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EXPERIMENTAL PAPER



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Soluble Glass, an Efficient Promoter for the Cascade Addition-Cyclization Reaction of 4-Hydroxycoumarins to Chalcone Derivatives

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The combination of two or more pharmacophoric subunits is an important and efficient strategy in organic synthesis for the construction and architecture of more effective drugs. With the use of this strategy, the original characteristics of the parent molecules remain, but the progeny molecules may also show entirely new medicinal aspects. Pyrano[3,2-c]chromen-5-one skeletons are good examples: they were designed by combining a coumarin moiety and an α,β -unsaturated carbonyl structure. Various α,β -unsaturated carbonyl compounds are used as anti-cancer, anti-inflammatory, anti-tuberculosis, and anti-fungal materials.¹⁻² On the other hand, naturally extracted compounds such as the coumarins are accessible scaffolds for constructing anticancer, antimicrobial, antiviral, antioxidant, and anti-inflammatory drugs.³⁻⁷

Among the methods that have been used for the synthesis of pyrano[3,2-c]chromene derivatives, the combination of 4-hydroxycoumarin and α,β -unsaturated carbonyl compounds is of great interest due to the simplicity and efficiency of the method and this was reported by Moreau and coworkers using a Brønsted acidic catalyst.⁸ Subsequently, AuCl₃/Ag(OTf)₃,⁹ SiO₂-ZnCl₂,¹⁰ I₂/H₂SO₄,¹¹ Cu(OTf)₂,¹² Al(OTf)₃,¹³ DDQ,¹⁴ molecular iodine,¹⁵ ZnCl₂,¹⁶ and alkaline earth metal¹⁷ catalytic systems have been developed for this transformation. This becomes all the more important in the light of the fact that compounds comprised of pyran and coumarin structural units are known for anticancer, anti-HIV, anti-inflammatory, and anti-hepatitis activities. Thus, significant efforts have been expended to make combinations of these nuclei.^{18–29} Recognizing the drug potential of pyrano[3,2-c]chromene core structures has encouraged us to provide a new method for the reaction of 4-hydroxycoumarins and α,β -unsaturated carbonyl compounds.

In this, soluble glass is used to promote the production of 2,4-diaryl-4H,5H-pyr-ano[3,2-c]chromen-5-one derivatives in aqueous media (Scheme 1).

We evaluated the feasibility of the synthesis of 1,3-diphenylpyrano[2,3-c]chromen-5(1H)one using soluble glass under different conditions. To begin this, 4-hydroxycoumarin (1 mmol) and chalcone (1 mmol) were combined with soluble glass in several solvents (CH₂Cl₂, hexane, EtOAc, EtOH and water) under reflux conditions for 3h. None of the desired product was detected in CH₂Cl₂ and hexane. The product was obtained in EtOAc

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Scheme 1. Synthesis of pyrano[3,2-c]coumarin derivatives.

