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# CuO-CeO<sub>2</sub> nanocomposite catalyzed efficient synthesis of aminochromenes

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## Abstract

CuO-CeO<sub>2</sub> nanocomposite is reported as an efficient and green recyclable catalyst for the synthesis of chromene derivatives under solvent-free conditions. The catalyst was synthesized by co-precipitation method and characterized by X-ray diffraction, Brenauer-Emmett-Teller specific surface area, FESEM and energy dispersive spectroscopy analysis. This catalyst can be recovered by simple filtration and recycled up to 10 consecutive runs without loss of its activity. This process provides some advantages such as simple work-up, clean procedure, short reaction times and high yields of the products.

**Keywords:** CuO-CeO<sub>2</sub> nanocomposite; Aminochromenes; Resorcinol; Malononitrile; Solvent-free conditions

## Background

In recent years, development of new catalysts by nanoscale design has emerged as a fertile field for research and innovation [1]. The ability of the nanotechnology to enhance catalytic activity opens the potential to replace expensive catalysts with lower amounts of inexpensive nanocatalysts [2]. In particular, nanocrystalline oxides have proved to be useful to chemists in the laboratory and industry due to the good activation of adsorbed compounds and reaction rate enhancement, selectivity, easier work-up, recyclability of the supports and the eco-friendly reaction conditions [3-7]. Also the practical applications of nanocomposite metal oxides as the catalysts in organic synthesis have been increased due to their high catalytic activity because of the high surface area [8,9].

Aminochromenes are important class of compounds that received significant attention from many pharmaceutical and organic chemists because of the broad spectrum of their biological and pharmaceutical properties such as antisterility and anticancer agents [10]. Furthermore, these compounds are the main constituents of many natural products and used as cosmetics, pigments, and potential biodegradable agrochemicals [11]. Therefore, the synthesis of aminochromenes is very

important in organic chemistry. The most important synthetic procedure for the synthesis of these compounds involves the multi-component condensation of aldehyde, malononitrile and phenol derivatives. Although various methods have been reported for the synthesis of aminochromenes [12-27], however, some of these methods involve the use of expensive reagents, toxic solvents, tedious workup, long reaction times and harsh reaction conditions. Therefore, introduction of new methods and catalysts for the synthesis of these important compounds in terms of potential simplicity, high activity, low cost, high yields and short reaction times are still in demand.

Recently, we have reported the preparation of CuO-CeO<sub>2</sub> nanocomposite and its catalytic activity for the synthesis of aryl-14*H*-dibenzo[*a-j*]xanthenes and benzopyran derivatives [28,29]. This catalyst is safe, easy to handle and environmentally benign. In continuation of these studies and our studies on the synthesis of benzochromene derivatives [30], herein, we wish to report the applicability of CuO-CeO<sub>2</sub> nanocomposite as a green, efficient and recyclable catalyst for the synthesis of aminochromenes via the one-pot three-component reaction of resorcinol, aromatic aldehydes and malononitrile under solvent-free conditions (Scheme 1).

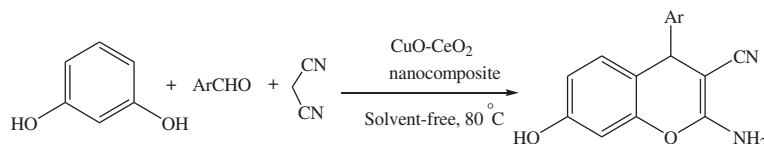
## Results and discussion

The CuO-CeO<sub>2</sub> nanocomposite oxide catalyst was prepared by a coprecipitation method. BET specific

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**Scheme 1** CuO-CeO<sub>2</sub> nanocomposite catalyzed synthesis of aminochromene.

surface area, emission scanning electron microscopy (ESEM) (Figure 1), X-ray diffraction (XRD) (Figure 2), and energy dispersive spectroscopy (EDS) (Figure 3), were used to characterize the catalyst [28].

