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ORIGINAL PAPER

# Solvent-free synthesis of amidoalkyl naphthols in the presence of MWCNTs@$SiO_2$/$SO_3H$ as effective solid acid catalyst

**Masoumeh Ahmadi[1] · Leila Moradi[1] · Masoud Sadeghzadeh[2]**

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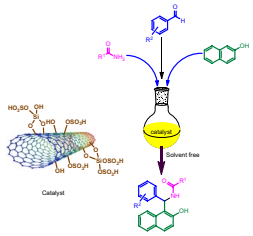
## Abstract

Multiwalled carbon nanotubes (MWCNTs) were modified with sulfonic acid groups through a new method. In the first step, MWCNTs' surfaces were hydroxylated using $KMNO_4$ as oxidating agent and a surfactant (TPABr). Second, $SiO_2$-coated MWCNTs were prepared through the reaction of hydroxylated CNTs with tetraethyl orthosilicate, and in last step, MWCNTs@$SiO_2$/$SO_3H$ was prepared using the reaction of MWCNTs@$SiO_2$ with $ClSO_3H$. Obtained catalyst was used as efficient solid acid catalyst for one-pot three-component condensation reaction of 2-naphthol, benzaldehyde, and amide derivatives to afford the corresponding amidoalkyl naphthols. The reaction was performed under solvent-free conditions and products were obtained in high to excellent yields (80–98%). Prepared solid acid catalyst was characterized using FT-IR, BET, TGA, SEM, and EDX techniques. Presented methodology has several advantages such as simple procedure, excellent yields of products, effective reusable solid acid catalyst, and eco-friendly reaction conditions.

### Graphical abstract



**Keywords** MWCNTs@$SiO_2$/$SO_3H$ · Amidoalkyl naphthols · Multicomponent reaction · Solvent-free · Green protocol · Solid acid catalyst

## Introduction

Multicomponent reactions (MCRs) can be considered as an important method for the synthesis of complex and diverse organic compounds through the formation of carbon–carbon and carbon–heteroatom bonds [1–4].

*Ortho*-quinone methides (*o*-QMs) are interesting molecules due to their toxicological properties against cancerous cells and also proposed intermediary in the formation of many biologically important polymers [5] and antitumor agents [6]. Tandem reaction involves the in situ preparation of *o*-QMs, and then, reaction with amides is one of the preparation methods of amidoalkyl naphthols [7, 8].

✉ Leila Moradi
L_moradi@kashanu.ac.ir

[1] Department of Organic Chemistry, Faculty of Chemistry, University of Kashan, P.O. Box 8731753153, Kashan, Islamic Republic of Iran

[2] School of Chemistry, College of Science, University of Tehran, PO Box 141556455, Tehran, Islamic Republic of Iran

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A variety of natural products containing 1,3-amino-oxygenated functional groups exist in biologically significant natural products and effective drugs including antitumor [9, 10], antibiotics [11], antianginal [12–14], antimalarial [15, 16], and HIV protease inhibitors [17–19]. These compounds have attracted much attention and considerable interests toward medical application, since they can be converted into hypotensive and bradycardiac activities by amide hydrolysis reactions [20, 21]. Various catalysts have been applied for the synthesis of amidoalkyl naphthols, such as montmorillonite K10 [22], *p*-TSA [7], $K_5CoW_{12}O_{40}{\cdot}3H_2O$ [23], $HClO_4$–$SiO_2$ [24], $Fe(HSO_4)_3$ [25], $FeCl_3$–$SiO_2$ [26], BrØnsted acidic ionic liquid [27], imidazolium salt [28], $P_2O_5$ [14], cyanuric chloride [29], thiamine hydrochloride [30], trityl chloride [31], [FemSILP] L-prolinate [32], silica sulfuric acid [33, 34], 1-hexanesulfonic acid sodium salt [35], and zwitterionic-type molten salt [36]. The efficiency of some reported procedures comes down with long reaction times [7, 37], low yields of products [22, 38], high temperatures [7, 22, 37, 38], use of toxic reagents, and tedious workup procedures [7, 39, 40]. Therefore, development of catalytic systems for the synthesis of 1-amidoalkyl naphthols is still one of the main interest fields for researchers.

Sulfuric acid is an essential and efficient catalyst for the production of industrial important chemicals. Large amounts of sulfuric acid are consumed annually as homogeneous and unrecyclable catalyst which need to costly and ineffective separation processes [41]. Application of reusable solid acids in organic reactions is often considered to follow the green chemistry principle that catalyzed processes must consume a minimum of energy and reagents or auxiliaries and minimize waste [42].

Since the discovery of carbon nanotubes (CNTs) in 1991 by Iijima [43], CNTs have an essential role in various fields of science and technology. Because of their supernatural and unique properties, CNTs have attracted considerable attentions in wide fields of industry and academic society.