| Entry | R^1 | R ² | R ³ | R^4 | R⁵ | Product | Time (h) | Yield (%) ^a | m.p. (°C) [Lit. m.p.] ^{Ref.} |
|-------|------------------|----------------|----------------|------------------|-------------|---------|----------|------------------------|---------------------------------------|
| 1 | Н | Н | Н | Н | Н | 3a | 3 | 87 | 172-174 [173-175] ¹¹ |
| 2 | Н | Н | Н | CH₃ | Н | 3b | 4 | 85 | 193-195 [192-194] ¹³ |
| 3 | Н | CH₃ | Н | Н | Н | 3c | 6 | 83 | 183-185 [184-186] ¹³ |
| 4 | Н | Н | Н | OCH ₃ | Н | 3d | 4.5 | 79 | 133-135 [132-138] ¹³ |
| 5 | Н | Н | Н | Cl | Н | 3e | 2.5 | 88 | 196-198 [196-197] ¹¹ |
| 6 | Н | Н | Cl | Н | Н | 3f | 2.5 | 80 | 201-203 |
| 7 | Н | Cl | Н | Н | Н | 3g | 3 | 77 | 189-191 |
| 8 | Н | Н | NO_2 | Н | Н | 3h | 2.5 | 89 | 227-229 [-] ¹² |
| 9 | Н | Н | Н | NO_2 | Н | 3i | 2.5 | 86 | 232-234 [230-231] ⁹ |
| 10 | Н | Н | Н | Br | Н | 3j | 3 | 96 | 212-214 [210-211] ¹¹ |
| 11 | Н | Н | Н | Н | CH₃ | 3k | 3.5 | 78 | 155-157 [156-158] ⁹ |
| 12 | Н | Н | Н | Cl | CH₃ | 31 | 3 | 88 | 226-228 [225-227] ⁹ |
| 13 | Н | Н | Н | Н | $C(CH_3)_3$ | 3m | 3 | 88 | 209-211 |
| 14 | Н | Н | Н | OCH ₃ | Cl | 3n | 3 | 94 | 221-223 [-] ¹² |
| 15 | CH₃ | Н | Н | Н | Н | Зо | 3 | 90 | 213-215 [212-213] ¹⁴ |
| 16 | Cl | Н | Н | Н | Н | 3р | 2.5 | 88 | 233-235 [231-232] ⁹ |
| 17 | Н | Н | Н | Cl | Cl | 3q | 2.5 | 92 | 243-245 [239-241] ⁹ |
| 18 | CH₃ | Н | Н | CH₃ | Н | 3r | 4 | 89 | 208-210 [210-211] ⁹ |
| 19 | Н | Н | OCH₃ | Н | Н | 3s | 4 | 82 | 146-148 [144-146] ¹¹ |
| 20 | Н | Н | Н | Н | Br | 3t | 3 | 86 | 203-205 [202-203] ⁹ |
| 21 | CH₃ | Н | Н | OCH ₃ | Н | 3u | 5 | 87 | 220-222 [218-219] ⁹ |
| 22 | OCH ₃ | Н | Н | Н | Н | 3v | 4 | 80 | 205-207 [206-207] ⁹ |

Table 1. Synthesis of pyrano[3,2-c]coumarin derivatives (3a-z) using liquid glass as a catalyst.

^alsolated yields.

and EtOH in low yields (EtOAc (20%), EtOH (40%)). Interestingly, the product was prepared in an aqueous environment with high yields (87%). In solvent-free media, a complex mixture was formed, and only 15% of product was obtained following workup.

Next, a series of chalcone derivatives were subjected to this approach and the results are summarized in Table 1. In each case, the desired product was produced in good to excellent yields. We observed that, when the donor groups are substituted, the rate of the reaction is slowed.

In continuation of our research,³⁰ we examined cognate preparations of 5-amino-7-aryl-7,8-dihydro-[1,2,4]triazolo[4,3-a]pyrimidine-6-carbonitrile derivatives (Scheme 2, Table 2, products **7a-7j**). All reactions were completed in short reaction times with high yields. A series of aldehyde derivatives containing different substitutions were subjected to the reaction. In all of these reactions the catalyst had satisfactory performance and high efficiency.

In summary, we have demonstrated a cascade addition-cyclization reaction of 4hydroxycoumarins with chalcone derivatives for the synthesis of functionalized 2,4-



Scheme 2. Synthesis of 5-amino-7-aryl-7,8-dihydro-[1,2,4]triazolo[4,3-a]pyrimidine-6-carbonitrile derivatives (7a-j).

