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Application of Silica Gel-supported Polyphosphoric Acid (PPA/SiO_2) as a Reusable Solid Acid Catalyst for One-Pot Multi-Component Synthesis of 3,4,5-substituted furan-2(5H)-ones

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Abstract: 3,4,5-substituted furan-2(5H)-ones were One-Pot Multi-Component synthesized by the reaction of aldehydes, amines and dialkyl acetylenedicarboxylates using silica gel-supported polyphosphoric acid (PPA/SiO_2) at room temperature. This reaction provides a convenient and efficient synthetic method of furan-2(5H)-one derivatives, and also reusability of catalyst.

Keywords: PPA/SiO_2, 3,4,5-substituted furan-2(5H)-ones, aldehydes, amines, dialkyl acetylenedicarboxylates.

1. INTRODUCTION

Highly substituted furans play an important role in organic chemistry, not only as key structural units in many natural products, common subunits in pharmaceuticals [1-7] and flavors [8] but also as useful building blocks in synthetic chemistry [9-13]. They have also found utility as synthetic intermediates or synthons for numerous functional groups, inter alia, carboxylic acids, α-keto-esters, and aromatics [14]. For this reason, the efficient synthesis of multiple substituted furans continues to attract the interest of synthesis chemists [15-16]. Accordingly, many strategies have been developed for the preparation of furans [17-20]. The above-mentioned methodologies have certain limitations like multistep strategies, use of costly metal catalysts and highly volatile solvents.

Recently, a methodology has been developed for the synthesis of furan-2(5H)ones via a multicompunent reaction of aromatic amines, aldehydes and dialkyl acetylenedicarboxylate. Multi-component reactions (MCR) make possible the speedy synthesis of molecular libraries that have a high degree of structural diversity. Combinations of different starting materials can produce a variety of products with facility, a process that is of great value in the search for new drugs and chemical compounds [21].

Narayana et al. described the synthesis of 2(5H)-furanone by b-cyclodextrin [22]. Ramesh and Nagarajan reported the preparation of 2(5H)-furanone via a three-component reaction of a series of aldehydes (aromatic, heterocyclic), dialkylacetylenedicarboxylates and 9-alkyl-9H-carbazol-3-amines in the presence KOH [23]. Recently, we reported an acetic acid catalyzed synthesis of 2(5H)-furanone via a three-component reaction of a series of aldehydes (aromatic, heterocyclic), dialkylacetylenedicarboxylates and amines [24].

Recently, PPA/SiO_2 has been used as an efficient heterogeneous catalyst for many organic transformations. PPA/SiO_2 has some advantages including its low cost, ease of preparation, and ease of handling. In addition, the catalyst can be easily separated from the reaction mixture by simple filtration and is reusable. Due to our interest in the synthesis of of furan [27-33] and in continuation of our previous works on the synthesis of α-amino phosphonates using PPA/SiO_2 [34] herein we wish to report a novel and convenient method for the synthesis of 3,4,5-substituted furan-2(5H)-ones derivatives using PPA/SiO_2 as a reusable catalyst.

RESULTS AND DISCUSSION

We have introduced a one-pot three-component condensation reaction (MCR) that is one of the most useful methods for the synthesis of organic compounds in an optimistic time, room temperature, PPA/SiO_2 catalyst and involving only a single step. Moreover, as previously mentioned, reaction most often leads to product that can be easily separated and purified by simple filtering and washing out with a regular solvent. Substrates benzaldehyde, aniline, with dimethyl acetylenedicarboxylate were taken as model compounds to be investigated for the optimization of reaction conditions. The reaction of benzaldehyde, aniline,with dimethyl acetylenedicarboxylate was initially carried out in ethanol using different catalysts (Table **1**). As can be seen, PPA/SiO_2 was found to be most effective in catalyzing the reaction under room temperature.

To optimize the catalyst loading, the above model reaction was carried out under different amounts of PPA/SiO_2 (0.01, 0.02, 0.03, 0.05, 0.07, 0.1, 0.15 and 0.2 g). It was observed that 0.15 g loading of PPA/SiO_2 provided the maximum yield at the lowest time. Higher percentage loading of the catalyst did not affect the reaction time and amount of yield. The data in Table **2** outline the optimization amount of PPA/SiO_2 for for the synthesis of furan-2(5H)-ones .under room tempreture conditions.

