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Majid M. Heravi [a] , Mansoureh Daraie [a] , Farahnaz K. Behbahani [b] & Reihaneh Malakooti [c]

[a] Department of Chemistry, School of Sciences, Azzahra University, Vanak, Tehran, Iran

[b] Department of Chemistry, School of Sciences, Azad University, Karaj Branch, Karaj, Iran

[c] Department of Chemistry, University of Birjand, Birjand, South Khorasan, Iran

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GREEN AND NOVEL PROTOCOL FOR ONE-POT SYNTHESIS OF β-ACETAMIDO CARBONYL COMPOUNDS USING Mn(bpdo)$_2$Cl$_2$/MCM-41 CATALYST

Majid M. Heravi,[1] Mansoureh Daraie,[1] Farahnaz K. Behbahani,[2] and Reihaneh Malakooti[3]
[1]*Department of Chemistry, School of Sciences, Azzahra University, Vanak, Tehran, Iran*
[2]*Department of Chemistry, School of Sciences, Azad University, Karaj Branch, Karaj, Iran*
[3]*Department of Chemistry, University of Birjand, Birjand, South Khorasan, Iran*

Aromatic aldehydes were reacted in a one-pot reaction with enolizable ketones, acetonitrile, and acetyl chloride in the presence of Mn(bpdo)$_2$Cl$_2$/MCM-41 under reflux condition or at 80 °C to afford the corresponding β-acetamido ketones in good yields.

Keywords: β-Acetamido carbonyl compounds; heterogeneous catalysis; Mn(bpdo)$_2$Cl$_2$/MCM-41

Multicomponent reactions (MCRs) are a promising and vital field of chemistry because they are rapid and efficient and can synthesize complicated molecules without isolation of any intermediate. They require minimum effort, which minimizes environmental loading and is acceptable from a green chemistry point of view. The synthesis of β-acetamido carbonyl compounds has gained considerable attention in organic synthesis. The β-acetamido carbonyl skeletons exist in a number of biologically or pharmalogically important compounds.[1,2] The best known procedure for the synthesis of these compounds is the Dakin–West reaction,[3] which involves the condensation of an α-amino acid with acetic anhydride in the presence of a base via an azalactone intermediate.[4] Iqbal and coworkers introduced both CoCl$_2$[5] and montmorillonite K-10 clay[6] catalyzed MCRs involving an aldehyde, enolizable ketone, or keto ester in acetonitrile and acetyl chloride for the one-pot synthesis of β-acetamido carbonyl compounds. Afterward, a number of catalysts such as Cu(OTf)$_2$/Sc(OTf)$_3$,[7] silica-supported sulfuric acid,[8] BiOCl,[9] ZrOCl$_2 \cdot$ 8H$_2$O,[10] H$_6$P$_2$W$_{18}$O$_{62}$,[11] H$_3$PW$_{12}$O$_{40}$,[12] I$_2$,[13] Amberlyst-15,[14] CeCl$_3 \cdot 7$H$_2$O,[15]

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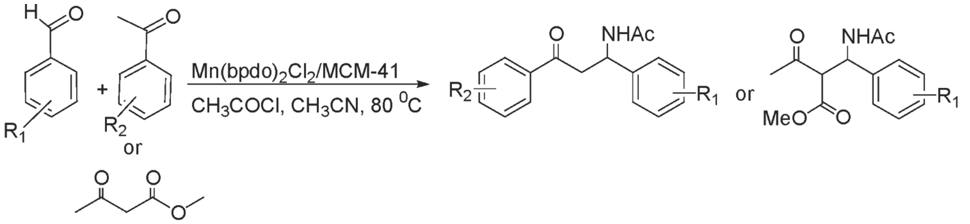
Address correspondence to Majid M. Heravi, Department of Chemistry, School of Sciences, Azzahra University, Vanak, Tehran, Iran. E-mail: mmh1331@yahoo.com



Scheme 1. $R_1 = H$, 4-nitro, 3-nitro, 4-methyl, 4-methoxy, 4-chloro; $R_2 = H$, 4-nitro.

and $FeCl_3 \cdot 6H_2O$[16] were reported as effective catalysts for the synthesis of β-acetamido carbonyl compounds.

Herein, we report an important three-component reaction in the presence of $Mn(bpdo)_2Cl_2$/MCM-41 as a green and nano-catalyst. β-Acetamido carbonyl compounds were synthesized using $Mn(bpdo)_2Cl_2$/MCM-41, aromatic aldehydes derivatives, acetophenone, acetyl chloride, and acetonitrile under reflux conditions (Scheme 1).

