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Synthesis of 2,5-dihydro-3-furans using nano-$CoAl_2O_4$

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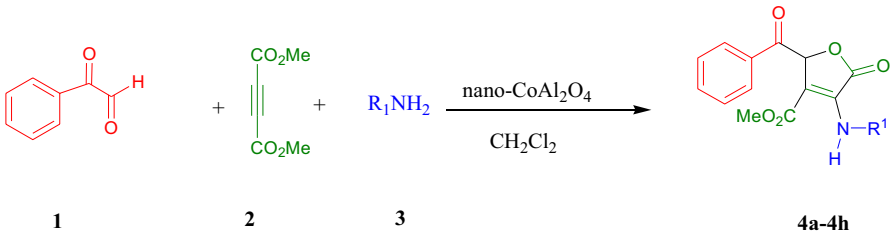
Abstract

Nano-$CoAl_2O_4$ has been used as an efficient catalyst for the preparation of 5-oxo-2,5-dihydro-3 furancarboxylates by multi-component reactions of phenylglyoxal, dimethyl acetylenedicarboxylate and primary amines. The best results were obtained in the presence of 4 mg of nano-$CoAl_2O_4$ in CH_2Cl_2 at room temperature. The structures of the products were deduced from their ^{1}H NMR, ^{13}C NMR, FT-IR and elemental analyses. Experimental simplicity, wide range of products, great yields in concise times, the retrievable of the nanocatalyst and low catalyst loading are some of the substantial features of this method.

Graphic abstract



1 **2** **3** **4a-4h**

Keywords Phenylglyoxal · Nanocatalyst · One-pot · Furancarboxylate

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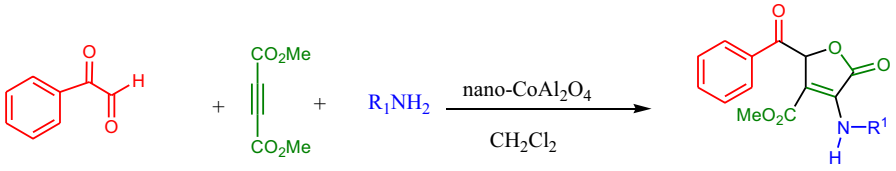
Introduction

Furans show anti-oxidation [1], antibacterial, [2], antimalarial [3], anticancer [4], anti-AIDS [5], anti-inflammatory [6] and anti-diabetic [7] activities. The detection effective procedure for the synthesis of furans is a serious challenge. The preparation of furans has been studied using catalysts including $K[Al(SO_4)_2]0.12H_2O$ [8], N-methyl 2-pyrrolidonium hydrogen sulfate [9], formic acid [10], $SnCl_2{\cdot}2H_2O$ [11], β-cyclodextrin [12], tetra-*n*-butylammonium bisulfate [13], $Al(HSO_4)_3$ [14], HY Zeolite [15] and Vitamin B12 [16]. Each of these catalysts may have its own advantages but also suffer from such apparent drawbacks as high times [12–14], complicated work-up [12], low efficiency [12, 16], or unwanted reaction conditions [11, 15]. Despite the use of these procedures, there remains a need for further new methods for the preparation of furans. Cobalt(II) compounds have been used extensively as a heterogeneous catalyst in many reactions, containing synthesis of hexahydroquinolines [17], soot combustion with NO_x/O_2 [18], Fischer–Tropsch synthesis [19] combination CO_2 reforming [20] and annulation of styrenes with α-bromoacetic acids [21]. Cobalt(II) aluminate has many advantages containing good selectivity with high stability, reusability and easy recyclability [22, 23]. Recently, nanoparticles as catalyst have appeared as an alternative way for the progression of many organic reactions [24–26]. Herein, we report the use of nano-$CoAl_2O_4$ as an efficient catalyst for the preparation of furans by multi-component reactions of phenylglyoxal, dimethyl acetylenedicarboxylate and primary amines (Scheme 1).

