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Catalytic evaluation of newly prepared GO-SB-H_2PMo as an efficient and reusable nanocatalyst for the neat synthesis of amidoalkyl naphthols

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Abstract

The catalytic activity of newly prepared functionalized graphene oxide nanosheets, denoted as GO-SB-H_2PMo, has been investigated in the synthesis of amidoalkyl naphthols through the one-pot, three-component reaction of *β*-naphthol with various aryl aldehydes and acetamide. The reactions occur under solvent-free conditions and the process is operative with various aryl aldehydes, giving the corresponding products in high yields over short reaction times. Moreover, the catalyst could be easily recovered from the reaction mixture and reused so that the considerable catalytic activity can still be achieved after the fifth run.

Keywords: GO-SB-H_2PMo; $H_3PMo_{12}O_{40}$; Schiff base; amidoalkyl naphthols.

Introduction

Over the last few decades, the problems associated with most homogeneous catalysts, such as their environmental hazards and difficult recovery have led both academic and industrial researchers to develop alternative chemical processes based on heterogeneous catalysis [1-7]. Heterogeneous catalysts have often the advantages of simple separation of the catalyst from liquid reaction media, reusability, easy handling, and low corrosion [8-12]. However, the main drawback to the bulk heterogeneous catalysts is their low specific surface area. In order to increase the surface area or even to increase the number of accessible active sites, the catalyst is usually deposited on the surface of a solid support with high surface area and suitable mechanical strength [13-17]. In recent years, graphene oxide (GO) nanosheets, which can be easily prepared from graphite powder using modified Hummers method [18], have attracted great interest as promising low-cost ideal support for a number of nanoparticles, metals, and organic compounds due to their extremely high specific surface area, high thermal conductivity, high hydrophilic nature, high thermal and

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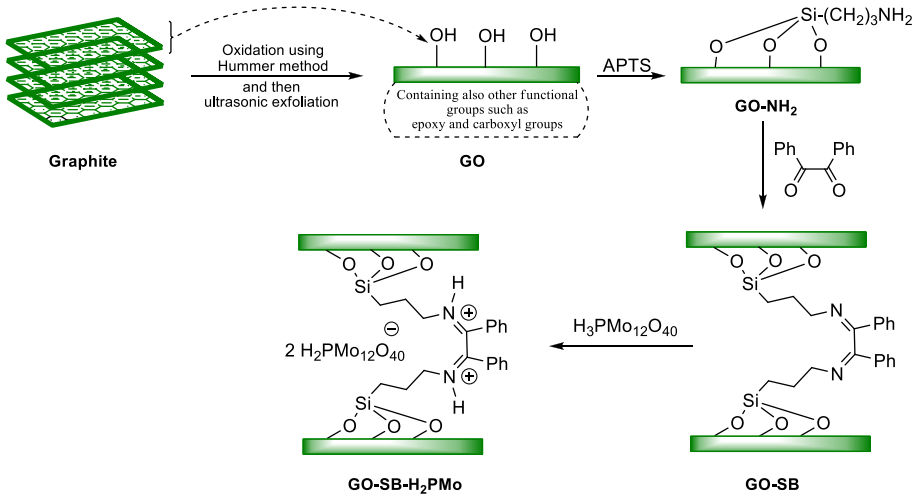
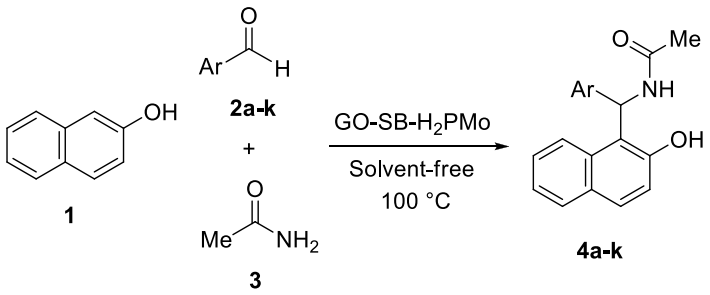
chemical stability, and good accessibility [19-22]. Functionalized GO-based materials have diverse applications in various fields such as solar cells [23], adsorption of organic dye [24], and also as efficient heterogeneous catalysts in various chemical transformations [25-27]. Among the several methods developed for functionalization of GO including esterification reaction between the carboxylic acid group of GO and alcohols [28], reaction between the hydroxyl and carboxyl groups of GO and isocyanates [29], amidation reaction between the carboxylic acid group of GO and amine [30], and silylation of the hydroxyl groups of GO using 3-aminopropyltriethoxysilane (APTS) as a silane coupling agent [31], the last one has gained considerable attention to covalently modify the surface of GO [26,31,32].

