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SYNTHESIS OF CARBAMATOALKYL NAPHTHOLS USING Fe_3O_4@ZrO_2-SO_3H AS HIGHLY EFFICIENT AND MAGNETICALLY RECYCLABLE NANO-CATALYST

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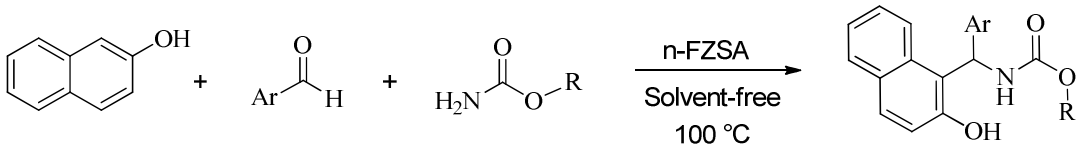
ABSTRACT
Nano-Fe_3O_4@ZrO_2-SO_3H (n-FZSA), as magneticcatalyst for the synthesis of carbamatoalkylnaphthols which prepared by a one-pot three component condensations of *β*-naphthol, aldehydes, and methyl/ethyl/benzyl carbamates in solvent-free condition has been reported. The results showed that n-FZSA showed exhibited catalytic activity towards the synthesis of carbamatoalkylnaphthols following high yield, simple work-up procedure, and avoids the use of harmful organic solvents. Furthermore, the catalyst is recyclable using an external magnet and could be reused at least four times without any discernible loss in its catalytic activity.

KEYWORDS: Carbamatoalkylnaphthols; Fast and green synthesis; Magnetic catalyst; Fe_3O_4@ZrO_2-SO_3H.

INTRODUCTION
Multi-component reactions (MCRs) have attracted much interest and are highly regarded in chemistry and discovery and synthesis of natural products because they are one-pot processes that bring together three or more components and show high atom economy and high selectivity[i-ii]. Organic compounds bearing 1,3-amido oxygenated functional groups are ubiquitous to a variety of biologically important natural products and potent drugs including a number of nucleoside antibiotics and HIV protease inhibitors such as ritonavir, lopinavir, and the hypotensive[iii]. In addition, 1-carbamatoalkyl-2-naphthols can convert to 1-aminomethyl-2-naphthols by a carbamate hydrolysis reaction. 1-aminomethyl-2-naphthols have been reported to show cardiovascular activity[iv]. Number of methods were known about the synthesis of carbamatoalkylnaphthols by three-component reaction of *β*-naphthol, an aldehyde, and a carbamate in the presence of a variety of catalysts such as Zwitterionicsalt[v],triethylbenzylammoniumchloride[vi], SiO_2-$NaHSO_4$[vii], $Mg(HSO_4)_2$[viii], Brønsted-acidic ionic liquids[ix], ($HClO_4$-C)[x], SiO_2-$HClO_4$[xi], $[MeC(OH)_2]^+ClO^{4-}$[xii], ionic liquid $[NMP]^+HSO_4^-$[xiii], P_2O_5/SiO_2[xiv], $Mg(OOCCF_3)_2$[xv], and $[Dsim]HSO_4$[xvi], and cerium ammonium nitrate (CAN)[xvii]. However, most of these methods suffer from drawbacks including low

yields, expensive reagents and catalysts. Therefore, the development of high yielding and less expensive catalytic method is desired.

The current presentation is the development of our earlier studies of reusable catalysts for the synthesis of organic compounds[xviii-xxxii], and as a result of global interest in the ongoing research towards the development of environmentally friendly methods for the synthesis of organic compounds, we reported herein facile and efficient green synthesis of carbamatoalkylnaphthols**4a-4h** with short reaction time by the three-component condensation of *β*-naphthol**1**, aromatic aldehydes **2**, and methyl/ethyl/benzyl carbamates**3**, using n-FZSA, as magnetic heterogeneous catalysts with high catalytic activity under solvent-free condition in high yield (Scheme 1).



1 **2** **3** **4a-4h**

Scheme 1.n-FZSA catalyzed synthesis of carbamatoalkylnaphthols.

EXPERIMENTAL SECTION

Chemicals and apparatus

All chemicals were available commercially and used without additional purification. The catalyst was synthesized according to the literature[xxxiii]. Melting points were recorded using a Stuart SMP3 melting point apparatus. The FT-IR spectra of the products were obtained with KBr disks, using a Tensor 27 Bruker spectrophotometer. The ^{1}H NMR (400 and 500 MHz) spectra were recorded using Bruker spectrometers.