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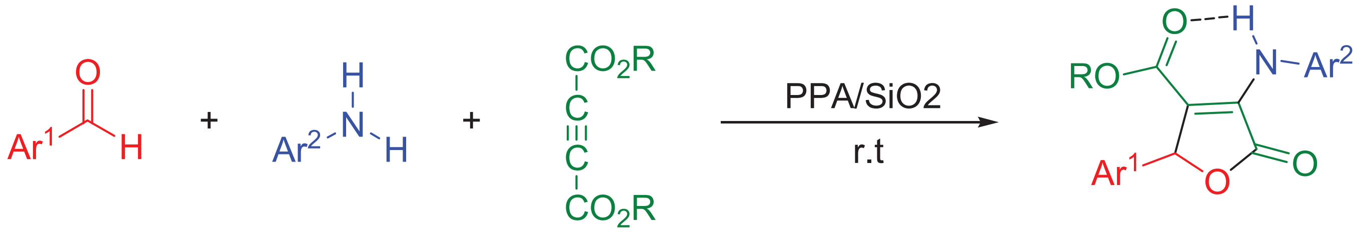


Scheme 1. Synthesis of furan-2(5H)-one derivatives.

Table 1. Optimization of Catalyst for the Synthesis of furan-2(5*H*)-ones at Room Temperature.

Entry	Catalyst	Time (h)	Yield(%)[a]
1	TiO_2	15	25
2	$Zn(SO_4)_2.7H_2O$	15	25
3	$Zr(NO_3)_4$	12	30
4	$ZrCl_4$	12	50
5	$HClO_4$-SiO_2	12	20
6	PPA/SiO_2	1	90

[a] Isolated yield.

Table 2. Optimization Amount of PPA/SiO$_2$ for the Synthesis of furan-2(5H)-ones. Under Room Tempreture Conditions.

Entry	PPA/SiO_2 (g)	Time (h)	Yield(%)[a]
1	0.01	10	85
2	0.02	9	85
3	0.03	5	85
4	0. 05	3.45	86
5	0.07	3.15	88
6	0.1	2	88
7	0.15	1	90
8	0.2	1	90

[a] Isolated yield.

Under the optimized reaction conditions, the generality of the reaction was investigated by using a variety of aldehydes, anilines and dialkyl acetylenedicarboxylate to produce furan-2(5H)-one derivatives. The results are summarized in Table **3**. These results indicate the effectiveness of electron-withdrawing and electron-donating groups on the time and yield of the reaction. Benzaldehydes with electron-withdrawing groups react with aniline better than electron-donating groups for generation of furan-2(5H)-ones in good to high yields. In our research work, aliphatic aldehyde and amine such as propanal and 1-buthyl amin did not practically did not tolerate the reaction.

The speculative proposed mechanism for the formation 4 is shown in Scheme **2**.

The structures of new compounds in Table **3** were deduced on the basis of IR, ^{1}H, and ^{13}C NMR spectroscopy, mass spectrometry, and elemental analysis. The mass spectrum of *Methyl 2,5-dihydro-5-oxo-4-(phenylamino)-2-p-tolylfuran-3-carboxylate* (Compound 4b) displayed the molecular ion peak at *m/z* = 323, which is consistent with the proposed structure. The ^{1}H NMR spectrum of this product, exhibited a singlet at $\delta = 2.29$ ppm for methyl protons, a singlet at $\delta = 3.77$ ppm for methyl protons of the carboxylate group and one sharp singlet arising from benzylic proton at $\delta = 5.73$ ppm. The aromatic protons of product were observed at $\delta = 7.07$ -7.49 ppm. A broad singlet for the NH group at $\delta = 8.9$ ppm indicated intramolecular hydrogen bond formation with the vicinal carbonyl group. The ^{13}C NMR spectrum of this product was showed 15 distinct resonances in agreement with the proposed structure. The IR spectrum indicated one sharp peak at 3220 cm^{-1} for NH within product.

The reusability of the catalyst is an important factor from economical and environmental point of views and has at-

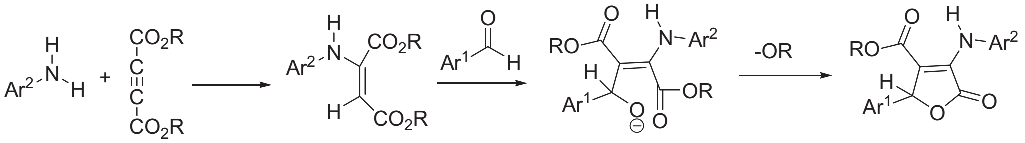
Table 3. Synthesis of furan-2(5*H*)-one Derivatives.