To overcome the agglomeration of CNTs resulted from strong van der Waals force between the nano tubes structures, chemical modification of CNT surfaces is thought to be an effective way. Functionalization of CNT surfaces with organic, organometallic, and polymer matrixes improved the application and superior properties of these materials in various fields of industry, catalyst, drug delivery, and so on [44–50].

In continuation of our studies to develop the mild and facile procedures for functionalization of MWCNTs and their applications as heterogeneous catalyst [51, 52], herein, we report an efficient and rapid method for the synthesis of 1-amidoalkyl naphthols using MWCNTs@$SiO_2$/$SO_3H$ as a mild, reusable, and effective solid acid catalyst under solvent-free conditions (Scheme 1).

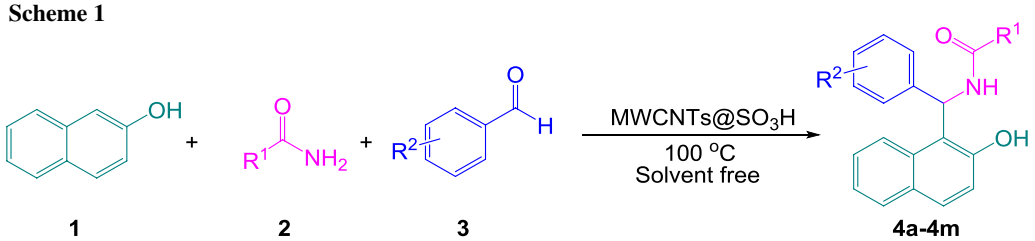
## Results and discussion

Preparation of catalyst was started from the attachment of hydroxy groups on MWCNTs surfaces using $KMnO_4$ with the aid of tetrapropylammonium bromide (TPABr). In the second step, silica-coated MWCNT was successfully prepared by deposition of silica onto nanoparticles surface using tetraethyl orthosilicate (TEOS). Finally, MWCNTs@$SiO_2$ was used as support for the immobilization of $SO_3H$ groups by simple mixing of silica-coated MWCNTs and chlorosulfonic acid in $CH_2Cl_2$ (Scheme 2).

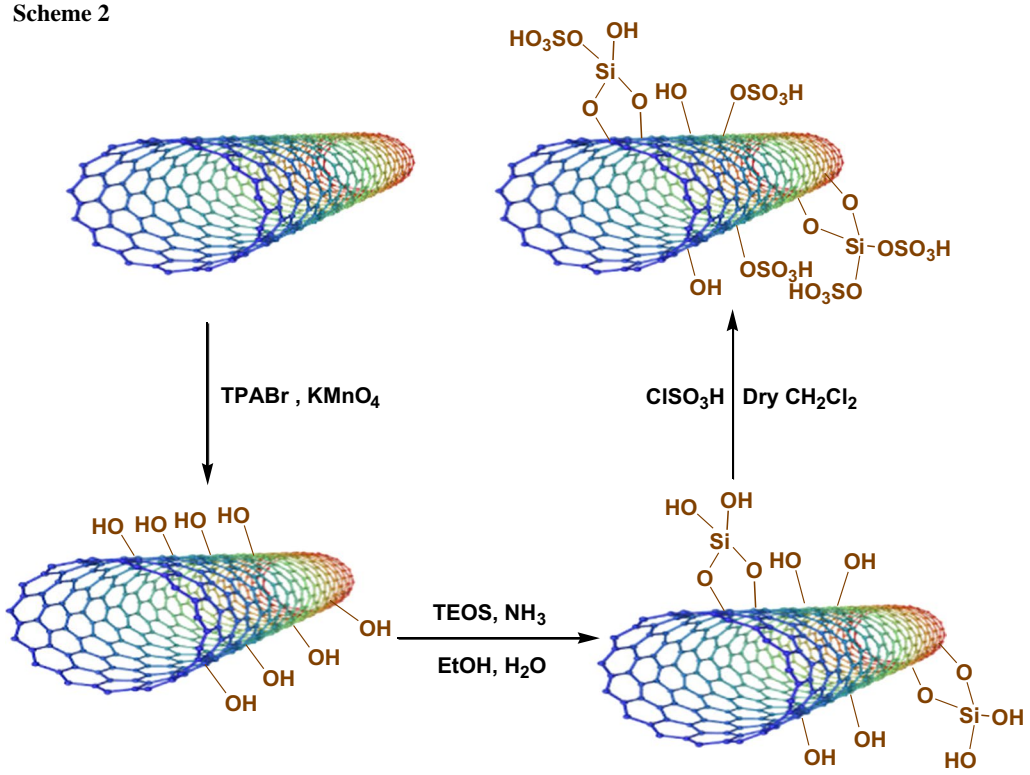
Figure 1 shows FT-IR spectra of raw and modified MWCNTs. For raw sample, the weak band around 1630 $cm^{-1}$ is from the graphitic structure of MWCNT. Hydroxylated MWCNT shows O–H-stretching vibration at 3428 $cm^{-1}$ also, and C–O-stretching vibration appeared between 1000 and 1100 $cm^{-1}$ (Fig. 1b). In FT-IR spectrum of MWCNTs@$SiO_2$ and catalyst, peaks at 455, 576, and 884 $cm^{-1}$ are from the asymmetric stretching, symmetric stretching, and bending vibration of Si–O–Si groups, respectively. The strong and wide peak at about 3383 $cm^{-1}$ demonstrates that there are Si–OH groups in the MWCNTs@$SiO_2$ and catalyst structure (Fig. 1c, d). The presence of sulfonyl groups in catalyst is authenticated by 1172 and 1071 $cm^{-1}$ bands that were covered by a stronger absorption of Si–C and Si–O bonds. In addition, the increase in the intensities of the band at 2800–3700 $cm^{-1}$ suggests that there are more OH groups on the catalyst surfaces after the sulfonation. All of these observations demonstrate that the –$SO_3H$ groups have chemically attached to the surfaces of MWCNTs.