Amidoalkyl naphthols, which can be converted to important biologically active aminoalkyl naphthol derivatives by amide hydrolysis, are generally synthesized *via* the one-pot three-component reaction of *β*-naphthol, an aldehyde, and an amide in the presence of various catalysts, such as iodine [33], $K_5CoW_{12}O_{40}$-$3H_2O$ [34], molybdophosphoric acid [35], $Al(H_2PO_4)_3$ [36], $Yb(OTf)_3$ [37], thiamine hydrochloride [38], copper *p*-toluenesulfonate [39], nano-sulfated zirconia [40], carbon-based solid acid [41], nano silica phosphoric acid [42], $ZrOCl_2 \cdot 8H_2O$ [43], nano Al_2O_3 [44], zirconocene dichloride (Cp_2ZrCl_2) [45], and copper complex supported on magnetic reduced graphene oxide ($RGO/CoFe_2O_4$@Cu(II)) [46]. The synthesis using ultrasound irradiation in the presence of $Cu(ClO_4)_2.6H_2O$ [47] has been also reported for these compounds. Though each of these methods has its own advantage, the majority suffer from at least one of the following disadvantages: the use of toxic halogenated solvents or catalysts, long reaction time, the use of relatively expensive catalysts or unsatisfactory yields. Thus, the exploration of novel methodologies using new efficient and reusable catalysts is still ongoing.

Considering the unique properties of functionalized GO nanosheets, very recently, a novel Schiff base (SB) functionalized GO containing phosphomolybdic counter-anion $H_2PMo_{12}O_{40}^-$ (H_2PMo), denoted as GO-SB-H_2PMo, was successfully prepared by grafting of 3-aminopropyltriethoxysilane (APTS) on GO nanosheets followed by condensation with benzil and finally reaction with phosphomolybdic acid ($H_3PMo_{12}O_{40}$, denoted as H_3PMo) and fully characterized in our group (Scheme 1). This new reusable catalyst performed well and showed a high level of catalytic activity in the synthesis of tetrahydrobenzo [*a*] xanthene-11-ones [48]. Prompted by these facts and as part of our research on the development of environmentally friendly methods for the synthesis of organic compounds using reusable catalysts [49-59], we report here another application of GO-SB-H_2PMo as catalyst in the synthesis of amidoalkyl naphthols **4a-k** *via* the reaction of *β*-naphthol **1** with aryl aldehydes **2a-k** and acetamide **3** under neat conditions (Scheme 2).



Scheme 1. Preparation of GO-SB-H_2PMo nanosheets



Scheme 2. GO-SB-H_2PMo nanosheets catalyzed synthesis of amidoalkyl naphthols

Experimental

All chemicals were purchased from Merck and Aldrich and used without further purification. Melting points were recorded with a Stuart SMP3 melting point apparatus. Ultrasonication was performed using a Soltec sonicator at a frequency of 40 kHz and a nominal power of 260 W. Fourier transform infrared (FT-IR) spectra were obtained using a Tensor 27 Bruker spectrophotometer as KBr disks. The ^{1}H NMR (300 MHz) spectra were recorded on a Bruker 300 FT spectrometer, in DMSO-d_6 as the solvent using tetramethyl silane (TMS) as internal standard. Field emission scanning electron microscopy (FESEM) analyses was done using a TESCAN BRNO-MIRA3 LMU. Transmission electron microscopy (TEM) analysis was performed using a Leo 912 AB microscope with an accelerating voltage of 120 kV. The amount of molybdenum and phosphorus in the catalyst was determined using inductively coupled plasma optical emission spectroscopy (ICP-OES) conducted with a Spectro Arcos model spectrometer.

