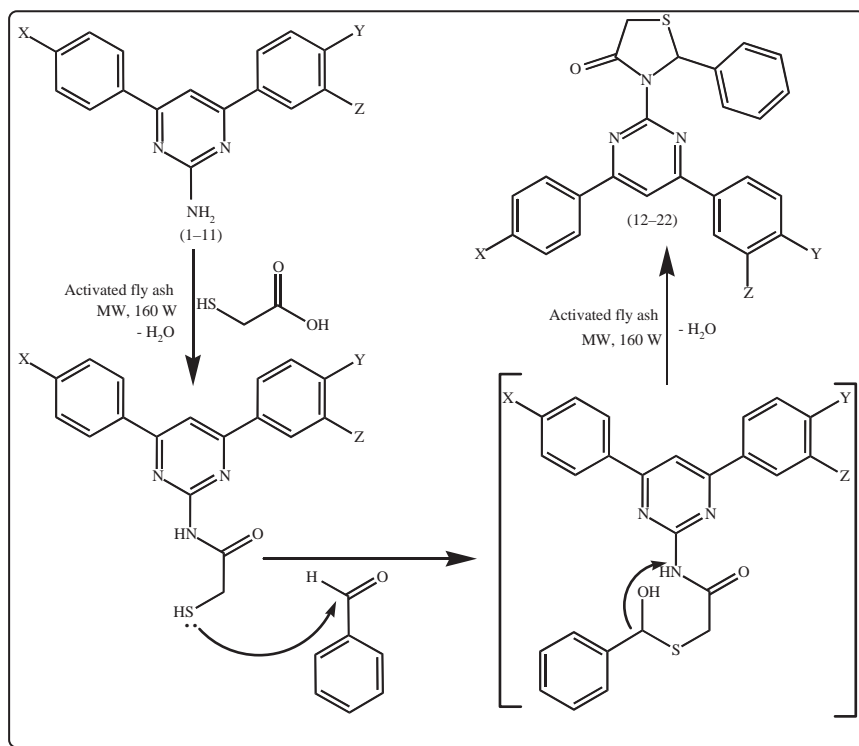
3-(4'-(3''-chlorophenyl)-6'-phenylpyrimidin-2'-yl)-2-phenylthiazolidin-4-one

14 IR (KBr) (cm^{-1}): 3115, 3033, 2927, 2850, 1714, 1627, 1575, 1344, 894, 767, 690, 648; ^1H NMR (δ ppm): 3.22 (d, 1H, CH_{2a} at H_{5a} , $J=15.36$ Hz), 3.39 (d, 1H, CH_{2e} at H_{5e} , $J=15.37$ Hz), 5.27 (s, 1H, CH at H_2), 7.21–8.24 (m, 15H, H_{arom}). A singlet for CH proton at position 5' of pyrimidine moiety is merged with aromatic protons. ^{13}C NMR (δ ppm): 33.9 C-5, 62.4 C-2, 108.9 C-5', 124.6–129.0 $-\text{C}_{\text{arom}}$, 130.6 *ipso* C, 131.5 C-2'', 139.0 C-4'', 141.8 C-6'', 164.9 C-4', 165.5 C-6', 162.7 C-2', 170.6 C-4.

Table 1. Physical and analytical data of 2-phenyl-3-(4,6-diarylpyrimidin-2-yl) thiazolidin-4-ones **12–22**.

Entry	X	Z	Y	Yield (%)	Reaction time (s)	m.p.°C	Elemental analysis (%)			m/z (M+1) ⁺ Molecular formula
							C Found (calculated)	H Found (calculated)	N Found (calculated)	
12	H	H	H	92 (65)	180 (620)	145	73.31 (73.35)	4.60 (4.64)	10.23 (10.26)	410 C ₂₅ H ₁₉ N ₃ OS
13	H	H	Cl	90 (52)	240 (900)	162	67.65 (67.66)	4.04 (4.06)	9.41 (9.46)	444 C ₂₅ H ₁₈ Cl N ₃ OS
14	H	Cl	H	85 (49)	300 (720)	130	67.62 (67.66)	4.01 (4.06)	9.44 (9.46)	444 C ₂₅ H ₁₈ Cl N ₃ OS
15	H	H	OCH ₃	95 (60)	120 (600)	110	71.03 (71.08)	4.72 (4.78)	9.51 (9.56)	440 C ₂₆ H ₂₁ N ₃ O ₂ S
16	H	H	CH ₃	90 (65)	180 (600)	162	73.72 (73.76)	4.92 (4.96)	9.89 (9.92)	424 C ₂₆ H ₂₁ N ₃ OS
17	H	H	F	85 (70)	240 (720)	104	70.23 (70.27)	4.18 (4.21)	9.79 (9.83)	428 C ₂₅ H ₁₈ FN ₃ OS
18	Cl	H	H	85 (65)	300 (900)	114	67.64 (67.66)	4.01 (4.05)	9.41 (9.46)	444 C ₂₅ H ₁₈ Cl N ₃ OS
19	OCH ₃	H	H	90 (58)	240 (780)	137	71.02 (71.08)	4.72 (4.78)	9.55 (9.56)	440 C ₂₆ H ₂₁ N ₃ O ₂ S
20	Cl	H	CH ₃	85 (55)	300 (600)	125	68.17 (68.21)	4.32 (4.36)	9.15 (9.17)	458 C ₂₆ H ₂₀ ClN ₃ OS
21	Cl	H	Cl	88 (60)	240 (600)	130	62.71 (62.78)	3.52 (3.55)	8.76 (8.78)	479 C ₂₅ H ₁₇ Cl ₂ N ₃ OS
22	Cl	H	F	90 (65)	240 (660)	103	64.98 (65.02)	3.65 (3.68)	9.04 (9.09)	462 C ₂₅ H ₁₇ ClFN ₃ OS

