

## Silica-Supported LiHSO<sub>4</sub> as a Highly Efficient, Heterogeneous and Reusable Catalytic System for the Solvent-Free Synthesis of $\beta$ -Enaminones and $\beta$ -Enamino Esters

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A highly efficient, simple and green procedure for the synthesis of  $\beta$ -enaminones and  $\beta$ -enamino esters is described. The reaction of aromatic and aliphatic amines with  $\beta$ -dicarbonyl compounds using catalytic amount of silica-supported LiHSO<sub>4</sub> (LiHSO<sub>4</sub>/SiO<sub>2</sub>) under solvent-free conditions at 80 °C affords the title compounds in high to excellent yields and in short reaction times.

**Keywords:** LiHSO<sub>4</sub>/SiO<sub>2</sub>, Solvent-free,  $\beta$ -Enaminone,  $\beta$ -Enamino ester,  $\beta$ -Dicarbonyl compound, Green chemistry

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### INTRODUCTION

In recent years, the use of solid-supported catalysts has gained considerable attention both in industrial and academia research due to their unique properties such as enhanced reactivity as well as selectivity, straightforward workup, recyclability of the catalyst and the eco-friendly reaction conditions [1,2]. Silica gel is one of the most interesting solid-supports because it has surface properties that suggest that very rich organic reactions may be at work. SiO<sub>2</sub> is an inexpensive, reusable, commercially available and environmentally benign which, accompanied with different catalysts, has been used in various organic transformations [2]. Nevertheless, most of the existing processes in organic synthesis involve toxic and volatile organic solvents as reaction media which are environmentally unacceptable from the green chemistry view point. One of the most effective techniques to solve this problem is solvent-free conditions

which makes synthesis simpler, saves energy, and prevents solvent waste, hazards, and toxicity [3]. Consequently, a safe catalysis in combination with solventless conditions would provide for a more suitable technique to facilitate the so-called "ideal synthesis". In this paper, we wish to introduce silica-supported LiHSO<sub>4</sub> as a new catalyst for organic synthesis. The strong oxophilicity of Li<sup>+</sup> as well as H<sup>+</sup> of HSO<sub>4</sub><sup>-</sup> powerfully activate oxygen-containing electrophiles for nucleophilic attack [4].

$\beta$ -Enaminone and  $\beta$ -enamino ester derivatives are significant because they have been extensively applied as synthons for the synthesis of various biologically active compounds, such as anti-epileptic [5], antibacterial [6a,b], anti-inflammatory [6b], anticonvulsant [6b,c], antitumor [6b,d], and other therapeutic agents [7]. Therefore, there is a great deal of interest in the synthesis of this class of compounds. The direct condensation of amines with  $\beta$ -dicarbonyl compounds has been used as a useful synthetic route toward  $\beta$ -enaminones and  $\beta$ -enamino esters [8-23]. Several catalysts have been applied to achieve this transformation, including NaAuClO<sub>4</sub> [8], Bi(TFA)<sub>3</sub> [9],

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Sc(OTf)<sub>3</sub> [10], Zn(ClO<sub>4</sub>)<sub>2</sub>.6H<sub>2</sub>O/MgSO<sub>4</sub> [11], Yb(OTf)<sub>3</sub> [12], CeCl<sub>3</sub>.7H<sub>2</sub>O [13], [EtNH<sub>3</sub>]NO<sub>3</sub> [14], ZrOCl<sub>2</sub>.8H<sub>2</sub>O [15], HClO<sub>4</sub>.SiO<sub>2</sub> [16], I<sub>2</sub> [17], silica chloride [18], LaCl<sub>3</sub>.7H<sub>2</sub>O [19], Bi(TFA)<sub>3</sub>/tetrabutylammonium bromide [20], heteropoly acids [21], ceric(IV) ammonium nitrate [22] and phosphotungstic acid [23]. There are also some synthetic approaches to  $\beta$ -enaminones including condensation of methyl ketones with dimethylformamide dimethylacetal [24], cyclization of amino acids [25], aminolysis of dithioacetals [26], reductive cleavage of isoxazoles [27] and substitution of the imidoylbenzotriazoles with trimethylsilyl (TMS) enol ethers [28]. However, these reported methods are associated with one or more of the following drawbacks: (i) unsatisfactory yield, (ii) long reaction time, (iii) low selectivity, (iv) no generality, (v) the use of non-available and expensive reagents, (vi) tedious work-up procedure, and (vii) lack of agreement with the green chemistry protocols. Therefore, it seems highly desirable to find an efficient, simple, general, green, and cheap protocol for the synthesis of  $\beta$ -enaminones and  $\beta$ -enamino esters.

