Research on Chemical Intermediates
https://doi.org/10.1007/s11164-020-04197-6

Graphene oxide anchored with sulfonic acid-functionalized glycerin: production, characterization and catalytic performance for the synthesis of *N*,*N*′-alkylidene bisamides

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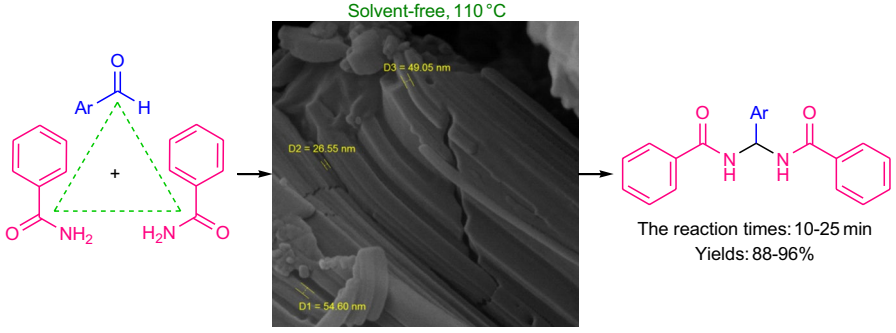
Received: 25 March 2020 / Accepted: 16 June 2020

Abstract

A novel nanosheet-structured material, namely graphene oxide anchored with sulfonic acid-functionalized glycerin (GO@Gl-SO_3H), was prepared and characterized by FTIR, EDS, FESEM, TEM and XRD analyses. Afterward, its catalytic performance was examined for the synthesis of *N*,*N*′-alkylidene bisamides through the reaction of benzamide (2 eq.) and arylaldehydes (1 eq.) under solvent-free conditions. GO@Gl-SO_3H was a highly effective catalyst for the reaction and afforded the products in high yields and short reaction times.

Graphic abstract



The nanosheet-structured catalyst: graphene oxide anchored with sulfonic acid-functionalized glycerin

Keywords Graphene oxide anchored with sulfonic acid-functionalized glycerin (GO@Gl-SO_3h) · Nanosheet-structured material · *N*,*N*′-alkylidene bisamide · Solvent-free

Electronic supplementary material The online version of this article (https://doi.org/10.1007/s11164-020-04197-6) contains supplementary material, which is available to authorized users.

Published online: 26 June 2020

Introduction

Graphene oxide (GO) is formed from flat sheets wherein carbon atoms with sp^2 and sp^3 hybridization are packed into a honeycomb lattice. In these sheets, carboxylic acid and hydroxyl groups have located at the edges, and epoxide and hydroxyl groups have located on the basal surface. Because of existing carboxylic acid and hydroxyl groups on the GO sheets, it can be easily functionalized by organic and inorganic moieties to produce a wide range of GO derivatives. Due to its unique properties such as proper thermal and chemical stability, high adsorption capability, high thermal conductivity, high carrier mobility, high structural strength, high hydrophilic nature, appropriate mechanical property, good catalytic activity, easy separation and aptitude to functionalize for different uses, many research groups have been focused on production and application of functionalized graphene oxides in different scientific, pharmaceutical and industrial fields [1–23]. Some applications of functionalized GO and graphene derivatives include SERS sensing and imaging [1], removal of heavy metal ions from water [2], nanofiltration [3], high-performance faradaic energy storage [4], wireless humidity sensing [5] and biofunctionalization of protein in anticancer therapy [6], and utilization as supercapacitor [7–9], anticorrosive agent [10], antifouling [11], sorbents for enrichment of estrogens [12], drug carrier [13, 14] and catalysts in organic transformation [15–23].

Solvent-free condition has several advantages relative to solution condition in chemical reactions, including (1) consuming fewer energy, (2) affording aimed product in shorter reaction time, and in higher yield and selectivity (often), (3) possessing safer reaction medium, (4) easier workup and purification of product, (5) preventing or decreasing by-products/waste formation and (6) being more economic [24–27].

