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Graphene oxide supported dicationic ionic liquid: an efficient catalyst for the synthesis of 1-carbamatoalkyl-2-naphthols

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Abstract

This article describes the synthesis, characterization, and catalytic application of graphene oxide (GO) supported imidazolium based dicationic ionic liquid (DIL@GO). The DIL@GO was successfully synthesized through stepwise functionalization of GO. The as prepared DIL@GO was characterized by different microscopic and spectroscopic techniques such as XRD, SEM, TEM, TGA, FT-IR, EDX and CHNS analysis. The catalytic efficiency of the DIL@GO was explored for the synthesis of 1-carbamatoalkyl-2-naphthol derivatives through condensation of aromatic aldehydes, alkyl carbamates and 2-naphthol. The influence of catalyst amount and reaction temperature is examined to optimize the performance of catalytic reactions. It is found that DIL@GO offers a highly efficient solvent-free reaction path for the synthesis of 1-carbamatoalkyl-2-naphthols. Based on this, a plausible mechanism for the conversion is proposed. Additionally, the catalyst showed good stability, facile separation and reusability up to five reaction cycles without significant loss of catalytic activity.

Keywords Graphene oxide · Dicationic ionic liquid · Heterogeneous catalyst · Solvent free

Introduction

Ionic liquids (ILs) have drawn tremendous attention of the scientific community due to their potential applications in different disciplines such as electrochemistry [1], organic synthesis [2], chemical separation [3], and material science [4]. ILs are

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composed of organic cations and inorganic or organic anions. Judicious selection of cation–anion combination can provide the task specific physiochemical properties of ILs. Compared to conventional solvents, ILs have unique properties such as low vapour pressure, high thermal stability, non-flammability, remarkable solubility, high conductivity, and broad liquid range [5]. Recently, there is an increasing research interest in dicationic ionic liquids (DILs) due to flexibility in tuning their physiochemical properties as compared to monocationic ionic liquids. Generally, DILs consist of dications which are linked with either flexible or rigid spacers (generally an alkyl chain) and two monoanions [6]. There are countless possibilities to tune the physiochemical properties of DILs through judicious selection of cations and anions [7]. DILs are extensively studied for their potential applications as a solvent [8], lubricant [9], nanoparticles coating [10] and catalysis [11]. Despite many attractive properties, their application in an industrial process is limited due to their homogeneous nature. To overcome this drawback, its immobilization on solid support is the best solution. There are many solid supports such as silica, polymer, metal ions, zeolites, carbon nano tubes (CNTs), etc., have been widely studied for the heterogenization of ILs [12]. Heterogenization facilitates its easy separation after completion of the reaction, effortless purification, storage and handling as compared to its homogeneous counterpart.

Nowadays, graphene oxide (GO) emerged as a promising carbon material for the heterogenization of the homogeneous catalyst due to its unique structural properties [13]. GO can be obtained through exfoliation of graphite oxide and, hence, possesses a large surface area for the immobilization of homogeneous moiety [14]. Additionally, GO possesses good thermal and chemical stability [15]. Besides, GO is a flexible layered carbon material having different functional groups, such as epoxy, hydroxyl and carboxyl on the basal plane and edges [16–19]. These functional sites are available for effective anchoring of homogeneous species through covalent functionalization [20]. Further, these hydrophobic groups present on the GO surface may assist substrate adsorption and, hence, speed up the catalytic reactions [21]. The immobilization of DILs on the surface of GO can result in an enhanced heterogeneous catalytic system with the translation of inherent characteristics of the GO to the catalyst.

On the other hand, 1-carbamatoalkyl-2-naphthols are the molecules of huge interest as their hydrolyzed derivatives 1-aminoalkyl-2-naphthols possesses interesting biological activities as hypotensive and bradycardia agents [22, 23]. These compounds having 1,3-amino-oxygenated functional groups are also present in many natural products and drugs [24]. There are many reports that have been published for the synthesis of 1-carbamatoalkyl-2-naphthols by a one-pot multicomponent reaction between aromatic aldehydes, 2-naphthols, and alkyl carbamates using different catalysts [25–29]. Nevertheless, some of the synthesis routes suffer from drawbacks such as harsh reaction conditions, longer reaction time, lower yield, use of an excessive amount of catalyst, and use of expensive metal catalysts. Therefore, development of a highly efficient process for the synthesis of 1-carbamatoalkyl-2-naphthols is of considerable importance.

