

Original Article: DTP/SiO₂: An Efficient and Reusable Heterogeneous Catalyst for synthesis of Dihydropyrano[3,2-c]chromene-3-Carbonitrile Derivatives



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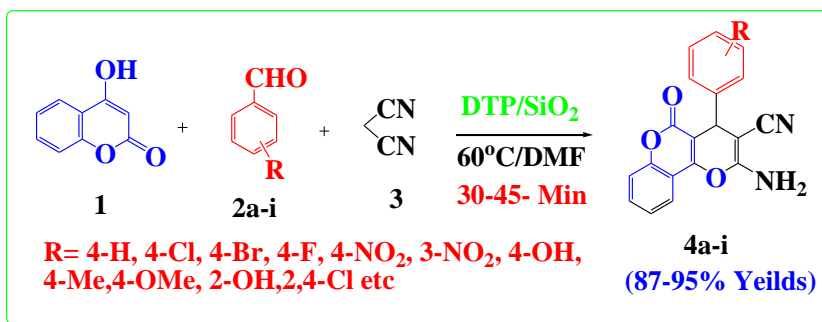
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ABSTRACT

An efficient and convenient method has been developed for the synthesis of 2-amino-5-oxo-4-phenyl-4,5-dihydropyrano[3,2-c]chromene-3-carbonitrile derivatives from a one-pot multi-component reaction between 4-hydroxy-2H-chromen-2-one. Aromatic aldehydes and malononitrile were catalyzed by DTP/SiO₂ as an efficient and reusable heterogeneous catalyst. The current method provides advantages over reported method viz simple operational procedure, easy isolation and recyclability of the catalyst, environmental benign, reduced reaction time and superior yield.



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Introduction

Silica-supported DTP/SiO₂ is simple to prepare and shows good acidic characteristics. The acidic properties of DTP/SiO₂ can be controlled by activation temperature and have shown significant catalytic activity [1]. DTP/SiO₂ exhibits efficient heterogeneous catalytic properties for the synthesis of a wide variety of important organic building blocks such as α -aminophosphonate [2]. Moreover, it is successfully employed as catalyst for the many organic transformations *via* C-H activation and functionalization of nitrogen-containing aromatic heterocycles [3, 4], Fries rearrangement [5], Friedel-Crafts benzylolation of anisole [6].

The pyrans are considered as an important building block for the synthesis of several natural products [7] and photochromic materials [8]. The heterocyclic entities containing pyrans ring show many medicinal and pharmacological properties and are involved in many biochemical reactions [8]. Furthermore, pyrans serve as important synthetic intermediates for the synthesis of biologically important compounds such as pyrano-pyridines [9], poly-azanaphthalenes [10], pyrano[2-*c*]pyrimidines [11], and pyridin-2-ones [12]. Hence, the synthesis of heterocyclic compounds containing pyran nucleus has attracted the attention of many synthetic and medicinal chemists. Moreover, the heterocyclic compounds containing pyrano[3,2-*c*]chromene nucleus is a class of important heterocycle with broad spectrum of biological activities [13] involving spasmolytic, diuretic, anti-coagulant, anti-cancer and anti-anaphylactic activity [14]. The chromene building block with fused ring system has proved to expand the biological spectrum with superior anti-bacterial profile against numerous microbes such as bacteria and fungi [15]. The fused chromene containing heterocycles has shown the excellent biological properties viz antiproliferative [16], sexpheromonal [17], mutagenic [18], anti-tumor [19], anti-viral [20] and CNS depressant activities [21].

There are many methods available in the literature for the synthesis of dihydropyrano[3,2-*c*]chromene compounds via one-pot multi-component reaction (MCR) between 4-hydroxycoumarin with aldehydes and malononitriles such as H₆P₂W₁₈O₆₂/18H₂O [22], sodium dodecyl sulfate (SDS) [23], DBU [24], Tetrabutylammonium bromide (TBAB) under solvent-free and in aqueous condition [25], ionic liquid [26], sulfonic acid functionalized silica (SiO₂PrSO₃H) [27], poly(N,N'-dibromo-N-ethyl-benzene-1,3-disulfonamide) [PBBS] and N,N,N',N'-tetrabromobenzene-1,3-disulfonamide [TBBDA] [28], trisodium citrate [29], Biguanide-functionalized Fe₃O₄/SiO₂ magnetic nanoparticles [30], inorganic-organic hybrid magnetic nanocatalyst Fe₂O₃ [31] Ru(II) phosphine complexes [32], Silica-bonded n-propylpiperazine sodiumn-propionate [33], 2-hydroxyethylammonium formate (ionic liquid) [34], bleaching earth clay [35] etc. However, these reported methods have been found to be inadequate in terms of longer reaction time, lower practical yields, ease of handling of hazardous chemicals, isolation of the product, lack of catalytic reusability etc. Taking into account the limitation of the reported methods, we can still have a scope to develop new method for the synthesis of dihydropyrano[3,2-*c*]chromene derivatives. To address the shortcomings of reported methods, herein we reported DTP/SiO₂ as efficient, recyclable heterogeneous catalysts for the synthesis of dihydropyrano[3,2-*c*]chromene derivatives.