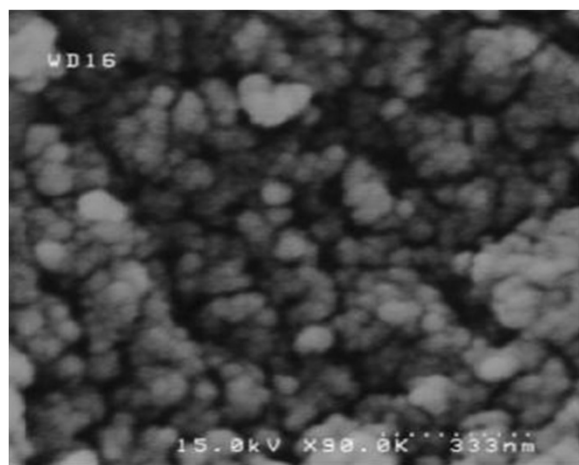
At first, for the optimization of the reaction conditions, a mixture of resorcinol, benzaldehyde and malononitrile was investigated as a model and its behavior was studied under a variety of conditions. The best result was achieved by carrying out the reaction of resorcinol, benzaldehyde and malononitrile (with 1:1:1 mol ratio) in the presence of 0.05 g of CuO-CeO<sub>2</sub> nanocomposite at 80°C under solvent-free conditions (Table 1, entry 1). Catalytic activity results reveal that the CuO-CeO<sub>2</sub> nanocomposite exhibited efficient activity during the synthesis of aminochromenes. Under the optimized conditions, the reaction of a variety of differently substituted aromatic aldehydes, containing electron-donating and electron-withdrawing groups, was explored. In all cases, aromatic aldehydes reacted successfully and desired products achieved in good to high yields. It can be found that the kind of group on aromatic ring has no significant effect on the reaction. Aliphatic aldehydes stay intact under the similar reaction conditions. Therefore, this method can be useful for the chemoselective synthesis of aminochromene derivatives from aromatic aldehydes in the presence of aliphatic ones. All reactions

were performed under solvent-free and entirely heterogeneous reaction conditions. Workup procedure is so simple and all products were cleanly isolated by simple filtration and evaporation of the solvent. Another advantage of this process is that CuO-CeO<sub>2</sub> nanocomposite is safe, easy to handle and its preparation is simple. Furthermore, very low amount of the catalyst is needed and it can be recovered simply by filtration. Products were easily recrystallized from hot ethanol and were obtained during the short reaction times. Furthermore, our procedure is environmentally friendly as it does not use any toxic auxiliary or solvent. A distinct characterization of this method, illustrated in this work is the synthesis of corresponding products without by-product.

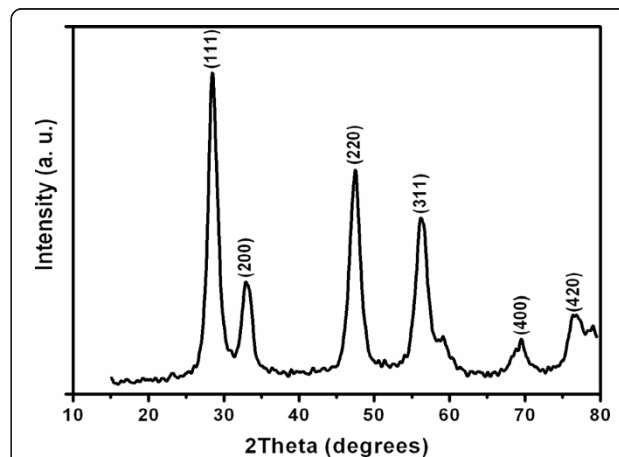
The recyclability of the catalyst is also important. To investigate of this property, the reaction of resorcinol with benzaldehyde and malononitrile was selected again as a model (Table 2). After completion of the reaction, the recovered catalyst was washed with hot ethanol and after dryness was reused in the next similar run. This procedure was repeated for eight consecutive runs and the desired product was obtained in good to high yields after one to eight runs.

## Conclusion

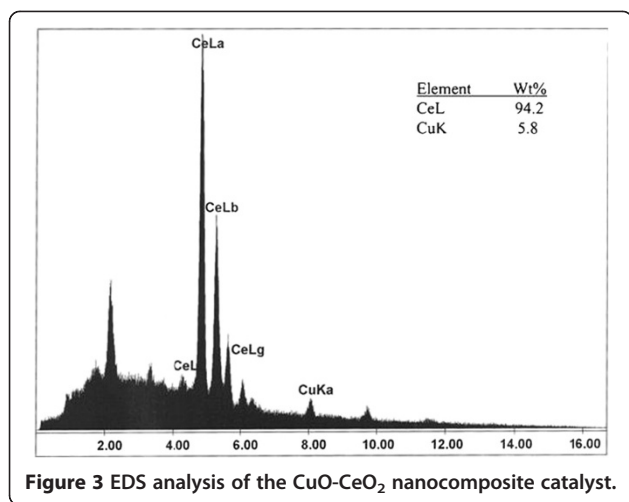
In conclusion, we have described a highly efficient method for the synthesis of aminochromenes catalyzed by CuO-



**Figure 1** ESEM image of the CuO-CeO<sub>2</sub> nanocomposite catalyst.



**Figure 2** XRD pattern of the CuO-CeO<sub>2</sub> nanocomposite catalyst.



CeO<sub>2</sub> nanocomposite as a green recyclable catalyst under solvent-free conditions. This catalytic system is stable and can promote the yields and reaction times over eight runs without loss of its activity. Moreover, simple work-up, high yields of products, short reaction times, heterogeneous nature of the reaction conditions, and clean procedure, will make this procedure a useful addition to the available methods.