In this study, we presented a graphene oxide supported –SO_3H functionalized imidazolium based dicationic ionic liquid (DIL@GO) as an efficient catalyst for the synthesis of 1-carbamatoalkyl-2-naphthols under solvent free reaction conditions (Scheme 1). After the reaction, DIL@GO can be easily recovered by centrifugation and reused five times without significant loss of its catalytical activity.

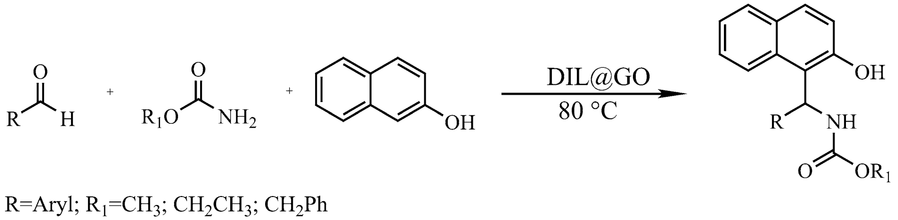
Experimental

Materials and methods

All the chemicals were purchased from Sigma-Aldrich (India) and SDFCL. Melting points were determined by a capillary method using VMP-D melting point apparatus and were uncorrected. FT-IR spectra were recorded on a Thermo Nicolet 6700 spectrometer (KBr). X-ray diffraction (XRD) patterns were recorded on a Bruker D-2 Phaser XRD instrument. EDX was done by a scanning electron microscope with EDS (ESEM-EDAX XL-30, Phillips, Netherlands). Thermogravimetric analysis (TGA) was done on a Mettler-Toledo Thermal Analyzer. Scanning electron microscopy (SEM) images were taken on a JEOL, JSM-6010LA instrument. Transmission electron microscopy (TEM) images were taken on a JEOL, JEM 2100 instrument. ^{1}H NMR spectra were recorded on a BRUKER 400 MHz. CHNS analysis was done by an elementar vario MICRO CUBE analyzer.

Synthesis of (3-chloropropyl)trimethoxysilane functionalized graphene oxide (ClPr-Si@GO)

Graphene oxide (GO) was synthesized by Hummers method [30]. Afterwards, synthesized GO was treated with (3-chloropropyl)trimethoxysilane (CPTMS) to get functionalized GO [31]. In a typical process, CPTMS (25 mmol) was added to the GO suspension in toluene (50 mL, 10 mg mL^{-1}), and the mixture was stirred at reflux temperature under nitrogen atmosphere. After 24 h, the reaction mixture was cooled to room temperature, and solid was separated. The resultant solid was washed with toluene, ethanol, and water followed by drying at 60 °C to get functionalized ClPr-Si@GO.



Scheme 1 Synthesis of 1-carbamatoalkyl-2-naphthols using DIL@GO

Synthesis of graphene oxide supported dicationic ionic liquid (DIL@GO)

Dicationic ionic liquid brush was generated through stepwise covalent grafting of imidazole under reflux condition. Initially, sodium imidazole salt was prepared through treatment of imidazole (12.5 mmol) with sodium hydride (12.5 mmol) in dry toluene (100 mL) for 2 h. After that, ClPr-Si@GO (500 mg) was added to the flask and stirred under reflux for 24 h. The obtained imidazole functionalized GO was treated with 1,4-dibromobutane (12.5 mmol) in toluene (100 mL) under reflux, followed by further treatment with sodium imidazole salt for 24 h. The obtained solid was dispersed in toluene (50 mL) and treated with 1,4-butanesultone (1.70 mL) at 100 °C for 6 h, followed by treatment with conc. H_2SO_4 (0.5 mL) at 50 °C for 8 h. The obtained DIL@GO was washed thoroughly with ethanol and dried under vacuum.

General procedure for the catalytic synthesis of 1-carbamatoalkyl-2-naphthols

In a typical procedure, a mixture of aromatic aldehyde (1 mmol), 2-naphthol (1 mmol), alkyl carbamate (1.1 mmol), and DIL@GO (0.1 g) was added to the round bottom flask and stirred at 80 °C. The progress of reaction was monitored through TLC. After the completion of reaction, crude product was washed thoroughly with water to remove unreacted starting material. After that, ethanol was added to solid product and catalyst was separated through centrifugation. After separation, catalyst was washed with ethanol and dried under vacuum for further use. Pure 1-carbamatoalkyl-2-naphthols were obtained through solvent evaporation followed by recrystallization. All synthesized compounds were characterized by physical and spectral methods and found in agreement with reported.