Amide and bisamide functional groups are present in the structure of numerous biological, medical and industrial compounds [28–39]. Bisamides have been utilized in the production of peptidomimetic materials [28]. Furthermore, they have been applied as fundamental fragments to introduce *gem*-diaminoalkyl moieties in retro-inverse pseudo-peptide compounds [29], and as ligands in the Ullmann reaction to produce potentially bioactive materials [30]. Bisamides have been also used as antimicrobial [31], anti-inflammatory [31], antitumor [32] and drug release [33] agents. Bisamides coordinated with metal ions have been utilized for removal of organic dyes [34], MRI blood-pool contrasting [35] and bimodal imaging (optical/magnetic resonance) [36]; they have been also used as inhibitor against α-glucosidase [37] and as reagents in organic synthesis [38, 39]. *N*,*N*′-Alkylidene bisamides, as a class of bisamide derivatives, could be prepared by the reaction of primary amides (2 eq.) with aldehydes (1 eq.) in the presence of a catalyst [40–48].

Considering the above-mentioned themes, it is highly desirable to introduce a novel effective GO-based catalyst for the solvent-free preparation of *N*,*N*′-alkylidene bisamides; in this work, we have done this. Herein, we have synthesized graphene oxide anchored with sulfonic acid-functionalized glycerin (GO@

Gl-SO_3H) as a novel nanosheet-structured material and characterized it by FTIR, EDS (energy-dispersive X-ray spectroscopy), FESEM (field emission scanning electron microscopy), TEM (transmission electron microscopy) and XRD (X-ray diffraction) analyses. Then, we have applied GO@Gl-SO_3H as an efficacious catalyst for the synthesis of *N*,*N*′-alkylidene bisamides by the reaction of benzamide (2 eq.) and arylaldehydes (1 eq.) under solvent-free conditions.