General experimental procedure

To a mixture of *β*-naphthol (1 mmol), an aldehyde (1 mmol), and a carbamate (1.2 mmol), n-FZSA (0.08 g) was added. The reaction mixture was magnetically stirred on a preheated oil bath at 100 ^{o}C. After completion of the reaction (monitored by TLC), hot ethanol was added and stirred for 5 min. The catalyst was separated using an external magnet and washed with hot ethanol (5 ml). The reaction mixture was allowed to stand at room temperature. The precipitated solid was collected by filtration, and recrystallized from ethanol 96% to give desired compounds in high yields.

Methyl ((2-chlorophenyl)(2-hydroxynaphthalen-1-yl)methyl)carbamate (4a)

FT-IR (ν, cm^{-1}KBr disc): 3435, 3230, 1688, 1523, 1339, 1063, 826, 752, 709 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d$_6$): δ 9.89 (s, 1H, OH), 8.11 (d, 1H, *J* = 8.1 Hz, NH), 7.82 (d, 1H, *J* = 7.1 Hz, aromatic-H), 7.77 (dd, 2H, *J* = 17.2, 8.4 Hz, aromatic-H), 7.49 (d, 1H, *J* = 4.4 Hz, aromatic-
H), 7.42 (dd, 2H, *J* = 12.4, 6.6 Hz, aromatic-H), 7.25 (d, 3H, *J* = 13.6 Hz, aromatic-H), 7.13 (d, 1H, *J* = 8.4 Hz, aromatic-H), 6.89 (d, 1H, *J* = 7.6 Hz, CH), 3.54 (s, 3H, OCH$_3$).

Methyl ((2,4-dichlorophenyl)(2-hydroxynaphthalen-1-yl)methyl)carbamate (4b)

FT-IR (ν, cm^{-1}KBr disc): 3413, 3258, 1673, 1522, 1112, 854, 763, 723 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d$_6$): δ 9.99 (s, 1H, OH), 8.11 (d, 1H, *J* = 8.4 Hz, NH), 7.92 (d, 1H, J = 7.7 Hz, ArH), 7.76 (dd, 2H, *J* = 17.6, 8.5 Hz, ArH), 7.55-7.35 (m, 4H, aromatic-H), 7.31 (t, 1H, *J* = 7.4 Hz, aromatic-H), 7.14 (d, 1H, *J* = 8.5 Hz, aromatic-H), 6.91 (d, 1H, *J* = 8.1 Hz, CH), 3.58 (s, 3H, OCH$_3$).

Methyl ((2-hydroxynaphthalen-1-yl)(o-tolyl)methyl)carbamate (4c)

FT-IR (ν, cm^{-1}KBr disc): 3421, 3269, 1672, 1528, 1069, 859, 762, 723 cm^{-1}; ^{1}H NMR (500 MHz, DMSO-d$_6$): δ 9.94 (s, 1H, OH), 7.89 (d, 1H, *J* = 8.6 Hz, aromatic-H), 7.76 (d, 1H, *J* =

8.1Hz, aromatic-H), 7.73 (d, 1H, *J* = 8.6Hz, aromatic-H), 7.66 (br., 1H, NH), 7.32 (t, 1H, *J* = 7.5 Hz, aromatic-H), 7.23 (t, 1H, *J* = 7.5 Hz, aromatic-H), 7.20-7.08 (m, 4H, aromatic-H), 6.98 (t, 1H, *J* = 6.5 Hz, aromatic-H), 6.76 (d, 1H, *J* = 8.5 Hz,CH), 3.48 (s, 3H, OCH_3), 2.19 (s, 3H, CH_3).

Methyl ((2-hydroxynaphthalen-1-yl)(2-nitrophenyl)methyl)carbamate (4d)

FT-IR (ν, cm^{-1}KBr disc): 3436, 3272, 1671, 1530, 1058, 842, 757, 718 cm^{-1}; ^{1}H NMR (500 MHz, DMSO-d_6): δ 9.80 (s, 1H, OH), 7.89 (d, 2H, *J* = 8.2 Hz, aromatic-H), 7.84 (br., 1H, NH), 7.78 (d, 1H, *J* = 8.2 Hz, aromatic-H), 7.75 (t, 2H, *J* = 8.4 Hz, aromatic-H), 7.61 (t, 1H, *J* = 7.5 Hz, aromatic-H), 7.57 (d, 1H, *J* = 7.6 Hz, aromatic-H), 7.46 (t, 1H, *J* = 7.5 Hz, aromatic-H), 7.40 (t, 1H, *J* = 7.8 Hz, aromatic-H), 7.33-7.25 (m, 2H, aromatic-H), 7.12 (d, 1H, *J* = 8.6 Hz, CH), 3.52 (s, 3H, OCH_3);