## Methods

### General

Chemicals were purchased from Merck Chemical company (Whitehouse Station, NJ, USA). All the NMR spectra were recorded on a Bruker Advance 400 MHz. Melting points were recorded on a Barnstead Electrothermal

**Table 2** Recyclability study of CuO-CeO<sub>2</sub> nanocomposite

Run	1	2	3	4	5	6	7	8
Time (min)	8	8	8	10	10	12	15	15
Yield (%) <sup>a</sup>	94	93	93	92	92	92	90	90

<sup>a</sup>Isolated yield.

9100BZ melting point apparatus (St. Louis, MO, USA). Yields refer to isolated pure products.

### Catalyst preparation

CuO-CeO<sub>2</sub> nanocomposite was prepared by co-precipitation method using aqueous solution of cerium and copper nitrates and drop-wise KOH as precipitant agent under vigorous mixing while temperature and pH was fixed at unique values. Then acquired sample was filtered, washed and calcined to obtain final catalyst to be used in the synthesis of aminochromenes.

### Catalyst characterization

The catalyst structure characterization was performed by XRD, using a Bruker AXS D8 advanced diffractometer equipment (Madison, WI, USA) with CuK $\alpha$  radiation ( $\lambda = 1.5406 \text{ \AA}$ ). The Debye-Scherrer equation is used to determine average crystallite size of nanoparticles. ESEM and EDS carried out by a Hitachi S4160 instrument to see the morphology, evaluation of cluster size and metal composition of the catalyst. BET specific surface area was estimated from the N<sub>2</sub> adsorption/desorption isotherms, measured at 77 K using a Quantachrome CHEMBET-3000 instrument (Boynton Beach, FL, USA). The infrared (IR) spectra were recorded on a PerkinElmer 781 spectrophotometer (Waltham, MA, USA).

**Table 1** CuO-CeO<sub>2</sub> nanocomposite catalyzed synthesis of aminochromene

Entry	Aldehyde	Time (min)	Yield (%) <sup>a</sup>	MP (°C)	
				Found	Reported
1	C <sub>6</sub> H <sub>5</sub> CHO	10	94	232 to 235	231 to 233 [24]
2	4-ClC <sub>6</sub> H <sub>4</sub> CHO	7	93	162 to 164	161 to 162 [23]
3	2-ClC <sub>6</sub> H <sub>4</sub> CHO	10	91	188 to 190	188 to 190 [11]
4	2-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> CHO	12	90	163 to 164	162 to 163 [24]
5	3-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub> CHO	15	90	170 to 171	169 to 170 [24]
6	4-BrC <sub>6</sub> H <sub>4</sub> CHO	8	92	224 to 226	224 to 226 [24]
7	4-FC <sub>6</sub> H <sub>4</sub> CHO	15	94	189 to 191	190 to 192 [11]
8	4-MeC <sub>6</sub> H <sub>4</sub> CHO	15	90	183 to 185	184 to 186 [24]
9	4-MeOC <sub>6</sub> H <sub>4</sub> CHO	20	90	111 to 113	110 to 112 [24]
10	4-HOC <sub>6</sub> H <sub>4</sub> CHO	35	88	251 to 253	252 to 254 [11]
11	4-NMe <sub>2</sub> C <sub>6</sub> H <sub>4</sub> CHO	30	88	194 to 196	193 to 195 [24]
12	4-CNC <sub>6</sub> H <sub>4</sub> CHO	17	91	109 to 110	-
13	2,4-ClC <sub>6</sub> H <sub>3</sub> CHO	8	93	257 to 259	-
14	4-(Me) <sub>2</sub> CH-C <sub>6</sub> H <sub>4</sub> CHO	20	90	216 to 218	-

MP, melting point. <sup>a</sup>Isolated yield.