Preparation of GO nanosheets

GO nanosheets were prepared from natural graphite using Hummers method [18] with some modification. A mixture of graphite powder (5.0 g), sodium nitrate ($NaNO_3$, 2.5 g), and concentrated sulfuric acid (115 mL, 98% H_2SO_4) was stirred in an ice bath at 0-5 °C for 15 min and then potassium permanganate ($KMnO_4$,15.0 g) was slowly added. The stirring was continued for 2 h while the temperature was kept in the range of 0-10 °C. The mixture was then transferred to a water bath and stirred at 35 °C for 30 min, forming a brownish grey thick paste. Afterwards, deionized water (230

mL) was slowly added to the paste and the suspension, now brown in color, was stirred at 95-98 °C for 15 min. The suspension was further diluted with warm deionized water (700 mL, 40 °C), followed with a drop by drop addition of 30% hydrogen peroxide (H_2O_2, 50 mL). The mixture was centrifuged and the isolated yellow-brown cake was washed with diluted HCl (5 wt%) and deionized water several times until the pH became neutral. The solid graphite oxide was separated by centrifugation and dried at 60 °C under vacuum for 12 h. The obtained graphite oxide (0.4 g) was dispersed in distilled water (400 mL) and sonicated in an ultrasonic bath cleaner (100 W) for 1 h to exfoliate graphitic oxide. The complete exfoliation of graphite oxide is confirmed with the formation of light brown coloured homogeneous dispersion GO. Afterwards, the GO solution was centrifuged for 20 min and then the supernatant was removed (to remove any unexfoliated graphitic oxide). The GO nanosheets were obtained after drying the sediment in a vacuum oven at 80 °C for 24 h.

Preparation of GO-NH_2

The GO-NH_2 was prepared by grafting of APTS on GO nanosheets according to methods cited in the literature [31]. Briefly, the synthesized GO (1.0 g) was ultrasonically dispersed in anhydrous toluene (30 mL) at room temperature for 30 min and then APTS (3.0 mmol, 0.66 g) was added. The mixture was heated under a N_2 atmosphere at 110 °C for 24 h. The resultant solid was filtered and washed with toluene (3 × 10 mL), and dried at 50 °C under vacuum for 24 h to give black powder of GO-NH_2.

Preparation of GO-SB

The resulting GO-NH_2 (0.5 g) was dispersed in absolute ethanol (30 mL) using an ultrasonic bath at room temperature for 30 min. This is followed by addition of benzil (1.5 mmol, 0.32 g) and a few drops of glacial acetic acid. The mixture was heated under reflux for 5 h. The solid was filtered and washed with absolute ethanol (3 × 5 mL) to remove the non-reacted benzil, and then dried at 40 °C under vacuum for 24 h to give GO-SB [48].

Preparation of GO-SB-H_2PMo

GO-SB (0.25 g) was ultrasonically dispersed in absolute ethanol (10 mL) at 60 °C for 20 min. H_3PMo (1 mmol, 1.83 g) was added to the suspension and sonication continued for another 1 h at the same temperature. The mixture was then refluxed for 10 h. After cooling to room temperature, the solid was collected by filtration and repeatedly washed with absolute ethanol and dried under vacuum at 60 °C for 12 h to form GO-SB-H_2PMo [48].

General procedure for the synthesis of amidoalkyl naphthols 4a-k catalyzed by GO-SB-H_2PMo

A mixture of *β*-naphthol **1** (1.0 mmol, 0.14 g), an aryl aldehyde **2a-k** (1.0 mmol), acetamide **3** (1.2 mmol, 0.07 g), and GO-SB-H_2PMo (0.05 g) was heated in an oil bath at 100 °C for 2-10 min and monitored by TLC. Upon completion of the transformation, the reaction mixture was cooled to room temperature and hot ethanol was added. The catalyst was insoluble in hot ethanol and it could therefore be recycled by a simple filtration. The product was then collected from the filtrate after cooling to room temperature and recrystallized from 96% ethanol to give compounds **4a-k** in high yields.

Results and discussion

To begin our study, GO-SB-H_2PMo was prepared according to the method reported in our previous work [48]. The FESEM and TEM images of the GO-SB-H_2PMo nancatalyst are shown in Figure

1. A nanosheet-like structure in disordered phase with crumpled and wrinkled edges can be seen in FESEM image. These folded edges owing to sp^3-carbon in GO-SB-H_2PMo, bear SB-H_2PMo on both sides of the GO nanosheets. These sites may serve as ideal templates for reactant molecules to accomplish the desired chemical transformation. On the other hand, many granular-like particles that were distributed on the surfaces of GO nanosheets in FESEM and TEM images, confirm efficient bonding of the organic moieties on the surfaces of the GO nanosheets.

