

GREEN ONE-POT SYNTHESSES of 1*H*- AND 4*H*-CHROMENES DERIVATIVES USING SiO₂-Pr-SO₃H AS CATALYST

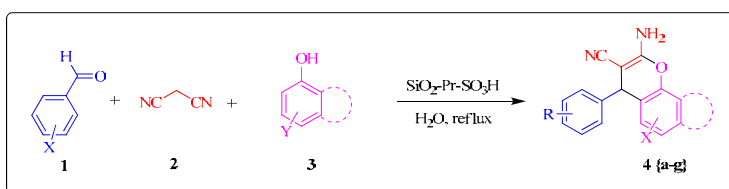
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Synthesis of 2-benzylidenemalononitrile and 1*H*- and 4*H*-chromenes derivatives was improved using sulfonic acid functionalized silica (SiO₂-Pr-SO₃H) as an efficient heterogeneous solid acid catalyst. 1*H*- and 4*H*-Chromenes prepared via a simple green one-pot procedure from the reaction of aromatic aldehydes, α -naphthol (or β -naphthol) and malononitrile using SiO₂-Pr-SO₃H in water media in high yields.



INTRODUCTION

The chromene or benzopyran substructure is frequently found in naturally occurring heterocycles that exhibit biological activity^{1,2} such as spasmolytic, diuretic, anticoagulant, anticancer and anti anaphylactic activity.³⁻⁵ They can be used for the treatment of neurodegenerative diseases, including Alzheimer's disease, amyotrophic lateral sclerosis, Huntington's disease, Parkinson's disease, AIDS connected dementia and Down's syndrome, and then treatment of schizophrenia and myoclonus.⁶

A few methods for the synthesis of chromene derivatives have been reported in literature such as employing aluminum oxide,⁷ H₁₄[NaP₅W₃₀O₁₁₀],⁸ methan sulfonic acid⁹ and ammonium cerium (IV) nitrate as catalyst.¹⁰

In recent years, much attention was afforded to the heterogeneous catalysts due to economic and environmental considerations.¹¹ These acid

catalysts are generally more reactive, eco-friendly, and convenient to handle, with shorter reaction times, simpler work up, high yield of products and better recoverability than most of other catalysts.¹²⁻¹⁶ Application of these catalysts is a grateful approach in green organic synthesis. Thus, in continuation of our studies in this field,¹⁷⁻²⁰ we explored the catalytical activity of sulfonic acid functionalized silica (SiO₂-Pr-SO₃H) as a highly efficient heterogeneous acid catalyst toward the synthesis of 1*H*- and 4*H*-chromenes and 2-benzylidenemalononitrile derivatives.

RESULTS AND DISCUSSION

We originally considered the solvent effects in condensation of aromatic aldehydes **1** with α -naphthol **3** (or β -naphthol **5**) and malononitrile **2** in the presence of SiO₂-Pr-SO₃H to produce 1*H*- and

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4*H*-chromene derivatives (Scheme 1). As a model of reaction, the condensation of α -naphthol, 4-NO₂-benzaldehyde and malononitrile was applied in different solvents such as in H₂O and EtOH, and in solvent-free condition (Table 1). Among the tested solvents, the best result was obtained in aqueous media in excellent yield and in other conditions, the main product was just benzylidenemalononitrile. So, this reaction was developed with different aldehydes in aqueous media and the results were demonstrated in the Table 2. This reaction was completed within 1.5-5.5 hour in high yields. After completion of the reaction (monitored by thin layer chromatography (TLC)), the crude product was dissolved in methanol, the heterogeneous solid catalyst was removed simply by filtration, and after

cooling of the filtrate, the pure crystals of products were obtained. The acid catalyst can be reactivated by simple washing subsequently using diluted acid solution, acetone and water, and then reused without considerable loss of reactivity. The new products were characterized by IR and NMR spectroscopy data for new compounds. Melting points are compared with reported values in literature as shown in Table 2.

The reaction of aromatic aldehydes **1** and malononitrile **2** in the presence of SiO₂-Pr-SO₃H at 120 °C under solvent-free conditions was performed to afford various alkenes in excellent yields (Scheme 2). The results were summarized in Table 3.

