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One-Pot Synthesis of β-acetamido Ketones Using Silica-Supported Preyssler Nanoparticles (SPNPs) as Green and Reusable Catalyst

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Abstract: We describe an efficient method for the synthesis of β-acetamido ketones through the condensation of an aryl (aromatic) aldehyde, an acetophenone, acetyl chloride, and acetonitrile in the presence of silica-supported Preyssler nanoparticles (SPNPs) at room temperature. High efficiency, easy availability and reusability are some advantages of silica-supported Preyssler nanoparticles (SPNPs) as green and reusable catalyst.

Keywords: β-acetamido, Catalyst, Synthesis, Nanoparticles, Preyssler

1. Introduction

The formation of carbon-carbon bonds is crucial to the development of organic molecules such as medicines, biodegradable plastics and natural products and a great deal of research has been focused in this area recently. Mannich reaction plays a vital role in the construction of variety of organic molecules [1]. The products of the Mannich reaction are used for the synthesis of amino alcohols, peptides and lactams and as precursors to synthesize amino acids. β-Acetamido carbonyl compounds are valuable intermediates for a large number of pharmaceutically [2] important compounds examples being for the preparation of 1,3-aminoalcohols [3,4] antibiotic nikkomycin or neopolyoximes [5,6]. Therefore, the synthesis of acetamido carbonyl compounds continues to be a challenging endeavor. MCRs contribute to the requirements of an environmentally friendly process by reducing the number of synthetic steps, energy consumption and waste production. Researchers have transformed this powerful technology into one of the most efficient and economic tools for combinatorial and parallel synthesis [2,3]. Multicomponent reactions (MCRs) consist of two or more synthetic steps, which are performed without isolation of any intermediates, thus reducing time and saving both energy and raw materials. MCRs are powerful tools in the modern drug discovery process and allow fast, automated, and high throughput generation of organic compounds. Furthermore, a field of increasing interest is the synthesis of useful synthetic building blocks via MCRs chemistry. For this reason, the discovery of novel MCRs is of interest [7-9]. β-acetamido ketones are versatile intermediates in that their skeletons exist in a number of biologically or pharmacologically important compounds [2]. The best known route for the synthesis of these compounds is the Dakin-West reaction [10], which involves the condensation of anamino acid with acetic anhydride in the presence of a base via an intermediate azlactone to give the acetamido ketones [11]. Bhatia et al. proposed another procedure for the formation of these compounds through the condensation of an aryl aldehyde, acetophenone, and acetyl chloride in acetonitrile in the presence of $CoCl_2$ [12] or montmorillonite K-10 clay [13]. Other catalysts such as heteropolyacids [14], $HClO_4$-SiO_2 [15], $CeCl_3$ [16], ZnO [17], cyanuric chloride [18], Amberlyst-15 [19], and $POCl_3$/Borax [20] have been used. Although these methods are valuable, most of them employ expensive catalysts, long reaction times, or harsh reaction conditions. Therefore, the introduction of new and efficient methods for this multicomponent reaction is still necessary. As known, solid heteropolyacids (HPAs) and their

derivatives have been intensively studied because they can be used as excellent acidic, redox, and bifunctional catalysts in catalytic reactions [21-23]. Preyssler HPA for a broad range of organic syntheses and environmentally benign catalysis during the past few years [24-25]. Good yields, high selectivity, economical convenience, ease of work up, high hydrolytic and thermal stability, and high catalytic activity of Preyssler HPA [26-32] have indicated high potential for nano-catalysis in organic synthesis and environmentally benign catalysis.

2. Experimental

2.1. Chemicals and Apparatus

The organic materials were purchased from Sigma-Aldrich and Merck and were used without any additional purification. All compounds gave satisfactory spectroscopic data. Melting points were measured by using the capillary tube method with an electro thermal 9200 apparatus. IR spectra were recorded on a 8700 Shimadzu Fourier transform spectrophotometer. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AVANCE 200-MHz instrument using TMS as an internal reference. All products were identified by comparing their NMR and IR data with those reported in the literature.