 Table 2.
 Synthesis of 5-amino-7-aryl-7,8-dihydro-[1,2,4]triazolo[4,3-a]pyrimidine-6-carbonitrile derivatives using liquid glass as a catalyst.

| Entry | Ar | Product | Time (min) | Yield (%) ^a | m.p. (°C) [Lit. m.p.] ^{Ref.} |
|-------|---------------------------------------|---------|------------|------------------------|---------------------------------------|
| 1 | Ph | 7a | 50 | 85 | 235-237 |
| 2 | 4-CIC ₆ H ₄ | 7b | 45 | 90 | 259-261 [257-258] ³¹ |
| 3 | 2-CIC ₆ H ₄ | 7c | 50 | 86 | 262-264 [263-266] ³¹ |
| 4 | 4-MeC ₆ H ₄ | 7d | 70 | 79 | 247-249 [245-246] ³¹ |
| 5 | 4-MeOC ₆ H ₄ | 7e | 70 | 85 | 222-224 [218-219] ³¹ |
| 6 | 4-BrC ₆ H ₄ | 7f | 45 | 94 | 267-269 [264-266] ³¹ |
| 7 | 3,4-diMeOC ₆ H₃ | 7g | 80 | 81 | 239-241 [235-237] ³¹ |
| 8 | $4-NO_2C_6H_4$ | 7ĥ | 45 | 89 | 245-247 [245-247] ³¹ |
| 9 | 2,4-diClC ₆ H ₃ | 7i | 50 | 86 | 276-278 [274-277] ³¹ |
| 10 | 3-CIC ₆ H ₄ | 7j | 45 | 90 | 251-253 |

^alsolated yield.

diaryl-4H,5H-pyrano[3,2-c]chromen-5-ones using soluble glass as a promoter in aqueous media. This methodology offers a straightforward and rapid access to the pyrano[3,2-c]chromen-5-one structures. We hope that our results will stimulate further investigations of these useful compounds in drug design and discovery.

Experimental section

All reagents were purchased from Merck and used without further purification. The NMR spectra were recorded on a Bruker Avance DPX 400 MHz instrument in DMSO- d_6 as a solvent. Melting points were determined in open capillaries with a BUCHI 510 melting point apparatus and are uncorrected. TLC was performed on silica gel Polygram SIL G/UV 254 plates. Elemental analysis was performed on a Heraeus CHN-O-Rapid analyzer.

Synthesis of pyrano[3,2-c]coumarin derivatives (3a-v); General procedure

A mixture of 4-hydroxycoumarin (1.0 mmol), chalcone (1.0 mmol), and five drops of soluble glass (10% w/v) was refluxed in water for the appropriate time (Scheme 1). The progress of the reaction was monitored by TLC (silica gel, eluent: ethyl acetate/hexane 1/4). After completion of the reaction, the reaction mixture was cooled to room temperature and filtered. The solid was purified by recrystallization from EtOH. Known compounds were identified by matching their melting points with those of the literature cited in the tables and/or were fully characterized. All novel compounds were fully characterized.

2,4-Diphenyl-4H,5H-pyrano[3,2-c]chromen-5-one (3a)

¹H-NMR (400 MHz, DMSO- d_6): $\delta = 4.68$ (d, J = 5.1 Hz, 1H, CH-sp³), 5.85 (d, J = 5.3 Hz, 1H), 7.24 (t, J = 7.7 Hz, 1H), 7.28–7.47 (m, 9H), 7.57 (t, J = 7.8 Hz, 1H), 7.75 (d, J = 7.8 Hz, 2H), 8.02 (d, J = 7.8 Hz, 1H) ppm; ¹³C-NMR (100 MHz, DMSO- d_6): $\delta = 36.8$, 103.9, 104.4, 115.3, 116.9, 123.1, 124.2, 125.2, 127.4, 128.6, 128.9, 129.1, 129.5, 131.6, 132.3, 143.2, 147.3, 152.7, 156.1, 161.3 ppm.

Anal. Calcd for C₂₄H₁₆O₃: C, 81.80; H, 4.58. Found: C, 81.73; H, 4.48.