Spectral data for some representative compounds

Methyl ((2-hydroxynaphthalen-1-yl)(3-nitrophenyl)methyl)carbamate (4c) IR (KBr, cm^{-1}): 3420, 3292, 3088, 2951, 1686, 1629, 1526, 1476, 1364, 1278, 1194, 1043, 970, 827, 752, 679, 517; ^1H NMR (400 MHz, DMSO-d_6): δ (ppm) 3.60 (s, 3H), 6.94 (d, $J=8.8$ Hz, 1H), 7.20 (d, $J=9.2$ Hz, 1H), 7.28 (t, $J=7.6$ Hz, 1H), 7.40 (s, 1H), 7.54–7.63 (m, 2H), 7.80–7.84 (m, 2H), 7.95 (s, 2H), 8.06 (d, $J=8$ Hz, 1H), 8.11 (s, 1H), 10.25 (s, 1H).

Methyl ((4-fluorophenyl)(2-hydroxynaphthalen-1-yl)methyl)carbamate (4e) IR (KBr, cm^{-1}): 3419, 3194, 3074, 2953, 1682, 1629, 1511, 1438, 1335, 1272, 1157, 1068, 1043, 939, 854, 748, 605, 514; ^1H NMR (400 MHz, DMSO-d_6): δ (ppm) 3.56 (s, 3H), 6.81 (d, $J=8.8$ Hz, 1H), 7.06–7.11 (m, 2H), 7.19–7.29 (m, 4H), 7.39 (s, 1H), 7.73–7.82 (m, 3H), 7.88 (d, $J=7.6$ Hz, 1H), 10.15 (s, 1H).

Benzyl ((2-hydroxynaphthalen-1-yl)(2-nitrophenyl)methyl)carbamate (4 g) IR (KBr, cm^{-1}): 3421, 3246, 3064, 2949, 1699, 1628, 1524, 1437, 1362, 1273, 1184, 1062, 961, 940, 832, 778, 697, 608, 557; ^1H NMR (400 MHz, DMSO-d_6): δ (ppm) 5.02 (d, $J=12.8$ Hz, 1H), 5.08 (d, $J=12.8$ Hz, 1H), 7.03 (d, $J=8.8$ Hz, 1H), 7.26–7.34 (m, 8H), 7.39 (t, $J=7.6$ Hz, 1H), 7.47 (t, $J=7.6$ Hz, 1H), 7.58–7.66 (m, 2H), 7.72–7.80 (m, 2H), 7.89 (d, $J=8.4$ Hz, 1H), 8.13 (d, $J=8.8$ Hz, 1H), 9.81 (s, 1H).

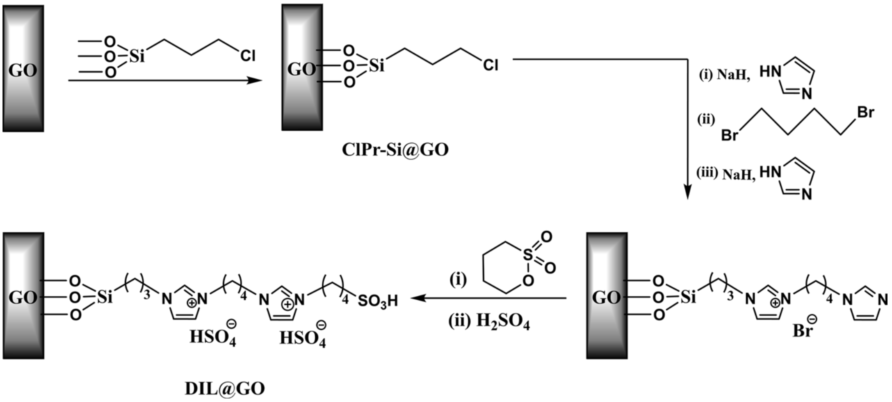
Benzyl ((3-chlorophenyl)(2-hydroxynaphthalen-1-yl)methyl)carbamate (4j) IR (KBr, cm^{-1}): 3433, 3242, 3069, 2965, 1674, 1628, 1511, 1439, 1326, 1271, 1227, 1182, 1063, 949, 813, 787, 617, 541; ^1H NMR (400 MHz, DMSO-d_6): δ (ppm) 5.02 (d, $J=12.8$ Hz, 1H), 5.10 (d, $J=12.8$ Hz, 1H), 6.88 (d, $J=8.4$ Hz, 1H), 7.13 (d, $J=7.2$ Hz, 1H), 7.20 (d, $J=8.8$ Hz, 1H), 7.24–7.42 (m, 10H), 7.78 (d, $J=9.2$ Hz, 1H), 7.81 (d, $J=7.6$ Hz, 1H), 7.90–7.96 (m, 2H), 10.20 (s, 1H).