In this paper, we report a new, clean and highly efficient method for the synthesis of  $\beta$ -enaminones and  $\beta$ -enamino esters *via*  $\beta$ -enamination of  $\beta$ -dicarbonyl compounds using LiHSO<sub>4</sub>/SiO<sub>2</sub> as a new, non-toxic, readily producible, heterogeneous and reusable catalytic system (Scheme 1).

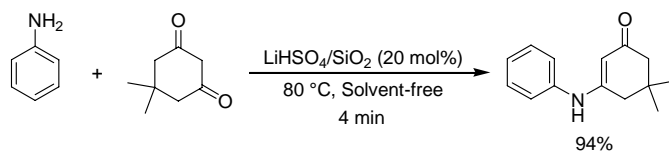
## EXPERIMENTAL

### Chemicals and Apparatus

All chemicals were purchased from Merck or Fluka Chemical Companies. Plate silica gel, (Mesh 60, 15-40  $\mu$ m) was used as support. All known compounds were identified by comparison of their melting points and <sup>1</sup>H NMR data with those in the authentic samples. The <sup>1</sup>H NMR (250 MHz) and <sup>13</sup>C NMR (62.5 MHz) were run on a Bruker Avance DPX-250, FT-NMR spectrometer. Microanalysis was performed on a Perkin-Elmer 240-B microanalyzer. Melting points were recorded on a Stuart Scientific Apparatus SMP3 (UK) in open capillary tubes.

### General Procedure for the Preparation of LiHSO<sub>4</sub>

A mixture of LiCl (0.848 g, 20 mmol) and H<sub>2</sub>SO<sub>4</sub> (2.002 g, 20 mmol) was stirred under N<sub>2</sub> gas for 120 min to give



Scheme 1

LiHSO<sub>4</sub> (2.080 g) as a white powder. The HCl gas produced was trapped with NaOH solution.

### General Procedure for the Preparation of LiHSO<sub>4</sub>/SiO<sub>2</sub> Catalytic System

A mixture of LiHSO<sub>4</sub> (0.5 g) and SiO<sub>2</sub> (9.5 g) was ground vigorously to give LiHSO<sub>4</sub>/SiO<sub>2</sub> catalytic system as a white powder (10 g).

### General Procedure for the Synthesis of $\beta$ -Enaminone and $\beta$ -Enamino Ester Derivatives

To a mixture of amine (1 mmol) and  $\beta$ -dicarbonyl compound (1 mmol) in a 10 ml round-bottomed flask connected to a reflux condenser was added LiHSO<sub>4</sub>/SiO<sub>2</sub> (0.416 g, 20 mol%) and the resulting mixture was stirred in an oil-bath (80 °C). After completion of the reaction (monitored by TLC), the reaction mixture was cooled to room temperature. To the resulting mixture, was added EtOAc (15 ml), stirred for 5 min and filtered to separate the catalyst. The solvent of the filtrate was evaporated and the crude product was purified by recrystallization from EtOH/H<sub>2</sub>O (1/1). The recycled catalyst was washed by EtOAc, dried and re-used.

### Selected Spectral Data of the Products

**4-(5,5-Dimethyl-3-oxocyclohex-1-enylamino)phenyl acetate (Table 3, entry 4).** White solid; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta$  (ppm) 1.13 (s, 6H), 2.10 (s, 2H), 2.24 (s, 2H), 2.76 (s, 3H), 5.73 (s, 1H), 7.02 (d, *J* = 7.1 Hz, 2H), 7.12 (s, 1H), 7.84 (d, *J* = 7.1 Hz, 2H); <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\delta$  (ppm) 26.4, 28.1, 32.8, 43.6, 50.3, 100.6, 115.8, 121.2, 140.3, 143.2, 158.8, 196.8, 198.4; Anal. Calcd. for C<sub>16</sub>H<sub>19</sub>NO<sub>3</sub>: C, 70.31; H, 7.01; N, 5.12. Found: C, 70.14; H, 7.13; N, 5.02.

**3-(Furan-2-ylmethylamino)-5,5-dimethylcyclohex-2-enone (Table 3, entry 9).** White solid; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta$  (ppm) 1.05 (s, 6H), 1.90 (s, 2H), 1.97 (s, 2H), 4.23 (s, 2H), 4.88 (s, 1H), 5.06 (s, 1H), 6.25 (d, *J* = 7.7 Hz, 1H), 6.31 (m, 1H), 7.34 (d, *J* = 5.6 Hz, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\delta$  (ppm)

27.3, 31.5, 40.1, 43.3, 50.2, 96.3, 107.8, 110.5, 142.3, 149.8; 165.6, 196.8; Anal. Calcd. for C<sub>13</sub>H<sub>17</sub>NO<sub>2</sub>: C, 71.21; H, 7.81; N, 6.39. Found: C, 71.10; H, 7.68; N, 6.47.