Experimental section

Materials and characterization

Reagent grade chemicals were purchased from Sigma-Aldrich and Merck and were used without further purification. All melting points were determined in a capillary tube on Boetius melting point microscope. NMR spectra were obtained on Bruker 400 MHz spectrometer with $CDCl_3$ as solvent using tetramethylsilane (TMS) as an internal standard, the chemical shift values are in δ. FT-IR spectrum was recorded on Magna-IR, spectrometer 550 Nicolet in KBr pellets in the range of 400–4000 cm^{-1}. The elemental analysis (C, H, N) was obtained from a Carlo ERBA Model EA 1108



1 2 3 4a-4h

Scheme 1 Synthesis of furans catalyzed by nano-$CoAl_2O_4$

analyzer. Powder X-ray diffraction (XRD) was carried out on a Philips diffractometer of X'pert Company with mono-chromatized Cu Kα radiation ($\lambda = 1.5406$ Å). The SEM images were prepared by MIRA3- TESCAN. The energy-dispersive X-ray spectroscopy (EDS) measurement was carried out with the SAMX analyzer. Surface area was carried out using nitrogen adsorption measurement (Micrometrics ASAP-2000). DLS was performed using a Malvern apparatus.

Synthesis of nano-$CoAl_2O_4$

$CoAl_2O_4$ was synthesized using sol–gel technique by citric acid. At first, a determined amount of $Al(NO_3)_3{\cdot}9H_2O$ (0.10 mol) and $Co(NO_3)_2{\cdot}6H_2O$ (0.05 mol) was solved in deionized water (100 mL). Afterward, an appropriate amount of citric acid was added (molar ratio of citric acid to metal ions was 2–1). Subsequently, the solution was stirred for 60 min and heated at 80 °C until a gel was created. The gel was dried in oven at 110 °C and fired at 500 °C with a heating rate of 10 K/min for 5 h in air.

General procedure for the preparation of furans (4a–h)

A mixture of amine (1 mmol) dimethyl acetylenedicarboxylate (1 mmol), phenylglyoxal (1 mmol) and nano-$CoAl_2O_4$ (4 mg) was stirred in dichloromethane (10 mL) at room temperature. After completion, as indicated by TLC (EtOAc-petroleum ether, 2:8), the nanocatalyst was separated from the mixture using filtration. The solvent was evaporated under vacuum and the product (Yellow oil) was washed with ethanol to give pure product. The characterization data of the compounds are given below.

Methyl 2-benzoyl-4-[(2-methoxybenzyl) amino]-5-oxo-2,5-dihydro-3-furancarboxylate (4a)

Yellow oil, FT-IR (KBr): 3402, 3052, 3004, 1771, 1705, 1675, 1604, 1476, 1122 cm^{-1}; ^1H NMR δ 8.04–6.92 (m, 9H, ArH), 6.25 (s, 1H, CH), 5.02–4.95 (m, 2H, CH_2), 3.87, 3.56 (2 s, 6H, 2MeO), 2.20 (s, 1H, NH). ^{13}C NMR δ 192.4, 164.5, 164.4, 157.3, 136.4, 135.2, 129.5, 129.4, 128.4, 128.1, 127.4, 126.7, 124.3, 110.6, 105.3, 76.1, 56.9, 52.5, 42.8. Anal. Calcd. for $C_{21}H_{19}NO_6$: C 66.13, H 5.02, N 3.67. Found: C 66.16, H 4.95, N 3.71.

Methyl 2-benzoyl-4-[(4-methoxybenzyl) amino]-5-oxo-2,5-dihydro-3-furancarboxylate (4b)

Yellow oil, FT-IR (KBr): 3302, 3105, 3006, 1752, 1702, 1675, 1602, 1478, 1468, 1379, 1102 cm^{-1}; ^1H NMR δ 8.09–6.92 (m, 9H, ArH), 6.42 (s, 1H, CH), 4.92–4.80 (m, 2H, CH_2), 3.84, 3.52 (2 s, 6H, 2MeO), 2.69 (s, 1H, NH), ^{13}C NMR δ 192.6, 169.1, 162.4, 159.2, 156.4, 155.2, 154.1, 153.2, 135.2, 135.1, 107.2, 105.4, 76.1, 60.2, 57.4, 44.6. Anal. Calcd. for $C_{21}H_{19}NO_6$: C, 66.13; H, 5.02; N, 3.67;. Found: C, 66.17; H, 5.08; N, 3.74.

