Res Chem Intermed
DOI 10.1007/s11164-016-2476-0

Preparation of 3,4,5-substituted furan-2(5*H*)-ones using HY Zeolite nano-powder as an efficient catalyst

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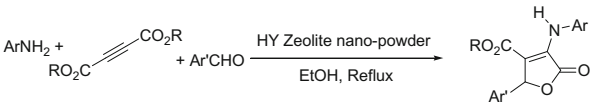
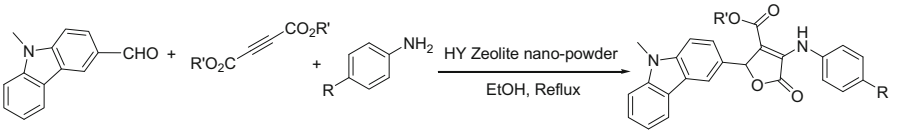
Received: 15 November 2015 / Accepted: 3 February 2016

Abstract Some 3,4,5-substituted furan-2(5*H*)-one derivatives, were obtained in good yields and from the corresponding three-component reaction of aromatic amines, aldehydes, and acetylenic esters by HY Zeolite nano-powder as an efficient catalyst.

Graphical Abstract





Keywords 3,4,5-Substituted furan-2(5*H*)-one · HY Zeolite nano-powder · Acetylenic esters · Butenolides · 9-Methyl-9*H*-carbazole-2-carbaldehyde

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Published online: 12 February 2016

Introduction

Functionalized furan-2(5*H*)-ones, generally called butenolides, constitute an important class of heterocyclic compounds, which are broadly found in many natural products and drugs [1–7]. Butenolides are of synthetic interest, since they show useful biological activities such as antimicrobial activity [8, 9], antifungal [10, 11], anti-inflammatory [12], anticancer [13, 14], and HIV-1 anti-viral activities [15, 16]. On the other hand, butenolides are versatile building blocks for natural product syntheses. Therefore, the synthesis of the butenolide scaffolds has gained great importance in organic synthesis, and many organic and medicinal chemists have devoted their efforts to the development of novel and efficient methods for the synthesis of furan-2(5*H*)-one derivatives.

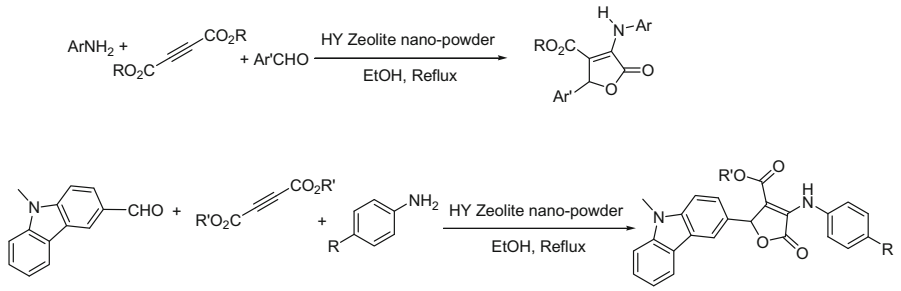
Among of the various methods established for the synthesis of different butenolides, the three-component reaction of aromatic amines, aldehydes, and acetylenic esters is considered as one of the most versatile and newly routes for the preparation of 3,4,5-substituted furan-2(5*H*)-one derivatives. Murthy et al. [17] first discovered this method and used β-cyclodextrin as an efficient catalyst. Similar catalytic systems later reported involving the use of $SnCl_2$ [18], ZnO nano-particles [19], $Al(HSO_4)_3$ [20] tetra-*n*-butylammonium bisulfate [21], and SnO [22]. Although these methods show good yields of substituted furan-2(5*H*)-ones, further development of efficient and simple strategies for the preparation of substituted furan-2(5*H*)-ones with cheap catalysts is highly desirable.