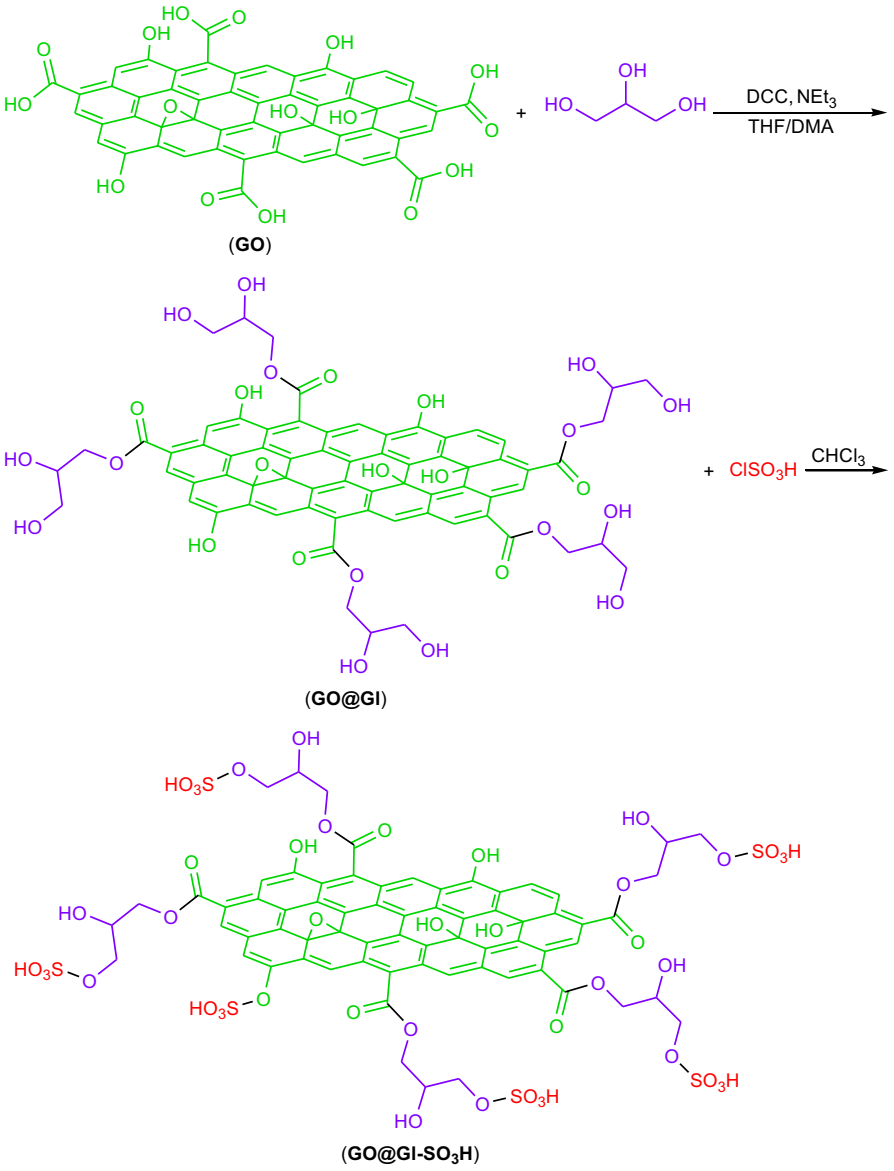
Experimental

Chemicals and instruments

The utilized starting materials and solvents were bought from Merck or Fluka Chemical Companies. Thin-layer chromatography (TLC) using silica gel SIL G/UV 254 plates was used for the observation of the reactions progress. Measurement of melting points was done by a Buchi B-545 apparatus in open capillary tubes. FTIR spectra were recorded using a Shimadzu IR-60 apparatus. A Bruker Avance DPX FT-NMR spectrometer was used for running ^{1}H and ^{13}C NMR spectra. EDS was performed by SAMx-EDS (France system) instrument. A FESEM device, model MIRA3TESCAN-XMU, and a TEM apparatus, model Zeiss EM900, 80 keV were applied for the characterization of morphologies, thickness and surface of the GO sheets. XRD analysis was done by Cu Kα radiation ($\lambda = 1.5408$, model: X'Pert PRO MPD, PANalytical, the Netherlands).

Production of graphene oxide anchored with sulfonic acid-functionalized glycerin (GO@Gl-SO_3H)

Graphene oxide (GO) was prepared by oxidization of graphite via a modified Hummers' method [49, 50]. GO (1.00 g) was added to a mixture of THF (20 ml) and dimethyl acetamide (DMA) (10 ml) and sonicated for 30 min at room temperature to give a homogeneous emulsion. *N,N'*-Dicyclohexylcarbodiimide (DCC) (5 mmol, 1.03 g) and triethylamine (6 mmol, 0.84 ml) were added to the emulsion and stirred for 3 h at room temperature, and then glycerin (0.5 ml) was added, and stirring at room temperature was continued for 48 h, and then it was centrifuged. The obtained precipitate was separated by decanting, washed repeatedly with hot DMSO, deionized water and ethanol and dried under vacuum at 80 °C to provide GO@Gl (Scheme 1). The produced GO@Gl (1.00 g) was added to dried chloroform (20 ml) and sonicated at room temperature for 30 min; then, chlorosulfonic acid (8 mmol, 0.53 ml) was added gradually to it and stirred for 24 h at room temperature. After centrifuging and washing the resulting precipitate by dry chloroform (3 × 10 ml), and drying, GO@Gl-SO_3H was obtained (Scheme 1).



Scheme 1 Preparation of GO@Gl-SO_3H

General procedure for the synthesis of N,N′-alkylidene bisamides

A mixture of benzamide (2 mmol, 0.242 g), aldehyde (1 mmol) and GO@Gl-SO_3H (0.04 g) was powerfully stirred by a small rod at 110 °C. After consuming the reactants (as observed by TLC), the mixture was cooled to room temperature, EtOAc (15 ml) was added and then it was stirred accompanied with refluxing for 2 min. The

resulting mixture was centrifuged and decanted to isolate the unsolvable catalyst. The obtained EtOAc after the decanting was evaporated, and the precipitate (crude product) was purified by recrystallization from EtOH/H_2O (95/5).

Note: Selected spectral data and original spectrums of the prepared bisamides are presented in supplementary material.

Results and discussion

Characterization of the catalyst

The novel nanosheet-structured catalyst, namely graphene oxide anchored with sulfonic acid-functionalized glycerin (GO@Gl-SO_3H), was characterized by FTIR, EDS, FESEM, TEM and XRD analyses.