2-Phenyl-4-(p-tolyl)-4H,5H-pyrano[3,2-c]chromen-5-one (3b)

¹H NMR (400 MHz, DMSO- d_6): $\delta = 2.32$ (s, 3H, CH₃), 4.70 (d, J = 4.9 Hz, 1H), 5.82 (d, J = 5.0 Hz, 1H), 7.11 (d, J = 8.0 Hz, 2H), 7.26-7.47 (m, 7H), 7.55 (d, J = 8.0 Hz, 1H), 7.71 (d, J = 7.8 Hz, 2H), 8.02 (d, J = 7.8 Hz, 1H) ppm.

Anal. Calcd for C₂₅H₁₈O₃: C, 81.95; H, 4.95. Found: C, 81.90; H, 4.88.

4-(4-Methoxy-phenyl)-2-phenyl-4H-pyrano[3,2-c]chromen-5-one (3d)

¹H NMR (400 MHz, DMSO- d_6): $\delta = 3.76$ (s, 3H, OCH₃), 4.69 (d, J = 5.1Hz, 1H), 5.81 (d, J = 5.3 Hz, 1H), 6.89 (d, J = 8.0 Hz, 2H), 7.28-7.47 (m, 7H), 7.62 (t, J = 7.8 Hz, 1H), 7.71 (d, J = 7.8Hz, 2H), 8.04 (d, J = 7.9 Hz, 1H) ppm.

Anal. Calcd for C₂₅H₁₈O₄: C, 78.45; H, 4.70. Found: C, 78.38; H, 4.61.

4-(4-Chlorophenyl)-2-phenyl-4H,5H-pyrano[3,2-c]chromen-5-one (3e)

¹H-NMR (400 MHz, DMSO- d_6): $\delta = 4.73$ (d, J = 5.1 Hz, 1H, CH-sp³), 5.87 (d, J = 5.3 Hz, 1H), 7.24 (t, J = 7.6 Hz, 1H), 7.29–7.48 (m, 9H), 7.59 (t, J = 7.7 Hz, 1H), 7.75 (d, J = 7.6 Hz, 2H), 8.02 (d, J = 8.0 Hz, 1H) ppm; ¹³C-NMR (100 MHz, DMSO- d_6): $\delta = 36.9$, 104.6, 110.4, 115.3, 116.4, 123.2, 123.6, 126.2, 128.5, 128.9, 129.0, 129.7, 129.9, 131.3, 136.7, 141.3, 147.8, 152.7, 156.3, 161.3 ppm.

Anal. Calcd for C₂₄H₁₅ClO₃: C, 74.52; H, 3.91. Found: C, 74.44; H, 3.83.

4-(3-Chlorophenyl)-2-phenyl-4H,5H-pyrano[3,2-c]chromen-5-one (3f)

¹H-NMR (400 MHz, DMSO- d_6): $\delta = 4.70$ (d, J = 5.4 Hz, 1H, CH-sp³), 5.87 (d, J = 5.3 Hz, 1H), 7.18-7.46 (m, 9H), 7.58 (t, J = 8.0 Hz, 1H), 7.74 (d, J = 7.8 Hz, 2H), 8.03 (d, J = 8.0 Hz, 1H) ppm; ¹³C-NMR (100 MHz, DMSO- d_6): $\delta = 36.7$, 104.7, 110.3, 115.4, 116.4, 123.2, 123.7, 126.2, 127.8, 128.0, 128.7, 129.0, 129.1, 129.6, 129.9, 130.8, 131.3, 141.4, 148.2, 152.7, 156.5, 161.4 ppm.

Anal. Calcd for C₂₄H₁₅ClO₃: C, 74.52; H, 3.91. Found: C, 74.48; H, 3.86.