Entry	Ar^1	Ar^2	R	Products	Time (h)	Yield(%)^a	Ref^b
1	Ph	Ph	CH_3	4a	1	90	—
2	4-Me-C_6H_4	Ph	CH_3	4b	2	80	—
3	3-NO_2-C_6H_4	Ph	CH_3	4c	1.5	87	—
4	4-CN-C_6H_4	Ph	CH_3CH_2	4d	1	92	—
5	Ph	Ph	CH_3CH_2	4e	1	91	[22]
6	Ph	4-Me-C_6H_4	CH_3CH_2	4f	2	85	[22]
7	4-Me-C_6H_4	Ph	CH_3CH_2	4g	2	80	[22]
8	4-Cl-C_6H_4	Ph	CH_3CH_2	4h	3.5	81	[22]
9	4-OMe-C_6H_4	Ph	CH_3CH_2	4i	8	62	[22]
10	1- naphtyl	Ph	CH_3CH_2	4j	8	60	[22]
11	Ph	4-F-C_6H_4	CH_3	4k	7	67	[24]
12	Ph	4-Cl-C_6H_4	CH_3	4l	6	75	[24]
13	Ph	3-NO_2-C_6H_4	CH_3	4m	7	80	[24]
14	4-OMe-C_6H_4	Ph	CH_3	4n	9	61	[24]
15	4-NO_2-C_6H_4	Ph	CH_3	4o	4	90	[24]

[22,24] Yields refer to the pure isolated products

[a] The new compounds synthesized in this work.

derivatives



Scheme 2. The speculative proposed mechanism for the formation of furan-2(5H)-one derivatives.

Table 4. Investigation of Reusability of the Catalyst in the Reaction between Benzaldehyde, Aniline, and Dimethyl Acetylenedicarboxylate in the Presence of PPA-SiO_2 at Room Temperature.

Entry	Run	Yield of 4a (%)
1	1	90
2	2	88
3	3	86
4	4	81
5	5	79
6	6	77
7	7	77

Isolated yield.

tracted much attention in recent years. PPA/SiO_2 is easily recovered from the reaction mixture by filtration. The recovered PPA/SiO_2 is regenerated by washing and drying, and can be used for a subsequent reaction. The results of recycling are summarized in Table **4**. As can be seen from Table **3**, it was possible to recycle PPA/SiO_2 up to seven times and 4a was obtained in satisfactory yield (Table **4**, entry 7).

EXPERIMENTAL

Melting points and IR spectra of all compounds were measured on an Electrothermal 9100 apparatus and a JASCO FT/IR-460 Plus spectrometer respectively. The ^{1}H and ^{13}C spectra were obtained on Bruker DRX-250 and 400 Avance instruments with $CDCl_3$ as solvent. Elemental analyses were performed using a Heraeus CHN-O-Rapid analyzer. Mass spectra were recorded on an Agilent Technology (HP) spectrometer operating at an ionization potential of 70 eV. All reagents and solvents obtained from Fluka and Merck were used without further purification.

General procedures for synthesis of furan-2(5H)-one 4. The mixture of aldehyde (1 mmol), amine (1 mmol), dialkylacetylenedicarboxylate (1 mmol) and silica gel-supported polyphosphoric acid (0.15g) was stirred in ethanol as solvent at room temperature. After completion of the reaction (monitored by thin-layer chromatograohy, TLC) CH_2Cl_2 was added and the mixture stirred for 5 min. The catalyst was filtrated and the filtrate dichloromethane solution was concentrated. The obtained solid was washed with ethanol to obtain a pure product.