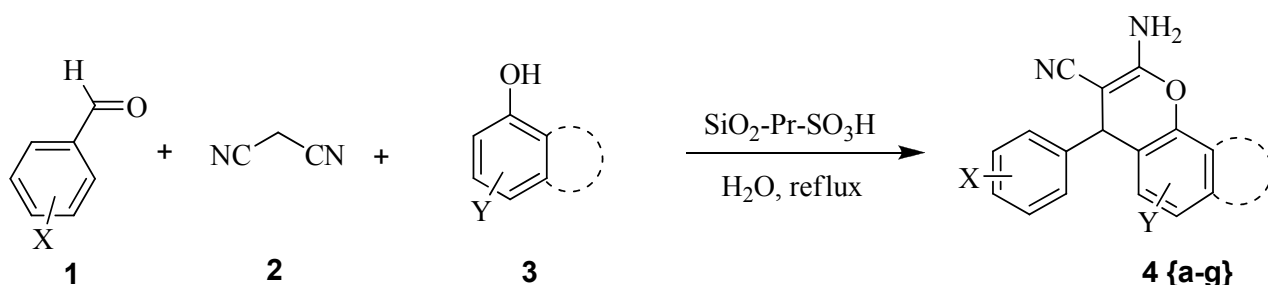


Table 1

The optimization of model reaction conditions in the synthesis of 4*H*-chromenes

No	Product	Solvent	Yield (%)
1		-	98
2		EtOH	95
3		H ₂ O	90

Table 2

Synthesis of 4*H*-chromenes **4** /1*H*-chromenes **6** catalyzed by SiO₂-Pr-SO₃H in water

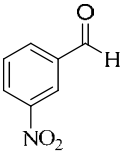
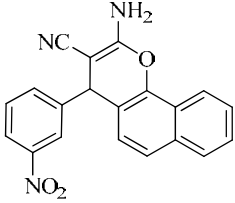
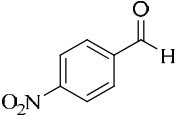
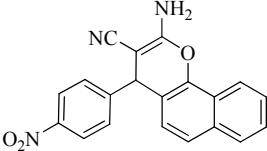
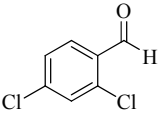
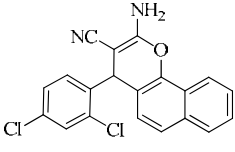
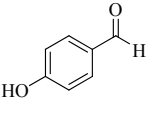
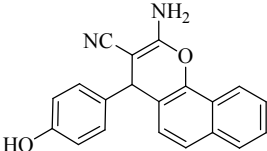
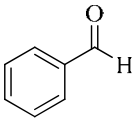
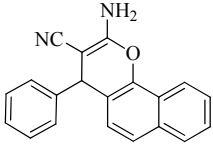
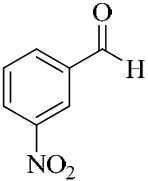
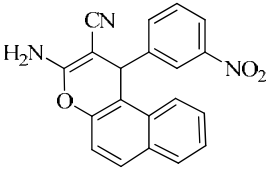
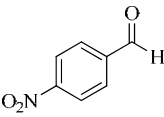
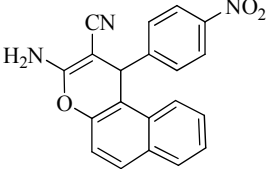
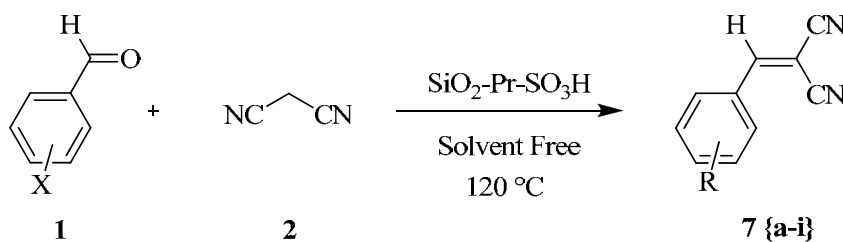
Entry	Phenol	Aldehyde	Product	Time (h)	Yield (%)	mp (°C)	mp (Lit)
1	α -Naphthol		 4a	5	95	211-212	212-214 ²¹
2	α -Naphthol		 4b	4.5	90	242-244	239-241 ²¹
3	α -Naphthol		 4c	5.5	91	214-215	212-214 ²¹
4	α -Naphthol		 4d	5	95	248-249	247-249 ²¹
5	α -Naphthol		 4e	5	89	180-181	178-180 ²¹
6	β -Naphthol		 6a	4	93	248	248 ²¹
7	β -Naphthol		 6b	4.5	95	184-185	185-186 ²¹