Methyl 2-benzoyl-4-[(4-methylbenzyl)amino]-5-oxo-2,5-dihydro-3-furancarboxylate (4c)

Yellow oil, FT-IR (KBr): 3455, 3054, 3005, 1738, 1704, 1605, 1473,1468, 1105 cm^{-1}; ^{1}H NMR δ 8.06–7.10 (m, 9H, ArH), 6.25 (s, 1H, CH), 4.82–4.92 (m, 2H, CH_2), 3.65 (s, 3H, OCH_3), 2.95 (s, 1H, NH), 2.28 (s, 3H, CH_3). ^{13}C NMR δ 192.8, 168.2, 164.7, 138.2, 137.5, 136.7, 130.2, 129.8, 128.4, 127.8, 127.5, 127.4, 105.6, 76.1, 50.2, 48.6, 22.4. Anal. Calcd. for $C_{21}H_{19}NO_5$: C 69.03, H 5.24, N 3.83. Found: C 69.09, H 5.20, N 3.89.

Methyl 2-benzoyl-4-(benzylamino)- 5-oxo-2,5 dihydro-3-furancarboxylate (4d)

Yellow oil, FT-IR (KBr): 3455, 3058, 3004, 1752, 1706, 1608, 1477, 1155 cm^{-1}; $^{1}HNMR$ δ 8.35–7.12 (m, 10H, arom), 6.19 (s, 1H, CH), 4.94–4.90 (m, 2H, CH_2), 3.57 (s, 3H, OCH_3), 2.60 (s, 1H, NH). ^{13}C NMR δ 193.9, 169.8, 166.2, 138.2, 137.9, 137.6, 135.2, 129.8, 128.6, 128.5, 128.4, 128.3, 105.4, 76.1, 50.2, 46.9. Anal. Calcd. for $C_{20}H_{17}NO_5$: C 68.37, H 4.88, N 3.99. Found: C 68.30, H 4.80, N 4.03.

Methyl-2-benzoyl-4-[(4-fluorobenzyl)amino]-5-oxo-2,5-dihydro-3-furancarboxylate (4e)

Yellow oil, FT-IR (KBr): 3405, 3075, 3008, 1772, 1754, 1708, 1606, 1478, 1155 cm^{-1}; ^{1} H NMR δ 8.19–6.50 (m, 9H, arom), 6.84 (s, 1H, CH), 4.90–4.82 (m, 2H, CH_2), 3.51 (s, 3H, OCH_3), 2.62 (s, 1H, NH). ^{13}C NMR δ 192.3, 169.8, 167.2, 139.8, 138.2, 138.1, 132.2, 131.4, 129.4, 129.2, 125.6, 118.4, 104.8, 76.2, 51.4, 47.4. Anal. Calcd. for $C_{20}H_{16}FNO_5$: C 65.04, H 4.37, N 3.79. Found: C 65.01, H 4.31, N 3.82.

Methyl 2-benzoyl-5-oxo-4-(propylamino)-2,5 dihydro-3-furancarboxylate (4f)

Yellow oil, yield: FT-IR (KBr): 3352, 3055, 3004, 2972, 1776, 1702, 1652, 1474, 1122 cm^{-1}; ^{1}H NMR δ 8.15–7.25 (m, 5H, ArH), 6.32 (s, 1H, CH), 3.74 (t, $J=7.0$, 2H, CH_2), 3.62 (s, 3H, OCH_3), 2.42 (s, 1H, NH), 1.66–1.60 (m, 2H, CH_2), 0.92 (t, $J=7.0$, 3H, CH_3). ^{13}C NMR δ 193.4, 168.2, 165.4, 135.4, 129.8, 129.4, 128.8, 126.5, 105.8, 76.2, 53.5, 51.2, 24.2, 12.8. Anal. Calcd. for $C_{16}H_{17}NO_5$: C 63.36, H 5.65, N 4.62. Found: C 63.30, H 5.69, N 4.56.