Ethyl ((2,4-dichlorophenyl)(2-hydroxynaphthalen-1-yl)methyl)carbamate (4o) IR (KBr, cm^{-1}): 3408, 3366, 3070, 2981, 1683, 1628, 1517, 1438, 1335, 1269, 1242, 1187, 1053, 937, 860, 797, 744, 598; ^1H NMR (400 MHz, DMSO-d_6): δ (ppm) 1.11 (t, $J=6.8$ Hz, 3H), 3.96–4.00 (m, 2H), 6.79 (d, $J=8.4$ Hz, 1H), 7.09 (d, $J=8.8$ Hz, 1H), 7.26 (t, $J=7.2$ Hz, 1H), 7.37–7.45 (m, 2H), 7.51–7.53 (m, 2H), 7.73 (d, $J=9.2$ Hz, 1H), 7.79 (d, $J=7.2$ Hz, 1H), 7.86 (d, $J=8.4$ Hz, 1H), 7.98 (d, $J=8.8$ Hz, 1H), 9.92 (s, 1H).

Results and discussion

Preparation and characterization of the catalyst (DIL@GO)

GO supported dicationic ionic liquid catalyst was prepared according to the procedure shown in Scheme 2. GO was synthesized by Hummers method [30]. The as prepared GO possess oxygen-containing functional groups (such as epoxy, hydroxyl and carboxyl) available for further functionalization. Post-syntyhetic modification of GO was done by immobilization of (3-chloropropyl)trimethoxysilane [31]. Silylation of GO has been researched by several research groups, which provides a wide range of transformation possibilities for the designing of a novel catalytic system [32–34]. This silane functionalized GO was then treated with



Scheme 2 Synthesis of graphene oxide supported dicationic ionic liquid (DIL@GO)

sodium salt of imidazole and 1,4-dibromobutane to generate dicationic ionic liquid brush. The obtained product was reacted with 1,4-butane sultone followed by conc. H_2SO_4 to get GO immobilized $–SO_3H$ functionalized dicationic ionic liquid catalyst (DIL@GO). The synthesized DIL@GO was then characterized by different spectroscopic and microscopic methods such as Fourier transform infrared spectroscopy (FT-IR), X-ray Diffraction (XRD), Transmission electron microscopy (TEM), Scanning electron microscopy (SEM), Thermogravimetric analysis (TGA), Energy dispersive X-ray (EDX) spectroscopy and CHNS elemental analysis.

FT-IR spectra of GO, ClPr-Si@GO, and DIL@GO are shown in Fig. 1. In the FT-IR spectrum of GO is shown a broad band at 3429 cm^{-1} for the –OH stretching vibrations [35, 36]. The bands at 1713 and 1614 cm^{-1} correspond to C=O and C=C vibrations of the aromatic ring, respectively [37, 38]. The band at 1384 cm^{-1}corresponds to skeletal vibrations of C–OH [39, 40]. The band at 1229 and 1048 cm^{-1} are due to C-O (epoxy) and C-O (alkoxy) stretching vibrations, respectively [41–43]. The spectrum of ClPr-Si@GO shows two bands around 1098 and 1015 cm^{-1} due to Si–O–Si stretching vibrations [44, 45]. The bands around 2949 and 2945 cm^{-1} are due to C–H stretching of the alkyl chain. In the spectrum of DIL@GO, the band around 1634 cm^{-1}corresponds to the C=C absorption peak. The bands at 1568 and 1441 cm^{-1} are due to an imidazole ring's vibration [46]. The band at 1055 $cm^{-1}$is due to $–SO_3$ stretching vibration [47–49]. The band at 1206 cm^{-1} was attributed to absorbing of S=O stretching vibrations [50].

X-ray diffraction (XRD) pattern (Fig. 2) of pure GO shows a sharp peak at $2\theta = 9.5°$, which corresponds to the crystalline nature of the pristine graphene oxide. The crystallite size of GO was determined using the Scherrer equation and found to be 10.4 nm. After immobilization of ionic liquid, the crystalline state of graphene oxide transforms into an amorphous state and thus the sharp peak diminishes, and a broad diffraction peak appears in the XRD pattern of DIL@GO.