Table 2 (continued)

8	β -Naphthol		5.5	97	278-280	278-279 ²³
9	Resorcinol		5.5	93	161-162	162-163 ²¹
10	Resorcinol		1.5	98	111-112	114-115 ²¹



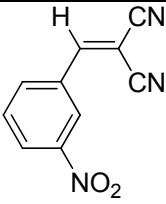
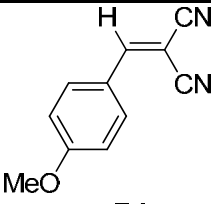
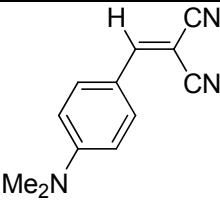
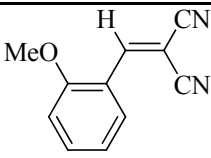
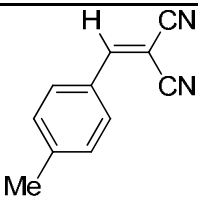
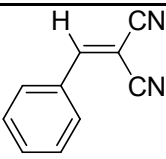
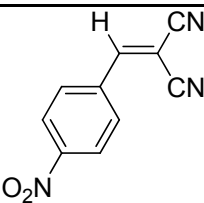
Scheme 2

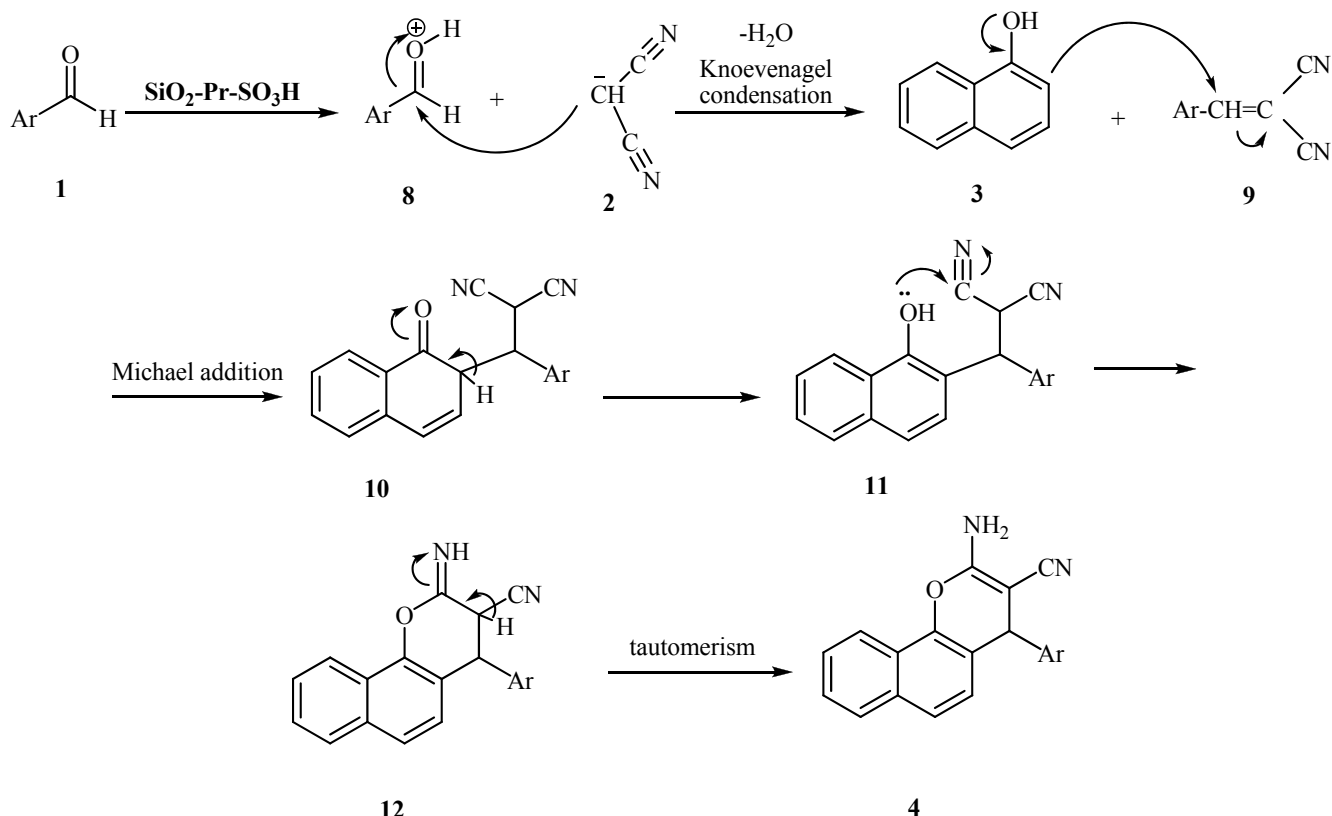
Table 3

Synthesis of 2-benzylidenemalononitrile derivatives 7 using $\text{SiO}_2\text{-Pr-SO}_3\text{H}$ under solvent free conditions

Entry	Benzaldehyde	Product	Time (h)	Yield (%)	mp ($^\circ\text{C}$)	mp (Lit)
1	4-Chloro-		5	95	161-162	167-168 ²⁴
2	4-Hydroxy-		4.5	90	185-186	188 ²⁵

Table 3 (continued)

3	3-Nitro-		5.5	91	98-99	100-101 ²⁶
		7c				
4	4-Methoxy-		5	95	114	115 ²⁴
		7d				
5	4-Dimethylamino-		4/5	95	176-177	179-180 ²⁷
		7e				
6	2-Methoxy-		4	95	77	78-80 ²⁴
		7f				
7	4-Methyl-		5	91	128-129	129 ²⁴
		7g				
8	-		4	95	82-84	84 ²⁴
		7h				
9	4-Nitro		5	98	157	159-160 ²⁴
		7i				



Scheme 3

Table 4

Comparable synthesis of 1*H*- and 4*H*-chromenes

Entry	Catalyst	Solvent	Condition	Time (h)	Yield (%)	Year	Ref
1	Aluminum Oxide	H ₂ O	Heating	3	96	2004	28
2	H ₁₄ [NaP ₃ W ₃₀ O ₁₁₀]	H ₂ O	Reflux	2.75-4.5	93	2007	8
3	Methane sulfonic acid	CH ₃ CN	Reflux	3	91	2008	9
4	SiO ₂ -Pr-SO ₃ H	H ₂ O	Reflux	1.5-5.5	90-98	This work	-

The suggested mechanism for the SiO₂-Pr-SO₃H catalyzed one-pot three component reaction was illustrated in Scheme 3. At first, solid acid catalyst protonates the carbonyl group to facilitate preparation of compound **9** through the Knoevenagel condensation. The Michael addition of α -naphthol **3** to compound **9** followed by cyclization and tautomerisation affords the desired final product **4** in high yield.