Methyl-2-benzoyl-4-[((furan-2-yl)methane) amino]-5-oxo-2,5-dihydro-3-furancarboxylate (4g)

Yellow oil, FT-IR (KBr): 3408, 3070, 3002, 1770, 1754, 1703, 1604, 1475, 1152 cm^{-1}; ^{1} H NMR δ 8.29–7.02 (m, 8H, ArH), 6.24 (s, 1H, CH), 4.98–4.90 (m, 2H, CH_2), 3.52 (s, 3H, OCH_3), 2.40 (s, 1H, NH). ^{13}C NMR δ 192.5, 168.9, 163.2, 151.4, 142.6, 138.4, 137.8, 136.5, 129.8, 129.6, 128.5, 112.6, 110.5, 76.2, 51.2, 40.4. Anal. Calcd. for $C_{18}H_{15}NO_6$: C 63.34, H 4.43, N 4.10, Found: C 63.28, H 4.38, N 4.02.

Methyl-2-benzoyl-4-[(3,4-dichlorobenzyl) amino]-5-oxo-2,5-dihydro-3-furancarboxylate (4h)

Yellow oil, FT-IR (KBr): 3505, 3045, 3014, 1754, 1708, 1612, 1479, 1158 cm^{-1}; ^{1}HNMR δ 8.35–7.12 (m, 8H, ArH), 6.25 (s, 1H, CH), 4.94–4.90 (m, 2H, CH_2), 3.57 (s, 3H, OCH_3), 2.90 (s, 1H, NH). ^{13}C NMR δ 192.1, 168.4, 165.7, 141.2, 139.4, 138.2, 137.8, 137.2, 136.1, 135.4, 131.2, 131.0, 129.6, 128.1, 107.6, 76.1, 52.4, 45.7. Anal. Calcd. for $C_{20}H_{15}Cl_2NO_5$: C, 57.16; H, 3.60; N, 3.33;. Found: C, 57.10; H, 3.52; N, 3.28.

Results and discussion

XRD of nano-$CoAl_2O_4$ is presented in Fig. 1. This shape displays excellent phase purity of nano-$CoAl_2O_4$ that has a complete agreement with the recorded XRD for nano-$CoAl_2O_4$ (spinel-type structure, JCPDS 44-0160) [18]. Average crystalline size of the nano-$CoAl_2O_4$ using Scherrer's formula was computed to be 15–25 nm. The BET surface area of the nano-$CoAl_2O_4$ catalyst was obtained 75 m^2/g.

The size and morphology of nano-$CoAl_2O_4$ was considered using SEM (scanning electron microscope) image (Fig. 2). The results display particles with diameters in the size of nanometers. The size distribution of nano-$CoAl_2O_4$ was determined using DLS measurements, (Fig. 3) that is centered at a value of 37.5 nm. The elemental composition of nano-$CoAl_2O_4$ was investigated by EDS (Energy-Dispersive X-ray Spectroscopy) (Fig. 4) that showed the cobalt, aluminium and oxygen in the nano-$CoAl_2O_4$ structure.

At first, to find the optimum conditions, the one-pot reaction of 2-methoxybenzylamine, dimethyl acetylenedicarboxylate and phenylglyoxal in the presence of the diverse catalysts and solvents was selected as model reaction for the preparation of 5-oxo-2,5-dihydro-3-furancarboxylates. Several reactions were scrutinized using various solvents such as EtOH, CH_3CN, DMF, $CHCl_3$ and CH_2Cl_2. In this reaction, the use of aprotic solvents favors the reaction mechanism. The model reaction was carried out in the presence of various catalysts such as Et_3N (base), *p*-TSA (acid), ZrO_2, $InCl_3$, Co_3O_4, nano-CuI, nano-ZnO and nano-$CoAl_2O_4$.