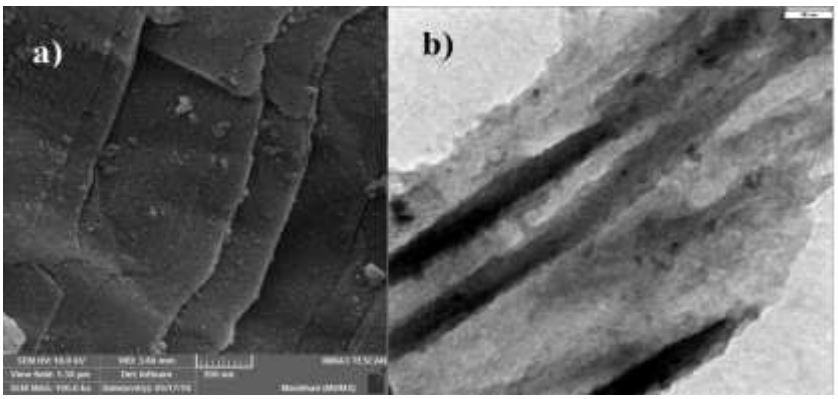


Figure 1. FESEM (a) and TEM (b) images of GO-SB-H_2PMo

According to ICP-OES result, the amounts of phosphorus and molybdenum incorporated into the catalyst GO-SB-H_2PMo are 2.076% and 7.131%, respectively, which confirms successful immobilization of Keggin heteropolyacid in the structure of the catalyst. The amount of the catalyst (mol%) in Table 1 has been calculated based on phosphorus amount.

After successful preparation of GO-SB-H_2PMo, the catalytic efficiency of this catalyst was evaluated in the synthesis of amidoalkyl naphthols. To determine the appropriate reaction conditions, initially, a model study was carried out on the synthesis of compound **4d** by reaction of *β*-naphthol **1** (1 mmol), 4-chlorobenzaldehyde **2d** (1 mmol), and acetamide **3** (1.2 mmol) in different sets of reaction conditions. The results are summarized in Table 1. Because of advantages of solvent-free conditions in chemical reactions including formation of cleaner products, simplification of work-ups, and environmental compatibility, we decided to investigate the efficiency of GO-SB-H_2PMo in the model reaction under solvent-free conditions in different catalyst amounts and temperatures. No significant yield of the product **4d** was obtained in the absence of the catalyst (Entry 1) under solvent-free conditions at high temperature, indicating that the catalyst is necessary for the reaction. Next, the reaction was performed in the presence of GO-SB-H_2PMo under solvent-free conditions at elevated temperature leading to a high yield of the product **4d**. As can be seen from Table 1, 0.05 g (3.33 mol%) of the catalyst under solvent-free conditions at 100 °C has been found to be the best of reaction conditions (Entry 13). The higher amount of the catalyst or temperature had no significant effect on the yield and reaction time. Subsequently, our investigation showed that the compound **4d** can be obtained in low to good yield in different solvents including EtOH, MeOH, H_2O, THF, $CHCl_3$, CH_2Cl_2, and CH_3CN. However, as depicted, solvent-free conditions proved to be much better than the others

in terms of reaction time and yield. For comparison and to show the merit of the GO-SB-H_2PMo catalyst, the effect of GO and H_3PMo was also investigated in the model reaction under optimized conditions. The results are shown in Table 1 (Entries 23 and 24). As is evident, GO-SB-H_2PMo proved to be a better catalyst than others in terms of reaction time and yield.

Moreover, in order to study the heterogeneous nature of GO-SB-H_2PMo nanocatalyst, the hot filtration test has been carried out on model reaction under the optimized conditions. In this test, a mixture of GO-SB-H_2PMo (0.05 g), *β*-naphthol **1** (1 mmol), 4-chlorobenzaldehyde **2d** (1 mmol) and acetamide **3** (1.2 mmol) was stirred at 100 °C in solvent-free conditions for 1 min. After 50% conversion, the catalyst was separated from the reaction mixture and then the reaction was continued for an additional time (1 min). No further product formation was observed which was monitored by TLC. This result clearly demonstrates that no leaching of Keggin heteropolyacid took place during the reaction, and GO-SB-H_2PMo is truly heterogeneous in nature.