The syntheses of 1*H*- and 4*H*-chromenes have been studied with several catalysts and solvents in literature as shown in Table 4. In contrast with other existing methods, the present methodology offers several advantages such as excellent yields, a simple procedure, short reaction times, easy synthesis, simple work-up and greener conditions using sulfonic acid functionalized silica (SiO₂-Pr-SO₃H) as an efficient catalyst.

EXPERIMENTAL

General information

IR spectra were recorded from KBr disk using a FT-IR Bruker Tensor 27 instrument. Melting points were measured by the capillary tube method with an electro thermal 9200 apparatus. The ¹H NMR (250 MHz) was run on a Bruker DPX, 250 MHz. Gas chromatography-mass spectrometry (GC-MS) analysis was performed on an Agilent 6890-5973 GC/MS detector.

Preparation of catalyst

Synthesis of 3-Mercaptopropylsilica (MPS) and its oxidation: To 20 g of SiO₂ in dry toluene, 25 mL of (3-mercaptopropyl) trimethoxysilane was added, and the reaction mixture was heated at reflux for 24 h. After this period, the mixture was filtered to obtain 3-mercaptopropylsilica (MPS), which was washed with acetone and dried under vacuum. 3-Mercaptopropylsilica (MPS) (20 g) was oxidized with H₂O₂ (50 mL) and one drop of H₂SO₄ in methanol (20 mL) for 24 h at room temperature and then the mixture was filtered and

washed with H₂O, and acetone to obtain the SiO₂-Pr-SO₃H catalyst. The modified SiO₂-Pr-SO₃H was dried and used as a solid acid catalyst in the organic synthesis.