As a part of our ongoing program for multi-component reactions [23–26], herein we wish to report an HY Zeolite nano-powder catalyzed synthesis of 3,4,5-substituted furan-2(5*H*)-ones via a multi-component reaction of aromatic amines, aldehydes, and acetylenic esters. This procedure developed a new, efficient, and green approach for the preparation of substituted furan-2(5*H*)-ones in good yields (Scheme 1).

Experimental

Reagents and instrumentation

All reagents were purchased from Merck and Aldrich and used without further purification. Field emission scanning electron microscope (FE-SEM) images were



Scheme 1 Preparation of 3,4,5-substituted furan-2(5*H*)-one derivatives

obtained on a HITACHI S-4160. N_2 adsorption measurements of the catalyst were carried out using Micrometrics adsorption equipment (Quantachrome instrument, model Nova 2000, USA), N_2 (99.99 %) as the analysis gas and the catalyst samples were slowly heated to 120 °C for 3 h under nitrogen atmosphere. The total pore volume was obtained from the maximum amount of nitrogen gas adsorbed at partial pressure $P/P_0 = 0.999$. Dynamic light scattering (DLS) measurement was done using a Malvern Zetasizer Nano ZS (ZEN 3600) instrument. The NMR spectra were recorded on a Bruker Avance DPX 400 MHz instrument. The spectra were measured in DMSO-d_6 relative to TMS (0.00 ppm). Melting points were determined in open capillaries with a BUCHI 510 melting point apparatus. TLC was performed on silica gel Polygram SIL G/UV 254 plates.

General procedure

Aromatic amine (1 mmol), and acetylenic esters (1 mmol) in ethanol (5 mL), HY Zeolite nano-powder (0.05 g) was added to a mixture of aldehyde (1 mmol), and the mixture was stirred for appropriate time at reflux condition. Progress of the reaction was monitored by TLC. Upon completion, the solvent was concentrated and the reaction mixture was diluted with CH_2Cl_2 (20 mL). The solid catalyst was removed by the filter paper. After evaporation of the solvent (CH_2Cl_2), the residue was washed with diethyl ether to afford the pure product.

Selected data

*Methyl 2,5-dihydro-2-(9-methyl-9H-carbazol-3-yl)-5-oxo-4-(phenylamino)furan-3-carboxylate (**18a**)* ^{1}H-NMR (400 MHz, DMSO-d_6): 3.74 (s, 3H, OCH_3), 3.77 (s, 3H, N-CH_3), 6.41 (s, 1H), 7.08 (t, $J = 7.8$ Hz, 1H), 7.20–7.29 (m, 3H), 7.48–7.61 (m, 7H), 7.90 (s, 1H), 8.94 (s, 1H, NH) ppm; ^{13}C-NMR (100 MHz, DMSO-d_6): 30.1, 52.1, 62.1, 109.7, 110.8, 119.5, 120.9, 121.3, 121.8, 123.5, 123.6, 124.7, 125.2, 126.7, 129.2, 129.8, 131.1, 137.4, 141.2, 142.4, 143.1, 163.6, 166.1 ppm; Found: C, 72.97; H, 5.03; N, 6.90 $C_{25}H_{20}N_2O_4$; requires: C, 72.80; H, 4.89; N, 6.79 %.

*Ethyl 2,5-dihydro-2-(9-methyl-9H-carbazol-3-yl)-5-oxo-4-(phenylamino)furan-3-carboxylate (**19a**)* ^{1}H-NMR (400 MHz, DMSO-d_6): 1.22 (t, $J = 6.9$ Hz, 3H), 3.76 (s, 3H, N-CH_3), 4.16 (q, $J = 6.9$ Hz, 2H), 6.40 (s, 1H), 7.08 (t, $J = 7.8$ Hz, 1H), 7.21–7.29 (m, 3H), 7.48–7.61 (m, 7H), 7.90 (s, 1H), 8.91 (s, 1H, NH) ppm; ^{13}C-NMR (100 MHz, DMSO-d_6): 14.6, 30.0, 52.1, 62.2, 109.7, 110.7, 119.4, 120.9, 121.3, 121.8, 123.5, 123.6, 124.7, 125.2, 126.7, 129.2, 129.8, 131.1, 137.3, 141.1, 142.4, 143.1, 163.5, 166.3 ppm; Found: C, 73.35; H, 5.31; N, 6.70 $C_{26}H_{22}N_2O_4$; requires: C, 73.23; H, 5.20; N, 6.57 %.