Figure 2 shows SEM images of the raw and sulfonated MWCNTs. It can be seen that the pristine MWCNTs are tangled together (Fig. 2a). Most of the MWCNTs are not

**Scheme 1**



**Scheme 2**



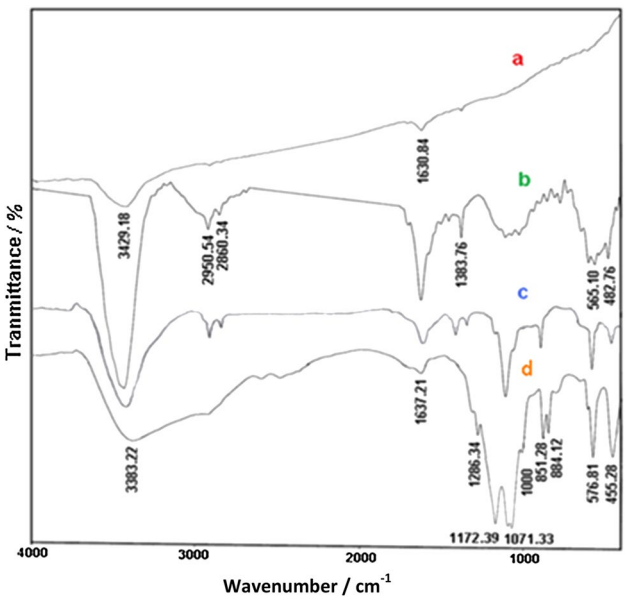




**Fig. 1** FT-IR spectrum of **a** raw MWCNTs, **b** MWCNTs-OH, **c** MWCNTs@$SiO_2$, and **d** MWCNTs@$SiO_2$/$SO_3H$

straight due to some defects and show a few local kinks and bends. SEM image of catalyst (Fig. 2b) indicated that CNT surfaces were functionalized and no damage occurred on CNT structures after chemical treatments.

The components of the MWCNT@$SO_3H$ were analyzed by energy-dispersive X-ray spectroscopy (EDX) technique. Figure 3 shows the presence of C, O, Si, and S atoms in catalyst structure and confirms the successful preparation of solid acid catalyst.

Thermogravimetric analysis (TGA) provided further evidence for evaluation and characterization of catalyst. TGA measurements carried out in air at a rate of 5 °C/min from 50 to 800 °C. As can be seen in Fig. 4a, there is no significant weight loss in raw MWCNTs between 50 and 800 °C. TGA graph of the MWCNTs@$SiO_2$/$SO_3H$ was divided into several regions corresponding to different mass loss ranges (Fig. 4b). The first region, which occurred below 200 °C, shows a mass loss that was from the removing of adsorbed water from the catalyst. The weight loss at higher temperature (250–600 °C) could be mainly attributed to decomposition of –$SO_3H$ and –SiOH groups. Obtained results show that catalyst is contains about 30% of functional groups and demonstrated that attachment of $SO_3H$ groups on CNT surfaces was occurred successfully through the mentioned process.