The FTIR spectrums of GO@Gl and GO@Gl-SO_3H (Scheme 1) are illustrated in Fig. 1, and the obtained data from the spectrum of GO@Gl-SO_3H are reported in Table 1. As Table 1 shows, the peaks related to all expected functional groups

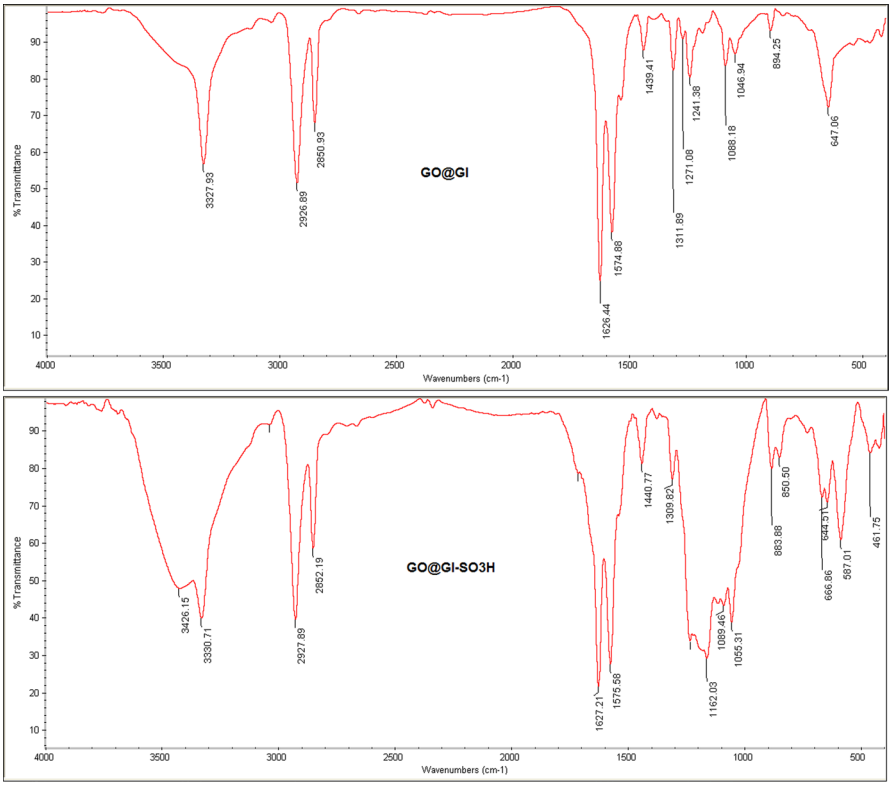


Fig. 1 FTIR spectrums of GO@Gl and GO@Gl-SO_3H

Table 1 FTIR data of GO@Gl-SO_3H

Peak (cm^{-1})	Functional group or bond
587	SO_2 (bending)
1055	C–O (stretching vibration)
1162	SO_2 (symmetric stretching)
~1233	SO_2 (asymmetric stretching)
1576	C=C (stretching vibration)
1627	C=O of GO (stretching vibration)
~1718	C=O of ester group (stretching vibration)
2852	sp^3 C–H of glycerol chain (symmetric stretching vibration)
2928	sp^3 C–H of glycerol chain (asymmetric stretching vibration)
~3040	sp^2 C–H (stretching vibration)
3000–3730	OH group of the SO_3H, and OH groups of GO (stretching)

and bonds in the structure of GO@Gl-SO_3H were seen in its spectrum; interpretation of the FTIR data is in agreement with those reported for the similar functional groups and bonds [19, 23, 42]. In another study, to confirm successful anchoring of SO_3H with the GO-based material, the FTIR data of GO@Gl-SO_3H were compared with those of GO and GO@Gl (the FTIR spectrum of GO have been reported in the literature [51–53]). The peaks related to the same bonds and functional groups in GO, GO@Gl and GO@Gl-SO_3H were observed in the three spectrums (i.e., C–O, C=C, C=O, sp^3 C–H of glycerol chain, sp^2 C–H and OH groups of GO). However, the peaks corresponding to SO_3H were seen only in the spectrum of GO@Gl-SO_3H (i.e., 587, 1162, 1233 and 3000–3730 cm^{-1}); this