2.2. Catalyst Synthesis Procedure

$H_{14}[NaP_5W_{30}O_{110}]$ was prepared according to the literature [33-35]. To a solution of the surfactant, sodium bis (2-ethylhexyl) sulphosuccinate, in cyclohexane (0.2 mol L^{-1}), a solution of Preyssler acid in a specified amount of water was added. The molar ratio of water to surfactant was selected to be 3, 5 and 7. Tetraethoxysilane was then added to the micro-emulsion phase. After mixing for various times (8, 12, 18, 25 and 30 h) at room temperature, dispersed Preyssler acid/SiO_2 nanostructures were centrifuged (1500 rpm) and the particles were rinsed with acetone (4 times) and dried in a vacuum oven. The optimum ratio of water to surfactant was 3:1 and the optimum time was 30 h.

2.3. General Procedure for the Synthesis of β-acetamido Ketones

To a stirred suspension of silica-supported Preyssler nanoparticles (SPNP) (0.05 g) in acetonitrile (2 mL) were added an aldehyde (2 mmol), an enolizable ketone (2 mmol) and acetyl chloride (0.5 mL). The reaction mixture was stirred at ambient temperature for 12 h. The progress of the reaction was monitored by TLC. After 12 h, 3 mL of ethanol was added to the cold reaction mixture, stirred for 2 min, and the catalyst was recovered by filtration and washed with ethanol. Then, 50 mL of cold water was added to the filtrate, the precipitated product was filtered and recrystallized from a mixture of ethanol and water. The precipitated solid was filtered off. The residue was washed with water (20 mL) and the crude product recrystallized from ethyl acetate/n-hexane.

2.4. The Compounds Spectral Data

β-Acetamido-*β*-(phenyl)propiophenone (Table 1, entry 1), IR (KBr, cm^{-1}): 3272, 3093, 1690, 1643, 1557, 1451, 1347, 1295, 993, 754., ^{1}H NMR (200 MHz, $CDCl_3$): δ_H 2.03 (s, 3H), 3.46 (dd, J = 6.0 and 16.8 Hz, 1H), 3.75 (dd, J = 5.2 and 16.8 Hz, 1H), 5.54-5.61 (m, 1H), 6.94 (d, J = 6.3 Hz, 1H), 7.23-7.37 (m, 5H), 7.44 (t, J = 8 Hz, 2H), 7.55 (t, J =7.6 Hz, 1H), 7.91 (d, J = 7.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ_c 23.34, 43.19, 49.98, 126.51, 127.48, 128.13, 128.7, 133.56, 136.56, 140.87, 169.72, 198.52.

β-Acetamido-*β*-(4-nitrophenyl)-4-nitropropiophenone (Table 1, entry 2), IR (KBr, cm^{-1}): 3277, 3078, 1699, 1660, 1527, 1408, 1348, 1235, 1196, 1109, 997, 851, 751., ^{1}H NMR (200 MHz, $CDCl_3$): δ_H 2.09 (s, 3H), 3.58 (dd, J = 5.8 and 17.8 Hz, 1H), 3.90 (dd, J = 4.9 and 17.8 Hz, 1H), 5.65-5.71 (m, 1H), 6.74 (d, J = 7. 7 Hz, 1H), 7.54 (d, J = 8.6 Hz, 2H), 8.09 (d, J = 8. 8 Hz, 2H), 8.20 (d, J = 8.7 Hz, 2H), 8.32 (d, J = 8.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ_c 23.39, 43.36, 46.15, 124.0, 124.09, 127.52, 129.18, 140.33, 147.28, 147.77, 150.73, 169.83, 196.34.

β-Acetamido-*β*-(2-chlorophenyl)-4-nitropropiophenone (Table 1, entry 3), IR (KBr, cm^{-1}): 3295, 3077, 1699, 1654, 1525, 1345, 1245, 997, 849, 753., ^{1}H NMR (200 MHz, $CDCl_3$): δ_H 2.13 (s, 3H), 3.53 (dd, J = 4.8 and 16.7 Hz, 1H), 3.85 (dd, J = 4.0 and 17.53 Hz, 1H), 5.85 (s, 1H), 6.79 (s, 1H), 7.19-7.46 (m, 4H), 8.08 (d, J = 8.5 Hz, 2H), 8.30 (d, J = 8.35 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ_c 23.39, 43.36, 46.15, 124.0, 124.09, 127.52, 129.18, 140.33, 147.28, 147.77, 150.73, 169.83, 196.34.