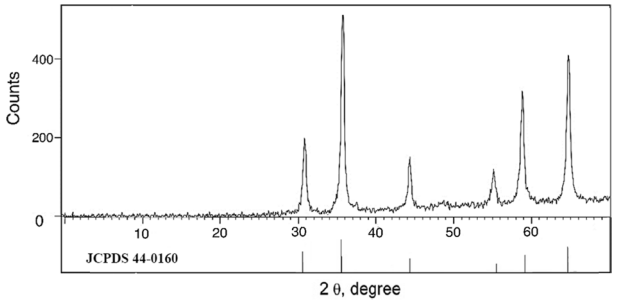


Fig. 1 XRD of nano-$CoAl_2O_4$

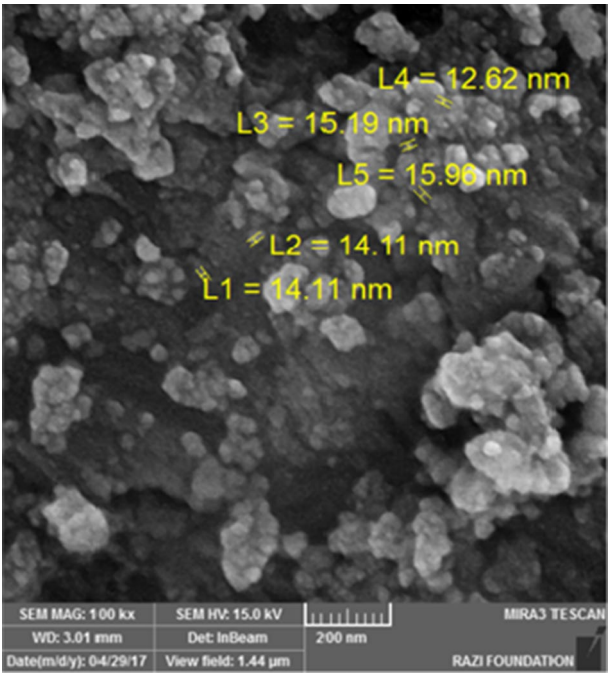


Fig. 2 SEM image of $CoAl_2O_4$ nanoparticle

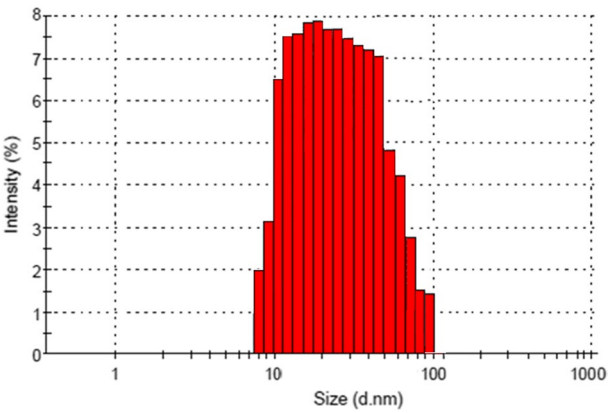


Fig. 3 DLS of nano-$CoAl_2O_4$

When the reaction was carried out using nano-CuI, Co_3O_4 and nano-$CoAl_2O_4$ as the catalyst, the product could be obtained in moderate to good yield and short times. As expected, the increased surface area due to small particle size increased reactivity of catalyst. The best results were gained in dichloromethane, and we found that the reaction gave convincing results in the presence of nano-$CoAl_2O_4$ (4 mg) at room temperature (Table 1). In the cases that the yield was not