*Methyl 4-(p-tolylamino)-2,5-dihydro-2-(9-methyl-9H-carbazol-3-yl)-5-oxofuran-3-carboxylate (**20a**)* ^{1}H-NMR (400 MHz, DMSO-d_6): 2.24 (s, 3H, CH_3), 3.75 (s, 3H, OCH_3), 3.76 (s, 3H, N-CH_3), 6.39 (s, 1H), 7.09–7.16 (m, 4H), 7.21–7.30 (m, 3H), 7.49–7.60 (m, 3H), 7.90 (s, 1H), 8.90 (s, 1H, NH) ppm; ^{13}C-NMR (100 MHz,

DMSO-d_6): 21.3, 29.9, 51.9, 62.3, 110.0, 110.7, 113.1, 119.5, 120.8, 121.2, 123.7, 123.9, 125.3, 126.7, 129.3, 129.9, 131.4, 132.5, 137.6, 141.0, 142.1, 142.7, 163.1, 166.1 ppm; Found: C, 73.33; H, 5.29; N, 6.68 $C_{26}H_{22}N_2O_4$; requires: C, 73.23; H, 5.20; N, 6.57 %.

*Ethyl 4-(p-tolylamino)-2,5-dihydro-2-(9-methyl-9H-carbazol-3-yl)-5-oxofuran-3-carboxylate (**21a**)* ^{1}H-NMR (400 MHz, DMSO-d_6): 1.27 (t, $J = 6.9$ Hz, 3H, CH_3), 2.23 (s, 3H, CH_3), 3.76 (s, 3H, N-CH_3), 4.16 (q, $J = 6.9$ Hz, 2H, CH_2), 6.38 (s, 1H), 7.09–7.16 (m, 4H), 7.21–7.33 (m, 3H), 7.49–7.60 (m, 3H), 7.79 (s, 1H), 8.89 (s, 1H, NH) ppm; ^{13}C-NMR (100 MHz, DMSO-d_6): 14.4, 21.3, 29.6, 60.7, 62.5, 110.1, 110.5, 113.0, 119.5, 120.7, 121.0, 123.7, 123.9, 125.2, 126.4, 129.6, 129.9, 131.3, 132.5, 137.5, 141.0, 142.4, 142.6, 163.2, 166.0 ppm; Found: C, 73.74; H, 5.57; N, 6.48 $C_{27}H_{24}N_2O_4$; requires: C, 73.62; H, 5.49; N, 6.36 %.

*Methyl 4-(4-chlorophenylamino)-2,5-dihydro-2-(9-methyl-9H-carbazol-3-yl)-5-oxofuran-3-carboxylate (**22a**)* ^{1}H-NMR (400 MHz, DMSO-d_6): 3.73 (s, 3H, OCH_3), 3.75 (s, 3H, N-CH_3), 6.40 (s, 1H), 7.20–7.30 (m, 3H), 7.44 (d, $J = 7.9$ Hz, 2H), 7.49–7.61 (m, 3H), 7.77 (d, $J = 7.9$ Hz, 2H), 7.80 (s, 1H) 8.98 (s, 1H, NH) ppm; ^{13}C-NMR (100 MHz, DMSO-d_6): 29.7, 52.3, 62.7, 110.2, 110.7, 119.5, 120.1, 120.7, 121.0, 123.3, 123.7, 125.2, 126.4, 129.4, 129.7, 130.0, 131.4, 137.4, 141.1, 142.0, 142.6, 163.5, 166.3 ppm; Found: C, 67.31; H, 4.44; N, 6.37 $C_{25}H_{19}ClN_2O_4$; requires: C, 67.19; H, 4.29; N, 6.27 %.