Previously, MCM-41 had been used in several organic transformation such as MCM-41-supported bidentate phosphine palladium(0) complex for the Sonogashira reaction of aryl iodides,[17] Al-MCM-41 for *N*-alkylation of aniline,[18] anchored palladium bipyridyl complex in nanosized MCM-41 for the Kumada–Corriu reaction,[19] vapor-phase oxidation of tetralin over Cr- and Fe-substituted MCM-41 molecular sieves,[20] MCM-41-supported bidentate phosphine palladium(0) complex for the heterogeneous Suzuki reaction,[21] and 12-tungstophosphoric acid supported on MCM-41 for esterification of fatty acid.[22] Recently, Mn-MCM-41 has been also used as catalyst[23] for hydroxylation of benzene,[24] the oxidation of ethyl benzene to acetophenone,[28] the oxidation of cyclohexene to cyclohexenol and cyclohexenone,[29] and oxidative aromatization of Hantzsch 1,4-dihydropyridines[30] and epoxidation of styrene and stilbene.[32]

RESULTS AND DISCUSSION

First, to optimize of the best reaction conditions, we set up a model reaction with benzaldehyde (1 mmol), acetophenone (1 mmol), acetyl chloride (1.5 mmol), and acetonitrile (0.05 mL). Results of 50% yield occurred at 4.0, 6.0, and 8.0 h and at 80 °C. Increasing the time just promoted the synthesis of chalcone (entries 1–3, Table 1). By changing conditions (5 mL of acetonitrile, 4.0 h, reflux), we obtained β-acetamido carbonyl compound/chalcon in 80/5% and 80/10% yields (entries 4 and 5, Table 1). In these cases, prolonged reaction times did not affect the yield of β-acetamido carbonyl compound. The use of 2 and 0.5 mL of acetonitrile (entries 6 and 7, Table 1) gave similar results. Therefore, entry 7 of Table 1 was chosen as the optimized reaction.

In optimized reaction conditions, various aldehydes with electron-donating and electron-withdrawing groups and enolized ketones such as acetophenone and 4-nitro acetophnone were subjected to the one-pot, three-component synthesis of β-acetamido carbonyl compounds in the presence of $Mn(bpdo)_2Cl_2$/MCM-41 under solvent-free conditions (Table 2). The naturality of groups did not affect the reaction

Table 1. Optimization of reaction conditions using benzaldehyde (1 mmol), acetophenone (1 mmol), and acetyl chloride (1.5 mmol) in the presence of various amounts of acetonitrile

Entry	CH_3CN(mL)	Temp. (°C)	Time (h)	Yield[a] (%) of chalcone/product
1	0.05	80	4.0	20/50
2	”	”	6.0	30/50
3	”	”	8.0	40/50
4	5	Reflux	4.0	5/80
5	”	”	7.0	10/80
6	2	”	4.0	5/80
7	0.5	”	”	5/80

[a] Yields refer to the isolated pure products.

times and yields. The reaction with methyl aceto acetate was also made instead of acetophenone derivatives. The results are shown in Table 2.

In summary, we have revealed a simple, efficient, and green protocol for the preparation of β-acetamido carbonyl compounds using $Mn(bpdo)_2Cl_2$/MCM-41 as a reusable catalyst. The salient features of this protocol include operational simplicity, excellent yields of the products, avoidance of column chromatography, ready availability, low toxicity, and reusability of the catalyst.

EXPERIMENTAL

All the chemicals were purchased from Merck Company. Melting points were measured by using the capillary tube method with an Electrothermal 9200 apparatus. ^1H NMR spectra were recorded on a Bruker AQS Avance 300-MHz spectrometer using tetramethylsilane (TMS) as an internal standard ($CDCl_3$ solution). Infrared (IR) spectra were recorded on the Fourier transform (FT)-IR Bruker Tensor 27. All products were known compounds and identified by comparison of their spectral and physical data to the literature description (Table 2).

Table 2. Preparation of β-acetamido carbonyl compounds from aldehydes and enolizable ketones in the presence of acetyl chloride and acetonitrile catalyzed using $Mn(bpdo)_2Cl_2$/MCM-41

					Mp (°C)	
Entry	Aldehyde	Ketone	Time (h)	Yield (%)[a]	Observed	Found
1	Benzaldehyde	Acetophenone	4.0	87	105–106	102–104[38]
2	4-Nitro benzaldehyde	”	4.0	78	149–151	148–149[39]
3	3-Nitro benzaldehyde	”	4.15	79	115–117	112–115[38]
4	4-Methyl benzaldehyde	”	3.0	85	113–115	112[39]
5	Benzaldehyde	4-Nitro acetophenone	3.0	85	75–77	74–76[38]
6	4-Methyl benzaldehyde	”	3.2	78	84–86	83–85[38]
7	4-Methoxy benzaldehyde	”	3.5	80	88–90	87–89[38]
8	4-Chloro benzaldehyde	”	4.0	85	121–122	116–118[38]
9	Benzaldehyde	Methyl aceto acetate	4.3	85	136–138	129–131[40]
10	4-Chloro benzaldehyde	”	3.5	75	137–139	130–132[40]

[a] Yields refer to the isolated pure products.