**(Z)-4-(3-Oxo-1,3-diphenylprop-1-enylamino)phenyl acetate (Table 3, entry 18).** <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ (ppm) 2.49 (s, 3H), 5.28 (s, 1H), 6.77 (d, *J* = 7.5 Hz, 2H), 7.39-7.51 (m, 8H), 7.97 (d, *J* = 7.5 Hz, 2H), 8.10 (d, *J* = 7.0 Hz, 2H), 12.88 (s, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>): δ (ppm) 26.1, 99.0, 113.8, 121.6, 127.4, 128.9, 129.3, 130.1, 130.8, 131.8, 132.2, 135.5, 139.4, 144.1, 160.0, 190.3, 196.8; Anal. Calcd. for C<sub>23</sub>H<sub>19</sub>NO<sub>3</sub>: C, 77.29; H, 5.36; N, 3.92. Found: C, 77.08; H, 5.24; N, 4.06.

## RESULTS AND DISCUSSION

To optimize the reaction conditions, we first studied the

effect of different molar ratios of LiHSO<sub>4</sub>/SiO<sub>2</sub> on β-enamination of dimedone (1 mmol) with aniline (1 mmol) (Scheme 1) under solvent-free conditions at a range of 25-100 °C. The results are summarized in Table 1. As Table 1 indicates, the best results were observed when the reaction was performed in the presence of 20 mol% of LiHSO<sub>4</sub>/SiO<sub>2</sub> at 80 °C.

The efficiency and capacity of the solvent-free procedure, in comparison to solution condition, was also studied. For this purpose, a mixture of aniline (1 mmol), dimedone (1 mmol) and LiHSO<sub>4</sub>/SiO<sub>2</sub> (20 mol%) was stirred in some solvents (10 ml) at 80 °C or under reflux conditions (Table 2). As it can be seen from Table 2, the solvent-free method afforded higher yield and shorter reaction time.

After optimization of the reaction conditions, different

**Table 1.** Effect of Different Molar Ratios of LiHSO<sub>4</sub>/SiO<sub>2</sub> and Temperature on the Reaction of Aniline with Dimedone under Solvent-free Conditions

Entry	Molar Ratio of LiHSO <sub>4</sub> /SiO <sub>2</sub> (%)	Temperature (°C)	Time (min)	Yield (%) <sup>a</sup>
1	5	80	45	37
2	10	80	12	85
3	20	80	4	94
4	30	80	3	91
5	20	25 (r.t.)	120	Trace
6	20	50	60	18
7	20	70	15	83
8	20	90	4	94
9	20	100	3	89

<sup>a</sup>Isolated yield.

**Table 2.** Comparative the β-Enamination of Dimedone with Aniline using LiHSO<sub>4</sub>/SiO<sub>2</sub> in Solution Conditions vs. the Solvent-free Method

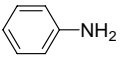
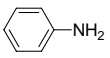
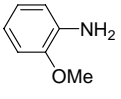
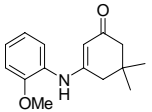
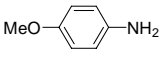
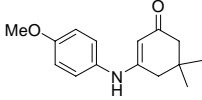
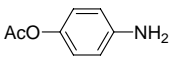
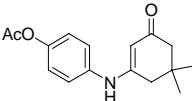
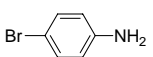
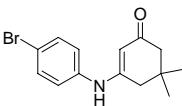
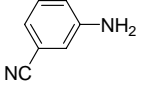
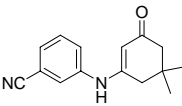
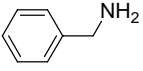
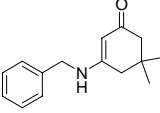
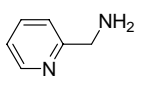
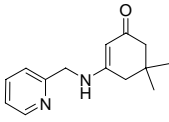
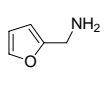
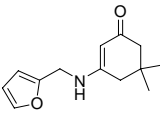
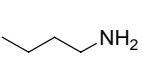
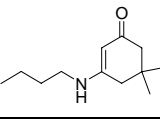
Entry	Solvent	Temperature (°C)	Time (min)	Yield (%) <sup>a</sup>
1	Solvent-free	80	4	94
2	EtOH	Reflux	60	61
3	CH <sub>2</sub> Cl <sub>2</sub>	Reflux	60	54
4	CH <sub>3</sub> CN	Reflux	60	35
5	THF	Reflux	60	24
6	H <sub>2</sub> O	80	120	-