Results from BET analysis (Table 1) indicate that functionalization process increases specific surface area of MWCNTs. In fact, functionalization treatment opens up the tube end caps and generate defects on the sidewall of nanotubes [17, 18], as well as debundling of CNTs. As a result, surface area has been increased after modification

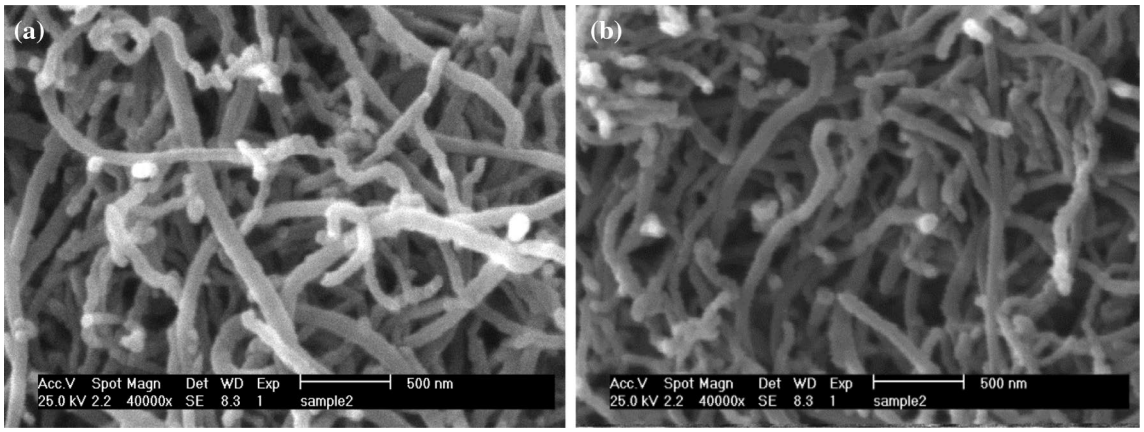



**Fig. 2** SEM image of **a** raw MWCNTs and **b** MWCNTs@$SiO_2/SO_3H$

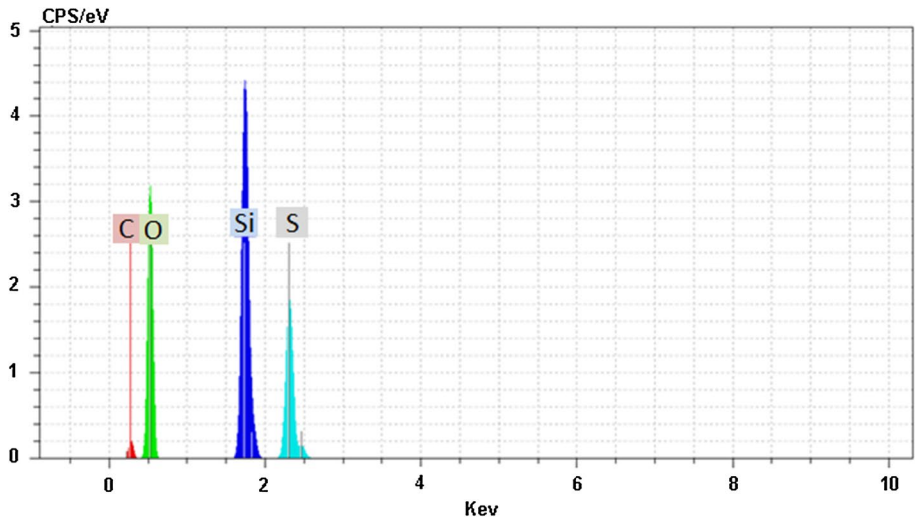



**Fig. 3** EDX of MWCNTs@$SiO_2/SO_3H$

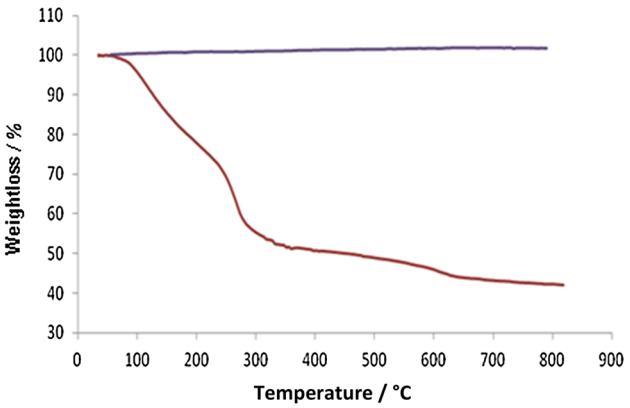



**Fig. 4** TGA curve of **a** raw MWCNTs, and **b** MWCNTs@$SiO_2/SO_3H$

**Table 1** BET surface area of pristine and functionalized CNTs

| Entry | Sample | Surface area/$m^2/g$ |
|---|---|---|
| 1 | Raw MWCNT | 42.74 |
| 2 | MWCNTs@$SiO_2/SO_3H$ | 74.42 |

and accessed into the CNT surfaces, and defects and cavity of the nanotubes can be achieved.