Ethyl ((2-hydroxynaphthalen-1-yl)(2-nitrophenyl)methyl)carbamate (4e)

FT-IR (ν, cm^{-1}KBr disc): 3418, 3291, 1674, 1528, 1332, 1048, 822, 741, 688 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.76 (s, 1H, OH), 7.84 (d, 1H, *J* = 8.4 Hz, NH), 7.80-7.71 (m, 4H, aromatic-H), 7.60-7.55 (m, 2H, aromatic-H), 7.43 (dt, 2H, *J* = 7.2, 6.2 Hz, ArH), 7.30-7.25 (m, 2H, aromatic-H), 6.99 (d,1H, *J* = 8.5 Hz, CH), 4.10 (q, 2H, *J* = 7.2 Hz, CH_2), 1.26 (t, 3H, J = 7.0 Hz, CH_3).

Ethyl ((2-hydroxynaphthalen-1-yl)(4-nitrophenyl)methyl)carbamate (4f)

FT-IR (ν, cm^{-1}KBr disc): 3430, 3185, 1685, 1518, 1351, 1049, 821, 739, 708 cm^{-1}; 1H NMR (400 MHz, DMSO-d6): δ 10.26 (s, 1H, OH), 8.22 (d, 2H, *J* = 8.6 Hz, aromatic-H), 7.95-7.78 (m, 4H, aromatic-H and NH), 7.50 (d, 2H, *J* = 8.4 Hz, aromatic-H), 7.45 (t, 1H, *J* = 7.3 Hz, aromatic-H), 7.33 (t, 1H, *J* = 7.3 Hz, aromatic-H), 7.25 (d, 1H, *J* = 8.6 Hz, aromatic-H), 6.97 (d, 1H, *J* = 7.5 Hz, CH), 4.12 (q, 2H, *J* = 6.5 Hz, CH_2), 1.19 (t, 3H, *J* = 6.5 Hz, CH_3);

Benzyl ((2-chlorophenyl)(2-hydroxynaphthalen-1-yl)methyl)carbamate (4g)

FT-IR (ν, cm^{-1}KBr disc): 3426, 3162, 1713, 1521, 1321, 1047, 811, 741, 689 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.92 (s, 1H, OH), 8.11 (d, 2H, *J* = 7.6 Hz, aromatic-H), 7.80-7.76 (m, 2H, aromatic-H and NH), 7.50-7.30 (m, 10H, aromatic-H), 7.26 (d, 2H, *J* = 7.2 Hz, aromatic-H), 6.96 (d, 1H, *J* = 5.5 Hz, CH), 5.13 (d, 1H, *J* = 12.1 Hz, CH_2), 4.97 (d, 1H, *J* = 12.1 Hz, CH_2);

Benzyl ((4-chlorophenyl)(2-hydroxynaphthalen-1-yl)methyl)carbamate (4h)

FT-IR (ν, cm^{-1}KBr disc): 3402, 3200, 1681, 1515, 1321, 1042, 812, 746, 696 cm-1; 1H NMR (400 MHz, DMSO-d_6): δ 10.11 (s, 1H, OH), 7.91 (d, 1H, *J* = 8.1 Hz, NH), 7.80-7.75 (m, 3H, ArH), 7.40-7.25 (m, 12H, ArH), 6.84 (d, 1H, *J* = 8.2 Hz, CH), 5.13 (d, 1H, *J* = 12.4 Hz, CH_2), 4.98 (d, 1H, *J* = 12.4 Hz, CH_2).

