

Synthesis of spirooxindole derivatives catalyzed by GN/SO_3H nanocomposite as a heterogeneous solid acid

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Received: 11 May 2018 / Accepted: 12 July 2018 © Springer Nature B.V. 2018

Abstract

In this study, we synthesized a GN/SO₃H solid acid nanocomposite by immobilization of chlorosulfonic acid onto the surface of $g-C_3N_4$. The nanocomposite was characterized by different techniques (transmission electron microscopy, Fourier transform infrared spectroscopy, thermal gravimetric analysis, atomic absorption spectroscopy, X-ray diffraction, and energy dispersive X-ray spectrometer). The GN/SO₃H nanocomposite was then applied to the synthesis of spirooxindole derivatives via multicomponent reaction of isatins, 1,3-dicarbonyl compounds, and malononitrile at reflux conditions in an ethanol/water solvent (1:1). The heterogeneous nanocomposite showed high catalytic activity in one-pot; the multi-component reaction of malononitrile, isatins, and 1,3-dicarbonyl compounds under reflux conditions and the corresponding spirooxindole derivatives were synthesized in good to excellent yields (70–95%). We found that the nanocomposite can be easily recovered and reused for several times with a minimal decrease of its catalytic performance.

Keywords Graphitic carbon-nitride · Heterocyclic compounds · Spirooxindole derivatives · Heterogeneous solid acid

Introduction

Sulfuric acid as a homogeneous Bronsted acid is a liquid acid which is widely used as a catalyst for the synthesis of chemical compounds in different industries. Despite high catalytic efficiency, atom and energy-inefficiency, tedious workup, equipment corrosion, environmental pollution due to the production of large-scale sulfate wastes and neutralization of this catalyst are some drawbacks of using sulfuric acid

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[1–3]. The design and production of active and stable heterogeneous solid sulfonic acids is a key to solving these problems. Some metal oxide nanomaterials and organic compounds are used for the construction of these desired solid acids, but such materials are usually expensive and have been shown to possess many fewer sulfuric acid active sites. Recently, an attractive field has opened by the use of carbon nanomaterials for the immobilization of $-SO_3H$ [4–7]. Carbon nitride as a new type of carbon nanomaterial; A 2-D graphene-like nanosheet can be a good candidate for the immobilization of organic and inorganic compounds developing various type of nanocomposites with a different type of active sites. These nanocomposites have special thermal and chemical stability as well as multiple applications, especially in the field of photocatalysis. Recently, several papers report the application of g-C₃N₄ and related compounds as catalysts in the oxidation of alcohols, amines, and alkenes with oxygen under visible light, conversion of carbohydrates into furanics products and the synthesis of biodiesel [8–12].

This compound has other important uses in the fields of material science, optics and electronics. Moreover, many metal and metal oxides, as well as organic compounds, have been supported onto the surface of $g-C_3N_4$;such metal and metal oxides and the catalytic activity of corresponding nanocomposites have been investigated in many fields, especially in the field of environmental pollution [13–21].

On a different note, heterocyclic compounds are valuable intermediates for the synthesis of medicinal drugs. The synthesis of oxindole derivatives as an important type of heterocyclic compounds is still a challenge in the field of organic chemistry and industrial science. Oxindole derivatives possess good biological and pharmaceutical activities [22, 23]. Therefore, the synthesis of these compounds is highly desirable for medicinal industries. Thus, in continuation of our attempt to develop novel and benign environmental synthetic protocols [24, 25], herein, we synthesized GN/SO₃H by immobilization of chlorosulfonic acid onto the surface of $g-C_3N_4$ and apply the corresponding nanocomposite to the synthesis of spirooxindole derivatives via multicomponent reaction of isatins, 1,3-dicarbonyl compounds, and malonon-itrile at reflux conditions in solvent (ethanol/water: 1:1) (Scheme 1).