*Ethyl 4-(4-chlorophenylamino)-2,5-dihydro-2-(9-methyl-9H-carbazol-3-yl)-5-oxofuran-3-carboxylate (**23a**)* ^{1}H-NMR (400 MHz, DMSO-d_6): 1.26 (t, $J = 6.9$ Hz, 3H, CH_3), 3.76 (s, 3H, N-CH_3), 4.17 (q, $J = 6.9$ Hz, 2H, CH_2), 6.39 (s, 1H), 7.20–7.30 (m, 3H), 7.45 (d, $J = 7.9$ Hz, 2H), 7.49–7.61 (m, 3H), 7.77 (d, $J = 7.9$ Hz, 2H), 7.79 (s, 1H) 8.97 (s, 1H, NH) ppm; ^{13}C-NMR (100 MHz, DMSO-d_6): 14.5, 29.6, 60.7, 62.6, 110.0, 110.6, 119.6, 120.2, 120.8, 121.0, 123.2, 123.6, 125.3, 126.4, 129.3, 129.6, 130.1, 131.3, 137.4, 141.0, 142.1, 142.5, 163.6, 166.5 ppm; Found: C, 67.92; H, 4.74; N, 6.21 $C_{26}H_{21}ClN_2O_4$; requires: C, 67.75; H, 4.59; N, 6.08 %.

*Methyl 4-(4-methoxyphenylamino)-2,5-dihydro-2-(9-methyl-9H-carbazol-3-yl)-5-oxofuran-3-carboxylate (**24a**)* ^{1}H-NMR (400 MHz, DMSO-d_6): 3.72 (s, 3H, OCH_3), 3.74 (s, 3H, OCH_3), 3.76 (s, 3H, N-CH_3), 6.40 (s, 1H), 6.67 (d, $J = 8.0$ Hz, 2H), 7.10 (d, $J = 8.0$ Hz, 2H), 7.20–7.30 (m, 3H), 7.49–7.60 (m, 3H), 7.80 (s, 1H), 8.92 (s, 1H, NH) ppm; ^{13}C-NMR (100 MHz, DMSO-d_6): 29.6, 52.7, 55.9, 62.8, 109.9, 110.4, 114.7, 119.7, 120.4, 120.7, 121.0, 123.2, 123.5, 125.2, 126.5, 129.7, 131.4, 137.5, 141.0, 142.2, 142.6, 157.4, 163.7, 166.2 ppm; Found: C, 70.74; H, 5.16; N, 6.50 $C_{26}H_{22}N_2O_5$; requires: C, 70.58; H, 5.01; N, 6.33 %.

*Ethyl 4-(4-methoxyphenylamino)-2,5-dihydro-2-(9-methyl-9H-carbazol-3-yl)-5-oxofuran-3-carboxylate (**25a**)* ^{1}H-NMR (400 MHz, DMSO-d_6): 1.27 (t, $J = 6.9$ Hz, 3H, CH_3), 3.76 (s, 3H, N-CH_3), 3.78 (s, 3H, OCH_3), 4.17 (q, $J = 6.9$ Hz, 2H, CH_2), 6.40 (s, 1H), 6.68 (d, $J = 8.0$ Hz, 2H), 7.11 (d, $J = 8.0$ Hz, 2H), 7.21–7.29 (m, 2H), 7.34 (d, $J = 8.0$ Hz, 1H), 7.49–7.60 (m, 3H), 7.79 (s, 1H),

