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Original article

Pseudo five-component process for the synthesis of functionalized tricarboxamides using CuI nanoparticles as reusable catalyst

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ABSTRACT

An efficient and multicomponent method has been developed for the synthesis of functionalized tricarboxamides at room temperature using CuI nanoparticles as catalyst. This method involved five-component coupling reactions of Meldrum's acid, isocyanides with aromatic aldehydes and amines at room temperature. Atom economy, wide range of products, excellent yields in short time and mild reaction conditions are some of the important features of this protocol. Notably, this catalyst could be recycled and reused for several times without significantly decreasing the catalytic activity.

1. Introduction

Multicomponent reactions (MCRs) have been proved to be an efficient method to approach complex structures in a single synthetic operation from simple building blocks. Advantages of MCRs are high selectivity, high atom economy, and procedural simplicity due to the formation of carbon–carbon or carbon–heteroatom bonds in one pot [1,2]. Typically, purification of products resulting from MCRs is also simple since all the organic reagents are consumed and converted into the target products [3–7].

Isocyanide-based multi-component reactions (IMCRs) are specifically interesting as they are more varied and versatile than other MCRs [8,9]. Isocyanides in multi-component reactions increase the diversity of available bond forming procedures, functional group tolerance, and levels of stereo-, chemo-, and regioselectivity. Therefore MCRs including isocyanides have emerged as suitable tools for the synthesis of structurally varied chemical libraries [10–12].

On the other hand, tricarboxamides and their analogs have displayed a wide range of important bioactivities, such as anti-tumor [13], anti-bacterial [14], anti-diabetic [15], neuroprotective [16] and anti-carcinogenic properties [17]. Carboxamides segments have an affinity to nucleic acids and cells, so their introduction into medicines can ease their interactions between cells and tissues and thereby provides a vigorous strategy to produce novel drugs or compounds.

The diversity of the structures encountered, as well as their pharmaceutical and biological relevance, has motivated research purposed at the development of convenient and efficient synthetic strategies, especially for the synthesis of substituted tricarboxamide scaffolds [18–22].

The chemical synthesis productivity can be increased by nano sized catalysts because of small size and high surface to volume ratios. Moreover, this productivity was improved by using heterogeneous catalysts for their simplicity of separation [23–27]. Recently, it was reported that CuI nanoparticles as heterogeneous catalysts offer huge opportunities for a wide range of applications in chemical synthesis and chemical manufacturing procedures [28,29].

In continuation of our efforts to develop syntheses of various biological compounds using reusable nano catalysts [30–32], we report here the use of CuI nanoparticles catalyzed five-component synthesis of tricarboxamides in mild conditions (Scheme 1).

2. Experimental

2.1. Preparation of copper iodide nanoparticles

The catalyst was prepared by ultrasonic irradiation. $CuSO_4$ was used as the Cu source. Firstly, the copper substrate (1 mmol) was ultrasonically cleaned for 20 s in acetone followed by repeated rinsing with distilled water. After drying, the substrate was dipped slowly into a solution of KI (1 mmol) in 40 mL of distilled water and

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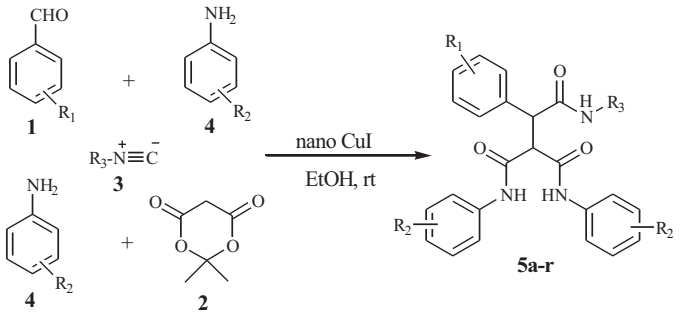


Scheme 1. Synthesis of tricarboxamides using CuI nanoparticles as catalyst.

sonicated to react for 30 min. When the reaction was completed, gray precipitate was obtained. The solid was filtered and washed with distilled water and dried.