The XRD profiles of raw and functionalized MWCNTs are presented in Fig. 5. It is clear that, the XRD pattern of sulfonated MWCNTs is as the same of raw sample with $2\theta$ of 25.8° and 43.42° correspond to 002 and 01 reflection of polyaromatic structure of CNTs and interlayer spacing between adjacent graphite layers. XRD profiles are in good agreement with the reported values [53]. It is clear that modified CNTs have the same interplanar spacing and cylinder wall structure after the sulfonation process. In

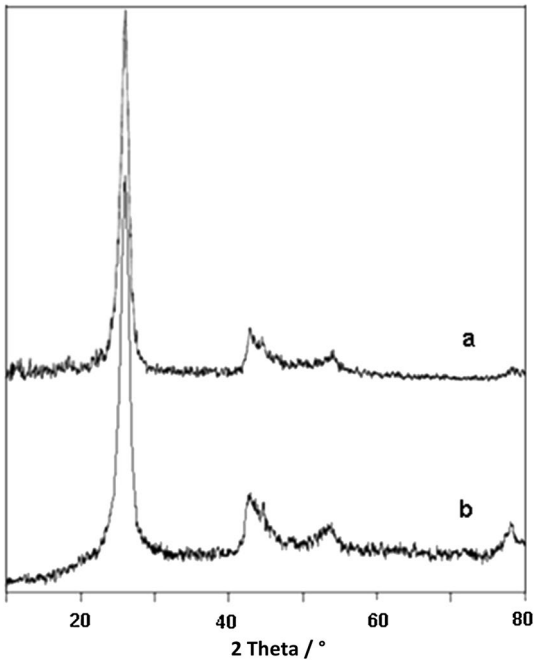



**Fig. 5** XRD pattern of **a** raw and **b** MWCNTs@$SiO_2$/$SO_3H$

**Table 2** Optimization of the catalyst amount and the reaction temperature for the preparation of amidoalkyl naphthols

| Entry | Catalyst/mg | Temperature/°C | Yield[a]/% |
|---|---|---|---|
| 1 | 5 | 100 | 78 |
| 2 | 8 | 100 | 82 |
| 3 | 10 | 100 | 85 |
| 4 | 13 | 100 | 90 |
| 5 | 15 | 100 | 90 |
| 6 | 18 | 100 | 90 |
| 7 | 13 | 90 | 86 |
| 8 | 13 | 110 | 90 |
| 9 | – | 100 | – |

Reaction conditions: benzaldehyde (1 mmol), 2-naphthol (1 mmol), and benzamide (1.2 mmol); time 15 min

[a]Isolated yield

fact, CNT structure was stable without any changes after the treatment (Fig. 5).

After the characterization of the solid acid catalyst, the catalyst activity of prepared solid acid catalyst has been tested in the preparation of amidoalkyl naphthol derivatives. In a typical experimental manner, a mixture of aldehyde (1 mmol), β-naphthol (1 mmol), benzamide (1.2 mmol), and CNT@$SiO_2$/$SO_3H$ (13 mg) as a model study was taken in round-bottom flask and stirred for a certain period of time as required to complete the reaction (indicated by TLC) at 100°C. As shown in Table 1, the best result was obtained by carrying out the reaction using 13 mg of MWCNTs@$SiO_2$/$SO_3H$ at 100 °C under solvent-free conditions (Table 2, entry 4). Furthermore, no reaction was occurred when the mixture was heated to 100 °C for 8 h in the absence of catalyst (Table 2, entry 9).

Using the optimized reaction conditions, the reaction was extended to substituted aromatic aldehydes, which was reacted with benzamide (or acetamide) and *β*-naphthol. As evident from the results (Table 3), it was shown that aromatic aldehydes with electron-withdrawing groups reacted faster (with higher yields) than those with electron-releasing groups and also sterically hindered aromatic aldehydes required longer reaction times (entry 3). Obtained results show the high activity and efficiency of solid acid catalyst.

In a plausible mechanism (Scheme 3) which is supported by the literature [27], aromatic aldehyde is activated by acidic group of catalyst to produce **I**. Then, 2-naphthol attacks to the carbonyl group of the activated aldehyde and gives intermediate **II**. Then, by removing $H_2O$ from **II**, *ortho*-quinone methide (*o*-QM, **III**) was prepared. Again, MWCNTs@$SiO_2$/$SO_3H$ activates intermediate **III** as a Michael acceptor. Afterward, Michael addition of amide to intermediate **IV** affords the expected amidoalkyl naphthol. According to proposed mechanism, the aromatic aldehydes with electron-withdrawing groups reacted faster than those having electron-donating groups.

A comparison between the efficacy of MWCNTs@$SiO_2$/$SO_3H$ catalyst for the preparation of *N*-[phenyl-(2-hydroxynapthalen-1-yl)-methyl]benzamide (**4a**) with some of those reported in the literatures is presented in Table 4. Although all of reaction conditions have considerable advantageous, but as can be seen, MWCNTs@$SiO_2$/$SO_3H$ is one of the high-efficient catalysts with respect to reaction times and yield of products. On the other hand, the advantageous of prepared catalyst in compared to zinc benzenesulfonate (with shorter time and higher yield, entry 14) is that MWCNTs@$SiO_2$/$SO_3H$ is a heterogeneous reusable catalyst and its recycling is simpler than ZBS. In fact, catalysts based on CNTs provided high-efficient, selective, and green conditions for organic synthesis reactions [49, 51, 52]. Furthermore, in presented method, low amount of catalyst (13 mg) with high yield of products provided a sufficient method for preparation of amidoalkyl naphthols and also can be used for other acid catalyzed organic reactions.