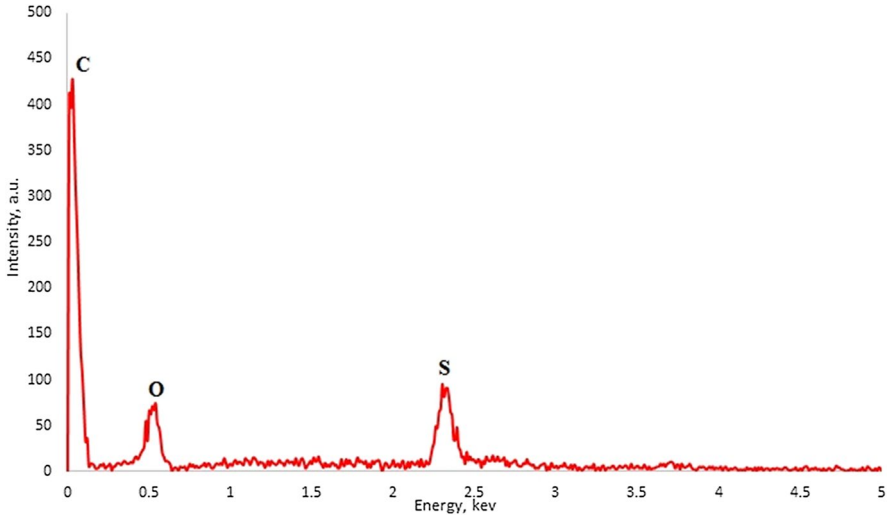


Fig. 2 The EDS spectrum of GO@Gl-SO_3H

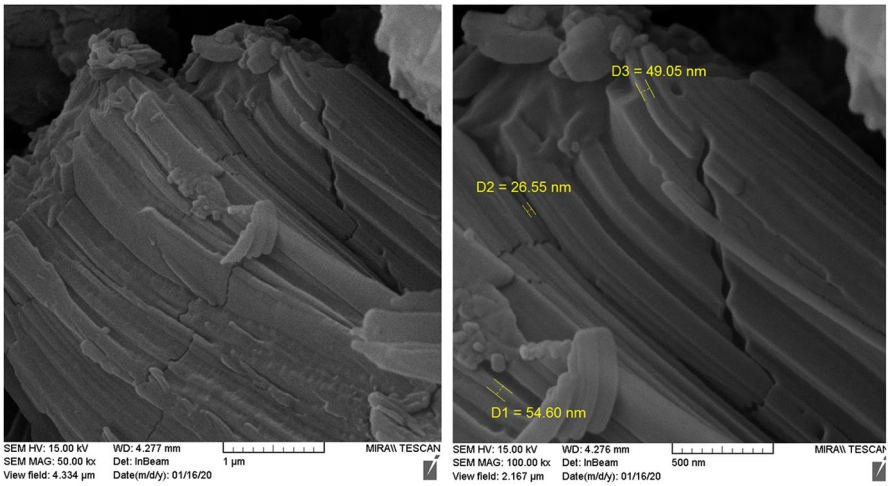


Fig. 3 FESEM micrographs of the GO-based material

subject confirmed successful functionalization of GO@Gl with SO_3H group, and formation of GO@Gl-SO_3H.

In the structure of graphene oxide anchored with sulfonic acid-functionalized glycerin, there are carbon, oxygen and sulfur; this theme was obviously observed in the EDS spectrum (Fig. 2).

The FESEM micrographs of GO@Gl-SO_3H are indicated in Fig. 3; the micrographs displayed homogeneous nanosheets with crumpled structure in one edge. The TEM micrographs of the nanosheets surface are shown in Fig. 4; according to the micrographs, the nanosheets surface was smooth with a slight fracture.