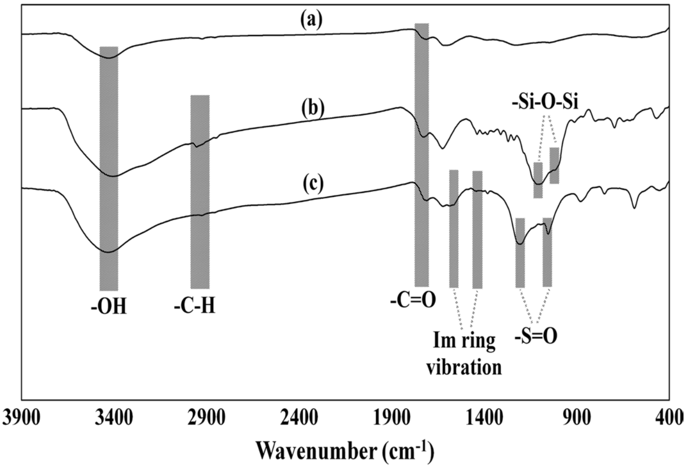


Fig. 1 FT-IR spectra of (a) GO, (b) ClPr-Si@GO and (c) DIL@GO

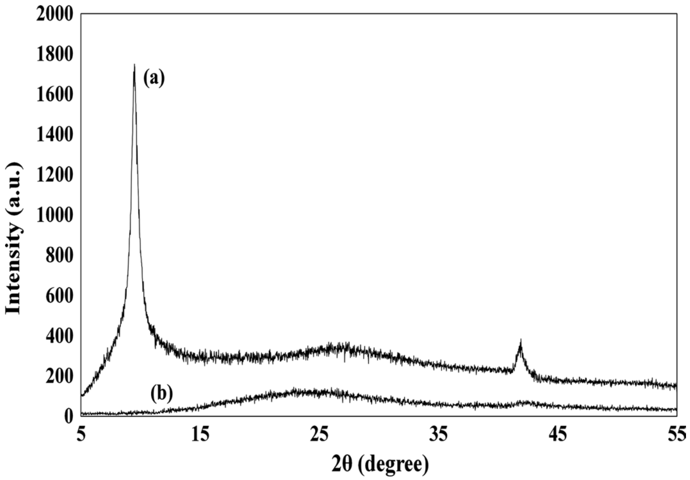


Fig. 2 X-ray diffraction patterns of (a) GO and (b) DIL@GO

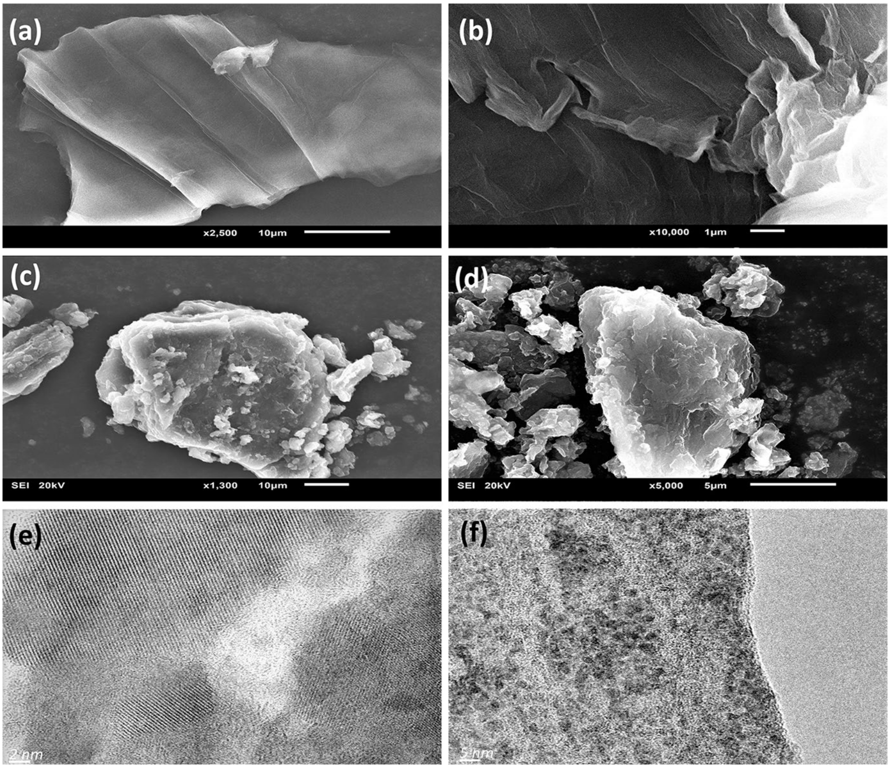


Fig. 3 SEM images of **a**, **b** GO and **c**, **d** DIL@GO; TEM images of **e** GO and **f** DIL@GO