Table 1. Effect of GO-SB-H_2PMo amount, solvent and temperature in the synthesis of compound **4d**[a]

Entry	Catalyst (g, mol%)	Solvent	T (°C)	Time (min)	Isolated Yield (%)	TON[b]	TOF (min^{-1})[c]
1	-----	-----	100	120	Trace	-----	-----
2	GO-SB-H_2PMo (0.02, 1.33)	-----	80	10	55	41.35	4.13
3	GO-SB-H_2PMo (0.02, 1.33)	-----	90	10	61	45.86	4.58
4	GO-SB-H_2PMo (0.02, 1.33)	-----	100	9	67	50.38	5.60
5	GO-SB-H_2PMo (0.03, 2.00)	-----	80	10	63	31.50	3.15
6	GO-SB-H_2PMo (0.03, 2.00)	-----	90	8	70	35.00	4.38
7	GO-SB-H_2PMo (0.03, 2.00)	-----	100	7	78	39.00	5.57
8	GO-SB-H_2PMo (0.04, 2.66)	-----	80	9	75	28.20	3.13
9	GO-SB-H_2PMo (0.04, 2.66)	-----	90	6	82	30.83	5.14
10	GO-SB-H_2PMo (0.04, 2.66)	-----	100	5	86	32.33	6.47
11	GO-SB-H_2PMo (0.05, 3.33)	-----	80	5	86	25.82	5.16
12	GO-SB-H_2PMo (0.05, 3.33)	-----	90	3	92	27.62	9.20
13	GO-SB-H_2PMo (0.05, 3.33)	-----	100	2	94	28.22	14.11
14	GO-SB-H_2PMo (0.05, 3.33)	-----	120	2	93	27.92	13.96
15	GO-SB-H_2PMo (0.06, 4.00)	-----	100	2	94	23.50	11.75
16	GO-SB-H_2PMo (0.05, 3.33)	EtOH	Reflux	30	75	22.52	0.75
17	GO-SB-H_2PMo (0.05, 3.33)	MeOH	Reflux	30	70	21.02	0.70
18	GO-SB-H_2PMo (0.05, 3.33)	H_2O	Reflux	60	30	9.01	0.15
19	GO-SB-H_2PMo (0.05, 3.33)	THF	Reflux	60	42	12.61	0.21
20	GO-SB-H_2PMo (0.05, 3.33)	$CHCl_3$	Reflux	30	60	18.02	0.60
21	GO-SB-H_2PMo (0.05, 3.33)	CH_2Cl_2	Reflux	30	73	21.92	0.73
22	GO-SB-H_2PMo (0.05, 3.33)	CH_3CN	Reflux	30	45	13.51	0.45
23	GO (0.05 g)	-----	100	75	36	-----	-----
24	H_3PMo (0.05 g)	-----	100	60	61	-----	-----

[a]Reaction conditions: *β*-naphthol **1** (1 mmol), 4-chlorobenzaldehyde **2d** (1 mmol), and acetamide **3** (1.2 mmol)
[b]Turn Over Number
[c]Turn Over Frequency

With successfully optimized conditions in hand, and in order to evaluate the scope of this catalytic transformation, a range of amidoalkyl naphthols was prepared by the reaction of various aryl aldehydes with *β*-naphthol and acetamide in the presence of GO-SB-H_2PMo under the optimized reaction conditions. The results are summarized in Table 2. As shown, a wide range of aryl aldehydes bearing either electron-donating or electron-withdrawing substituents reacted successfully and gave the products in

high yields within short reaction time and no undesirable side-products were observed, showing high catalytic activity of GO-SB-H_2PMo. However, it was found that the aromatic aldehydes with electron-withdrawing groups reacted faster than those with electron-donating groups and gave the higher yields of the products as would be expected.