β-Acetamido-*β*-(phenyl)-4-nitropropiophenone (Table 1, entry 4), IR (KBr, cm^{-1}): 3297, 3079, 1698, 1640, 1518, 1345, 1203, 996, 850, 749., ^{1}H NMR (200 MHz, $CDCl_3$): δ_H 2.06 (s, 3H), 3.49 (dd, J = 6.5 and 16.7 Hz, 1H), 3.86 (dd, J = 5.1 and 16.8 Hz, 1H), 3.54 (m, 1H), 6.66 (s, 1H), 7.27-7.35 (m. 5H), 8.07 (d, J = 8.7 Hz, 2H), 8.29 (d, J = 8.7 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ_c 23.28, 44.07, 50.35, 123.94, 126.60, 128.01, 128.95, 129.22, 140.04, 140.87, 169.92, 196.74.

β-Acetamido-*β*-(2-metoxyphenyl)-4-bromopropiophenone (Table 1, entry 5), IR (KBr, cm^{-1}): 3312, 3074, 1694, 1648, 1543, 1301, 1242, 1197, 1100, 811, 758, 516., ^{1}H NMR (200 MHz, $CDCl_3$): δ_H 2.02 (s, 3H), 3.43 (dd, J = 6.7 and 15.72 Hz, 1H), 3.55 (dd, J = 6.0 and 15.9 Hz, 1H), 3.89 (s, 3H), 5.71 (m, 1H), 6.86-6.94 (m, 5H), 7.59 (d, J = 8.5 Hz, 2H), 7.77 (d, J = 8.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ_c 23.62, 42.10, 56.12, 72.64, 112.11, 120.87, 126.12, 127.52, 127.75, 129.32, 129.85, 131.54, 131.55, 135.72, 156.54, 170.76, 200.11.

β-Acetamido-*β*-(4-bromophenyl)-4-bromopropiophenone (Table 1, entry 6), IR (KBr, cm^{-1}): 3269, 3061, 1684, 1638, 1586, 1541, 1300, 1073, 995, 826., ^{1}H NMR (200 MHz, $CDCl_3$): δ_H 2.03 (s, 3H), 3.35 (dd, J = 6.0 and 17.1 Hz, 1H), 3.71 (dd, J = 4.3 and 17.1 Hz , 1H), 5.47-5.53 (m, 1H), 6.80 (d, J = 7.8 Hz, 1H), 7.21 (d, J = 8.3 Hz, 2H), 7.44 (d, J = 8.4 Hz, 2H), 7.60 (d, J = 8.5 Hz, 2H), 7.76 (d, J = 8.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ_c 23.32, 42.89, 49.39, 121.47,

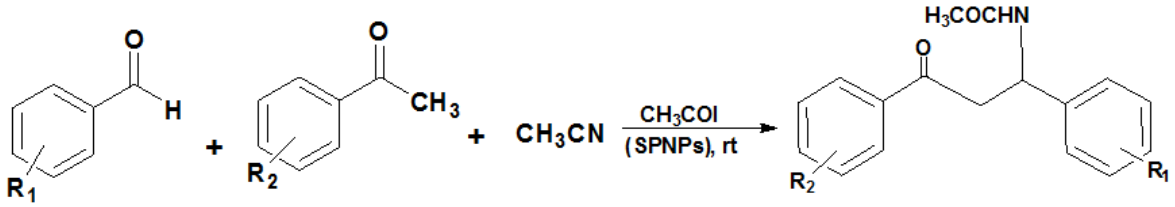
128.29, 129.06, 129.60, 131.80, 132.12, 135.07, 139.73, 169.78, 197.23.