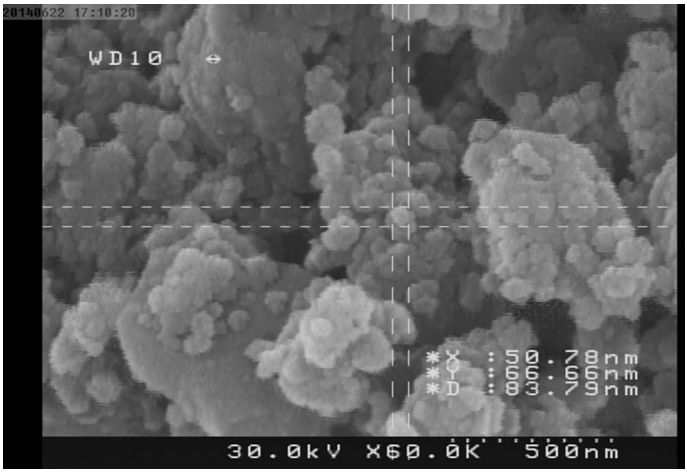


Fig. 1 FE-SEM micrograph of HY Zeolite nano-powder

Table 1 Specific surface area, total pore volume and average pore diameter of HY Zeolite nano-powder

Specific surface area (m^2/g)	Pore diameter (nm)	Pore volume (cc/g)
559	71.21	0.172

8.90 (s, 1H, NH) ppm; ^{13}C-NMR (100 MHz, DMSO-d_6): 14.7, 29.6, 55.7, 61.2, 62.2, 110.1, 110.6, 114.8, 119.9, 120.5, 120.8, 121.1, 123.3, 123.6, 125.3, 126.4, 129.5, 131.5, 137.6, 141.1, 142.3, 142.5, 157.0, 163.8, 166.1 ppm; Found: C, 71.21; H, 5.42; N, 6.25 $C_{27}H_{24}N_2O_5$; requires: C, 71.04; H, 5.30; N, 6.14 %.

Result and discussion

NaY zeolite nano-powder was prepared according to the procedures described by Wang and Yan [27]. The as prepared NaY zeolite nano-powder was converted to HY zeolite nano-powder following the procedure described by Ramli and Amin [28].

The morphological evolution of the HY Zeolite nano-powder was further studied using FE-SEM images of calcined powder and the results are revealed in Fig. 1. As shown in Fig. 1 the particles are relatively homogeneous in size and shape and are uniform spheres.

The specific surface area measurements of the HY Zeolite nano-powder were evacuated by BET method, and also the total pore volume and average pore diameter were determined at 200 °C for 300 min. The results are given in Table 1. These results show that the catalyst has a good specific surface area. The average diameter of the nanoparticles is comparable with those obtained from FE-SEM technique.

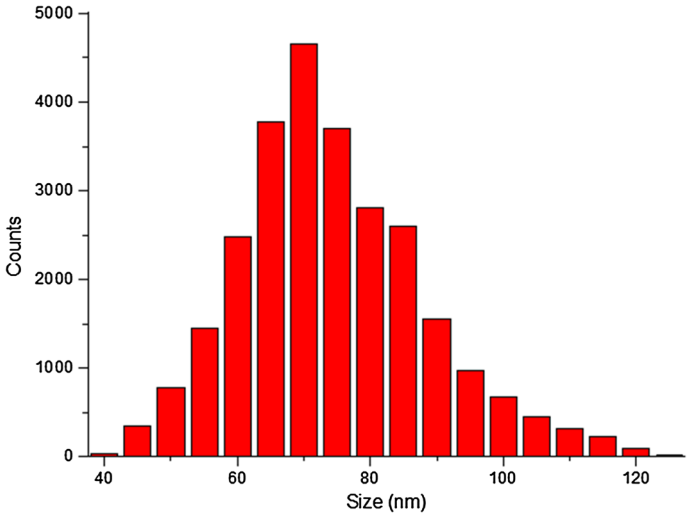


Fig. 2 Particle size distribution of HY Zeolite nano-powder

The dynamic light scattering (DLS) analysis was used to determine the particle size distribution of HY Zeolite nano-powder. Before analysis the sample was diluted in water (0.5 g L^{-1}) and sonicated for 2 h. Figure 2 shows the size distribution of nano-powder. Focusing on the results, the intensity-average diameter determined by DLS is about 73 nm.