Note: The values in parentheses are the reaction conditions in classical method.



Scheme 2. Proposed mechanism for the formation of pyrimidino thiazolidin-4-ones.

3-(4'-(4''-methoxyphenyl)-6'-phenylpyrimidin-2'-yl)-2-phenylthiazolidin-4-one

15 IR (KBr) (cm^{-1}): 3065, 3038, 2927, 2851, 1714, 1627, 1577, 1351, 700, 650, 649; ^1H NMR (δ ppm): 3.23 (d, 1H, CH_{2a} at H_{5a} , $J = 15.35$ Hz), 3.39 (d, 1H, CH_{2e} at H_{5e} , $J = 15.27$ Hz), 3.84 (s, 3H, OCH_3), 5.28 (s, 1H, CH at H_2), 7.21–8.21 (m, 15H, H_{arom}). A singlet for CH proton at position 5' of pyrimidine moiety is merged with aromatic protons. ^{13}C NMR (δ ppm): 34.5 C-5, 54.9 OCH_3 on aryl ring, 62.5 C-2, 108.7 C-5', 126.0–128.6 $-\text{C}_{\text{arom}}$, 129.1 C-2'', 139.1 C-4'', 141.5 C-6'', 164.0 C-4', 165.0 C-6', 162.3 C-2', 170.6 C-4.

3-(4'-(4''-methylphenyl)-6'-phenylpyrimidin-2'-yl)-2-phenylthiazolidin-4-one

16 IR (KBr) (cm^{-1}): 3060, 3033, 2926, 2852, 1715, 1627, 1579, 1350, 714, 700, 643.; ^1H NMR (δ ppm): 2.32 (s, 3H, CH_3), 3.23 (d, 1H, CH_{2a} at H_{5a} , $J = 15.34$ Hz), 3.40 (d, 1H, CH_{2e} at H_{5e} , $J = 15.36$ Hz), 5.27 (s, 1H, CH at H_2), 7.20–8.24 (m, 15H, H_{arom}). A singlet for CH proton at position 5' of pyrimidine moiety is merged with aromatic protons. ^{13}C NMR (δ ppm): 24.5 CH_3 on aryl ring, 34.1 C-5, 62.6 C-2, 108.4 C-5', 126.0–131.4 $-\text{C}_{\text{arom}}$, 133.1 C-2'', 135.9 *ipso* C, 138.7 C-4'', 139.1 C-6'', 164.9 C-4', 165.5 C-6', 162.6 C-2', 170.8 C-4.

3-(4'-(4''-fluorophenyl)-6'-phenylpyrimidin-2'-yl)-2-phenylthiazolidin-4-one

17 IR (KBr) (cm^{-1}): 3071, 3027, 2928, 2852, 1712, 1626, 1575, 1352, 836, 769, 698; ^1H NMR (δ ppm): 3.20 (d, 1H, CH_{2a} at H_{5a} , $J = 15.24$ Hz), 3.37 (d, 1H, CH_{2e} at H_{5e} , $J = 15.28$ Hz), 5.26 (s, 1H, CH at H_2), 6.64–8.19 (m, 15H, H_{arom}). A singlet for CH proton at position 5' of pyrimidine moiety is merged with aromatic protons. ^{13}C NMR (δ ppm): 34.5 C-5, 62.9 C-2, 108.9 C-5', 127.3–143.1 $-\text{C}_{\text{arom}}$, 143.6 C-2'', 145.1 C-4'', 146.1 C-6'', 166.8 C-4', 167.0 C-6', 163.9 C-2', 171.4 C-4.

3-4'-phenyl-(6'-(4''-chlorophenyl) pyrimidin-2'-yl)-2-phenylthiazolidin-4-one

18 IR (KBr) (cm^{-1}): 3071, 3027, 2926, 2852, 1721, 1627, 1576, 1398, 782, 730, 693, 582; ^1H NMR (δ ppm): 3.21 (d, 1H, CH_{2a} at H_{5a} , $J = 15.33$ Hz), 3.38 (d, 1H, CH_{2e} at H_{5e} , $J = 15.32$ Hz), 5.25 (s, 1H, CH at H_2), 7.15–7.93 (m, 15H, H_{arom}). A singlet for CH proton at position 5' of pyrimidine moiety is merged with aromatic protons. ^{13}C NMR (δ ppm): 34.1 C-5, 62.9 C-2, 108.9 C-5', 126.5–128.6 $-\text{C}_{\text{arom}}$, 129.2 C-2'', 139.1 C-4'', 141.9 C-6'', 164.4 C-4', 165.3 C-6', 162.5 C-2', 170.8 C-4.