β-Acetamido-*β*-(2-chlorophenyl)propiophenone (Table 1, entry 7), IR (KBr, cm^{-1}): 3290, 3079, 1692, 1653, 1547, 1441, 1356, 1296, 1229, 996, 749, 691, 620., ^{1}H NMR (200 MHz, $CDCl_3$): δ_H 2.04 (s, 3H), 3.46 (dd, *J* = 5.5 and 16.8 Hz, 1H), 3.76 (dd, *J* = 5.9 and 16.8 Hz, 1H), 5.80-5.87 (m, 1H), 7.22 (m, 5H), 7.50 (m, 3H), 7.90 (d, *J* = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ_c23.28, 44.07, 50.35, 123.94, 126.60, 128.01, 128.95, 129.22, 140.04.

β-Acetamido-*β*-(2-nitrophenyl)propiophenone (Table 1, entry 8), IR (KBr, cm^{-1}): 3304, 3066, 1699, 1653, 1600, 1527, 1349, 1296, 1230, 992, 751, 695., ^{1}H NMR (200 MHz, $CDCl_3$): δ_H 2.08 (s, 3H), 3.50 (dd, *J* = 5.5 and 17.6 Hz, 1H), 3.80 (dd, *J* = 5.1 and 17.6 Hz, 1H), 5.63-5.69 (m, 1H), 7.11 (d, *J* = 7.9 Hz, 1H), 7.44-7.63 (m, 5H), 7.89 (d, *J* = 8.5 Hz, 2H), 8.16 (d, *J* = 8.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ_c 23.28, 44.07, 50.35, 123.94, 126.60, 128.01, 128.95, 129.22, 140.04.

3. Result and Discussion

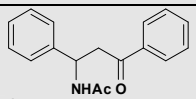
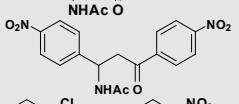
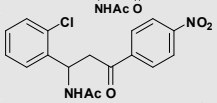
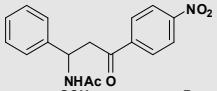
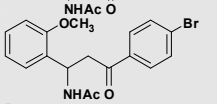
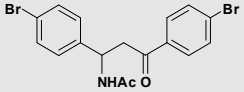
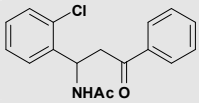
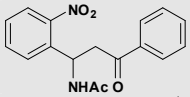
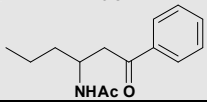
At the first, the reaction of benzaldehyde, acetophenone, acetyl chloride and acetonitrile was studied in presence of silica-supported Preyssler nanoparticles (SPNPs) catalyst (Scheme 1., Table 1).



Scheme 1. *Synthesis of β-acetamido carbonyl compounds using silica-supported Preyssler nanoparticles (SPNPs) catalyst.*

The results showed that the naturality of groups didn't affect upon the reaction time and yields (Table 1). The minimum yield and longer time was obtained with aliphatic aldehyde (Table 1, entry 9). The reaction was not completed even after 24 h (Table 1, entry 9). In generally, the catalyst is an effective component for thess reactions. We observed formation of *β*-acetamido carbonyl compounds with enhanced yield (87-98%) as shown in Scheme 1 and Table 1.

Table 1. *Synthesis of β-acetamido ketones using silica-supported Preyssler nanoparticles (SPNPs) at room temperature and in appropriate times.*

Entry	R_1	R_2	Product	Time (min)	[a]Yield (%)	Mp (ºC)
1	C_6H_5	C_6H_5		26	90 (90, 89, 89) [b]	104-106
2	4-$NO_2C_6H_4$	4-$NO_2C_6H_4$		40	87	176-179
3	2-ClC_6H_4	4-$NO_2C_6H_4$		27	94 (94,94,93) [b]	192-195
4	C6H5	4-$NO_2C_6H_4$		28	91	101-103
5	2-$CH_3OC_6H_4$	4-BrC_6H_4		33	92	161-162.5
6	4-BrC_6H_4	4-BrC_6H_4		28	98 (98, 98, 97) [b]	142-145
7	2-ClC_6H_4	C_6H_5		29	96 (96,96,96) [b]	155-157
8	2-$NO_2C_6H_4$	C_6H_5		55	89	148-150
9	C_3H_7	C_6H_5		134 and in (24 h)	30 and 30	143-146

[a] Isolated yield; [b]Catalyst was reused over three runs. all the products were fully characterized by IR spectroscopy ^{1}H NMR and ^{13}C NMR, and their data were compared with authentic data (Lit. 36-47).