The XRD diagrams of GO, GO@Gl and GO@Gl-SO_3H are shown in Fig. 5. In the GO diagram, a sharp peak was observed at $2\theta \approx 9.6°$, which is related to the crystalline nature of the pristine graphene oxide; this characteristic peak was seen at $2\theta \approx 9.3°$ for GO@Gl and GO@Gl-SO_3H. By anchoring graphene oxide with glycerol and sulfonic acid-functionalized glycerol, the peak intensity (at 2θ

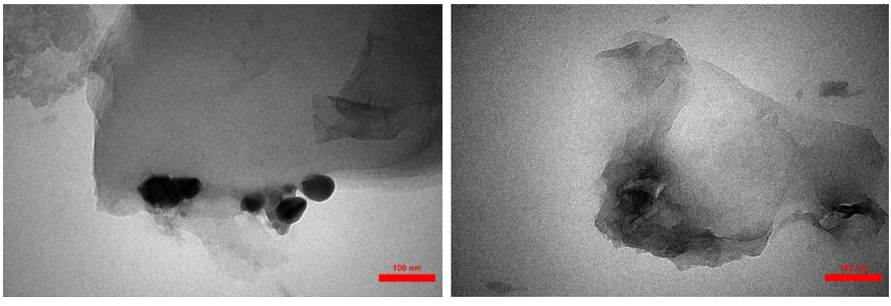


Fig. 4 TEM micrographs of GO@Gl-SO_3H

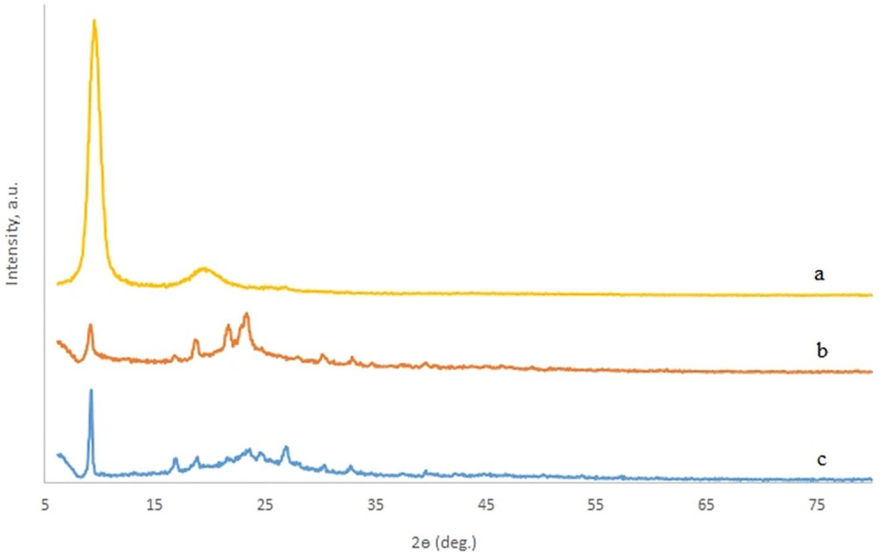
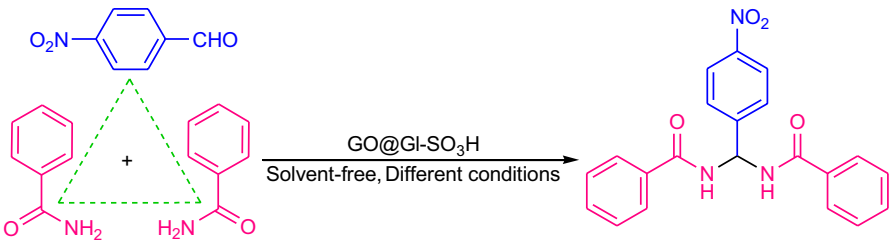


Fig. 5 The XRD diagrams of GO (**a**), GO@Gl (**b**) and GO@Gl-SO_3H (**c**)

≈ 9.3°) was decreased, and some peaks (with low intensity) appeared at $2\theta \approx$ 16.0°–40.0° (for GO@Gl: $2\theta \approx$ 16.9°, 18.8°, 21.5°, 23.4°, 24.8°, 27.9°, 30.1°, 32.9°, 34.6°, 39.5°, and for GO@Gl-SO_3H: $2\theta \approx$ 16.9°, 18.8°, 21.5°, 23.4°, 24.8°, 26.9°, 27.9°, 30.2°, 32.8°, 39.5°). Comparing the data of GO diagram with the two other spectrums revealed that by anchoring the mentioned groups with GO, some amount of its crystalline form is converted into amorphous state. Furthermore, most of the data obtained from the XRD patterns of GO@Gl and GO@Gl-SO_3H were similar; this topic can be attributed to unchanging the GO@Gl scaffold after functionalization by SO_3H, i.e., almost all the SO_3H groups have bonded with the hydroxyl groups of glycerin. The literature data verified these explanations [20].