Experimental methods

Materials

All chemical materials were purchased from Merck Company. The thermal gravimetric analysis (TGA), XRD, Fourier transform infrared spectroscopy (FT-IR), energy dispersive X-ray spectrometer (EDX), and transmission electron microscopy (TEM) were done using a Perkin-Elmer Diamond TG/DTA thermal analyzer, a Bruker D₈-Advance X-ray diffractometer, a Perkin Elmer FT-IR spectrometer (Spectrum 2), a JEOL JSM-6360 scanning electron microscope and a Philips CM10 instrument, respectively. ¹H and ¹³C-NMR spectra were also recorded by a Bruker DPX-250 MHZ Advance NMR instrument.



Scheme 1 The synthesis of spirooxindole derivatives catalyzed by GN/SO₃H nanocomposite

Synthesis of the g-C₃N₄ nanosheet

10 grams of melamine were added to an alumina crucible with a cover and the contents of the crucible were heated in a muffle furnace at air atmosphere at a rate of 2.5 °C/min to 550 °C and kept for 4 h at this temperature. After the time completed, $g-C_3N_4$ (yellow powder) was collected and stored for further use [24].

Synthesis of GN/SO₃H nanocomposite

1 g of $g-C_3N_4$ was dispersed in dichloromethane (20 mL) for 30 min. Then, a solution of 4 mL of chlorosulfonic acid in 10 mL dichloromethane was added dropwise to the solution and stirred for an appropriated time. After that, the GN/ SO₃H was separated, washed and dried in an Oven at 80 °C for 8 h.

General procedure for the synthesis of Spirooxindole derivatives

 GN/SO_3H (80 mg) was added to the 3 mL of EtOH/water (1:1) and dispersed for 30 min. Then, malononitrile (1 mmol), isatin (1 mmol), and dimedone (1 mmol) were added to the mixture and the mixture was heated to reflux and kept at this temperature for an appropriate time. After the time of reaction was completed, water was separated by centrifuging followed by extracting the crude product by ethyl acetate. The solvent was dried using Na₂SO₄ and removed from the reaction by evaporation with a rotary evaporator. Finally, the crude product was recrystallized in hot ethanol to achieve the pure product. The GN/SO₃H nanocomposite was washed and reused several times.

Selected spectral data

2-Amino-2',5-dioxo-5,6,7,8-tetrahydrospiro-[chromene-4,3'-indoline]-3-carbonitrile White solid, M.p. > 250 °C. ¹H-NMR (250 MHz, DMSO-*d*₆), ppm: 1.92 (2H, *t*, CH₂), 2.15 (2H, *m*, CH₂), 2.67 (2H, *t*, CH₂), 6.67 (1H, *d*, ArH), 6.91 (1H, *t*, ArH), 7.13 (1H, *d*, ArH), 7.23 (1H, *t*, ArH), 7.37 (2H, *s*, NH₂), 10.67 (1H, *s*, NH). ¹³C-NMR (62.9 MHz, DMSO-*d*₆), ppm: 19.86, 26.88, 36.41, 46.95, 57.65, 109.20, 111.93, 117.47, 121.86, 123.38, 128.57, 134.65, 142.2, 158.71, 166.14, 178.25, 195.14. IR/cm⁻¹: 3356 (N–H), 3295 (N–H), 3170 (C–H, *sp*²), 2953 (C–H, sp³), 2205 (CN), 1715 (C=O), 1652 (N–C=O), 1474 (C–H, Ar).