Experimental

General

All the physical constants were recorded in an open capillary tube and were uncorrected. The reagents, chemicals and solvents used were of synthetic grades and were used as obtained. The reactions were monitored by thin-layer chromatography on precoated sheets of alumina gel-G (Merk, Germany) using iodine vapours and or UV light for detection. The Infra-Red (IR) spectra were recorded on Shimadzu Spectrophotometer (KBr pellets). ¹H NMR (300MHz) and ¹³C NMR (100 MHz) spectra

were recorded in DMSO-d₆ or CDCl₃ using TMS an internal standard with an Avance spectrometer (Bruker, Germany). Mass spectra were determined on an EI-Schimidzu QP 2010+ GCMS system.

2.1. General procedure for the synthesis of 2-amino-5-oxo-4-phenyl-4,5-dihydropyrano[3,2-c]chromene-3-carbonitrile derivatives 4:

A mixture of 4-hydroxy-2H-chromen-2-one **1** (1 mol), aldehyde (2a-2n) (1.1 mol), malononitrile **3** (1.1 mmol), and DTP/SiO₂ (20 wt %) in DMF (10 mL) was heated to 60°C with stirring about 30-50 Minute (Table 2). The progress of reaction was checked by TLC. After completing the conversion of reactant into product (by TLC), the catalyst was filtered off and reaction mixture was allowed to cool at room temperature. To this cooled mixture, ice cold water (50 mL) was added and stirred mechanically for 5-10 min. The solid was separated out, filtered and recrystallized from ethanol to afford the pure products **4 a-n**.

2.1.1. Product 4a: Pale yellow powder; (purified by recrystallization with ethanol); IR (KBr) cm⁻¹: 3323, 3204, 2195, 1720, 1668, 1601, 1519, 1381, 1264, 1143, 1048, 761, 481; ¹H NMR (300 MHz, DMSO-d₆ TMS) δ ppm; 4.40 (1H, s, pyran-CH), 7.21-7.30 (5H, m, arom.), 7.36 (2H, s, NH₂), 7.40-7.48 (2H, m, arom.), 7.69 (1H, t, J = 7.2 Hz, arom.), 7.86 (1H, d, J = 7.2 Hz, arom.); ¹³C NMR (100 MHz, DMSO-d₆, TMS) δ ppm; 37.1, 57.9, 103.8, 112.9, 116.6, 119.2, 122.5, 124.7, 127.2, 127.7, 128.6, 133.0, 143.4, 152.2, 153.5, 158.1, 159.6.

2.1.2. Product 4b: Grayish solid; (purified by recrystallization with ethanol); IR (KBr) cm⁻¹: 3319, 3310, 3195, 2196, 1718, 1676, 1608, 1377, 1057, 954, 757, 506; ¹H NMR (300 MHz, DMSO-d₆ TMS) δ ppm; 2.21 (3H, s, CH₃), 4.36 (1H, s, CH), 7.05-7.11 (4H, m, arom.), 7.34 (2H, s, NH₂), 7.39-7.47 (2H, m, arom.), 7.66 (1H, t, J = 9.0 Hz, arom.), 7.86 (1H, d, J = 9.0 Hz, arom.); ¹³C NMR (100 MHz, DMSO-d₆, TMS) δ ppm; 20.7, 36.7, 58.2, 104.2, 113.1, 116.6, 117.8, 119.3, 122.5, 124.7, 127.6, 129.1, 132.9, 136.3, 140.5, 152.2, 153.3, 158.0, 159.6.