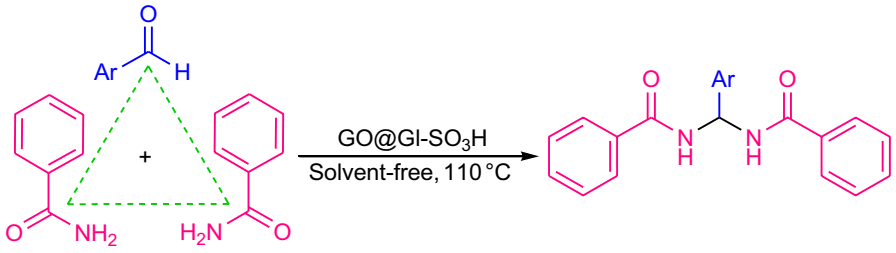
Scheme 2 The model reaction

Examining catalytic performance of GO@Gl-SO_3H for the synthesis of *N*,*N*′-alkylidene bisamides

To attain most appropriate reaction conditions for the production of *N*,*N*′-alkylidene bisamides, at first, the condensation of benzamide (2 mmol) with 4-nitrobenzaldehyde (1 mmol) was chosen as a model reaction (Scheme 2), and influence of the catalyst amount and temperature on it was studied under solvent-free conditions. For this purpose, the reaction was checked in the presence of different catalytic amounts of GO@Gl-SO_3H (0.02–0.05 g) at a range of 90–115 °C. According to the obtained reaction times and yields, the most appropriate amount of the catalyst and the reaction temperature were 0.04 g and 110 °C, respectively (time 10 min; yield 96%).

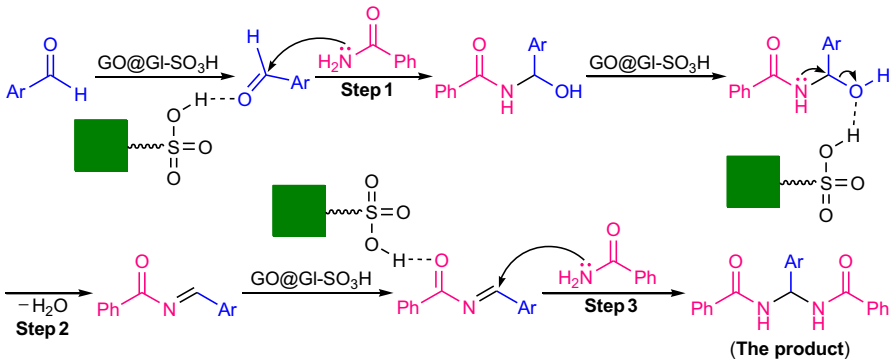
After obtaining the most appropriate conditions for the reaction, efficacy and generality of GO@Gl-SO_3H were assessed by reaction of benzamide with diverse arylaldehydes, consisting of benzaldehyde and aromatic aldehydes bearing halogen, electron-attracting and electron-donating substituents; the resulting reaction times and yields are illustrated in Table 2. As it can be seen in Table 2, aldehydes possessing bromo, chloro, nitro, methyl and methoxy substituents on ortho-, meta- and para-positions afforded the corresponding *N*,*N*′-alkylidene bisamides in short times and high yields. Thus, graphene oxide anchored with sulfonic acid-functionalized glycerin was a highly efficacious and general catalyst for the reaction.