### General procedure

A mixture of aromatic aldehyde (1 mmol), malononitrile (1 mmol), resorcinol (1 mmol) and CuO-CeO<sub>2</sub> nanocomposite (0.05 g) was heated in an oil bath (80°C) for the appropriate times (Table 1). After completion of the reaction as followed by TLC, the reaction mixture was cooled to room temperature and hot ethanol (10 ml) was added and the catalyst was filtered. Evaporation of the solvent from the filtrate and recrystallization of the solid residue from hot ethanol afforded the pure products in good to high yields. The spectral and analytical data for the new compounds are as follow (see Additional file 1):

- Table 1, entry 12: White solid, MP: 109°C to 111°C; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3,434, 3,340, 3,201, 2,808, 2,231, 2,185, 1,648, 1,505, 1,406, 1,306, 1,237, 1,158, 1,107, 1,050. Proton Nuclear Magnetic Resonance (<sup>1</sup>H NMR) (CDCl<sub>3</sub>, 400 MHz),  $\delta$  (ppm): 4.70 (s, 1H, CH), 5.22 (s, 2H, NH<sub>2</sub>), 6.53 to 6.58 (m, 2H, Ar), 6.67 (d, 1H,  $J = 8.36$  Hz), 7.28 (d, 2H,  $J = 8.16$  Hz, Ar), 7.58 (d, 2H,  $J = 8.20$  Hz, Ar), 9.07 (s, 1H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz): 39.36 (CH), 57.21 (CN), 102.03, 109.45, 110.78, 112.03, 117.66, 118.98, 127.48, 128.66, 131.40, 147.91, 149.39, 156.33, 158.74. Elem. Anal. Found: C, 70.71%; H, 3.89% N, 14.61% (calculated for C<sub>17</sub>H<sub>11</sub>N<sub>3</sub>O<sub>2</sub>: C, 70.58%; H, 3.83%; N, 14.52%).
- Table 1, entry 13: Yellow solid, MP: 257°C to 259°C; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3,476, 3,338, 3,271, 2,191, 1,641, 1,586, 1,504, 1,463, 1,409, 1,341, 1,312, 1,151, 1,105, 1,049, 854, 807. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 400 MHz),  $\delta$  (ppm): 5.11 (s, 1H, CH), 6.39 (s, 1H, Ar), 6.45 to 6.48 (m, 1H, Ar), 6.69 (d, 1H,  $J = 8.8$  Hz, Ar), 6.97 (s, 2H, NH<sub>2</sub>), 7.18 (d, 1H,  $J = 8.4$  Hz, Ar), 7.38 (d, 1H,  $J = 8.4$ , Ar), 7.57 (s, 1H, Ar), 9.76 (s, 1H, OH). <sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 100 MHz): 37.35 (CH), 54.90 (CN), 102.75, 112.36, 113.05, 120.61, 128.51, 129.55, 129.72, 132.62, 132.69, 133.27, 142.37, 149.48, 157.94, 160.96. Elem. Anal. Found: C, 57.74%; H, 3.08% N, 8.47% (calculated for C<sub>16</sub>H<sub>15</sub>N<sub>2</sub>O<sub>2</sub>Cl<sub>2</sub>: C, 57.68%; H, 3.02%; N, 8.40%).
- Table 1, entry 14: Orange solid, MP: 216°C to 218°C; IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3,476, 3,337, 3,270, 2,961, 2,190, 1,639, 1,587, 1,504, 1,463, 1,409, 1,341, 1,311, 1,246, 1,153, 1,105, 1,049, 963, 854, 807. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 400 MHz),  $\delta$  (ppm): 1.13 (d, 6H, 2CH<sub>3</sub>), 2.77 to 2.82 (m, 1H, CH), 4.56 (s, 1H, CH), 6.822 (m, 3H, Ar), 7.05 (s, 2H, NH<sub>2</sub>), 7.13 to 7.15 (m, 2H, Ar), 9.69 (s, 1H, OH). <sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 100 MHz): 25.47 (CH<sub>3</sub>), 31.09 (CH), 34.61 (CH), 57.97 (CN), 103.75, 113.95, 115.56, 122.37, 128.08, 128.79, 131.50, 145.44, 148.20, 150.45, 158.61, 161.85. Elem. Anal. Found: C, 74.48%; H, 5.97% N, 9.24% (calculated for C<sub>19</sub>H<sub>18</sub>N<sub>2</sub>O<sub>2</sub>: C, 74.39%; H, 5.92%; N, 9.18%).