We observed that the catalyst can be easily recovered by simple filtration after the reaction. The reusability of the catalyst was studied by the reaction of benzaldehyde, 2-naphthol, and benzamide. After completion of reaction, the catalyst was separated, washed with acetone and dried at 80 °C, and then reused for the next cycle. As shown in Fig. 6, the yield of the product decreased from the first run to fifth run (90–79%). Obtained results show the good efficiency of prepared solid acid catalyst. On the other hand,

**Table 3** Solvent-free synthesis of amidoalkyl naphthols in the presence of MWCNTs@$SiO_2$/$SO_3H$

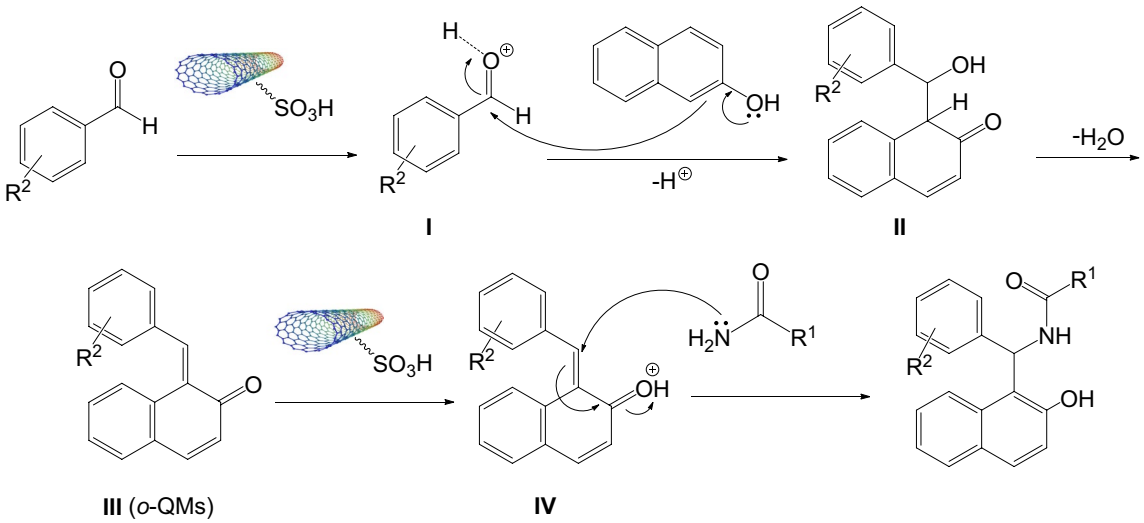
| Entry | Product | $R^1$ | $R^2$ | Time/min | Yield[a]/% | M.p./°C [Refs.] | TON[c] |
|---|---|---|---|---|---|---|---|
| 1 | **4a** | $C_6H_5$ | H | 15 | 90 | 244–246 [54] | 11,110 |
| 2 | **4b** | $C_6H_5$ | 4-Cl | 10 | 95 | 186–188 [55] | 11,730 |
| 3 | **4c** | $C_6H_5$ | 2-Cl | 10 | 93 | 266–269 [56] | 11,480 |
| 4 | **4d** | $C_6H_5$ | 4-$NO_2$ | 6 | 97 | 238–240 [57] | 11,970 |
| 5 | **4e** | $C_6H_5$ | 3-$NO_2$ | 6 | 98 | 234–236 [57] | 12,100 |
| 6 | **4f** | $C_6H_5$ | 4-$OCH_3$ | 25 | 84 | 208–210 [14] | 10,370 |
| 7 | **4g** | $C_6H_5$ | $SC_4H_3$[b] | 12 | 86 | 220–224 [58] | 10,610 |
| 8 | **4h** | $C_6H_5$ | 4-$(CH_3)_2N$ | 26 | 80 | 220–222 [15] | 9880 |
| 9 | **4i** | $C_6H_5$ | 4-CHO | 20 | 84 | 259–262 [59] | 10,370 |
| 10 | **4j** | $C_6H_5$ | 4-$CH_3$ | 20 | 85 | 190–191 [27] | 10,490 |
| 11 | **4k** | $CH_3$ | H | 15 | 93 | 240–243 [8] | 11,480 |
| 12 | **4l** | $CH_3$ | 4-$OCH_3$ | 22 | 88 | 184–286 [8] | 10,860 |
| 13 | **4m** | $CH_3$ | 4-$NO_2$ | 7 | 98 | 249–251 [8] | 12,100 |

[a]Isolated yield

[b]2-thiophenecarbaldehyde was used as aldehyde

[c]Moles of desired product/moles of catalyst

**Scheme 3**




because of turn over number (TON = moles of desired product/moles of catalyst) which is more crucial than good recycling ability of a catalyst [64], TON values were calculated for every recycling process. The TON value was in range of 11,110–9750, which demonstrated the good activity and stability of heterogeneous acidic catalyst.