<sup>a</sup>Isolated yield.

aromatic and aliphatic amines were condensed with some  $\beta$ -dicarbonyl compounds to furnish the desired  $\beta$ -enaminones and  $\beta$ -enamino esters in high to excellent yields and in short reaction times (Table 3). It was observed that the presence of

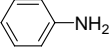
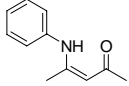
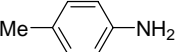
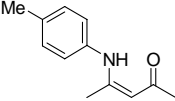
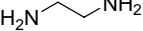
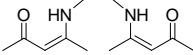
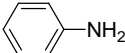
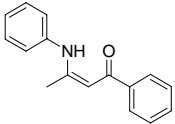
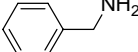
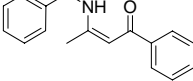
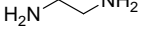
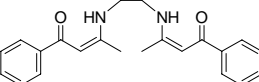
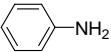
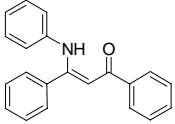

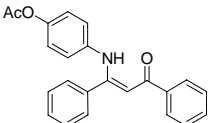
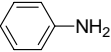
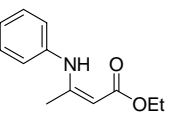

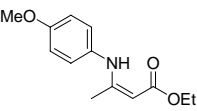
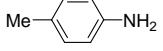
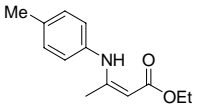
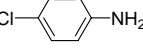
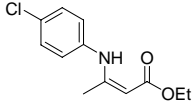
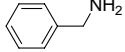
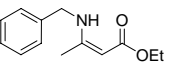
electron-releasing substituents or halogens on the aromatic ring of aromatic amines had no significant effect on the reaction results (Table 3, entries 2-5, 12, 18 and 20-22); however, electron-withdrawing substituents slightly decreased

**Table 3.** Synthesis of  $\beta$ -Enaminones and  $\beta$ -Enamino Esters using  $\text{LiHSO}_4/\text{SiO}_2$  under Solvent-Free Conditions at 80 °C

Entry	Amine	Product	Time (min)	Yield (%) <sup>a</sup>	M.p. °C (Lit.)
1			4	94	184-186 (185) [29]
2			8	89	126-128 (129-131) [30]
3			3	95	192-194 (195) [29]
4			7	92	225-227
5			6	93	219-220
6			10	87	182-184
7			4	95	128-129 (125) [29]
8			5	93	118-119
9			6	94	149-151
10			7	89	117-119

Silica-Supported LiHSO<sub>4</sub> as a Highly Efficient Catalytic System

Table 3. Continued

11			10	91	47-49 (47) [31]
12			8	93	66-68 (65-67) [32]
13			20	85	111-114 (112-114) [33]
14			12	91	109-111 (108) [12]
15			12	94	61-63 (59) [34]
16			30	87	182-184 (180) [9]
17			30	87	100-103 (99-101) [35]
18			40	84	149-150
19			27	88	Oil (Oil) [33]
20			18	91	45-47 (44.5-45) [31]
21			24	89	Oil (Oil) [33]
22			33	87	57-58 (53-54) [32]
23			25	86	Oil (Oil) [33]

<sup>a</sup>Isolated yield.

**Table 4.** The Condensation of Aniline with Dimedone in the Presence of Recycled LiHSO<sub>4</sub>/SiO<sub>2</sub>

Entry	Cycle	Time (min)	Yield (%) <sup>a</sup>
1	1 <sup>st</sup> use	4	94
2	2 <sup>nd</sup> use	5	94
3	3 <sup>rd</sup> use	8	93
4	4 <sup>th</sup> use	10	90
5	5 <sup>th</sup> use	15	89

<sup>a</sup>Isolated yield.

the yields and increased the reaction times (Table 3, entry 6). The condensation of aliphatic amines as well as diamines with  $\beta$ -dicarbonyl compounds was also efficiently performed (Table 3, entries 7-10, 13, 15, 16 and 23).

Ease of recycling of the catalyst is the most significant advantage of our method. For the reaction of aniline with dimedone no significant loss of the product yield was observed when LiHSO<sub>4</sub>/SiO<sub>2</sub> was reused after four times of recycling (please see Table 4).

## CONCLUSIONS

In summary, we have introduced a new and highly efficient catalyst for  $\beta$ -enamination of  $\beta$ -dicarbonyl compounds. The significant aspect of our methodology are efficiency, generality, high yield, short reaction time, ease of preparation of the catalyst, low cost, cleaner reaction profile, ease of product isolation, simplicity, potential for recycling of the catalyst, and finally agreement with the green chemistry protocols.

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