(Compound 4a). *Methyl 2,5-dihydro-5-oxo-2-phenyl-4-(phenylamino)furan-3-carboxylate* White solid: 0.284 g (92%); mp 195-196 °C; IR (KBr): 3260, 3208, 1702, 1661 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ :3.77 (s, 3H, OCH_3), 5.76 (s, 1H, benzylic), 7.13 (t, J = 7.3 Hz, 1H), 7.24-7.31 (m, 6H), 7.52 (d, J = 8 Hz, 2H), 8.90 (br, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.3 and 162.7 (ester CO), 156.3, 136.1, 134.9, 129.0, 128.7, 128.6, 127.4, 125.9, 122.3 and 112.8 (aromatic C), 61.6 (methoxy C), 52.1(benzylic C); MS m/z (%): 57(100), 97(75), 152(24), 213(51), 240(39), 250(33), 309(M$^+$, 44); 61.60, 52.10; MS (positive mode): Anal. calcd. for $C_{18}H_{15}NO_4$: C, 69.89; H, 4.89; N, 4.53. Found: C, 70.08; H, 4.97; N, 4.60

(Compound 4b). *Methyl 2,5-dihydro-5-oxo-4-(phenylamino)-2-p-tolylfuran-3-carboxylate*: White solid; 0.268 g (83%); mp 177-178 °C; IR (KBr): 3220, 1700, 1683 cm^{-1}.^{1}H NMR (400 MHz, $CDCl_3$) δ: 2.29 (s, 3H, CH_3), 3.78 (s, 3H, OCH_3), 5.73 (s, 1H, benzylic), 7.07-7.32 (m, 7H, aromatic), 7.49 (d, J = 8.4 Hz, 2H), 8.90 (br, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.3 and 162.8 (ester CO), 156.1, 138.3, 136.2, 131.8, 129.4, 128.9, 127.3, 125.8, 122.3 and 112.9 (aromatic C), 61.3 (methoxy C), 52.1 (benzylic C), 21.18 (methyl C). MS m/z (%): 93 (12), 135 (27), 144 (69), 145 (20), 172 (13), 203 (73), 236 (15), 264 (72), 294 (7), 323 (M$^+$, 100); Anal. calcd. for $C_{19}H_{17}NO_4$: C, 70.58; H, 5.30; N, 4.33. Found: C, 70.74; H, 5.39; N, 4.38.

(Compound 4c). *Methyl 2,5-dihydro-2-(3-nitrophenyl)-5-oxo-4-(phenylamino)furan-3-carboxylate*: White solid; 0.319 g (90%); mp 193-194 °C; IR (KBr): 3311, 3087, 2956, 1724, 1686, 1653, 1536 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 3.80 (s, 3H, OCH_3), 5.90 (s, 1H, benzylic), 7.17 (t, J = 7.2 Hz, 1H), 7.31-7.58 (m, 7H, aromatic), 8.13 (d, J = 8.4 Hz, 1H), 8.18(s, 1H, aromat), 8.88 (br, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 164.7 and 162.5 (ester CO), 156.4, 148.3, 137.6, 135.5, 133.2, 129.9, 129.3, 126.4, 123.8, 122.8, 122.3 and 112.0 (aromatic C), 60.6 (methoxy C), 52.3 (benzylic C). MS m/z (%): 93(5), 119 (11), 175 (46), 203 (28), 218 (30), 295 (38), 322 (10), 354 (M$^+$, 100); Anal. calcd. for $C_{18}H_{14}N_2O_6$: C, 61.02; H, 3.98; N, 7.91. Found: C, 61.19; H, 4.01; N, 7.94.

(Compound 4d). *Ethyl 2-(4-cyanophenyl)-2,5-dihydro-5-oxo-4-(phenylamino)furan-3-carboxylate*: White solid; 0.324 g (93%); mp 188-189 °C; IR (KBr): 3293, 2977, 2225, 1731, 1684, 1666, 1500 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.23 (t, J = 7.2 Hz, CH_3), 4.24 (q, J = 7.2 Hz, CH_2), 5.82 (s, 1H, benzylic), 7.17 (t, J = 7.2 Hz, 1H), 7.32-7.47 (m, 6H, aromatic), 7.59 (d, J = 8 , 2H), 9.0 (br, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 164.6 and 162.5 (ester CO), 156.8, 140.8, 135.7, 132.5, 129.2, 128.3, 126.3, 122.1, 118.1 and 112.6 (aromatic C), 112.2 (CN), 61.6 (ethoxy CH_2), 60.8 (benzylic C), 14.0 (ethoxy CH_3). MS m/z (%): 93 (17), 119 (9), 155 (70), 183 (29), 228 (13), 275 (59), 302 (14), 348 (M+, 100); Anal. calcd. for $C_{20}H_{16}N_2O_4$: C, 68.96; H, 4.63; N, 8.04. Found: C, 69.10; H, 4.69; N, 8.11.

CONFLICT OF INTEREST

The author(s) confirm that this article content has no conflict of interest.

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