To obtain a visual image of this catalyst, powder X-ray diffraction (XRD) and scanning electron microscopy (SEM) was carried out (see Figs. S1 and S2 in supporting information). As shown in the XRD pattern, all reflection peaks can be readily indexed to pure cubic crystal phase of nanocrystalline copper iodide. Crystallite size of CuI has been found to be 20 nm, which was calculated by the Debye-Scherrer equation ($D = K\lambda/\beta\cos\theta$) and confirmed by SEM. The increased surface area due to small particle size increased reactivity. This factor is responsible for the accessibility of the substrate molecules on the catalyst surface.

In addition, the specific surface area was measured by nitrogen physisorption (the BET method), the specific surface area was approximately 2.94 m^2/g.

2.2. General procedure for the preparation of tricarboxamides (**5a–r**)

A solution of aldehyde (2 mmol), Meldrum's acid (2 mmol), CuI nanoparticles (2 mol%) and ethanol (4 mL) was stirred for 30 min. Then, cyclohexyl isocyanide (2 mmol) and amine (4 mmol) was added and vigorously stirred for the appropriate time (Table 1, monitored by TLC). After completion of reaction, the solid was filtered off and washed with chloroform. The residue was dissolved in hot methanol and then filtered until the heterogeneous catalyst was recovered. The filtrate solution was recrystallized to afford the pure product in 89%–98% yield.

N^2,$N^{2\prime}$-Bis(4-chlorophenyl)-N^1-cyclohexyl-1-phenylethane-1,2,2-tricarboxamide **(5b)**: White solid; mp 309–311 °C, FT- IR (KBr, cm^{-1}): ν 3280, 3262, 1681, 1643, 1607; ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.11–1.62 (m, 10 H, $5CH_2$), 3.52 (s, 1H, NCH), 3.81 (d, 1H, J = 11.2 Hz, CH), 4.14 (d, 1H, J = 11.2 Hz, CH), 6.99–7.51 (m, 13H, Ar), 8.21 (brs, 1H, NH), 9.62 and 9.75 (2s, 2H, 2 NH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 173.2, 168.6, 167.1, 151.6, 150.9, 147.3, 147.1, 129.7, 128.3, 126.1, 125.6, 124.5, 122.3, 121.9, 118.7, 62.5, 49.4, 46.2, 33.4, 33.1, 26.8. Anal. Calcd. for $C_{29}H_{29}Cl_2N_3O_3$: C, 64.68; H, 5.39; N, 7.81. Found: C, 63.77; H, 5.16; N, 7.93. MS (EI) (m/z): 537 (M^+).

N^2,$N^{2\prime}$-Bis(4-bromophenyl)-1-(4-chlorophenyl)-N^1-cyclohexylethane-1,2,2-tricarboxamide **(5g)**: White solid; mp 314–318 °C, FT-IR (KBr, cm^{-1}): ν 3287, 3265, 1680, 1646, 1611; ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.13–1.57 (m, 10H, $5CH_2$), 3.55 (s, 1H, NCH), 3.79 (d, 1H, J = 11.4 Hz, CH), 4.17 (d, 1H, J = 11.3 Hz, CH), 7.06–7.68 (m, 12H, Ar), 8.19 (brs, 1H, NH), 9.63 and 9.77 (2s, 2H, 2 NH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 173.4, 168.1, 167.5, 151.8, 151.2, 148.0, 147.8, 146.5, 129.2, 127.4, 125.9, 124.3, 122.7, 122.1, 118.6, 62.6, 49.8, 46.7, 33.5, 33.0, 27.0. Anal. Calcd. for $C_{29}H_{28}Br_2ClN_3O_3$: C, 52.60; H, 4.23; N, 6.35. Found: C, 52.43; H, 4.16; N, 6.51. MS (EI) (m/z): 659 (M^+).

N^2,$N^{2\prime}$-Bis(4-methoxyphenyl)-1-(4-nitrophenyl)-N^1-cyclohexylethane-1,2,2-tricarboxamide **(5i):** Yellowish solid; mp 323–325 °C, FT-IR (KBr, cm^{-1}): ν 3288, 3268, 1682, 1650, 1617; ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.10–1.58 (m, 10H, $5CH_2$), 3.59 (s, 1H, NCH), 3.82 (d, 1H, J = 9.7 Hz, CH), 4.18 (d, 1H, J = 9.7 Hz, CH), 7.02–7.64 (m, 12H, Ar), 8.12 (brs, 1H, NH), 9.60 and 9.74 (2s, 2H, 2 NH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 173.2, 168.6, 167.1, 150.6, 149.8, 148.7, 145.4, 145.1, 129.9, 126.7, 125.4, 124.1, 122.9, 121.6, 118.5, 63.7, 50.1, 46.9, 33.7, 33.4, 26.4. Anal. Calcd. for $C_{31}H_{34}N_4O_7$: C, 64.81; H, 5.92; N, 9.76. Found: C, 64.55; H, 5.72; N, 9.94. MS (EI) (m/z): 574 (M^+).