General procedure for the preparation of 2-benzylidenemalononitrile derivatives

The SiO₂-Pr-SO₃H (0.1 g) was activated under vacuum and then after cooling to room temperature, malononitrile **2** (1 mmol) and aromatic aldehyde **1** (1 mmol) were added to it. The mixture was heated in solvent free condition at 120 °C for an appropriate time. The completion of reaction was indicated by thin layer chromatography, the resulting solid product was dissolved in ethyl acetate, filtered for removing the unsolvable catalyst and then the filtrate was cooled to give the pure product. The spectroscopic and analytical data for products are presented in the following part. Finally, the catalyst was washed with diluted acid solution, distilled water and then acetone, dried under vacuum and re-used for several times without loss of considerable activity.

1,1-Dicyano-2-(4-chlorophenyl) ethylene (7a): mp: 161-162 °C. IR (KBr): ν_{\max} = 3032, 2225, 1583, 1457, 1092, 825 cm⁻¹. Mass: m/z 188 [M+1]⁺, 153, 126, 87. ¹H NMR (DMSO-d₆) δ_{H} = 7.75 (s, 1H, H-C=C), 7.56 (d, 2H, Ar-H), 7.88(d, Ar-H, 2H).

1,1-Dicyano-2-(4-hydroxyphenyl) ethylene (7b): mp: 185-186 °C. IR (KBr) ν_{\max} = 3280, 3018, 2280, 1580, 1220 cm⁻¹. Mass: m/z 170 [M+1]⁺, 142, 119, 115, 39, 85, 75. ¹H NMR (CDCl₃) δ_{H} = 5 (s, 1H, OH), 7.72 (s, 1H, H-C=C), 6.92 (d, J=8 Hz, 2H, Ar-H), 7.89 (d, J=8.0Hz, Ar-H, 2H).

1,1-Dicyano-2-(3-nitrophenyl) ethylene (7c): mp: 98-99 °C. IR (KBr): ν_{\max} = 3032, 2225, 1583, 1554, 1082 cm⁻¹. Mass: m/z 199 [M+1]⁺, 169, 153, 126, 100, 75, 51. ¹H NMR (DMSO-d₆) δ_{H} = 8.60 (t, 1H, J=1.8 Hz, Ar-H), 8.3-8.4 (dd, 1H, J=0.8, J=9 Hz, Ar-H), 8.30 (d, 1H Ar-H).

1,1-Dicyano-4-(methoxyphenyl)ethylene (7d): mp: 114 °C. IR (KBr): ν_{\max} = 3015, 2983, 2280, 1560, 1210 cm⁻¹. Mass: m/z 184 [M+1]⁺, 169, 141, 114, 88, 57, 43. ¹H NMR (CDCl₃) δ_{H} = 7.62 (s, 1H, H-C=C), 7.01(d, J = 9.0 Hz, 2H, Ar-H), 7.90 (d, J = 9.0Hz, Ar-H, 2H), 3.90 (s, 3H, OCH₃).

1,1-Dicyano-2-(4-N,N-dimethylphenyl) ethylene (7e): mp: 176-177 °C. IR (KBr): ν_{\max} = 3021, 2206, 1608, 1518 cm⁻¹. Mass: m/z 197 [M+1]⁺, 196, 179, 153, 126, 99, 42. ¹H NMR (DMSO-d₆) δ_{H} = 3.33(s, 6H, Me), 8.05 (s, 1H, H-C=C), 6.87-6.87 (d, J=7.2 Hz, 2H, Ar-H), 7.82-7.86(d, J=7.2 Hz, Ar-H, 2H).

1,1-Dicyano-2-(methoxyphenyl) ethylene (7f): mp: 78 °C. IR (KBr): ν_{\max} = 3021, 2210, 1580, 1160 cm⁻¹. Mass: m/z 184 [M+1]⁺, 141, 114, 88, 39. ¹H NMR (CDCl₃) δ_{H} = 8.28 (s, 1H, H-C=C), 8.20 (d, J=9.5 Hz, 1H, Ar-H), 7.10 (t, J=9.0 HZ, Ar-H, 1H), 7.58 (t, 1H, J=7 Hz, Ar-H), 6.98 (d, 1H, J=8.5, Ar-H), 3.94 (s, 3H, OMe).