3-(4'-phenyl-(6'-(4''-methoxyphenyl) pyrimidin-2'-yl))-2-phenylthiazolidin-4-one

19 IR (KBr) (cm^{-1}): 3065, 3033, 2928, 2851, 1715, 1627, 1590, 1370, 835, 770, 699, 656; ^1H NMR (δ ppm): 3.20 (d, 1H, CH_{2a} at H_{5a} , $J=15.08$ Hz), 3.37 (d, 1H, CH_{2e} at H_{5e} , $J=15.34$ Hz), 3.86 (s, 3H, OCH_3), 5.26 (s, 1H, CH at H_2), 6.97–8.20 (m, 15H, H_{arom}). A singlet for CH proton at position 5' of pyrimidine moiety is merged with aromatic protons. ^{13}C NMR (δ ppm): 34.1 C-5, 55.2 OCH_3 on aryl ring, 62.5 C-2, 108.6 C-5', 114.1–129.1 $-\text{C}_{\text{arom}}$, 129.5 C-2'', 130.2 *ipso* C, 139.2 C-6'', 146.1 C-4'', 163.8 C-4', 164.4 C-6', 161.2 C-2', 170.7 C-4.

3-(4'-(4''-chlorophenyl)-6'-(p-tolylpyrimidin-2'-yl))-2-phenylthiazolidin-4-one

20 IR (KBr) (cm^{-1}): 3060, 3027, 2927, 2851, 1721, 1626, 1576, 1398, 781, 728, 694, 650; ^1H NMR (δ ppm): 2.40 (s, 3H, CH_3), 3.20 (d, 1H, CH_{2a} at H_{5a} , $J=15.04$ Hz), 3.37 (d, 1H, CH_{2e} at H_{5e} , $J=15.11$ Hz), 5.26 (s, 1H, CH at H_2), 7.18–8.33 (m, 14H, H_{arom}). A singlet for CH proton at position 5' of pyrimidine moiety is merged with aromatic protons. ^{13}C NMR (δ ppm): 25.2 CH_3 on aryl ring, 34.5 C-5, 62.9 C-2, 108.9 C-5', 131.4 C-2'', 126.1–130.4 $-\text{C}_{\text{arom}}$, 133.1 *ipso* C, 138.7 C-6'', 139.1 C-4'', 164.8 C-4', 165.0 C-6', 161.2 C-2', 170.8 C-4.

3-(4',6'-bis(p-chlorophenyl) pyrimidin-2'-yl)-2-phenylthiazolidin-4-one

21 IR (KBr) (cm^{-1}): 3060, 3027, 2927, 2852, 1727, 1627, 1575, 1400, 897, 787, 730, 693; ^1H NMR (δ ppm): 3.20 (d, 1H, CH_{2a} at H_{5a} , $J=15.21$ Hz), 3.36 (d, 1H, CH_{2e} at H_{5e} , $J=15.23$ Hz), 5.26 (s, 1H, CH at H_2), 7.28–8.20 (m, 14H, H_{arom}). A singlet for CH proton at position 5' of pyrimidine moiety is merged with aromatic protons. ^{13}C NMR (δ ppm): 34.5 C-5, 62.5 C-2, 108.7 C-5', 129.1 C-2'', 126.0–128.6 $-\text{C}_{\text{arom}}$, 139.1 C-6'', 141.8 C-4'', 164.1 C-4', 161.3 C-6', 165.3 C-2', 170.9 C-4.

3-(4'-(p-chlorophenyl)-6'-(p-fluorophenyl) pyrimidin-2'-yl)-2-phenylthiazolidin-4-one

22 IR (KBr) (cm^{-1}): 3065, 3027, 2926, 2853, 1719, 1627, 1576, 1394, 897, 833, 776, 728, 695; ^1H NMR (δ ppm): 3.18 (d, 1H, CH_{2a} at H_{5a} , $J=14.92$ Hz), 3.35 (d, 1H, CH_{2e} at H_{5e} , $J=14.92$ Hz), 5.26 (s, 1H, CH at H_2), 7.18–8.19 (m, 14H, H_{arom}). A singlet for CH proton at position 5' of pyrimidine moiety is merged with aromatic protons. ^{13}C NMR (δ ppm): 34.1 C-5, 62.6 C-2, 110.1 C-5', 133.2 C-2'', 127.3–143.1 $-\text{C}_{\text{arom}}$, 145.1 C-6'', 146.8 C-4'', 166.8 C-4', 167.0 C-6', 163.4 C-2', 171.8 C-4.

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