N^1-tert-Butyl-N^2,$N^{2\prime}$-bis(4-chlorophenyl)-1-phenylethane-1,2,2-tricarboxamide **(5k):** White solid; mp 317–319 °C, FT-IR (KBr, cm^{-1}): ν 3301, 3287, 1678, 1647, 1636; ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.18–1.27 (s, 9H, $3CH_3$), 3.77 (d, 1H, J = 10.5 Hz, CH), 4.19 (d, 1H, J = 10.6 Hz, CH), 7.09–7.63 (m, 13H, Ar), 8.32 (s, 1H, NH), 9.58 and 9.79 (2s, 2H, 2 NH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 173.4, 168.1, 166.9, 151.2, 151.0, 147.6, 147.1, 129.8, 128.1, 125.9, 125.5, 124.8, 122.8, 121.7, 119.2, 62.1, 49.6, 46.3, 29.9. Anal. Calcd. for $C_{27}H_{28}Cl_2N_3O_3$: C, 64.62; H, 5.18; N, 7.88. Found: C, 63.65; H, 5.11; N, 7.98. MS (EI) (m/z): 511 (M^+).

N^1-tert-Butyl-N^2,$N^{2\prime}$-bis(4-bromophenyl)-1-(4-chlorophenyl)ethane-1,2,2-tricarboxamide **(5p):** Yellowish solid; mp 320–322 °C, FT- IR (KBr, cm^{-1}): ν 3296, 3258, 1688, 1651, 1617; ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.21–1.29 (s, 9H, $3CH_3$), 3.81 (d, 1H, J = 11.0 Hz, CH), 4.16 (d, 1H, J = 10.9 Hz, CH), 7.09–7.73 (m, 12H, Ar), 8.16 (s, 1H, NH), 9.68 and 9.81 (2s, 2H, 2 NH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 173.7, 167.8, 167.1, 151.9, 151.0, 148.3, 147.5, 146.2, 128.8, 127.5, 126.3, 124.8, 123.1, 122.0, 118.9, 62.7, 50.1, 46.3, 29.2. Anal. Calcd. for $C_{27}H_{27}Br_2ClN_3O_3$: C, 51.03; H, 4.18; N, 6.61. Found: C, 52.37; H, 4.12; N, 6.48. MS (EI) (m/z): 633 (M^+).

N^1-tert-Butyl-N^2,$N^{2\prime}$-bis(4-methoxyphenyl)-1-(4-nitrophenyl)ethane-1,2,2-tricarboxamide **(5r):** Yellowish solid; mp 329–331 °C, FT-IR (KBr, cm^{-1}): ν 3296, 3271, 1690, 1664, 1621; ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.20–1.32 (s, 9H, $3CH_3$), 3.86 (d, 1H, J = 8.1 Hz, CH), 4.22 (d, 1H, J = 8.1 Hz, CH), 7.05–7.61 (m, 12H, Ar), 8.11 (s, 1H, NH), 9.57 and 9.76 (2s, 2H, 2 NH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 172.6, 168.9, 167.3, 151.1, 149.6, 148.8, 145.2, 145.1, 129.4, 126.3, 125.1, 124.5, 123.3, 121.2, 118.7, 63.9, 50.4, 46.7, 29.5. Anal. Calcd. for $C_{29}H_{33}N_4O_7$: C, 63.49; H, 5.88; N, 10.21. Found: C, 64.11; H, 5.78; N, 9.95. MS (EI) (m/z): 548 (M^+).

Table 1
Optimization of model reaction.