2-Amino-7,7-dimethyl-2',5-dioxo-5,6,7,8-tetrahydrospiro [chromene-4,3'-indoline]-3-carbonitrile White solid, M.p. > 250 °C. ¹H-NMR (250 MHz, DMSO-*d*₆) ppm: 0.97 (6H, *s*, 2CH₃), 2.14 (2H, *s*, CH₂), 2.42 (2H, *s*, CH₂), 6.72-7.25 (4H, ArH, 2H, NH₂, m), 10.37 (1H, *s*, NH). ¹³C-NMR (62.9 MHz, DMSO-*d*₆) ppm: 27.45, 28.06, 32.32, 47.27, 50.44, 57.90, 109.77, 111.24, 117.73, 122.42, 123.41, 128.55, 134.80, 142.40, 152.0, 159.35, 164.52, 178.45, 195.37. IR/cm⁻¹: 3374 (N–H), 3318 (NH), 3142 (C–H, *sp*²), 2924 (C–H, *sp*³), 2190 (CN), 1720 (C=O), 1657 (N–C=O), 1603 (C–H, Ar), 1472 (C–H, Ar).

2-Amino-1'-benzyl-7,7-dimethyl-2',5-dioxo-5,6,7,8-tetrahydrospiro [chromene-4,3'-indoline]-3-carbonitrile White solid, M.p. > 250 °C. ¹H-NMR (250 MHz, DMSO-*d*₆) ppm: 1.08 (3H, *s*, CH₃), 1.05 (3H, *s*, CH₃) 2.12–2.26 (2H, *m*, CH₂), 2.54 (2H, *s*, CH₂), 4.96 (2H, *s*, CH₂-Ar), 6.73 (1H, *d*, ArH), 6.85 (1H, *t*, ArH), 7.11–7.18 (2H, *m*, ArH), 7.20–7.33 (3H, *m*, ArH), 7.41 (2H, *s*, NH₂), 7.53 (2H, *d*, ArH). ¹³C-NMR (62.9 MHz, DMSO-*d*₆) ppm: 27.44, 28.17, 32.44, 43.72, 47.05, 50.35, 57.50, 109.33, 111.0, 117.95, 123.03, 123.45, 127.55, 127.63, 128.74, 128.84, 134, 136.65, 143.03, 159.42, 165.03, 177.23, 195. IR/cm⁻¹: 3324 (N–H), 3298 (NH), 3144 (C–H, *sp*²), 2923 (C–H, *sp*³), 2195 (CN), 1715 (C=O), 1660 (N–C=O), 1600 (C–H, Ar), 1465 (C–H, Ar).

2-Amino-2'0,5-dioxo-5,6-dihydro-spiro[pyrano[3,2-c]quinoline-4,3'-indoline]-3-carbonitrile White solid, M.p. > 250 °C. ¹H-NMR (250 MHz, DMSO-*d*₆), ppm: 6.82 (1H, *d*, ArH), 6.94 (1H, *t*, ArH), 7.22 (1H, *d*, ArH), 7.32 (1H, *d*, ArH), 7.40 (1H, *t*, ArH), 7.45 (1H, *t*, ArH), 7.51 (1H, *d*, ArH), 7.52 (2H, *s*, NH₂), 7.97 (1H, *d*, ArH), 10.70 (1H, *s*, NH). ¹³C-NMR (62.9 MHz, DMSO-*d*₆), ppm: 57.52, 70.21, 101.92, 109.90, 112.93, 117.0, 117.44, 122.55, 123.21, 124.55, 125.40, 129.30, 133.55, 134.17, 142.71, 152.43, 155.50, 155.75, 158.93, 177.40. IR/cm⁻¹: 3414 (NH), 3264 (N–H), 3145 (C–H, *sp*²), 2942 (C–H, *sp*³), 2205 (CN), 1715 (C=O), 1678 (N–C=O), 1602 (C–H, Ar), 1470 (C–H, Ar).

Results and discussion

To synthesize GN/SO_3H heterogeneous solid acid nanocomposite, at first, g-C₃N₄ was thermally synthesized and dispersed in dichloromethane and chlorosulfonic acid was added dropwise to the mixture and stirred for a definite time. Then, the

corresponding GN/SO_3H nanocomposite was characterized by several techniques including TGA, TEM, XRD, and FT-IR.