2.1.3. Product 4c: White solid; (purified by recrystallization with ethanol); IR (KBr) cm⁻¹:

3370, 3290, 3182, 2191, 1709, 1671, 1605, 1571, 1507, 1459, 1379, 1319, 1251, 1178, 1111, 1052, 1026, 951, 834, 756, 564, 529; ¹H NMR (300 MHz, DMSO-d₆ TMS) δ ppm; 3.68 (3H, s, OCH₃), 4.35 (1H, s, CH), 6.82 (2H, d, J = 8.4 Hz, arom.), 7.13 (2H, d, J = 8.4 Hz, arom.), 7.33 (2H, s, NH₂), 7.38-7.47 (1H, m, arom.), 7.63-7.69 (1H, m, arom.), 7.84 (1H, dd, J = 7.5 Hz, J = 1.2 Hz, arom.), 7.93 (1H, d, J = 9.0 Hz, arom.); ¹³C NMR (100 MHz, DMSO-d₆, TMS) δ ppm; 36.2, 55.1, 58.4, 104.3, 114.0, 115.3, 116.6, 119.4, 122.5, 124.8, 128.8, 132.9, 133.5, 135.5, 152.2, 153.1, 158.0, 159.6, 160.5.

2.1.4. Product 4e: Light yellow colored solid; (purified by recrystallization with ethanol); IR (KBr) cm⁻¹: 3402, 3323, 3204, 2197, 1714, 1670, 1604, 1509, 1379, 1264, 1143, 1047, 761, 481; ¹H NMR (300 MHz, DMSO-d₆ TMS) δ ppm; 4.46 (1H, s, CH), 7.23 (2H, d, J = 8.4 Hz, arom.), 7.43-7.50 (6H, m, NH₂ + arom.), 7.68-7.72 (1H, m, arom.), 7.88 (1H, d, J = 7.2 Hz, arom.); ¹³C NMR (100 MHz, DMSO-d₆, TMS) δ ppm; 36.4, 57.7, 103.6, 113.1, 116.6, 119.1, 122.6, 124.8, 128.6, 129.6, 131.7, 133.1, 142.4, 152.3, 153.6, 158.1, 159.7.

2.1.5. Product 4f: Yellow colored solid; (purified by recrystallization with ethanol); IR (KBr) cm⁻¹: 3385, 3305, 3188, 2191, 1712, 1674, 1606, 1375, 1060, 759, 510; ¹H NMR (300 MHz, DMSO-d₆ TMS) δ ppm; 5.12 (1H, s, CH), 7.17-7.23 (3H, m, NH₂ + arom.), 7.34 (3H, t, J = 8.7 Hz, arom.), 7.46 (4H, t, J = 10.1 Hz, arom.); ¹³C NMR (100 MHz, DMSO-d₆, TMS) δ ppm; 37.0, 56.6, 116.5, 116.9, 119.5, 120.7, 124.8, 125.1, 125.8, 129.8, 130.4, 131.9, 134.5, 142.5, 150.3, 154.1, 159.0.

2.1.6. Product 4j: Yellow colored solid; (purified by recrystallization with ethanol); IR (KBr) cm⁻¹: 3390, 3212, 3179, 2197, 1662, 1575, 1465, 1409, 1260, 1227, 746, 548; ¹H NMR (300 MHz, DMSO-d₆ TMS) δ ppm; 4.64 (1H, s, CH), 7.44 (2H, t, J = 7.5 Hz, arom.), 7.49-7.54 (2H, m, arom.), 7.57 (2H, s, NH₂), 7.69 (1H, t, J = 7.5 Hz, arom.), 7.87 (1H, d, J = 7.5 Hz, arom.), 8.14 (2H, d, J = 8.4 Hz, arom.); ¹³C NMR (100 MHz, DMSO-d₆, TMS) δ ppm; 22.3, 36.9, 43.9, 56.9, 102.9, 113.0, 116.7, 118.9, 122.7, 123.8, 124.8, 129.2, 133.2, 146.7, 150.8, 152.4, 154.0, 158.1, 159.6.