Entry	Solvent/condition	Catalyst (mol%)	Time (h)	Yield (%)[a]
1	MeCN/rt	CuI (12%)	10	62
2	CH_2Cl_2/rt	CuI (12%)	10	87
3	H_2O/rt	CuI (12%)	10	31
4	MeOH/rt	CuI (12%)	10	88
5	EtOH/rt	CuI (12%)	10	89
6	EtOH/reflux	CuI (12%)	10	54
7	EtOH/rt	None	9	82
8	EtOH/rt	MgO (15%)	10	81
9	EtOH/rt	$InCl_3$ (20%)	10	85
10	EtOH/rt	Nano CuI (1%)	4	94
11	EtOH/rt	Nano CuI (2%)	4	98
12	EtOH/rt	Nano CuI (3%)	4	98

[a] Isolated yields.

3. Results and discussion

In our initial experiments, the model reaction conditions were established based on the reactions between benzaldehyde (2 mmol), 4-chloroaniline (4 mmol), cyclohexyl isocyanide (2 mmol) and Meldrum's acid (2 mmol) in different solvents and catalysts. This reaction was carried out using the aprotic (Table 1, entries 1 and 2) and protic solvents (Table 1, entries 3–5). The best result was obtained in ethanol (Table 1, entry 5). Next, we studied the model reaction in ethanol at different temperatures (Table 1,

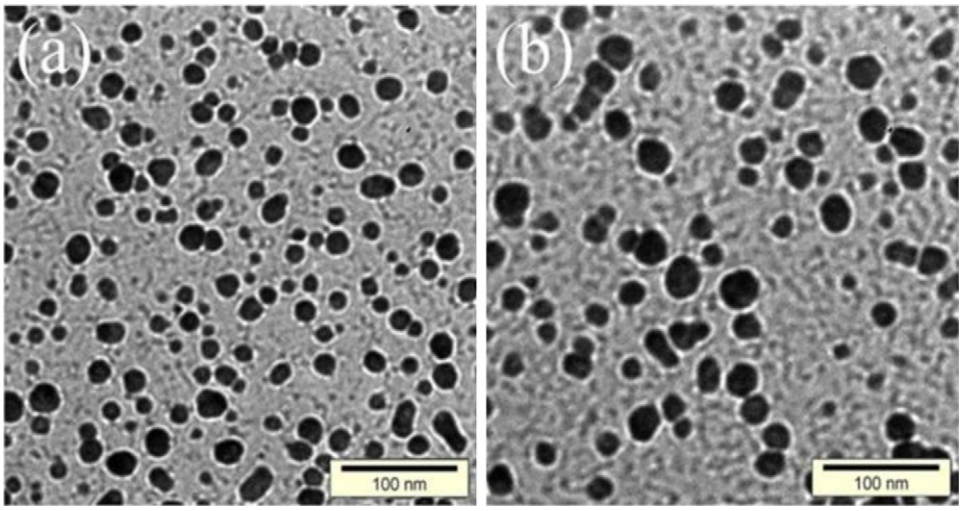


Fig. 1. TEM images of nano CuI before used (a), after four uses (b).

entries 5 and 6). The maximum yield was obtained at room temperature (Table 1, entry 5). The model reaction in ethanol at room temperature was also studied in absence of catalyst and using other types of catalysts (Table 1, entries 6–12). Although in the absence of catalyst, the reaction was carried out but in the presence of CuI nanoparticle we observed higher yield of product in shorter time (Table 1, entry 7). We believe that nano copper iodide surface chemistry plays an important role in this reaction. The best results were obtained with 2 mol% of nano CuI (Table 1, entry 11).

The same reaction was carried out many times in a row to check the reusability of catalyst. The catalyst could be reused four times with a minimal loss of activity (see Fig. S3 in supporting information). The characterization of the nano CuI before used and after four times reused, showed the same particle size by TEM (Fig. 1.). Interestingly, the shape and size of the nanoparticles remained the same before and after the reactions. We believe that, this is also the possible reason for the extreme stability of the CuI nanoparticles presented herein.

We have shown this reaction is possible under similar conditions with a wide range of aromatic aldehydes, amines, and isocyanides. Three substituents in the products could be varied freely of each other. The results were summarized in Table 2.

Aromatic aldehydes and amines possessing both electron-withdrawing and electron-donating substituents were converted into the corresponding tricarboxamides in good yields. In fact, the reaction was carried out well with all aldehydes and amines. However, aromatic aldehydes with low steric hindrance, and amines involving withdrawing groups showed excellent reactivity in this approach. Using benzaldehyde, 4-chloroaniline and cyclohexyl isocyanide gave the best yield (98%).