RESULTS AND DISCUSSION

Characterization of the catalyst

For our investigations, the n-FZSA catalyst was prepared according to the literature procedure[xxxiii]. The n-FZSA was characterized by FT-IR, X-ray diffraction (XRD), thermal gravimetric (TG), and pH analysis. The FT-IR spectrums of nano-ZrO_2, nano-Fe_3O_4, nano-Fe_3O_4@ZrO_2, and nano-Fe_3O_4@ZrO_2-SO_3H are shown in Figure 1. In Figure 1(a), the characteristic vibrational bands of the Zr–O bond at 578 and 755 cm^{-1}, as well band belonging to the Zr–OH group at 1627 cm^{-1} [xxxiv]. The characteristic absorption band of Fe_3O_4 appears at 593 cm^{-1} in Figure 1(b). The spectrum of the Fe_3O_4@ZrO_2 nanoparticles (Figure 1(c)) shows a new absorption peak related to the characteristic absorption of zirconia at 624 cm^{-1} which confirmed the successful formation of Fe_3O_4@ZrO_2nanoparticles[xxxv]. The FT-IR spectrum of the n-FZSA catalyst prepared in the current study revealed new bonds at 825–1325 and 2500–3500 cm^{-1} corresponding to the characteristic absorption of the O=S=O, S-O and O-H stretching vibration of the sulfonic groups, respectively[xxxiii].

The XRD patterns of the prepared nano-Fe_3O_4, nano-Fe_3O_4@ZrO_2, and nano-Fe_3O_4@ZrO_2-SO_3H are presented in Figure 2. In Figure 2(a), the signals at the values of 2θ equal to 30.23 (220), 35.10 (311), 43.26 (400), 53.51 (422), 56.06 (511) and 63.11 (440) corresponds to cubic structure of $Fe_3O_4$and has good agreement with (JCPDS file PDF no. 65-3107)[xxxvi]. The XRD pattern of the nano-Fe_3O_4@ZrO_2 sample shows peaks at 31.02˚ and 36.23˚ belong to Fe_3O_4 which have shifted from 30.23˚ and 35.10˚, respectively. Besides the peaks for Fe_3O_4, two small nonmagnetic related peaks located in 50.21˚ and 60.52˚ are found which can be indexed to the diffraction of (112) and (211) planes of the standard data for ZrO_2 (JCPDS file no. 88-1007)[xxxvii]. The peaks position of nano-Fe_3O_4@ZrO_2-SO_3H unchanged during modification by chlorosulfonic acid shows that the crystalline structure of the core-shell nanomagnetic is essentially maintained after functionalization.

The TG curves of nano-Fe_3O_4@ZrO_2,and nano-Fe_3O_4@ZrO_2-SO_3H are shown in Figure 3. In the TG curve of nano-Fe_3O_4@ZrO_2 (Figure 3(a)) Two-stage decomposition is seencorresponding to different mass lose ranges. In the first region, a mass loss approximately 1% weight occurred below 120 °C is attributable to the loss of trapped water, organic solvents, and surface hydroxyl groups. A mass loss of approximately 1% weight occurred lower than 750 °C possibly related to the slow mass loss of dehydroxylation of ZrO_2. The TG curve of nano-Fe_3O_4@ZrO_2-SO_3H (Figure 3(a)) was divided into several regions relating to different mass lose ranges. The first region, which occurred below 136 °C, shown a mass loss 2% weight that is attributable to the evaporation of the H_2O, and organic solvents molecules adsorbed onto the surface and the release of the structural water resulted from the bonded hydroxyl groups. The mass loss of approximately 3% weight occurred between 145 and 360 °C is related to the slow mass loss of SO_3H groups. Finally, the mass loss of approximately 22 % weight occurred between 500 and 700 °C is related to the sudden loss of SO_3H groups. This mass loss confirms the coating of sulfonic acid groups on ZrO_2[xxxviii]. From the TG, it can be concluded that the prepared catalyst could be safely used in organic reactions at temperatures up to 140 °C.

The density of the SO_3H groups was measured using NaOH (0.1 N) as titrant by acid-base potentiometric titration. The amount of SO_3H in the catalyst was 4.83 mmol/g.