4-(2-Chlorophenyl)-2-phenyl-4H,5H-pyrano[3,2-c]chromen-5-one (3g)

¹H-NMR (400 MHz, DMSO- d_6): $\delta = 4.71$ (d, J = 5.3 Hz, 1H, CH-sp³), 5.86 (d, J = 5.2 Hz, 1H), 7.29-7.47 (m, 9H), 7.57 (t, J = 7.9 Hz, 1H), 7.73 (d, J = 7.8 Hz, 2H), 8.00 (d, J = 8.0 Hz, 1H) ppm; ¹³C-NMR (100 MHz, DMSO- d_6): $\delta = 36.5$, 104.6, 110.3, 115.5,

116.5, 123.2, 123.6, 126.3, 128.3, 128.7, 129.1, 129.4, 129.8, 130.3, 130.6, 131.3, 132.9, 148.2, 152.6, 156.6, 161.3 ppm.

Anal. Calcd for C₂₄H₁₅ClO₃: C, 74.52; H, 3.91. Found: C, 74.45; H, 3.88.

4-(3-Nitro-phenyl)-2-phenyl-4H-pyrano[3,2-c]chromen-5-one (3h)

¹H-NMR (400 MHz, DMSO- d_6): $\delta = 4.89$ (d, J = 4.9 Hz, 1H, CH-sp³), 5.84 (d, J = 5.1 Hz, 1H), 7.37–7.55 (m, 6H), 7.64 (t, J = 7.6 Hz, 1H), 7.78 (d, J = 7.8 Hz, 2H), 7.83 (d, J = 7.8 Hz, 1H), 8.04 (d, J = 7.9 Hz, 1H), 8.12 (d, J = 7.9 Hz, 1H), 8.26 (d, J = 7.9 Hz, 1H) ppm.

Anal. Calcd for $C_{24}H_{15}NO_5$: C, 72.54; H, 3.80; N, 3.52. Found: C, 72.48; H, 3.76; N, 3.45.

4-(4-Bromophenyl)-2-phenyl-4H,5H-pyrano[3,2-c]chromen-5-one (3j)

¹H-NMR (400 MHz, DMSO- d_6): $\delta = 4.71$ (d, J = 4.8 Hz, 1H, CH-sp³), 5.80 (d, J = 4.9 Hz, 1H), 7.25-7.49 (m, 9H), 7.62 (t, J = 7.8 Hz, 1H), 7.72 (d, J = 7.8 Hz, 2H), 8.02 (d, J = 7.9 Hz, 1H) ppm.

Anal. Calcd for C₂₄H₁₅BrO₃: C, 66.84; H, 3.51. Found: C, 66.75; H, 3.46.

4-(4-(tert-Butyl)phenyl)-2-phenyl-4H,5H-pyrano[3,2-c]chromen-5-one (3m)

¹H-NMR (400 MHz, DMSO-*d*₆): $\delta = 1.27$ (s, 9H), 4.69 (d, J = 5.1 Hz, 1H, CH-sp³), 5.85 (d, J = 5.0 Hz, 1H), 7.02 (d, J = 7.8 Hz, 2H), 7.16 (d, J = 7.8 Hz, 2H), 7.29-7.46 (m, 5H), 7.55 (t, J = 7.8 Hz, 1H), 7.72 (d, J = 7.8 Hz, 2H), 8.02 (d, J = 7.9 Hz, 1H) ppm; ¹³C-NMR (100 MHz, DMSO-*d*₆): $\delta = 31.3$, 34.4, 36.4, 104.2, 110.4, 115.5, 116.5, 123.2, 123.7, 125.6, 125.9, 126.3, 126.9, 128.8, 129.1, 129.7, 131.3, 141.4, 152.3, 153.4, 156.7, 159.7, 161.4 ppm. *Anal.* Calcd for C₂₈H₂₄O₃: C, 82.33; H, 5.92. Found: C, 82.27; H, 5.89.