2.1.7. Product 4k: Yellow colored solid; (purified by recrystallization with ethanol); IR (KBr) cm^{-1} : 3382, 3235, 3179, 2193, 1728, 1663, 1600, 1416, 1298, 1173, 1119, 1010, 753, 472; ^1H NMR (300 MHz, DMSO- d_6 TMS) δ ppm: 4.69 (1H, s, CH), 7.42 (1H, d, $J = 8.7$ Hz, arom.), 7.48 (1H, d, $J = 7.8$ Hz, arom.), 7.52 (2H, s, NH_2), 7.59 (1H, t, $J = 7.8$ Hz, arom.), 7.68 (1H, dt, $J = 8.0$ Hz, $J = 8.0$ Hz, $J = 1.4$ Hz, arom.), 7.76 (1H, t, $J = 7.8$ Hz, arom.), 7.87 (1H, d, $J = 7.2$ Hz, arom.), 8.08 (2H, d, $J = 7.8$ Hz, arom.), ^{13}C NMR (100 MHz, DMSO- d_6 , TMS) δ ppm; 22.3, 36.8, 43.9, 57.1, 103.0, 113.0, 116.7, 119.0, 122.5, 124.8, 130.2, 133.2, 134.8, 145.6, 148.0, 152.4, 154.0, 158.3, 159.7.

Result and Discussion

To pursue our work towards the development of efficient methods for the synthesis of important heterocyclic compounds adopting MCRs [35], herein we became interested in developing an environmental friendly method involving use of DTP/SiO₂ as an efficient, recyclable heterogenous catalyst for the synthesis of 2-amino-5-oxo-4-phenyl-4, 5-dihydropyrano[3,2-*c*]chromene-3-carbonitrile derivatives through a one-pot multi-component

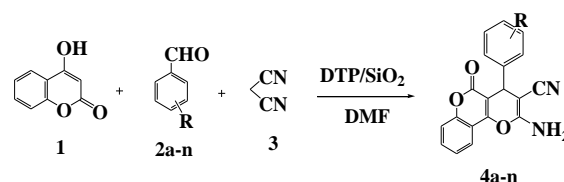
Table 1. Comparison of catalytic activity of various catalysts for synthesis of pyrano[3,2-*c*]chromene-3-carbonitrile derivatives

Entry	Solvent	Catalyst	Yield%
1	Methanol	DTP/SiO ₂	60
2	Ethanol	DTP/SiO ₂	64
3	DCM	DTP/SiO ₂	70
4	Acetonitrile	DTP/SiO ₂	75
5	DMF	DTP/SiO ₂	94
6	Water	DTP/SiO ₂	N R
7	DMF	20%DTP/SiO ₂	94
8	DMF	30%DTP/SiO ₂	94

^aIsolated yields

In order to optimize the reaction condition viz. catalyst loading and solvent, a model reaction was studied by varying the range of solvents including polar and non-polar solvents. In order to find out the appropriate solvent for the synthesis, the model reaction was carried out by using solvents such as methanol, ethanol, dichloromethane (DCM), acetonitrile, Dimethylformamide (DMF). However, the DMF solvent gave the preferred pyrano[3,2-

condensation reaction of 4-hydroxyquinolin-2(1H)-one, aldehydes, and malononitrile. By a preliminary experiment, we found that this three-component condensation reaction catalyzed by DTP/SiO₂ worked very well. Hence, inspired by the preliminary experiments, herein we have reported an efficient one-pot multi-component synthesis of 2-amino-5-oxo-4-phenyl-4, 5-dihydropyrano [3,2-*c*]chromene-3-carbonitrile derivatives in excellent yields (Scheme 1).



Scheme 1. Synthetic route of 2-amino-5-oxo-4-phenyl-4, 5-dihydropyrano[3,2-*c*]chromene-3-carbonitrile derivatives

Initially, we investigated the three-component condensation reaction of 4-hydroxy-2H-chromen-2-one **1**, benzaldehyde **2a**, and malononitrile **3** in the presence of various catalyst; the results are tabulated in Table 1.

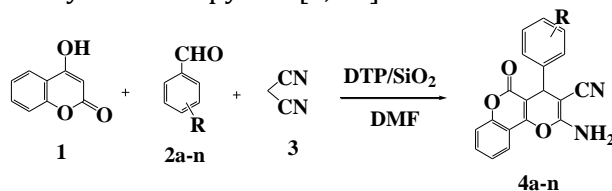
c]chromene-3-carbonitrile product in good yield (Table 1, entry 8), whereas methanol, ethanol, DCM and acetonitrile, respectively gave moderate yield (Table 1, entries 1–4). The formation of the preferred product was not observed using water as the solvent (Table 1, entry 6). This indicates that the solvent plays a key role in the activity and performance of the catalyst. The above observations indicate the reaction using polar protic solvent that shows