Evaluation of catalytic activity of n-FZSAin the synthesis of fluoroquinolone derivatives

The catalytic activity of n-FZSA was evaluated in the synthesis of carbamatoalkylnaphthols. For the beginning of this study, *β*-naphthol**1** (1 mmol), 4-nitrobenzaldehyde **2** (1 mmol), and ethyl carbamate**3** (1.2 mmol) was selected as a model reaction in different solvents such as H_2O, EtOH, MeOH, CH_2Cl_2, CH_3CN, and also solvent-free conditions at different temperature (80, 100, and 120 °C) and catalyst amount (0.02, 0.04, 0.06, 0.08, and 0.1). It was found that presence of the solvents were not so good for the reaction yields and times in all cases. However, the polar solvents were better than other non-polar and the yield of the productwas strongly affected by the catalyst amount and reaction temperature. On the other hand, the best results were obtained in solvent-free conditions. Low to moderate yields of the product was obtained in the absence of the catalyst at different temperature or in the presence of the catalyst at room temperature indicating that the catalyst and temperature are necessary for the reaction. The best amount of the catalyst and reaction temperature was obtained 0.08 g and 100 °C, respectively, whereas further increase in both catalyst amount and temperature did not improve the product yield and reaction time. Among the tested solvents and also solvent-free conditions in the presence of various amounts of the catalyst and temperature, the reaction was more facile and proceeded to give the highest yield (97%), and short reaction time (19 min), using 0.08 g of n-FZSA under solvent-free condition at 100 °C. All subsequent reactions were carried out in these optimized conditions.

According to these results, and in order to evaluate the generality of this model reaction, we extended the reaction of *β*-naphthol, and a carbamate, with a range of other various aldehydes under the optimized reaction conditions (Table 1). The n-FZSA efficiently catalyzed the reactions, giving the products 4a-4h in high yields over relatively short reaction times. Easy separation of obtained products from the catalyst makes this method useful for the synthesis of carbamatoalkylnaphthols. Purity checks with melting points, TLC and the ^{1}H NMR spectroscopic data reveal that only one product is formed in all cases and no undesirable side-products are observed. The structures of all known products 4a-4h were deduced from their ^{1}H NMR and FT-IR spectral data and a comparison of their melting points with those of authentic samples.

Table 1. Synthesis of carbamatoalkylnaphthols using n-FZSA[a]

Entry	Ar	R	Product	Time (min)	Yield (%)	m.p. (°C)	
						Found	Reported
1	2-ClC_6H_4	Me	**4a**	19	90	181-183	180-182[xiv]
2	2,4-$Cl_2C_6H_3$	Me	**4b**	21	92	193-195	194-196[ix]
3	2-MeC_6H_4	Me	**4c**	17	89	232-233	230-232[ix]
4	2-$O_2NC_6H_4$	Me	**4d**	18	93	240-242	241-242[ix]
5	2-$O_2NC_6H_4$	Et	**4e**	20	95	215-216	215-216[xvii]
6	4-$O_2NC_6H_4$	Et	**4f**	19	97	228-230	229-230[xvii]
7	2-ClC_6H_4	Bn	**4g**	20	91	161-163	163-165[vii]
8	4-ClC_6H_4	Bn	**4h**	17	92	180-182	179-181[xvii]

[a]**Reaction conditions:***β*-naphthol**1** (1 mmol), an aromatic aldehyde **2** (1 mmol), methyl/ethyl/benzyl carbamates**3** (1.2 mmol), n-FZSA (0.08 g), 100 °C, solvent-free.

We also used the model reaction under optimized reaction conditions to evaluate the reusability of the n-FZSA catalyst. After completion of the reaction, the catalyst was recovered as described in the experimental section. The separated catalyst was washed with hot ethanol and subsequently dried at 50 °C under vacuum for 1 h before being reused in a similar reaction. The catalyst could be used at least five times without significant reduction in its activity (97, 95, 94, 94, 93 % yields in first to fifth use, respectively) which clearly demonstrates the practical reusability of this catalyst. Furthermore, the FT-IR spectra of the fifth run recovered catalysts (Figure 1(e)) were almost identical to the spectrum of the fresh catalyst (Figure 1(d)), indicating that the structure of the catalyst was unchanged by the reaction.

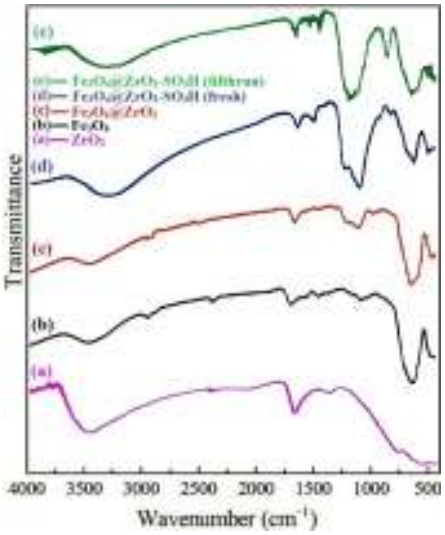


Figure 1. FT-IR spectra of nano-ZrO_2 (a) nano-Fe_3O_4 (b) nano-Fe_3O_4@ZrO_2 (c) nano-Fe_3O_4@ZrO_2-SO_3H (first run (d)) nano-Fe_3O_4@ZrO_2-SO_3H (fifth run (e))