## Conclusion

In conclusion, we have reported a new method for highly sulfonated MWCNTs as efficient solid acid catalyst for novel and eco-friendly synthesis of amidoalkyl naphthols under solvent-free conditions. Green and mild reaction conditions, short reaction times, easy workup, and inexpensive and recoverable of the catalyst make the procedure an attractive route to the existing methods for the synthesis of amidoalkyl naphthols.

## Experimental

Multiwalled carbon nanotubes were prepared from Shenzhen Nanotechnology Co., Ltd. (China). The Purity of the CNTs was about 90–95%, with diameters ranging between 20 and 40 nm and lengths 5–15 µm. All organic materials were purchased commercially from Sigma-Aldrich and Merck companies. FT-IR spectra were recorded with KBr

**Table 4** Comparison of MWCNTs@$SiO_2$/$SO_3H$ with other catalysts reported in the literature for the synthesis of compound **4a**

| Entry | Catalyst | Conditions | Time/min | Yield/% | References |
|---|---|---|---|---|---|
| 1 | Montmorillonite K10 | 0.1 g, 125 °C | 90 | 78 | [22] |
| 2 | *p*-TSA | 10 mol%; 125 °C | 900 | 89 | [7] |
| 3 | MNPs-$SO_3H$ | 0.02 g, 100 °C | 10 | 82 | [60] |
| 4 | $K_5CoW_{12}O_{40}{\cdot}3H_2O$ | 1 mol%, 125 °C | 120 | 80 | [38] |
| 5 | $H_4SiW_{12}O_{40}$ | 5 mol%, 110 °C | 20 | 88 | [61] |
| 6 | $P_2O_5$ | 10 mol%, 60 °C | 10 | 85 | [15] |
| 7 | Zwitterionic salt | 10 mol%, 80 °C | 120 | 80 | [36] |
| 8 | Cyanuric chloride | 10 mol%, 100 °C | 10 | 90 | [39] |
| 9 | SILP[a] | 50 mg, 100 °C | 5 | 82 | [32] |
| 10 | *β*-CD-BSA[b] | 0.02 mmol, 100 °C | 6 | 88 | [62] |
| 11 | MSNs-HPZ-$SO_3H$[c] | 10 mg, 120 °C | 95 | 84 | [63] |
| 12 | SILC[d] | 80 mg, 100 °C | 7 | 82 | [8] |
| 13 | $HClO_4$-C[e] | 0.075 mmol, 125 °C | 120 | 87 | [37] |
| 14 | ZBS[f] | 2 mol%, 80 °C | 15 | 92 | [40] |
| 15 | MWCNTs@$SiO_2$/$SO_3H$ | 13 mg, 100 °C | 15 | 90 | This work |

[a]Ferrocene-labeled supported ionic liquid phase

[b]*β*-Cyclodextrin-butanesulfonic acid

[c]Mesoporous silica nanoparticles functionalized with homopiperazine sulfamic acid

[d]Silica gel-supported –$SO_3H$

[e]Graphite-supported perchloric acid

[f]Zinc benzenesulfonate (as homogeneous catalyst)