The spectral and analytical data for the known compounds are as follow (see Additional file 2):

- Table 1, entry 1: IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3,450, 3,300, 3,200, 2,200, 1,650, 1,580, 1,510, 1,480, 1,380, 1,120, 1,060, 760, 700. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz),  $\delta$  (ppm): 4.61 (s, 1H, CH), 5.47 (s, 2H, NH<sub>2</sub>), 6.52 (t, 2H,  $J = 8.88$  Hz, Ar), 6.73 (d, 2H,  $J = 8.28$  Hz, Ar), 7.17 to 7.30 (m, 5H, Ar), 9.17 (s, 1H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz): 38.74, 57.55, 101.39, 111.31, 112.01, 119.23, 125.36, 126.30, 127.09, 128.46, 144.07, 147.65, 155.64, 158.42.
- Table 1, entry 2: IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3,450, 3,300, 3,200, 2,200, 1,650, 1,580, 1,510, 1,480, 1,380, 1,120, 1,060, 760, 690. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 400 MHz),  $\delta$  (ppm): 4.64 (s, 1H, CH), 6.38 (s, 1H, Ar), 6.45 (d, 1H,  $J = 8.00$  Hz, Ar), 6.75 (d, 1H,  $J = 8.4$  Hz, Ar), 6.90 (s, 2H, NH<sub>2</sub>), 7.15 (d, 2H,  $J = 8.00$  Hz, Ar), 7.33 (d, 2H,  $J = 8.4$  Hz, Ar), 9.71 (s, 1H, OH). <sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 100 MHz): 38.41, 56.30, 102.67, 112.92, 113.64, 120.95, 129.67, 129.01, 129.73, 130.34, 131.67, 145.77, 149.27, 157.66, 160.69.
- Table 1, entry 5: IR (KBr)  $\nu_{\max}/\text{cm}^{-1}$ : 3,400, 3,300, 3,200, 3,050, 2,200, 1,650, 1,580, 1,520, 1,380, 1,360, 1,120, 1,080, 760, 680. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz),  $\delta$  (ppm): 4.79 (s, 1H, CH), 5.61 (s, 2H, NH<sub>2</sub>), 6.55 to 6.58 (m, 2H, Ar), 6.70 (d, 1H,  $J = 8.20$  Hz, Ar), 7.50 (t, 1H,  $J = 7.92$ , Ar), 7.58 (d, 1H,  $J = 7.63$  Hz), 8.01 (s, 1H, Ar), 8.07 (d, 1H,  $J = 8.08$  Hz, Ar), 9.25 (s, 1H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz): 38.00 (CH), 58.95 (CN), 101.86, 110.58, 111.81, 118.90, 120.68, 121.29, 128.31, 128.44, 132.86, 146.33, 147.04, 147.76, 156.29, 158.78, 169.72.
- Table 1, entry 6: 3,450, 3,300, 3,200, 2,200, 1,650, 1,580, 1,510, 1,480, 1,380, 1,120, 1,060, 750. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz),  $\delta$  (ppm): 4.60 (s, 1H, CH), 5.34 (s, 2H, NH<sub>2</sub>), 6.54 (t, 2H,  $J = 8.44$  Hz, Ar), 6.70 (d, 1H,  $J = 8.36$  Hz, Ar), 7.06 (d, 2H,  $J = 8.2$  Hz, Ar), 7.40 (2H,  $J = 8.2$  Hz, Ar), 9.20 (s, 1H, OH). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz): 38.65, 57.56, 101.70, 111.50, 111.67, 119.23, 119.41, 128.35, 128.62, 130.37, 143.22, 147.74, 155.96, 158.49.
- Table 1, entry 7: 3,450, 3,300, 3,200, 2,200, 1,650, 1,600, 1,510, 1,380, 1,320, 1,120, 1,060, 760. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 400 MHz),  $\delta$  (ppm): 4.63 (s, 1H, CH), 6.37 (s, 1H, Ar), 6.45 (d, 1H,  $J = 8.00$  Hz, Ar), 6.74 (d, 1H,  $J = 8.4$  Hz, Ar), 6.87 (s, 2H, NH<sub>2</sub>), 7.10 (d, 2H,  $J = 8.4$  Hz, Ar), 7.16 (s, 3H, Ar), 9.70 (s, 1H, OH). <sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 100 MHz): 39.34, 56.63, 102.66, 112.91, 114.00, 115.66, 115.87, 121.04, 129.67, 129.75, 130.37, 143.02, 149.27, 157.61.

### Additional file

**Additional file 1:** Analytical data for new compounds synthesized in the presence of Cu-CeO<sub>2</sub> nanocomposite.

**Additional file 2: Analytical data for compounds synthesized in the presence of Cu-CeO<sub>2</sub> nanocomposite.**

#### Competing interests

The authors declare that they have no competing interests.

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