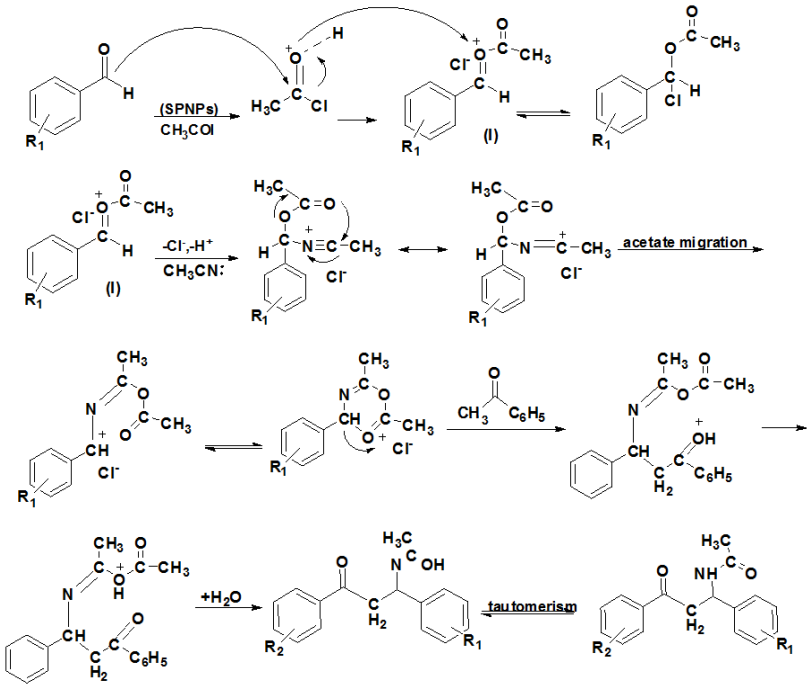
In an effort to develop an optimal catalytic system, various reaction parameters like effect of catalyst loading, and time for the synthesis of *β*-acetamido-*β*-(phenyl)propiophenone (Table 1, entry 1) were studied (Table 2). At the first, also, the reaction of benzaldehyde, acetophenone, acetyl chloride and acetonitrile was studied in the absence of catalyst (Table 2, entry 1).

We reported the results and effective of various catalysts in the synthesis of *β*-acetamido-*β*-(phenyl)propiophenone (Table 1, entry 1). The results listed in Table. 2 showed that the conversions were sensitive to the catalysts types. To further improve the yield and to optimize the reaction conditions, the same reaction was carried out in the presence of (0.05 g) of heteropolyacids catalysts under similar conditions. The percentage yield of the product with 0.01, 0.03, 0.04, 0.05, 0.09 and 0.1 g of silica-supported Preyssler nanoparticles (SPNPs) as the catalyst are 81%, 84%, 89%, 90%, 90% and 90%, respectively. Therefore, it was found that the use of 0.05 g of the catalyst was sufficient to promote the reaction, and higher amounts of the catalyst did not increase the yields significantly. In all contents, complete conversion was observed after appropriate time and the products were isolated in very high yields. To optimize the reaction conditions, the reaction of benzaldehyde, acetophenone, acetyl chloride, and acetonitrile was used as a model reaction with various catalysts (Table 2). After completion of the reaction, the catalyst silica-supported Preyssler nanoparticles (SPNPs) can easily be separated from the reaction mixture.