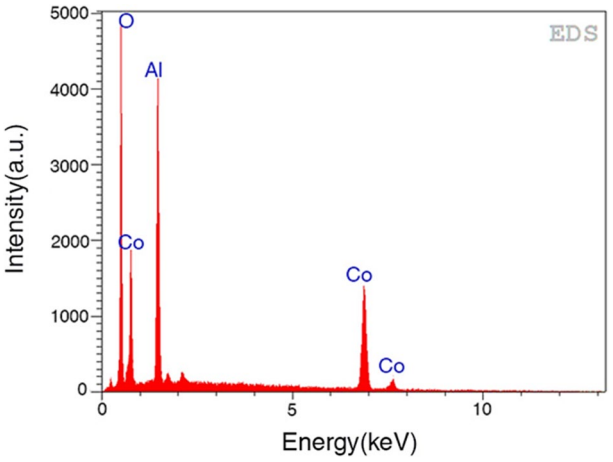


Fig. 4 EDS of nano-$CoAl_2O_4$

Table1 Optimization of reaction conditions[a]

Entry	Solvent	Catalyst (amount)	Time (min)	Yield[b] (%)
1	CH_2Cl_2	–	300	11
2	CH_2Cl_2	Et_3N (10 mol%)	300	43
3	CH_2Cl_2	ZrO_2(6 mol%)	300	26
4	CH_2Cl_2	*p*-TSA (10 mol%)	300	15
5	CH_2Cl_2	$InCl_3$ (7 mol%)	300	31
6	CH_2Cl_2	Co_3O_4 (5 mol%)	150	62
7	CH_2Cl_2	Nano-CuI (5 mg, 4 mol%)	200	52
8	CH_2Cl_2	Nano-ZnO (7 mg, 5 mol%)	200	39
9	CH_2Cl_2	Nano-$CoAl_2O_4$ (2 mg, 1 mol%)	50	85
10	CH_2Cl_2	Nano-$CoAl_2O_4$ (4 mg, 2 mol%)	50	93
11	CH_2Cl_2	Nano-$CoAl_2O_4$ (6 mg, 3 mol%)	50	93
12	CH_3CN	Nano-$CoAl_2O_4$ (6 mg, 3 mol%)	90	50
13	$CHCl_3$	Nano-$CoAl_2O_4$ (6 mg, 3 mol%)	60	82
14	DMF	Nano-$CoAl_2O_4$ (6 mg, 3 mol%)	90	60
15	EtOH	Nano-$CoAl_2O_4$ (6 mg, 3 mol%)	140	38

[a]Reaction conditions: 2-methoxybenzylamine (1 mmol) dimethyl acetylenedicarboxylate (1 mmol), phenylglyoxal (1 mmol)

[b]Isolated yield

satisfactory (Entry 1, 3, 4, 5 and 8), it was observed the presence of by-products and the recovering of the starting materials. After that, the obtained optimal conditions were applied to perform the reaction of different primary amines in the presence of nano-$CoAl_2O_4$ as catalyst, in order to afford the corresponding products in high to excellent yields (Table 2).

Table 2 Synthesis of 5-oxo-2,5-dihydro-3-furancarboxylates using nano-$CoAl_2O_4$[a]

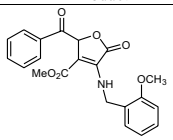
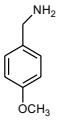
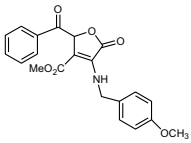
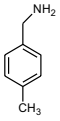
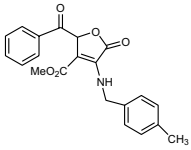
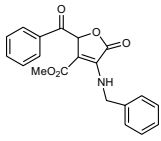
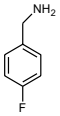
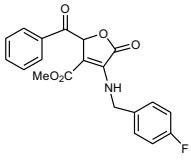
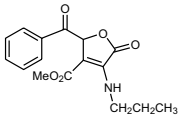
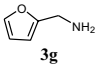
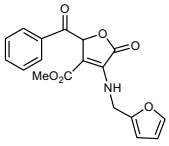
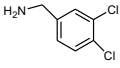
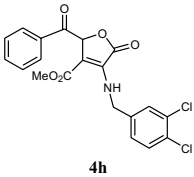
Entry	Primary amine	Product	Time (min)	Yield[b] %
1	 **3a**	 **4a**	50	93
2	 **3b**	 **4b**	50	91
3	 **3c**	 **4c**	60	89
4	 **3d**	 **4d**	70	88
5	 **3e**	 **4e**	70	83
6	$CH_3CH_2CH_2NH_2$ **3f**	 **4f**	70	82
7	 **3g**	 **4g**	70	86
8	 **3h**	 **4h**	70	84