Our initial aim was to develop an efficient one-pot procedure for the synthesis of 3,4,5-substituted furan-2(5*H*)-one derivatives through the reaction of aromatic amines, aldehydes, and acetylenic esters by employing HY Zeolite nano-powder. Accordingly, the transformation of 4-methylaniline, dimethylacetylenedicarboxylate, and benzaldehyde into methyl 4-(*p*-tolylamino)-2,5-dihydro-5-oxo-2-phenylfuran-3-carboxylate was investigated (Table 2). The reaction was carried out by the

Table 2 Optimization of the reaction condition in the synthesis of methyl 4-(*p*-tolylamino)-2,5-dihydro-5-oxo-2-phenylfuran-3-carboxylate

Entry	Catalyst (g)	*T* (°C)	Solvent (5 mL)	Time (h)	Yield (%)[a]
1	0.05	r.t.	*n*-Hexane	24	TRACE
2	0.05	r.t.	CH_2Cl_2	24	TRACE
3	0.05	r.t.	Et_2O	24	25
4	0.05	r.t.	EtOAc	24	65
5	0.05	r.t.	EtOH	15	76
6	0.05	r.t.	MeOH	15	75
7	0.05	r.t.	–	24	20
8	–	r.t.	EtOH	24	–
9	0.025	r.t.	EtOH	15	68
10	0.075	r.t.	EtOH	12	77
11	0.1	r.t.	EtOH	10	73

[a] Isolated yields

Table 3 Synthesis of 3,4,5-substituted furan-2(5*H*)-one derivatives (Scheme 1)

Product	Aldehyde	Amine	R	Time (h)	Yield (%)[a]	M.p. (°C) [lit M.p.][ref.]
1a	Benzaldehyde	4-Methylaniline	**Me**	15	76	176–178 [173–175][17]
2a	Benzaldehyde	4-Methylaniline	**Et**	15	80	181–183 [–][13]
3a	4-Methylbenzaldehyde	4-Methylaniline	**Me**	10	85	179–181 [–][16]
4a	4-Methylbenzaldehyde	4-Methylaniline	**Et**	12	90	189–191 [–][16]
5a	4-Chlorobenzaldehyde	4-Methylaniline	**Me**	15	76	203–205 [–][16]
6a	4-*tert*-Butylbenzaldehyde	4-Methylaniline	**Me**	10	89	211–213 [–][16]
7a	4-*tert*-Butylbenzaldehyde	4-Methylaniline	**Et**	12	85	216–218 [–][16]
8a	4-Methylbenzaldehyde	4-Chloroaniline	**Me**	15	85	174–176 [–][16]
9a	Benzaldehyde	4-Chloroaniline	**Me**	15	70	167–169 [–][16]
10a	4-Methylbenzaldehyde	4-Methoxyaniline	**Me**	10	88	186–188 [–][16]
11a	Benzaldehyde	Aniline	**Me**	15	80	197–199 [195–196][17]
12a	Benzaldehyde	Aniline	**Et**	15	74	201–203 [–][13]
13a	4-Methylbenzaldehyde	Aniline	**Me**	12	87	177–178 [–][16]
14a	4-Methylbenzaldehyde	Aniline	**Et**	15	84	180–182 [–][13]
15a	4-Chlorobenzaldehyde	Aniline	**Me**	15	77	150–152 [148–151][18]
16a	2-Chlorobenzaldehyde	Aniline	**Me**	20	70	274–276 [274–276][18]
17a	2,4-Dichlorobenzaldehyde	Aniline	**Me**	20	83	286–288 [–][16]
18a	9-Methyl-9H-carbazole-3-carbaldehyde	Aniline	**Me**	15	89	291–293 [new product]
19a	9-Methyl-9H-carbazole-3-carbaldehyde	Aniline	**Et**	17	84	283–285 [new product]
20a	9-Methyl-9H-carbazole-3-carbaldehyde	4-Methylaniline	**Me**	15	93	277–279 [new product]
21a	9-Methyl-9H-carbazole-3-carbaldehyde	4-Methylaniline	**Et**	15	87	285–287 [new product]
22a	9-Methyl-9H-carbazole-3-carbaldehyde	4-Chloroaniline	**Me**	20	79	>300 [new product]
23a	9-Methyl-9H-carbazole-3-carbaldehyde	4-Chloroaniline	**Et**	20	77	>300 [new product]
24a	9-Methyl-9H-carbazole-3-carbaldehyde	4-Methoxyaniline	**Me**	15	86	289–291 [new product]
25a	9-Methyl-9H-carbazole-3-carbaldehyde	4-Methoxyaniline	**Et**	15	80	290–292 [new product]