Table 2. GO-SB-H_2PMo catalyzed synthesis of amidoalkyl naphthols **4a-k**[a]

Entry	Ar	Product	Time (min)	Isolated Yields (%)	m.p. (°C)	
					Found	Reported
1	C_6H_5	**4a**	2	92	238-239	241-243 [34]
2	4-FC_6H_4	**4b**	4	91	229-231	230-232 [36]
3	2-ClC_6H_4	**4c**	5	93	212-214	213-215 [36]
4	4-ClC_6H_4	**4d**	2	94	230-232	228-229 [34]
5	3-BrC_6H_4	**4e**	5	90	242-245	245-247 [47]
6	4-BrC_6H_4	**4f**	8	91	227-229	230-232 [43]
7	2-$O_2NC_6H_4$	**4g**	8	89	212-213	211-213 [47]
8	3-$O_2NC_6H_4$	**4h**	4	92	240-242	241-242 [36]
9	4-$O_2NC_6H_4$	**4i**	2	93	243-245	242-244 [44]
10	4-MeC_6H_4	**4j**	10	87	212-214	213-215 [44]
11	4-$MeOC_6H_4$	**4k**	9	80	182-184	183-185 [44]

[a]Reaction conditions: *β*-naphthol **1** (1 mmol), an aryl aldehyde **2a-k** (1 mmol), acetamide **3** (1.2 mmol), GO-SB-H_2PMo (0.05 g, 3.33mol%), 100 °C, solvent-free.

In order to evaluate the overall utility of the current methodology, the results were compared with those of the other methods reported for the synthesis of amidoalkyl naphthols. This comparison is shown in Table 3. It is clear from the data that our procedure with GO-SB-H_2PMo as catalyst gave high yields of the products in shorter reaction times than the other conditions.

Table 3. Comparison of the efficiencies of various catalysts for the one-pot three -component synthesis of amidoalkyl naphthols

Catalyst	Conditions			Time (min)	Yield (%)	Ref.
	Solvent	T (°C)	Other			
Iodine	$ClCH_2CH_2Cl$	r.t.	-----	480-1440	62-93	[33]
$K_5CoW_{12}O_{40}$-$3H_2O$	-----	125	-----	120-180	74-90	[34]
molybdophosphoric acid	AcOEt	65	-----	180-240	86-97	[35]
$Al(H_2PO_4)_3$	-----	125	-----	15-240	55-93	[36]
$Yb(OTf)_3$	[bmim][BF_4]	80	-----	240-480	87-92	[37]
thiamine hydrochloride	EtOH	reflux	-----	240	75-93	[38]
copper *p*-toluenesulfonate	-----	80	-----	6-720	16-95	[39]
nano-sulfated zirconia	-----	120	-----	30-90	81-94	[40]
carbon-based solid acid	-----	130	-----	2-20	86-93	[41]
nano silica phosphoric acid	-----	80	-----	15-170	80-98	[42]
$ZrOCl_2 \cdot 8H_2O$	-----	80	-----	30-45	82-96	[43]
nano Al_2O_3	-----	110	-----	15-190	70-97	[44]
Cp_2ZrCl_2	$ClCH_2CH_2Cl$	r.t.	-----	300-540	42-94	[45]
RGO/$CoFe_2O_4$@Cu(II)	-----	120	-----	25-45	85-92	[46]
$Cu(ClO_4)_2.6H_2O$	-----	r.t.	Ultrasound	30-80	84-90	[47]
GO-SB-H_2PMo	-----	100	-----	2-10	80-94	This work

The recyclability and reusability of the catalyst GO-SB-H_2PMo were also examined in the same model reaction under the aforementioned optimized reaction conditions. The catalyst was readily recovered from the reaction mixture using the procedure outlined in the experimental section. The separated

catalyst was washed with hot ethanol and subsequently dried at 70 °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 (Figure 2). Furthermore, retention of the structure of the catalyst was confirmed by comparing the FT-IR spectrum of the recovered catalyst after the fifth run (Figure 3(b)) with that of the fresh catalyst (Figure 3(a)) for the model reaction. As shown, both spectra are almost identical.