1,1-Dicyano-2-(4-methylphenyl) ethylene (7g): mp: 128-129 °C. IR (KBr): ν_{\max} = 3020, 2250, 1560, 1590 cm⁻¹. Mass: m/z 168 [M+1]⁺, 153, 142, 116, 77. ¹H NMR (DMSO-d₆) δ_{H} = 7.75 (d, Ar-H), 7.65 (s, 1H, H-C=C), 7.27(d, Ar-H, 2H).

1,1-Dicyano-2-phenyl ethylene (7h): mp: 82-84 °C. IR (KBr): ν_{\max} = 3022, 3200, 2280, 1560, 1090 cm⁻¹. Mass: m/z 154 [M+1]⁺, 127, 103, 76. ¹H NMR (CDCl₃) δ_{H} = 7.48-7.68 (m, 3H, Ar-H), 7.76 (s, 1H, H-C=C), 7.90 (d, j=5.7 Hz, 2H, Ar-H).

1,1-Dicyano-2-(4-nitrophenyl) ethylene (7i): mp:157 °C. IR (KBr): ν_{\max} = 3038, 2230, 1582, 1518, 1344 cm⁻¹. Mass: m/z 199 [M+1]⁺, 157, 142, 127, 114, 100, 85, 75. ¹H NMR (CDCl₃) δ_{H} = 7.8 (s, 1H, H-C=C), 8.10 (d, J=9.0 Hz, 2H, Ar-H), 8.40 (d, J=9.0 Hz, Ar-H, 2H).

General procedure for the preparation of 4H-chromenes and 1H-chromenes derivatives

The SiO₂-Pr-SO₃H (0.1 g) was activated under vacuum and then after cooling to room temperature, aromatic aldehyde **1** (1 mmol), malononitrile **2** (1 mmol), α -naphthol **3** (or β -naphthol **5**) and 10 ml water as solvent was added to it. The mixture was refluxed for an appropriate time. The completion of reaction was indicated by thin layer chromatography, the resulting solid product and catalyst was filtered and then the filtrate was dissolved in ethanol, filtered for removing the unsolvable catalyst and then the filtrate again was cooled to afford the pure product. Finally, the catalyst was washed with diluted acid solution, distilled water and then acetone, dried under vacuum and re-used for several times without loss of considerable activity.

2-amino-4-(3-nitrophenyl)-4H-benzo[h]chromene-3-carbonitrile (4a): mp 211-212 °C. IR (KBr) ν_{\max} = 3460, 3335, 2196 cm⁻¹. ¹H NMR (DMSO-d₆) δ_{H} = 4.74 (s, 1H, H-4), 7.56 (s, 2H, NH₂), 7.44 (d, 1H, J = 6.7, HAr), 7.64 (t, 1H, HAr), 7.73 (dt, 1H, J=7.5, HAr), 7.90 (d, 1H, J= 6.8, HAr), 7.92 (dd, 1H, HAr), 8.12 (dd, 1H, J = 8.4, HAr), 8.14(1H, s, HAr) ppm.

2-amino-4-(4-nitrophenyl)-4H-benzo[h]chromene-3-carbonitrile (4b): mp 242-244 °C. IR (KBr) ν_{\max} = 3473, 3351, 2189, 1572, 1101 cm⁻¹. Mass: m/z 221 [M+1]⁺. ¹H NMR (DMSO-d₆) δ_{H} = 5.17 (s, 1H, H-4), 7.14 (d, 1H, H-5), 7.35 (s, 2H, NH₂), 7.35 (s, 1H, H-9), 7.54-7.57 (d, 2H, H-2', 6'), 7.60-7.70 (m, 3H, H-6, 8, 9), 7.90-7.93 (d, 1H, H-10 or 7), 8.19-8.23 (d, 1H, H-10 or 7), 8.26-8.30 (d, 2H, H-3', 5') ppm.