The structural and morphological analysis of DIL@GO was carried out by SEM and TEM (Fig. 3). The sheet-like structures can be seen in the SEM images of pristine GO (Fig. 3a, b). The SEM images of DIL@GO are shown in Fig. 3c, d, which exhibits less of sheet-like structure and more of a clustered or thicker structure, which indicates successful grafting of dicationic ionic liquid on the surface of graphene oxide. The TEM analysis of GO (Fig. 3e) indicates the flat planer layer of graphene oxide, and in the image of DIL@GO (Fig. 3f), the uniformly distributed dicationic ionic liquid can be observed.

The thermogravimetric analysis was done using a nitrogen atmosphere with a temperature increase rate of 10 °C/min. TGA curves of GO and DIL@GO are shown in Fig. 4. As shown in the curve of GO, the first weight loss appears near 100 °C, which can be attributed to the loss of physisorbed water by graphene oxide. Another weight loss observed between 165 and 245 °C due to the degradation of oxygen-containing functional groups on the surface of GO. The TGA curve of DIL@GO shows that the functionalized GO is much more thermally stable than GO. A large weight loss was observed at 550 to 610 °C temperature, which suggests the thermal decomposition of the immobilized dicationic ionic liquid on the surface of GO.

Elemental analysis was carried out by using energy dispersive X-ray (EDX) spectroscopy. The EDX spectrum for DIL@GO is shown in Fig. 5. The spectrum shows distinct peaks of carbon, nitrogen, oxygen, silicon, and sulfur, which confirms the successful grafting of –SO_3H functionalized dicationic ionic liquid on the surface of graphene oxide. Additional confirmation was obtained from CHNS analysis of the catalyst and found to be 43.37%C, 4.49%H, 2.36%N and 5.68%S.

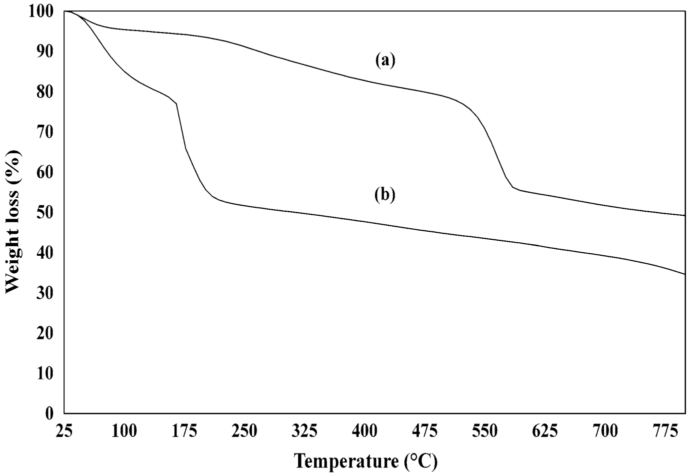


Fig. 4 TG analysis of (a) DIL@GO and (b) GO

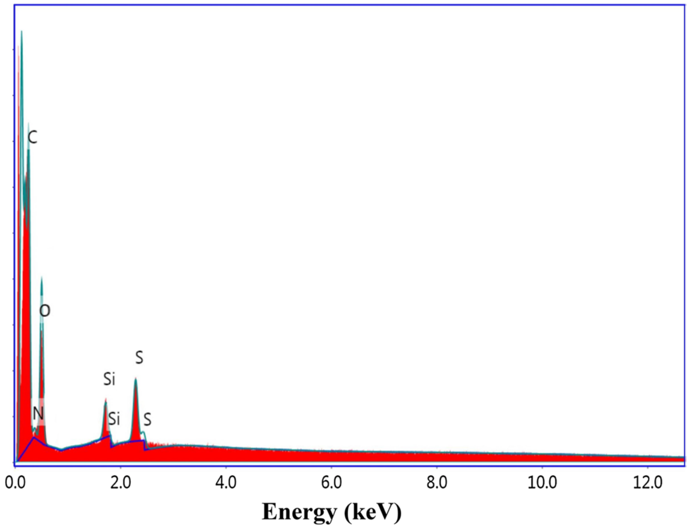


Fig. 5 EDX spectrum of DIL@GO

Catalytic activity of DIL@GO for the synthesis of 1-carbamatoalkyl-2-naphthol derivatives