Characterizations of catalyst

The IR Spectra of $g-C_3N_4$ and GN/SO_3H are shown in Fig. 1. The broad peaks ranging from 3000 to 3450 cm⁻¹ are assigned to the stretching vibration of the N–H bond. Heterocyclic C=N stretching vibration bond appeared at 1648 cm⁻¹. The vibration of C–N bond of tri-triazine showed vibration bonds at 1235, 1320, 1405, and 1545 cm⁻¹. In addition, the peak centered at 807 cm⁻¹ is assigned to the out-of-plane vibration of the triazine cycle. A new broad peak which ranges from 1050 to 1100 and 500 cm⁻¹ belongs to S=O and confirmed that $-SO_3H$ is supported onto the surface of $g-C_3N_4$ (Fig. 1b).

Figure 2, shows the XRD patterns of $g-C_3N_4$ and GN/SO_3H . In the XRD patterns of $g-C_3N_4$, the peaks at 13.1° and 27.4° are assigned to (100) and (002) (according to JCPDS 087-1526). These peaks show in-plane repeated units, an interlayer stacking peak, and the hexagonal phase of the nanosheet of $g-C_3N_4$, respectively (Fig. 2a). In Fig. 2b, despite the presence of $-SO_3H$, $g-C_3N_4$ still has a crystalline nature.

Figure 3a, b show the TEM images of $g-C_3N_4$ and GN/SO_3H , respectively. The TEM image of pure $g-C_3N_4$ nanosheets shows the agglomeration of the lamellar structure (Fig. 3a). Figure 3b shows the TEM image of GN/SO_3H , which demonstrate the presence of $-SO_3H$ on the surface of $g-C_3N_4$ nanosheets.

The thermal stability of $g-C_3N_4$ and GN/SO_3H were investigated (Fig. 4). Both samples have an initial weight loss of about 2–3% at a temperature range of about 100 °C, which belongs to the removal of absorbed water. The pure $g-C_3N_4$ is stable up to 550 °C and the sublimation or decomposition of $g-C_3N_4$ nanosheets



Fig. 1 FT-IR spectra of $g-C_3N_4$ (a) and Gn/SO_3H (b)





Fig. 4 TGA images of g-C₃N4 (a) and GN/SO₃H (b)

was started by increasing the temperature from 550 °C and completed at 600 °C (Fig. 4a). In the GN/SO₃H, the decomposition temperature increased to 650 °C, confirming that $-SO_3H$ has tight contact with g-C₃N₄ (Fig. 4b).

The EDX analysis was used for the distribution of elements in GN/SO_3H (Fig. 5). It was found that the percentage of C, N, O, and S are 31.37, 62.53, 5.58, and 0.52, respectively. The presence of S and O atoms confirm the successful synthesis of the composite.



Fig. 5 The EDX pattern of GN/SO₃H

Catalytic activity

A control experiment consisted of 1 mmol of isatin, malononitrile and 1,3cyclohexadione, confirming that in the absence of the catalyst only a trace amount of the corresponding product was detected (Table 1, entry 1). An additional experiment containing 1 mmol of isatin, malononitrile and 1, 3-cyclohexadione in the presence of $g-C_3N_4$ was conducted and the same results were obtained (Table 1, entry 8). The mixture was then heated at reflux conditions for 1 h and the reaction was not progressed. Then, a model reaction consisting of 1 mmol of isatin, malononitrile and 1, 3-cyclohexadione was selected for the catalytic activity of GN/ SO_3H . Catalyst in the amount of 0.04 g was dispersed in ethanol/water (1:1) and 1 mmol of each reactant was added to the mixture and heated at room temperature for 80 min; the prepared product was obtained in 20% yield. This model reaction was repeated at reflux conditions for the mentioned time and 58% product was isolated (Table 1, entry 2). These results showed that GN/SO₃H was essential for the progress of the reaction at reflux conditions. Then, the effect of the amount of catalyst was investigated on the yield of the reaction at reflux conditions and the results showed that 0.08 g of catalyst is the optimum condition for the model reaction (Table 1, entry 5). Further increase in the amount of catalyst did not show a considerable increase in the yields nor decrease the time of the reaction (Table 1, entries 6 and 7). Due to the important effect of solvent on the progress of the reactions, the progress of the reaction was investigated using different nonpolar, medium and high polar solvents; the mixture of ethanol and water (1:1) was chosen as the best solvent in terms of the yield and time of the model reaction (Table 2, Entry 10).