Table 2. *The synthesis of β-Acetamido-β-(phenyl)propiophenone (Table 1, entry 1) using various catalysts in appropriate times and at room temperature.*

Entry	Catalyst	mol % or g	Time (min)	[a] Yield (%)
1	silica-supported Preyssler nanoparticles (SPNPs)	0.01g	26	81
2	silica-supported Preyssler nanoparticles (SPNPs)	0.03g	26	84
3	silica-supported Preyssler nanoparticles (SPNPs)	0.04g	26	89
4	silica-supported Preyssler nanoparticles (SPNPs)	0.05g	26	90
5	silica-supported Preyssler nanoparticles (SPNPs)	0.09g	26	90
6	silica-supported Preyssler nanoparticles (SPNPs)	0.1g	26	90
7	Preyssler Heteropolyacid $H_{14}[NaP_5W_{30}O_{110}]$	0.05g	26	87
8	Trichloroisocyanuric acid	0.01 g	45	76
9	$Fe(CF_3CO_2)_3$	15	69	62
10	Silica chloride	0.04 g	78	64
11	Montmorilonite K-10	0.02 g	65	67
12	$Sc(OTf)_3$	2 g	62	71
13	$Cu(OTf)_2$	10	64	70
14	$Bi(OTf)_3$	10	60.5	73
15	I_2	10	71	61.5
16	Amberlyst-15	10	69	66
17	CuO	0.2 g	83	57.5
18	Fe_2O_3	30	81	48.5
19	ZnO	30	80	50
20	$Zn(HSO_4)_2$	30	76	55
21	$Fe(ClO_4)_3.6H_2O$	20	78	59
22	$Mg(HSO_4)_2$	20	82	54

[a] Isolated yield.



Scheme 2. *Proposed mechanim for the one-pot condensation of an aryl aldehyde, acetophenone, acetyl chloride, and acetonitrile in presence of silica-supported Preyssler nanoparticles (SPNPs).*

The mechanism involves acylation of the aldehyde with acetyl chloride and formation of α-chloroacetate as intermediate (I). Then, this intermediate reacts with acetonitrile to afford the corresponding α-acetoxy amide, which further combines with the enolate form of acetophenone to afford the imidate ester and, finally, the amide product. The protonation of the aldehyde activates the carbony group for nucleophillic attack. The acetyl group is displaced by alkyl/aryl nitrile followed by water addition leads to provide the product (Scheme 2).

Catalyst Rcovery

The separation of silica-supported Preyssler nanoparticles (SPNPs) catalyst from the reaction mixture becomes very easy at room temperature. Ease of recycling of the catalyst is one of the most advantages of our method. In our experiments, the reusability of the catalyst were examined by repetitive use of the catalyst. The wet catalyst was recycled and no appreciable change in activity was noticed after five cycles (Table 3). We have found that Preyssler catalyst can be reused several times without any appreciable loss of activity. The several time recoveries had only slightly decreased the catalytic activity, pointing to the stability and retention capability of this useful polyanion. Even after five runs for the reaction, the catalytic activity of silica-supported Preyssler nanoparticles (SPNPs), $H_{14}[NaP_5W_{30}O_{110}]/SiO_2$ was almost the same as that of the freshly used catalyst.

Table 3. *The Results of the synthesis of β-Acetamido-β-(phenyl)propiophenone (Table 1, entry 1) in the presence of recycled silica-supported Preyssler nanoparticles (SPNPs), $H_{14}[NaP_5W_{30}O_{110}]/SiO_2$.*

Entry	Run	Time (min)	[a]Yield (%)
1	1	6	90
2	2	6	90
3	3	6	89
4	4	6	88
5	5	6	88

[a]Isolated yield.

4. Conclusion

In summary, We have reported an efficient procedure for the synthesis of *β*-acetamido ketones using silica-supported Preyssler nanoparticles (SPNPs) as a reusable, eco-friendly and efficient heterogeneous catalyst. The advantages of this method are extremely mild reaction conditions, short reaction times, high yields, simple experimental and isolation procedures, and compliance with the green chemistry protocols. Catalytic activity of silica-supported Preyssler nanoparticles (SPNPs) as an easily producible and cheap material has been studied for the synthesis of *β*-acetamido ketones. A mild reaction condition, a simple experimental procedure, an easy work-up and improved yields of products are some major features of this reported method. It could also be recovered and reused for more than five reaction cycles without noticeable loss of reactivity.

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