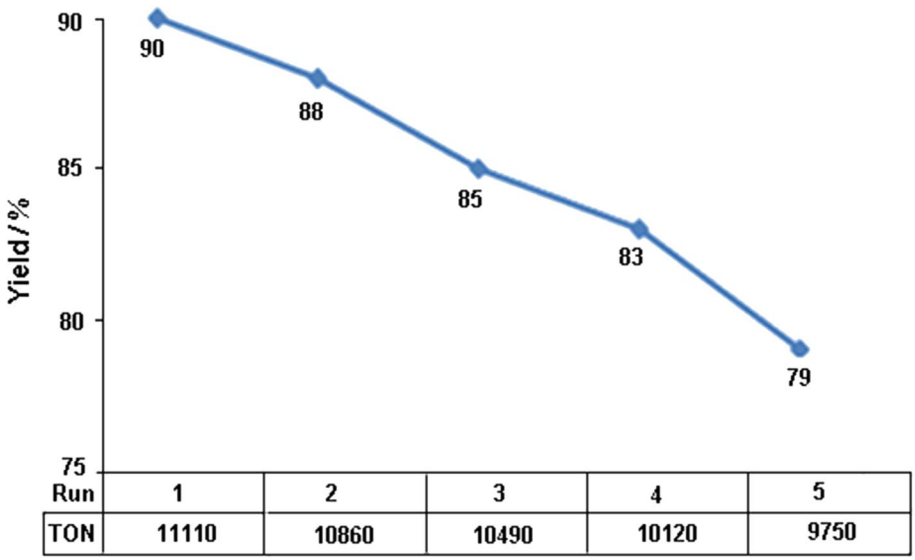



**Fig. 6** Recyclability of MWCNTs@$SiO_2$/$SO_3H$

disk using a Magna-IR, spectrometer 550 Nicolet. NMR spectra were recorded using a Bruker 400 MHz spectrometer with TMS as an internal standard and DMSO-$d_6$ as solvent. The thermogravimetric analysis (TGA) curves were carried out using a V5.1A DUPONT 2000. To investigate the morphology of the MWCNTs, FE-SEM images and EDX analysis were used by a Sigma ZEISS, Oxford Instruments Field Emission Scanning Electron Microscope. Brunauer–Emmett–Teller (BET) analysis was used to characterize the functionalized surfaces of CNTs by MicrotracBEL Corp instrument.

## Preparation of catalyst

### Hydroxylation of MWCNTs surfaces

Raw MWNTs (0.2 g) and 100 $cm^3$ methylene chloride were added into a 250 $cm^3$ flask and sonicated for 10 min. After

that, TPABr (1 g dissolved in 10 $cm^3$ $H_2O$), $KMnO_4$ (0.25 g dissolved in 5 $cm^3$ $H_2O$), and 10 $cm^3$ acetic acid were added. The mixture was stirred vigorously at 25 °C for 24 h. Then, the mixture was filtered through a 10-µm pore size polycarbonate filter paper and washed with HCl and methanol. After drying, MWCNTs functionalized with hydroxyl groups (MWCNTs-OH) were achieved [65].

#### Preparation of sulfonated MWCNTs (MWCNTs@$SiO_2$/$SO_3H$)

MWCNTs-OH (0.50 g) were dispersed in 50 $cm^3$ of ethanol (using bath sonicator for 10 min) followed by addition of 1.0 $cm^3$ 28 wt% concentrated ammonia aqueous solution ($NH_3 \cdot 3H_2O$), 9 $cm^3$ deionized water, and 0.50 $cm^3$ of tetraethyl orthosilicate (TEOS). The mixture was vigorously stirred at 25°C for 16 h. After that, MWCNTs@$SiO_2$ were filtered through a 10-µm pore size polycarbonate filter paper and washed with deionized water, ethanol, and acetone, and, finally, dried at 60 °C under vacuum.

For sulfonation of MWCNTs@$SiO_2$, a 500 $cm^3$ suction flask was equipped with a pressure equalizing dropping funnel-containing chlorosulfonic acid and gas inlet tube for conducting HCl gas over adsorbing the solution water. MWCNT@$SiO_2$ (500 mg) was debundled in dry $CH_2Cl_2$ using bath sonicator for 10 min, and then, chlorosulfonic acid (0.4 $cm^3$ in dry $CH_2Cl_2$) was added dropwise over a 30 min period. After the addition of $ClSO_3H$, the mixture was filtered through 10-µm pore size polycarbonate filter paper and MWCNTs@$SiO_2$/$SO_3H$ as a black solid was separated and washed with methanol before being dried in an oven at 70 °C.

#### Determination the acidity of catalyst

The acidity of prepared catalyst was quantitatively analyzed by back titration method. In a common route, 0.1 g of MWCNTs@$SiO_2$/$SO_3H$ was dispersed in 15 $cm^3$ of a 0.1 N NaOH using a bath sonicator (20 min) and then stirred overnight. After that, the mixture was filtered and the filtrate was titrated with a 0.1 N HCl solution to determine the excess NaOH. Obtained result shows that the concentration of $SO_3H$ groups on prepared solid acid surfaces was 6.2 mmol $g^{-1}$. Accordingly, the acidity of 13 mg of catalyst in synthesis of amidoalkyl naphthols was 0.081 mmol.

### General procedure for the synthesis of amidoalkyl naphthols

A mixture of arylaldehyde (1 mmol), β-naphthol (1 mmol), amide (1.2 mmol), and MWCNTs@$SiO_2$/$SO_3H$ (13 mg) were taken in a round-bottom flask and stirred and heated in an oil bath (100 °C). After the reaction was completed (monitored by TLC), the reaction mixture was cooled to room temperature and washed with hot water to remove the remained amide. Then, 10 $cm^3$ acetone were added to the reaction mixture and filtered off for separation the catalyst. After that, the filtrate was evaporated for obtaining the crude product and for further purification recrystallized from hot ethanol.

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