After the reaction parameters were conditioned, the generality of the method for the production of spirooxindoles was investigated. Three types of ketone consisting of 1,3-cyclohexadione, dimedone and aldehyde derivatives, as well as 1, 4-hydroxycoumarin, were reacted with isatin and malononitrile; the results are

Table 1 The effect of different amount of GN/SO ₃ H	Entry	GN/SO ₃ H (g)	Time (min)	Yield ^a
	1	_	80	Trace
	2	0.04	80	58
	3	0.06	50	90
	4	0.07	40	90
	5	0.08	40	95
	6	0.1	40	97
	7	0.11	40	98
	8 ^b	0.1	80	Trace

Isatin (1.0 mmol), malononitrile (1 mmol), 1,3-cyclohexadione (1 mmol), ethanol/water (1:1) (3 mL). Reflux conditions

^aIsolated yields

^bg-C₃N₄ as catalyst

 Table 2
 The effect of solvent

 on the yield of spirooxindoles

Entry	Solvent (3 mL)	Time (min)	Yield (%)
1	<i>n</i> -Hexane	120	18
2	CH ₂ Cl ₂	55	47
3	THF	50	60
4	CHCl ₃	50	40
5	CH ₃ CN	60	75
6	EtOAc	60	61
7	MeOH	60	77
8	EtOH	60	75
9	H ₂ O	60	49
10	EtOH/water (1:1)	40	95

Isatin (1.0 mmol), 1,3-cyclohexadione (1 mmol), malononitrile (1 mmol), GN/SO₃H (0.08 g). Reflux conditions

displayed in Table 3. Also, the effect of isatin derivatives was investigated. When 1,3-cyclohexadione and malononitrile were reacted with different types of isatins, the results showed the substituent groups of the benzene ring of isatin (electronneutral, electron-rich or electron-poor groups) have no considerable effect on the yield of the reaction, and the desired products were produced in high yields (Table 3, entries 1–9, 90–98%). By using dimedone instead of 1,3-cyclohexadione, the progress of the reaction was slightly increased (Table 3, entries 10-13). In the case of 4-hydroxycoumarin as a substrate, the reactions were proceeded more slowly and lower yields were observed, which may be due to the lower activity of carbonyl group of 4-hydroxycoumarin (Table 3, entries 14-22). The effect of benzaldehyde derivatives was also investigated in the reactions; the results showed that the yields and times of the reactions were emphasized, and longer times with lower yields were observed (Table 3, entries 23-27). The efficiency of the catalyst was compared with those reported in the literature [26-33]. As can be seen in Table 4, this catalyst showed good catalytic activity and excellent yields (95%) in shorter times compared to most of the reported works (Table 4, entries 1-5).

In addition, a reasonable mechanism for preparation of spirooxindole derivatives was introduced (Scheme 2). GN/SO₃H can activate the carbonyl group of isatin, and after that malononitrile, nucleophilic attack to activated carbon generate intermediate 2. This intermediate generates intermediate 3 via elimination by Konevenagel condensation; after addition of dimedone to this intermediate the corresponding product was synthesized [34].

Reusability of the catalyst is important for industries. Therefore, after each experiment, the nanocomposite was separated and applied for another reaction and, as the results showed, no considerable decrease in the yields were observed even after 10 cycles, which confirms the stability and efficiency of the catalyst (Fig. 6).