2-amino-4-(2,4-dichlorophenyl)-4H-benzo[h]chromene-3-carbonitrile (4c): mp 212-214 °C. IR (KBr) ν_{\max} = 3453, 3332, 2190 cm⁻¹. Mass: m/z 366 [M+1]⁺, 350, 331, 265, 221. ¹H NMR (DMSO-d₆) δ_{H} = 5.47 (s, 1H, H-4), 7.30 (s, 2H, NH₂), 7.37 (d, 1H, J= 9 H-9 or H-10), 6.98 (d, 1H, H-6'), 7.59-7.61 (m, H-3', 5'), 7.39 (d, 1H, H-5), 7.69-7.89 (m, 3H, H-6,8, 9), 8.03 (d, 1H, H-7), 8.25 (d, 1H, H-10) ppm.

2-amino-4-phenyl-4H-benzo[h]chromene-3-carbonitrile (4e): mp 180-181 °C. IR (KBr): ν_{\max} = 3449, 3302, 2195, 1654, 1605 cm⁻¹. ¹H NMR (CDCl₃) δ_{H} = 4.76 (s, 2H, NH₂), 4.90 (s, 1H, CH), 7.04 (d, J = 8.8 Hz, 1H, ArH), 7.25-7.36 (m, 5H, ArH), 7.52-7.62 (m, 3H, ArH), 7.81 (d, J = 8 Hz, 1H, ArH), 8.20 (d, J = 8.4 Hz, 1H, ArH) ppm.

3-amino-1-(3-nitrophenyl)-1H-benzo[f]chromene-2-carbonitrile (6a): mp 248 °C; IR (KBr): ν_{\max} = 3464, 3355, 2189, 1661 cm⁻¹. Mass: m/z 343 [M+1]⁺, 326, 221, 192, 176, 166, 139, 76, 81. ¹H NMR (DMSO-d₆) δ_{H} = 5.65 (s, 1H, H-4), 7.19 (s, 2H, NH₂), 7.39 - 7.49 (m, 2H, HAr), 7.56-7.64 (t, 2H, HAr), 7.68-7.71 (d, 2H, HAr), 7.90 (d, 1H, J= 6.8, HAr), 7.92 (d, 1H, HAr), 8.12 (m, 2H, HAr), 8.14(m, 2H, HAr) ppm.

3-amino-1-(4-nitrophenyl)-1H-benzo[f]chromene-2-carbonitrile (6b): mp 184-185 °C. IR (KBr): ν_{\max} = 3430, 3325, 2190 cm⁻¹. Mass: m/z 221 [M+1]⁺. ¹H NMR (DMSO-d₆) δ_{H} = 5.45 (s, 1H, H-4), 7.20 (s, 2H, NH₂), 7.38 (d, 1H, H-9), 7.40-7.52 (m, 2H, H-6, 7), 7.69-8.03 (m, 2H, H-5, 8), 7.98 (d, 1H, J= 9.2, H-10), 7.44 (d, 2H, H-2', 6'), 8.15 (d, 2H, H-3', 5') ppm.

3-amino-1-(phenyl)-1H-benzo[f]chromene-2-carbonitrile (6c): mp 278-280 °C. IR (KBr): ν_{\max} = 3435, 2185, 1669, 1560 cm⁻¹. ¹H NMR (300 MHz, CDCl₃) δ_{H} = 5.24 (s, 1H, H-4), 7.11(brs, 2H, NH₂), 7.19 (d, 1H, H-5), 7.25-7.81 (m, 11H, Ar-H).

2-amino-7-hydroxy-4-(4-methoxyphenyl)-4H-chromene-3-carbonitrile (4g): mp 111-112 °C. IR (KBr): ν_{\max} = 3420, 3210, 2198, 1677, 1595 cm⁻¹. ¹H NMR (DMSO-d₆) δ_{H} = 4.32 (s, 1H, H-4), 6.78(brs, 2H, NH₂), 7.02 (s, 1H, OH), 7.47-7.86 (m, 8H, Ar-H).

CONCLUSIONS

In summary, environmentally benign sulfonic acid functionalized silica (SiO₂-Pr-SO₃H) as a solid acid catalyst assisted us to improve the synthesis of 1*H*- and 4*H*-chromenes in a three component reaction of aromatic aldehydes, α -naphthol (or β -naphthol) and malononitrile under solvent free conditions. Simple procedure, reusability of the catalyst and non-chromatographic purification of products, *i.e.* simple recrystallization got our attention to this process.

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