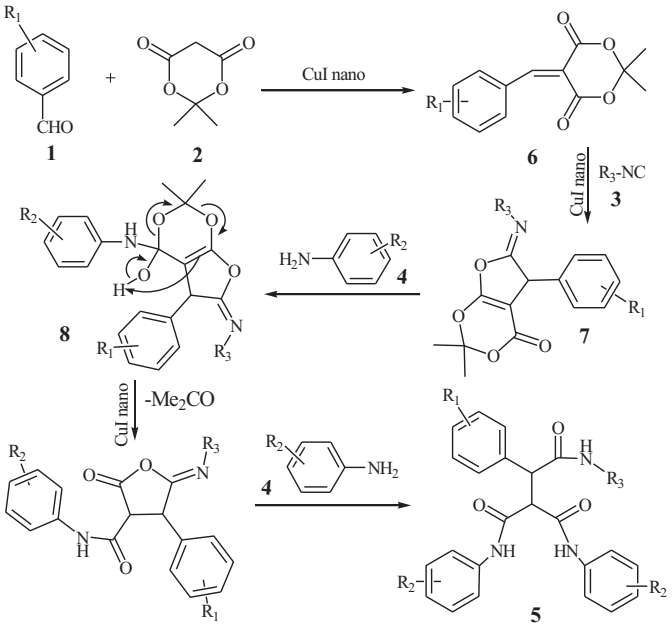
A proposed mechanism for this five-component reaction was outlined in Scheme 2 [19]. The first step of this reaction could be considered as nano CuI catalyzed Knoevenagel condensation between aldehyde **1** and Meldrum's acid **2** to afford intermediate **6**. Then **6** reacted with cyclohexyl isocyanide **3** following by a [1+4] cycloaddition, to give iminolactone **7** [33,34].

It was known that acylated Meldrum's acid readily was transformed into β-ketoamide by aminolysis [35]. Correspondingly,

Table 2
Synthesis of tricarboxamides using CuI nanoparticles.[a]

Entry	R_1	R_2	R_3	Product	Time (h)	Yield (%)[b]
1	H	H	Cyclohexyl	**5a**	4	97
2	H	4-Cl	Cyclohexyl	**5b**	3	98
3	H	2-Me	Cyclohexyl	**5c**	4	94
4	4-NO_2	H	Cyclohexyl	**5d**	4	95
5	4-Br	H	Cyclohexyl	**5e**	3	96
6	4-Me	H	Cyclohexyl	**5f**	4	93
7	4-Cl	4-Br	Cyclohexyl	**5g**	5	93
8	2-Cl	4-Me	Cyclohexyl	**5h**	6	92
9	4-NO2	4-OMe	Cyclohexyl	**5i**	6	91
10	H	H	*Tert*-butyl	**5j**	4	95
11	H	4-Cl	*Tert*-butyl	**5k**	4	97
12	H	2-Me	*Tert*-butyl	**5l**	5	94
13	4-NO_2	H	*Tert*-butyl	**5m**	5	93
14	4-Br	H	*Tert*-butyl	**5n**	4	95
15	4-Me	H	*Tert*-butyl	**5o**	4	92
16	4-Cl	4-Br	*Tert*-butyl	**5p**	5	91
17	2-Cl	4-Me	*Tert*-butyl	**5q**	5	90
18	4-NO2	4-OMe	*Tert*-butyl	**5r**	5	89

[a] Reaction and conditions: aldehyde/Meldrum's acid/isocyanide and amine = 2:2:2:4, 2 mol% nano CuI, EtOH; rt.
[b] Isolated yield.



Scheme 2. Possible mechanism for the formation of tricarboxamides.

it was reasonable to assume that the reaction of iminolactone with amine leaded to produce vinylogous carbonate **8**. Subsequently, in the presence of CuI, **8** reacted with another molecule of amine to afford product **5** after losing acetone.

4. Conclusion

In conclusion, we have demonstrated an efficient, clean, and one-pot procedure for the synthesis of tricarboxamides *via* five-component coupling of Meldrum's acid, isocyanides with aromatic aldehydes and amines over the high surface area of nano CuI as catalyst at room temperature. Wide range of products, mild reaction conditions, excellent yields of the products, reduced time of reaction, and a recyclable catalyst are advantages of this procedure over the previous reported ones.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.cclet.2013.01.049.

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