Table 2 Solvent-free synthesis of *N*,*N*′-alkylidene bisamides using GO@Gl-SO_3H



Product	Ar	Time (min)	Yield[a] (%)	M.p., °C found (reported)
1a	C_6H_5	15	91	215–217 (216–218) [42]
1b	3-BrC_6H_4	15	93	231–233 (226–228) [47]
1c	4-BrC_6H_4	15	90	252–254 (254–256) [48]
1d	2,4-$Cl_2C_6H_3$	15	89	194–196 (192–193) [43]
1e	4-ClC_6H_4	10	96	251–253 (252–254) [47]
1f	2-$O_2NC_6H_4$	15	92	254–256 (257–259) [40]
1g	3-$O_2NC_6H_4$	10	95	232–234 (230–232) [43]
1h	4-$O_2NC_6H_4$	10	96	261–263 (263–264) [41]
1i	4-$CH_3C_6H_4$	15	90	242–244 (241–244) [44]
1j	4-$CH_3OC_6H_4$	25	88	225–227 (223–225) [42]

[a]Isolated yield



Scheme 3 The proposed mechanism

Table 3 Comparing the reaction conditions and the results of GO@Gl-SO_3H with those of some reported catalysts for the preparation of products **1a**, **1e** and **1j**

Catalyst	Conditions	Time (min) for products **1a/1e/1j**	Yield (%) for products **1a/1e/1j**	References
GO@Gl-SO_3H	Solvent-free, 110 °C	15/10/25	91/96/88	This work
Montmorillonite K10	Solvent-free, 100 °C	80/–[a]/180	85/–[a]/72	[40]
Boric acid	Toluene, 120 °C	960/2880/–[a]	92/65/–[a]	[41]
Boric acid	CH_2Cl_2, microwave irradiation (650 W)	20/30/–[a]	85/60/–[a]	[41]
Nano-[DSPECDA][HSO_4][b]	Solvent-free, 90 °C	30/–[a] /30	91/–[a] /83	[42]
ZnO/KIT-6@$NiFe_2O_4$	Solvent-free, 60 °C	10/10/10	90/94/85	[43]
3D-network polymer -supported ionic liquid	Toluene, reflux	30/25/50	85/83/80	[44]
KH_2PO_4 supported on silica	Solvent-free, 80 °C	15/15/15	87/90/81	[45]
Catalyst-free	Solvent-free, 100 °C	5/10/30	85/80/85	[46]
$H_{14}[NaP_5W_{29}MoO_{110}]$	CH_3OH, reflux	90/70/–[a]	86/82/–[a]	[47]
$NiFe_2O_4$@SiO_2-PPA	CH_3OH, reflux	80/60/–[a]	86/86/–[a]	[48]

[a]In the work, this product has not been prepared

[b]Nano-2-[*N'*,*N'*-dimethyl-*N'*-(silica-*n*-propyl)ethanaminium chloride]-*N*,*N*-dimethylaminium bisulfate

Based on the literature [42, 45], a plausible mechanism was proposed for the synthesis of *N*,*N'*-alkylidene bisamides (Scheme 3). GO@Gl-SO_3H has catalyzed the reaction by its acidic group (SO_3H); the catalyst tasks are clearly illustrated in the mechanism, which include (1) activation of the carbonyl groups in steps 1 and 3, to accept nucleophilic attack of benzamide nitrogen, and (2) activation of the hydroxyl group in step 2 for removal of a H_2O molecule.

In another study, the reaction conditions and the results of GO@Gl-SO_3H for the preparation of compounds **1a**, **1e** and **1j** were compared with those of some reported catalysts (Table 3). As Table 3 indicates, GO@Gl-SO_3H afforded the products in

higher yields in comparison with the other catalysts. The reaction times of GO@Gl-SO_3H are also shorter than most of the other catalysts. Moreover, the reaction medium of our protocol (solvent-free conditions) is better than the reaction medium of the protocols in which toxic organic solvents have been used.

Conclusions

In conclusion, a novel nanosheet-structured catalyst (GO@Gl-SO_3H) was introduced for organic synthesis. In this work, it could catalyze a valuable organic transformation, i.e., the synthesis of *N*,*N*′-alkylidene bisamides. Several advantages are associated with this catalytic process, such as efficacy, generality, short reaction times, high yields, easy separation of the catalyst from the reaction medium, simple purification of the products (by recrystallization) and performing the reactions under solvent-free conditions.

Acknowledgements The support of this work by Research Council of Payame Noor University is gratefully acknowledged. Moreover, we appreciate Abdolkarim Zare (Payame Noor University) for helping us to do this research.

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