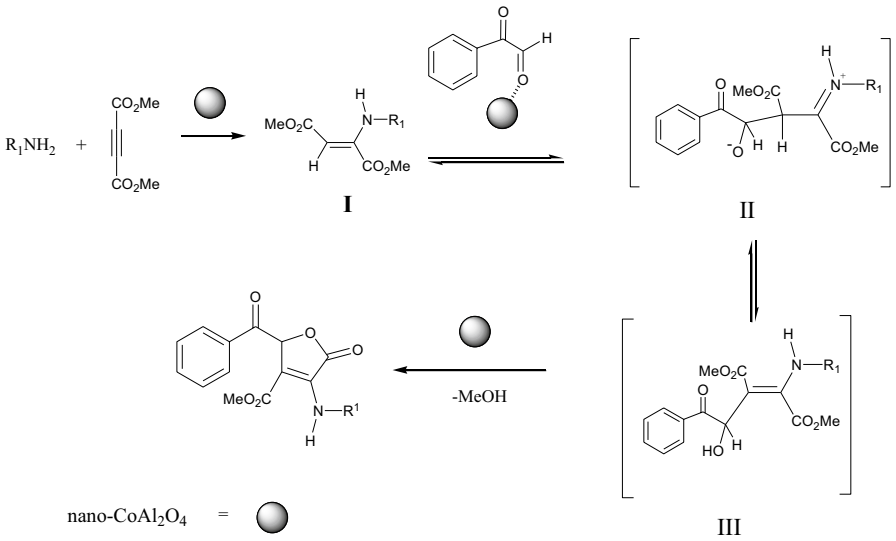
Table 2 (continued)

[a]Reaction conditions: primary amines (1 mmol) dimethyl acetylenedicarboxylate (1 mmol), phenylglyoxal (1 mmol)

[b]Isolated yield

The reusability of nanocatalyst was studied for the model reaction, and it was found that product yields lessened only to a very small extent on each reuse (run 1, 93%; run 2, 93%; run 3, 92%; run 4, 92%; run 5, 91%, run 6, 91%). After completion of the reaction (as determined by TLC), CH_2Cl_2 was added. The nano-$CoAl_2O_4$ was insoluble in CH_2Cl_2, and it could therefore be obtained by simple filtration. The catalyst was washed four times with ethanol and dried at room temperature for 15 h prior to re-use.

Scheme 2 shows a plausible mechanism for this reaction in the presence of nano-$CoAl_2O_4$. At first, the nucleophilic attack by the amine on dimethyl acetylenedicarboxylate generates the aminobutendioate **I** as an electron-rich enaminone. Subsequent nucleophilic attack of aminobutendioate **I** to the aldehyde carbonyl group of the phenylglyoxal would yield iminium–oxoanion intermediate **II**, which can be tautomerized to intermediate **III**. γ-Lactonization of intermediate **III** would produce the 5-oxo-2,5-dihydro-3-furancarboxylates.



Scheme 2 Proposed reaction pathway for the synthesis of **4a–h**

Conclusions

In conclusion, our procedure uses nano-$CoAl_2O_4$ at room temperature in dichloromethane for the synthesis of 5-oxo-2,5-dihydro-3-furancarboxylates. The structures of the products were deduced from their ^{1}H NMR, ^{13}C NMR, FT-IR and elemental analyses. The salient features of this protocol are: great yields in concise times, retrievability of the nanocatalyst, little nanocatalyst loading and easy separation of products.

Supporting information

The copies of the H NMR spectra of products are given in the Supporting Information available online.

Supplementary information The online version contains supplementary material available at https://doi.org/10.1007/s11164-021-04463-1.

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