[a] Isolated yields. All known products have been reported previously in the literature and were characterized by comparison of NMR spectra with authentic samples [17–21]

using of different solvents (Table 2, Entries 1–6) or solvent-free condition (Table 2, Entry 7) at room temperature. The lower yield of product was achieved under solvent-free condition. It was found that the best results were obtained when 0.05 g of HY Zeolite nano-powder in EtOH as a solvent was employed (Table 2, Entry 5).

To find the optimal amount of HY Zeolite nano-powder, the reaction was carried out by varying amount of the catalyst (Table 2, Entries 9–11). Maximum yield was obtained when 0.05 g of catalyst was used (Table 2, Entry 5). A further increase in the amount of HY Zeolite nano-powder in mentioned reaction did not have any significant effect on the product yield. The results are summarized in Table 2.

Next, the scope and efficiency of these procedures were explored for the synthesis of a wide variety of substituted 3,4,5-substituted furan-2(5*H*)-ones (Scheme 1; Table 3).

Generally, the results were excellent in terms of yields and product purity. A series of aromatic aldehydes and amines were investigated (Table 3, products **1a–25a**). In all cases aromatic aldehydes containing electron-donating groups gave shorter time and higher yields than that with electron-withdrawing groups. The results indicated that in contrast with *para*-substituted substrates, the reaction was more sluggish when *ortho*-substituted substrates such as 2-chlorobenzaldehyde was used as the starting materials (Table 3, products **16a and 17a**). The probable reason for this phenomenon may be explained that the ortho-position has more steric hindrance effects on the reaction centers. Furthermore, when 4-chloroaniline was examined the target compounds were obtained in good yields, but in longer reaction times (Table 3, products **8a and 9a**). In contrast with 4-methylaniline or 4-methoxyaniline, 4-chloroaniline is a considerably less reactive nucleophile. The reaction of 9-methyl-9H-carbazole-3-carbaldehyde and different aromatic amines proceeds smoothly to obtain product **18a–25a** in good yields.

The work-up procedure is very clear-cut; that is, the products were isolated and purified by simple filtration and washing with diethyl ether. Our protocol has been used from HY Zeolite nano-powder during the reaction process, making it superior to the reactions that use hazardous liquid acidic catalysts.

Conclusion

In summary, an efficient protocol for the preparation of 3,4,5-substituted furan-2(5*H*)-one derivatives was described. The reactions were carried out under ambient conditions with short reaction times and produce the corresponding products in good yields. The present methodology offers several advantages such as good yields, simple procedure, shorter reaction times, and milder conditions and the products were purified *without* resorting to chromatography.

Acknowledgments We are thankful to the Najafabad Branch, Islamic Azad University research council for partial support of this research.

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