The performance of the prepared DIL@GO as a catalyst was explored by the synthesis of 1-carbamatoalkyl-2-naphthols. Different parameters were applied for the optimization of the model reaction between 2-naphthol, benzaldehyde, and methyl carbamate under solvent-free condition (Table 1). Initially, the reaction was carried

Table 1 Optimization of reaction condition for the model reaction[a] and comparison with the reported catalysts

Entry	Catalyst (mg)	Temperature (°C)	Time (min)	Yield (%)
1	None	80	120	NR
2	DIL@GO (50)	60	10	86
3	DIL@GO (50)	80	8	89
4	DIL@GO (50)	100	8	90
5	DIL@GO (100)	80	3	97
6	DIL@GO (150)	80	3	97
7	[Dsim]HSO_4 [51]	80	11	98
8	Preyssler nanoparticles/SiO_2 [52]	90	3	84
9	4-(1-Imidazolium)-butanesulfonate [53]	80	120	78
10	$SnCl_4·5H_2O$ [54]	60	12	83
11	$NaHSO_4/SiO_2$ [55]	100	3.5	81

[a] 2-Naphthol (1 mmol), benzaldehyde (1 mmol), methyl carbamate (1.1 mmol); solvent free

NR no reaction

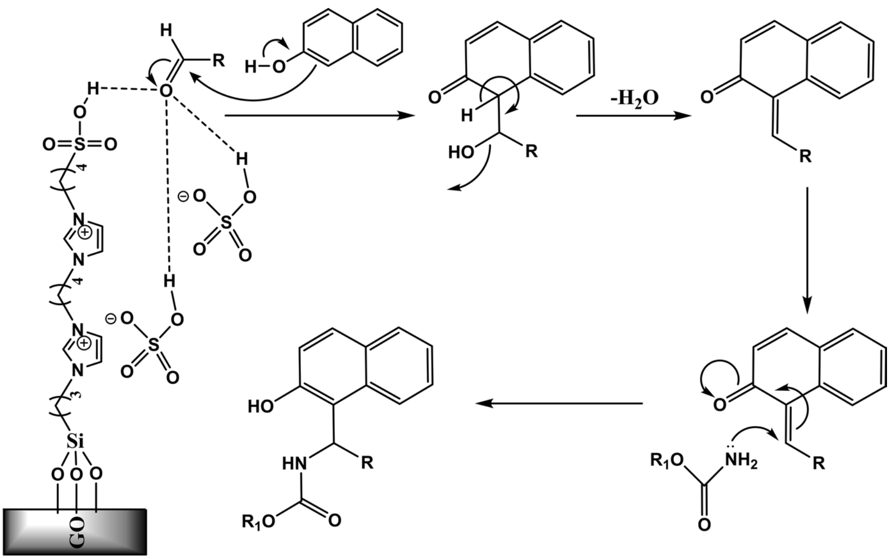
out without any catalyst (entry 1), and no reaction was observed over a period of 120 min. For the subsequent reactions, different amounts of catalyst at various temperatures and times were applied (entry 2–6). The optimal reaction condition was found at 80 °C with 100 mg of catalyst. The catalytic efficiency of DIL@GO was also compared with some reported catalysts (entry 7–11) and found to be more efficient for the model reaction. The efficiency and scope of the catalyst were determined by synthesizing derivatives of 1-carbamatoalkyl-2-naphthols under optimized conditions (Table 2). To check the substrate scope, differently substituted aromatic aldehydes were treated with methyl-, ethyl- and benzyl-carbamates. The catalyst was found suitable for all the substrates under this study. All the reactions were completed very smoothly, with short time and high to excellent isolated yields. Based on the above results, the plausible mechanism for the synthesis of 1-carbamatoalkyl-2-naphthols using DIL@GO is shown in Scheme 3. In this reaction, the DIL@GO activates the carbonyl group of aldehyde through the formation of hydrogen bonds, which then reacts with 2-naphthol to generate an intermediate. The nucleophilic addition of alkyl carbamate to this intermediate leads to the targeted 1-carbamatoalkyl-2-naphthol products.