Preparation of MCM-41

MCM-41 was prepared according to the procedure described.[23,31,33–35]

Synthesis of bpdo Ligand

2,2′-Bipyridine-1,1′-dioxide was prepared according to the procedure reported by Simpson and coworkers.[36]

Synthesis of $Mn(bdpo)_2Cl_2$

This compound was prepared according to the procedure described previously.[36]

Immobilization of $Mn(bdpo)_2Cl_2$ Nanoreactors of MCM-41

$Mn(bdpo)_2Cl_2$ (0.2 g) in 5 mL of ethanol was slowly added to a solution of 1 g of MCM-41 in 5 mL of ethanol. The resultant mixture was refluxed for 24 h under nitrogen gas. Upon filtration of the mixture, the solid was washed with ethanol. The atomic absorption spectrophotometry (AAS) determination showed the percentages of Mn and Cl were 4.4 and 5.72, respectively.[37]

Synthesis of β-Acetamido Ketone and Esters: General Procedure

A mixture of ketone or methyl aceto acetate (5 mmol), aldehyde (5 mmol), and acetyl chloride (1.5 mL) in aceto nitrile (0.5 mL) was treated with a catalytic amount of Mn(bpd)/2 (1 mol%) at 80 °C. The progress of the reaction was monitored by thin-layer chromatography (TLC). Upon completion of the reaction, ice water was added to the reaction mixture. The precipitate was obtained for some derivatives. After filtration, the residue was washed with diethyl ether (10 mL) and the catalyst was removed. After evaporating the solvent, the crude product was crystallyzed with ethanol–water or n-hexane–ethyl acetate. For some cases in which a solid was not obtained, we solved them in petroleum ether and put them in a refrigerator overnight. Over time, we observed a solid and filtered and collected the product.

For the other derivatives, after adding ice water, the oily products were obtained, extracted with dichloromethane (10 mL), and dried, and the solvent was evaporated under reduced pressure.

To investigate the reusability and recyclibility of the catalyst, the catalyst was washed using diethyl ether and dried. The recycled catalyst could be subjected in subsequent experiments and gave similar results (76, 74, 75%).

Spectroscopic Data for Some Compounds

β-Acetamido-β-(phenyl)propiophenone (Table 2, Entry 1). IR (KBr) ν_{max}/cm^{-1}: 3286, 1693, 1650; ^1H NMR (300 MHz; $CDCl_3$) δ_H 2.1 (s, 3H, CH_3), 3.49 (dd, $J = 6.2$ and 16.9 Hz, 1H, CH_2), 3.77 (dd, $J = 5.3$ and 16.9 Hz, 1H, CH_2), 5.61 (m, 1 H, methyne H), 6.92 (br, d, $J = 6.3$ Hz, 1H, NH), 7.25–7.60 (m, 8H, Ar-H), 7.94 (d, $J = 7.5$ Hz, 2H, Ar-H), δc (100 MHz, $CDCl_3$) 23.6, 43.2, 49.9, 126.6, 127.7, 128.3, 128.9, 133.7, 136.4, 140.7, 169.3, 198.3.

β-Acetamido-β-(4-nitrophenyl)propiophenone (Table 2, Entry 2). IR (KBr) ν_{max}/cm^{-1}: 3291, 1689, 1653; ^1H NMR (300 MHz; $CDCl_3$) δ_H 2.05 (s, 3 H, CH_3), 3.38 (dd, $J = 5.8$ and 17.4 Hz, 1 H, CH_2), 3.85 (dd, $J = 7.1$ and 17.4 Hz, 1 H, CH_2), 5.64 (m, 1H, methyne H), 7.19–7.54 (m, 7H, Ar-H) 7.80 (d, $J = 7.7$ Hz, 2H, Ar-H), 9.10 (br, 1H, NH). δc (100 MHz, $CDCl_3$) 23.5, 42.6, 49.2, 123.7, 127.2, 127.9, 128.7, 131.0, 133.9, 136.0, 138.4, 169.5, 197.8.

β-Acetamido-β-(3-nitrophenyl)propiophenone (Table 2, Entry 3). IR (KBr) ν_{max}/cm^{-1}: 3291, 1689, 1653; ^1H NMR (300 MHz; $CDCl_3$) δ_H 2.12 (s, 3 H, CH_3), 3.55 (dd, $J = 5.5$ and 17.6 Hz, 1 H, CH_2), 3.73 (dd, $J = 5.0$ and 17.5 Hz, 1 H, CH_2), 5.70 (m, 1H, methyne H), 7.21 (d, $J = 7.6$ Hz, 1H, NH), 7.46–7.58 (m, 3H, Ar-H), 7.65 (t, $J = 7.4$ Hz, 1H, Ar-H), 7.76 (d, $J = 7.5$ Hz, 1H, Ar-H), 7.91(d, $J = 8.3$ Hz, 2H, Ar-H), 8.14(d, $J = 8.3$ Hz, 1H, Ar-H), 8.26(s, 1H, Ar-H). δc (100 MHz, $CDCl_3$) 23.6, 42.9, 49.3, 121.5, 122.6, 128.3, 129.1, 129.8, 130.8, 133.1, 134.2, 136.4, 143.7, 169.9, 198.3.

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