2-(4-Chloro-phenyl)-4-(4-methoxy-phenyl)-4H-pyrano[3,2-c]chromen-5-one (3n)

¹H-NMR (400 MHz, DMSO- d_6): $\delta = 3.78$ (s, 3H, OCH₃), 4.69 (d, J = 5.0 Hz, 1H, CH-sp³), 5.84 (d, J = 5.2 Hz, 1H), 6.89 (d, J = 8.0 Hz, 2H), 7.31–7.46 (m, 6H), 7.63 (t, J = 7.6 Hz, 1H), 7.69 (d, J = 7.9 Hz, 2H), 8.03 (d, J = 7.9 Hz, 1H) ppm. Anal. Calcd for C₂₅H₁₇ClO₄: C, 72.03; H, 4.11. Found: C, 71.94; H, 4.03.

9-Methyl-2,4-diphenyl-4H,5H-pyrano[3,2-c]chromen-5-one (3o)

¹H-NMR (400 MHz, DMSO- d_6): $\delta = 2.44$ (s, 3H, CH₃), 4.71 (d, J = 4.8 Hz, 1H, CH-sp³), 5.80 (d, J = 4.9 Hz, 1H), 7.25-7.49 (m, 9H), 7.62 (t, J = 7.8 Hz, 1H), 7.72 (d, J = 7.8 Hz, 2H), 8.02 (d, J = 7.9 Hz, 1H) ppm.

Anal. Calcd for C₂₅H₁₈O₃: C, 81.95; H, 4.95. Found: C, 81.88; H, 4.87.

Synthesis of 5-amino-7-aryl-7,8-dihydro-[1,2,4]triazolo[4,3-a]pyrimidine-6carbonitrile derivatives (7a-j); General procedure

A mixture of 4H-1,2,4-triazol-3-amine (1.0 mmol), malononitrile (1.0 mmol), aldehyde (1.0 mmol) and soluble glass (30% w/v) was refluxed in water for the appropriate time (Scheme 2). The progress of the reaction was monitored by TLC (silica gel, eluent: ethyl acetate/hexane 1/3). After completion of the reaction, the reaction mixture was cooled to room temperature and filtered. The solid was purified by recrystallization from EtOH. Representative spectral data are given below.

5-Amino-7-phenyl-7,8-dihydro-[1,2,4]triazolo[4,3-a]pyrimidine-6-carbonitrile (7a)

¹H-NMR (400 MHz, DMSO- d_6): $\delta = 5.31$ (d, J = 3.5 Hz, 1H, CH-sp³), 6.98 (s, 2H, NH₂), 7.23-7.32 (m, 5H), 7.72 (s, 1H), 8.84 (d, J = 3.3 Hz, 1H, NH) ppm; ¹³C-NMR (100 MHz, DMSO- d_6): $\delta = 53.7$, 56.1, 119.1, 126.1, 128.4, 129.6, 137.6, 146.9, 152.3, 154.1 ppm.

Anal. Calcd for $C_{12}H_{10}N_6\!\!:$ C, 60.50; H, 4.23; N, 35.27. Found: C, 60.39; H, 4.14; N, 35.16.

5-Amino-7-(3-chlorophenyl)-7,8-dihydro-[1,2,4]triazolo[4,3-a]pyrimidine-6-carbonitrile (7j)

¹H-NMR (400 MHz, DMSO- d_6): $\delta = 5.34$ (d, J = 3.5 Hz, 1H, CH-sp³), 7.11 (s, 2H, NH₂), 7.37-7.54 (m, 4H), 7.723 (s, 1H), 8.96 (d, J = 3.3 Hz, 1H, NH) ppm; ¹³C-NMR (100 MHz, DMSO- d_6): $\delta = 53.8$, 56.2, 119.1, 127.3, 128.3, 130.5, 132.8, 137.7, 144.6, 146.9, 152.4, 154.2 ppm.

Anal. Calcd for C₁₂H₉ClN₆: C, 52.85; H, 3.33; N, 30.82. Found: C, 52.76; H, 3.25; N, 30.74.

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