Recyclability of the catalyst (DIL@GO)

Recyclability is one of the most important aspect of catalysis. Recyclability of DIL@GO was studied in a model reaction between 2-naphthol, benzaldehyde and methyl carbamate under optimized conditions for five cycles. The catalyst shows retention in

Table 2 Synthesis of 1-carbamatoalkyl-2-naphthols using DIL@GO at 80 °C under solvent-free conditions

Entry	R	R_1	Time (min)	Yield %	M.P. (°C)	
					Found	Reported
4a	C_6H_5	CH_3	3	97	222–224	224–226 [56]
4b	4-NO_2 C_6H_4	CH_3	3	96	203–205	202–204 [56]
4c	3-NO_2 C_6H_4	CH_3	3	96	244–246	248–250 [56]
4d	4-Cl C_6H_4	CH_3	5	97	197–199	196–198 [56]
4e	4-F C_6H_4	CH_3	6	95	202–204	203–205 [29]
4f	C_6H_5	$C_6H_5CH_2$	9	96	180–182	182–184 [56]
4 g	2-NO_2 C_6H_4	$C_6H_5CH_2$	7	95	198–200	198–200 [57]
4 h	3-NO_2 C_6H_4	$C_6H_5CH_2$	7	94	183–185	186–188 [56]
4i	4-F C_6H_4	$C_6H_5CH_2$	8	92	205–207	206–208 [56]
4j	3-Cl C_6H_4	$C_6H_5CH_2$	9	91	202–204	200–202 [29]
4 k	C_6H_5	CH_3CH_2	5	94	202–204	202–204 [56]
4 l	4-NO_2 C_6H_4	CH_3CH_2	4	94	223–225	226–228 [56]
4 m	4-F C_6H_4	CH_3CH_2	6	91	209–211	210–212 [56]
4n	4-Cl C_6H_4	CH_3CH_2	5	93	210–212	208–210 [56]
4o	2,4-Cl_2 C_6H_3	CH_3CH_2	6	92	207–209	208–210 [28]



Scheme 3 Plausible mechanism for DIL@GO catalyzed synthesis of 1-carbamatoalkyl-2-naphthol derivatives

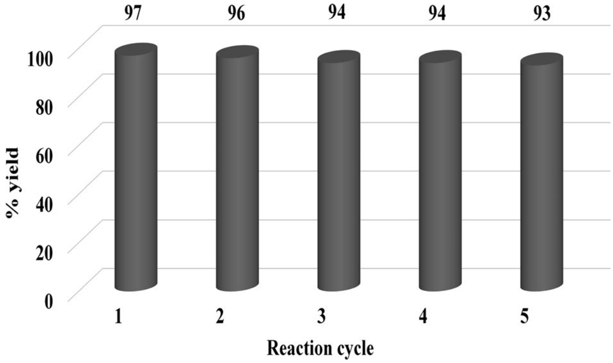


Fig. 6 Recyclability test for the DIL@GO

catalytic activity as there was no significant drop in the yield of final product during five cycles (Fig. 6). The comparison of the FT-IR spectra of fresh and used DIL@GO (Fig. 7) suggest no significant change in the functionality.

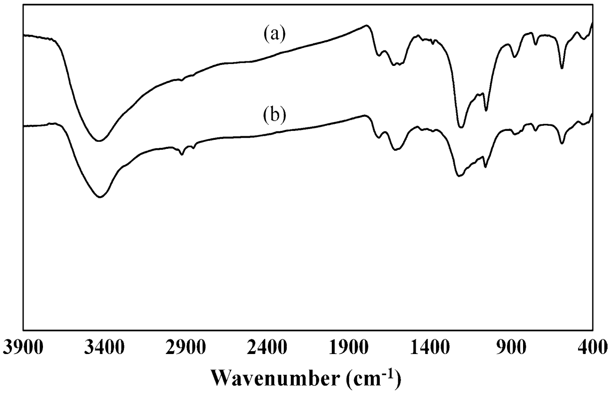


Fig. 7 FT-IR spectra of (a) fresh and (b) used DIL@GO

Conclusions

In conclusion, graphene oxide supported –SO_3H functionalized dicationic ionic liquid (DIL@GO) was found to be a highly efficient and environment friendly heterogeneous catalyst for the synthesis of 1-carbamatoalkyl-2-naphthol derivatives. The present methodology has some notable advantages such as recyclability, easy catalyst separation and handling, shorter reaction time, a solvent-free approach, and high yield.

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