an astonishing effect on the yield of the product. Thus, with the reaction in the presence of polar protic solvent, there was a negligible possibility for the substrate to come in contact with catalyst therefore, the yield of the product was found to be low, and reaction in polar aprotic solvent showed the highest yield. The promising results were observed using DMF as a solvent over a DTP/SiO₂ catalyst, which allowed us to further optimize the DTP/SiO₂ catalyst loading. The results in Table 1 (entry 7) reveal that the catalyst with 20 mole % of DTP/SiO₂ loading is excellent. Considering the catalyst using 30 mole % DTP/SiO₂ tested, there was no considerable rise in the yield of the product (Table 1, entry 8). The optimized reaction condition for the given reaction was

found to be using 20 mole% DTP/SiO₂ in DMF solvent. These results motivated us to explore the scope of the pyrano[3,2-c]chromene-3-carbonitrile derivatives synthesis from substituted 4-hydroxyquinolin-2(1H)-one, aldehydes, and malononitrile in the presence of a DTP/SiO₂ catalyst at optimized reaction conditions.

A series of aromatic aldehydes were selected to undergo the condensation in the presence of DTP/SiO₂ catalyst. As shown in Table 2, aromatic aldehydes **2** carrying either electron-donating or electron-withdrawing substituent reacted efficiently and gave excellent yields (Table 2, entries 1–14). The possible mechanism is depicted in scheme 2 in the supplementary file.

Table 2. DTP/SiO₂ catalyzed synthesis of pyrano[3,2-c]chromene-3-carbonitrile derivatives

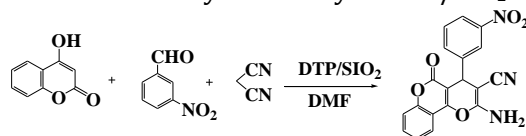


Product	Ar	Time (Min)	Yield ^a (%) Found(Reported)	MP°C Found(Reported)
4a	C ₆ H ₅ 2a	30	94 (10)	254-256 (253-255)[31]
4b	4-CH ₃ C ₆ H ₄ 2b	32	92 (80)	210-220 (219-220) [32]
4c	4-CH ₃ O-C ₆ H ₄ 2c	35	90 (78)	220-222 (220-222) [31]
4d	4-F-C ₆ H ₄ 2d	30	95 (96)	260–262 (260-262) [26]
4e	4-Cl-C ₆ H ₄ 2e	35	92 (88)	257--259 (256-258)[27]
4f	4-Br-C ₆ H ₄ 2f	35	91(80)	247-248 (247-249)[23]
4g	3-CH ₃ O-4-HOC ₆ H ₃ 2g	30	94	228-230
4h	4-OH-C ₆ H ₄ 2h	30	87	258-260
4i	3-OH-C ₆ H ₄ 2i	30	89	263-265
4j	4-NO ₂ -C ₆ H ₄ 2j	40	90 (85)	177-179 (177-178)[32]
4k	3-NO ₂ -C ₆ H ₄ 2k	45	87 (84)	257-258 (257-258) [31]
4l	2-Cl-C ₆ H ₄ 2l	45	93 (84)	262-263 (263-266 [23]
4m	2,4-Cl ₂ C ₆ H ₃ 2m	45	90 (86)	256-258 (255-257) [22]
4n	2-OH-C ₆ H ₄ 2n	40	88	280-282

^aIsolated yields.

Therefore, the nature of the substituents attached to the aromatic ring did not show a significant effect in this conversion. The experimental operations involved efficient, eco-friendly, convenient, rapid properties and showed the ability to endure a variety of electron releasing and electron-withdrawing

functional groups, such as methoxyl, nitro, hydroxyl, and halides. The recycling experiment revealed that the catalyst could be recycled for next 4-5 times without further purification of the catalyst. And it was observed that there was no significant loss in the yield of the product (Table-3).

Table 3. Catalytic activity of DTP/SiO₂.

Run	1 st	2 nd	3 rd	4 th	5 th
Time (Min)	45	45	45	45	45
Yield	87	87	86	86	86

Conclusion

In summary, we have reported a simple, rapid, efficient one-pot multi-component condensation of 4-hydroxyquinolin-2(1H)-one 1, aldehyde 2, malononitrile 3 catalyzed by efficient heterogeneous catalyst DTP/SiO₂ to offer 2-amino-5-oxo-4-phenyl-4,5-dihydropyrano [3,2-c] chromene-3-carbonitrile derivatives 4. The current method offers simple experimental procedure, easy isolation of catalyst, efficacy and reusability of the catalyst over the previously reported methods.

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