Entry	Product	Substrate 1	Substrate 2	Time (min)	Yield (%)
1	H ₂ N NC NC NC NC NC NC NC NC NC NC NC		O N H	40	95
2	CI NC O			40	92
3	O ₂ N NC O NC O H O			40	95
4	H ₂ N MeO_NC_O_O H_OO		MeO MEO MEO	40	90
5	Me NC			40	97
6	H ₂ N O ₂ N NC NC O			40	95
7	Br NC NC		Br C S S S S S S S S S S S S S S S S S S	40	95
8	Br, NC NC NC Me		Br C N N N N N N N N N N N N N N N N N N	40	95
9	H ₂ N NC NC NC NC NC O NC O NC O NC O NC		O N Ph	40	91

Table 3 Preparation of oxindole derivatives

Entry	Product	Substrate 1	Substrate 2	Time (min)	Yield (%)
10	H ₂ N NC NC NC NC NC NC NC NC NC NC NC			40	95
11	H ₂ N O ₂ N NC N O O			40	93
12	CI NC			40	91
13	NC - O NC - O H O			40	95
14	NH ₂ CN O NH	OH OH O O O		60	85
15	NH ₂ CN O O O O CI	OH OH		60	86
16	NH ₂ O O O O O O NH	OH OH O O O		60	75
17	NH ₂ CN CN N Ph	OH OH O O O		60	88

Table 3 continued

Entry	Product	Substrate 1	Substrate 2	Time (min)	Yield (%)
18	NH ₂ O O O O O O O O O	OH CCCO 0		60	70
19	NH ₂ NH NH MeO	OH OH OH		60	85
20	NH ₂ O O O O Me	OH OH		60	85
21	NH2 N-Me Br	OH OH	Br	60	75
22	NH2 CN O O O Br	OH OH		60	83
23				120	89
24		н	H C N	120	90

Table 3 continued

Table 3 continued



Reaction condition: Isatin (1 mmol), malononitrile (1 mmol), dimedone (1 mmol)

Entry	Catalyst	Solvent/ T (°C)	Time (min)	Yield (%)	References
1	Sodium stearate	H ₂ O/60 °C	180	95	[26]
2	TEBA	H ₂ O/90 °C	120	88	[27]
3	AIUM (10 mol%)	DMSO/60 °C	120	40	[28]
4	B-CD	H ₂ O/60 °C	480	88	[29]
5	DES (0.5 mL)	Urea: CHCl ₃ /80 °C	240	88	[30]
6	Ni NPS	Ethylene glycole	20	88	[31]
7	$SBA-PR-NH_2$	H ₂ O/ref.	5	80	[32]
8	NET ₃	EtOH/ref.	30	80	[33]
9	GN/SO ₃ H	EtOH/water (1:1), ref.	40	95	This work

Table 4 The comparison of the efficiency of GN/SO₃H with different catalysts

Isatin (1 mmol), malononitrle (1 mmol), dimedone (1 mmol)

Conclusions

In summary, we easily synthesized GN/SO_3H nanocomposite by immobilization of chlorosulfonic acid onto the g-C₃N₄ and characterized the nanocomposite by FT-IR, TGA, XRD, EDX, and TEM. To study the catalytic activity of as-prepared nanocomposite, the reactions of isatin derivatives with malononitrile and different carbonyl compounds were studied using GN/SO_3H via one-pot, three-component reaction at reflux conditions. The results showed that the products were produced at



Scheme 2 The reasonable mechanism for the synthesis of spirooxindole derivatives



Fig. 6 The reusability of GN/SO₃H for the synthesis of spirooxindole derivatives

excellent yields in a short time. Compared to 1,3-cyclohexadione and dimedone, 4-hydroxycoumarin gave lower yields in a longer time. Finally, this protocol has some advantageous including easy recovery and reusability and stability of the catalytic system, with eco-friendly as well as excellent yields.

Acknowledgements The authors are grateful from the University of Birjand for the financial support.

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