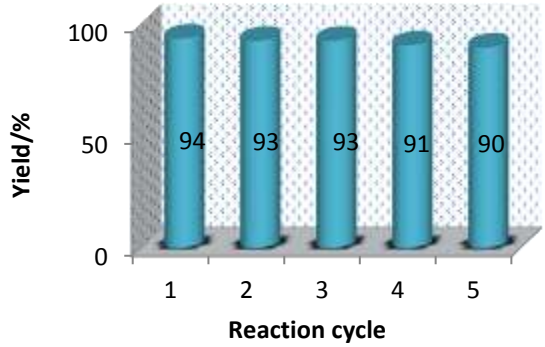


Figure 2. Reusability of GO-SB-H_2PMo for the synthesis of compound **4d**

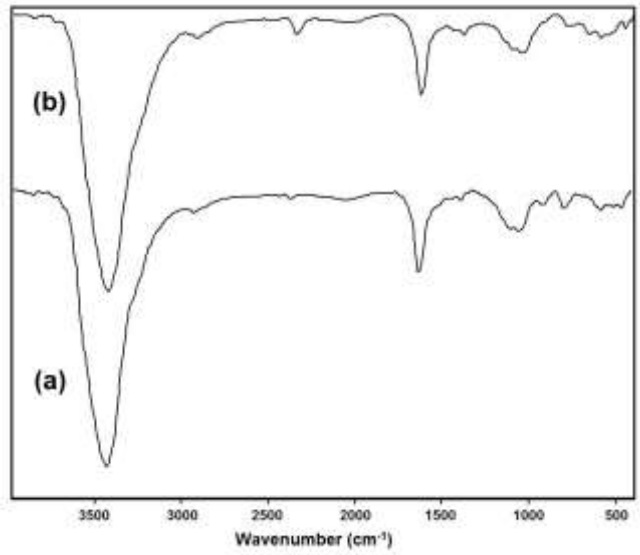
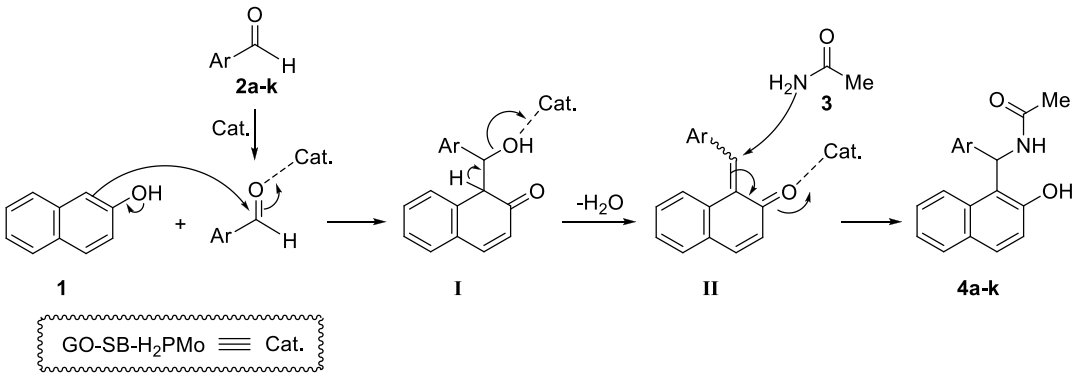


Figure 3. FT-IR spectra of fresh catalyst GO-SB-H_2PMo (a), and recovered catalyst after fifth run (b) for the synthesis of compound **4d**

Although we did not investigate the reaction mechanism, a plausible pathway for this reaction can be considered as depicted in Scheme 3. As shown, the catalyst, GO-SB-H_2PMo ≡ Cat., with several accessible Mo sites and P-O-H moieties plays a significant role in increasing the electrophilic character of the electrophiles in the reaction. The reaction occurs *via* the formation of *ortho*-quinone methide (*o*-QM) intermediate [**II**] prepared *in situ* by Knoevenagel condensation of *β*-naphthol **1** and aryl aldehydes **2a-k** *via* the intermediate [**I**]. Subsequent addition of acetamide to the intermediate [**II**] afforded the final products **4a-k**.



Scheme 3. Plausible pathway for the formation of amidoalkyl naphthols in the presence of GO-SB-H_2PMo as catalyst

Conclusion

In summary, we showed that newly prepared GO-SB-H_2PMo efficiently catalyzes the synthesis of amidoalkyl naphthols by one-pot three-component reaction of *β*-naphthol with several aryl aldehydes and acetamide under solvent-free reactions. The method was fast and high yielding but it was found that the aryl aldehydes with electron-withdrawing groups reacted faster than those with electron-donating groups and gave the higher yields of the products as would be expected. Furthermore, the catalyst could be recycled after a simple work-up, and used at least five times without significant reduction in its catalytic activity. The procedure is also advantageous in the sense that it is a solvent-free reaction and therefore operates under environmentally friendly conditions. Further applications of this new catalyst for other reaction systems are currently under investigation.

Acknowledgements

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