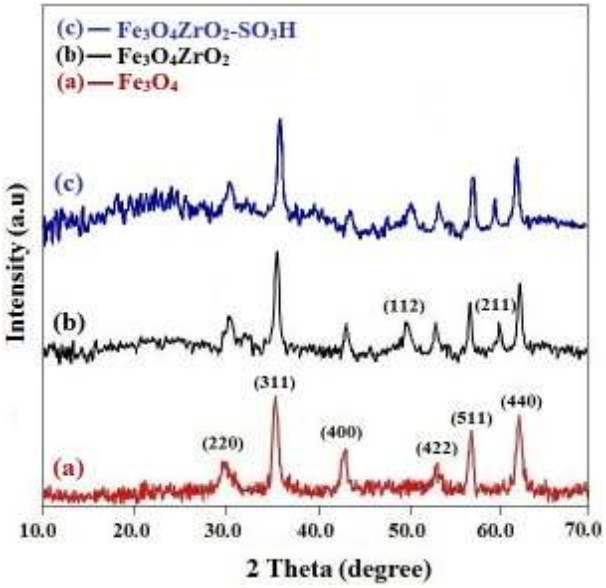


Figure 2. XRD patterns of nano-Fe_3O_4 (a) nano-Fe_3O_4@ZrO_2 (b) nano-Fe_3O_4@ZrO_2-SO_3H (c)

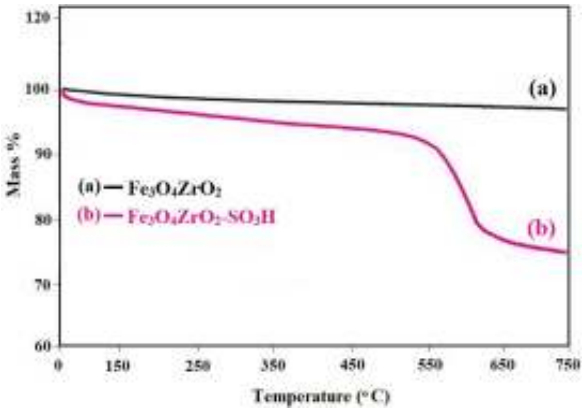
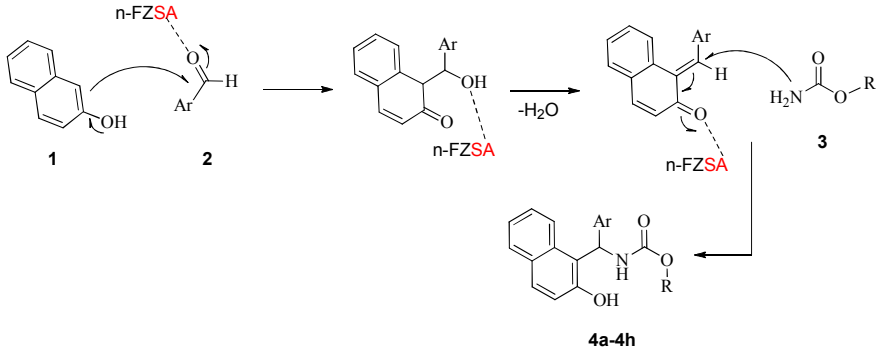


Figure 3. Thermal gravimetric (TG) analysis of the n-FZSA

In the reaction mechanism, the n-FZSAcould acts as Brönsted acid related to the $–SO_3H$ groups and therefore promote the necessary reactions. The catalyst would play a significant role in increasing the electrophilic character of the electrophiles in the reaction (Scheme 2).



Scheme 2. Plausible mechanism for the n-FZSA catalyzed formation of carbamatoalkylnaphthols.

CONCLUSIONS

In this paper we developed the synthesis of carbamatoalkylnaphthols in the presence of n-FZSA as a highly effective heterogeneous catalyst under solvent-free condition. This method provided these products in high yields over short reaction time, following a facile work-up process. The catalyst is inexpensive and easily obtained, stable and storable. Also, easy magnetic separation makes this catalyst attractive in view of green chemistry and catalysis science.

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