



Synthesis of benzodiazepines catalyzed by chitosan functionalized by triacid imide as a superior catalyst

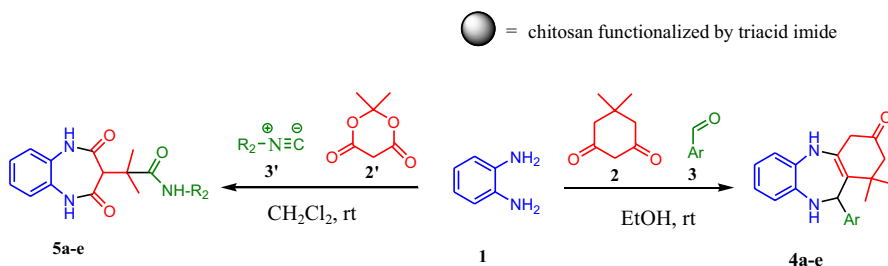
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Abstract

Chitosan functionalized by triacid imide has been applied as an effective catalyst for the synthesis of benzodiazepines by one-pot reactions of 1,2-phenylenediamine with dimedone and aryl aldehydes or isocyanides and Meldrum's acid. Chitosan functionalized by triacid imide was confirmed by FT-IR (infrared spectroscopy), XRD (X-ray diffraction), DSC (differential scanning calorimetry) and TGA (thermogravimetric analysis). To investigate the scope and limitation of this reaction, different aryl aldehydes and isocyanides were used as substrates. Good yields in short reaction times, experimental simplicity, extensive range of products, retrieval of the catalyst and low catalyst loading are some of the substantial aspects of this method.

Graphic abstract



Keywords Benzodiazepines · One-pot · Chitosan · Triacid imide

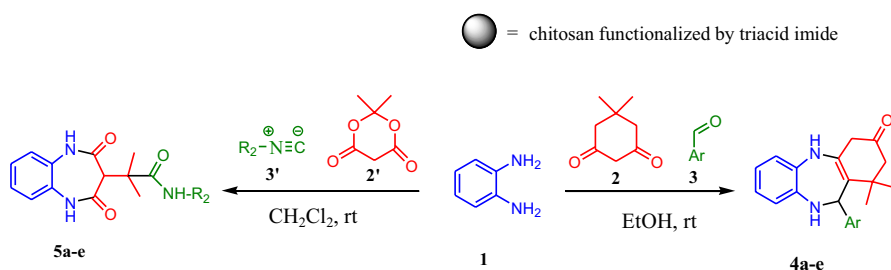
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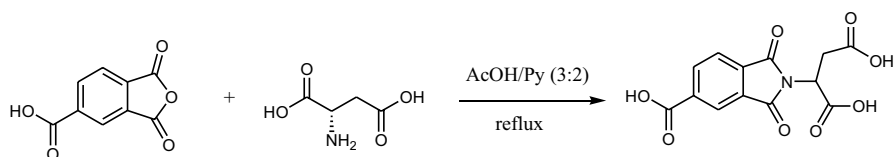
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Introduction

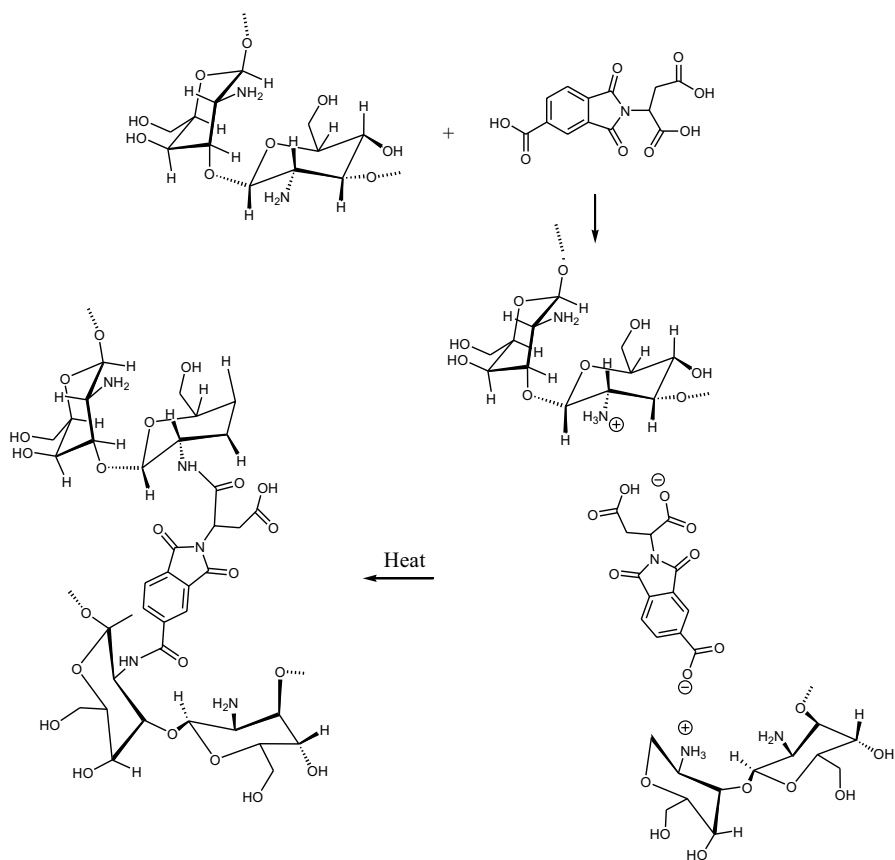
Benzodiazepines display anti-bacterial [1], anti-tumor [2], anti-anxiety [3], anti-AIDS [4] and anti-hepatic B virus [5] activities. The discovery of impressive methods for the preparation of benzodiazepines is a serious challenge. A number of ways have been improved for the synthesis of diazepines using mercury(II) trifluoromethanesulfonate [6], organic acid [7], ytterbium(III) trifluoromethanesulfonate [8], ceric ammonium nitrate [9] and scandium(III) trifluoromethanesulfonate [10]. Each of these procedures may have its own advantages but also suffer from such apparent drawbacks as prolonged reaction times, complicated work-up, low-yield or hazardous reaction conditions. The potential of chitosan-based structures to form open network has provided their use not only as macrochelating ligands, but also as a direct organocatalyst [11]. The chitosan can be applied as a support for the synthesis of new catalysts in the form of flakes, colloids, fibers, gel or functionalized on inorganic supports (alumina, titania, silica or other metal oxides) [12, 13]. Chitosan functionalized with sulfonic acid was utilized as a superior catalyst in the esterification of fatty acids with alcohol [14]. Amino and hydroxyl groups in chitosan make it suitable as an effective catalyst [15, 16]. Chitosan is a biopolymer that can be utilized as green catalyst in many reactions [17, 18]. Ideally, introducing neat processes and utilizing eco-friendly and green heterogeneous catalysts which can be simply recycled at the end of reactions have received substantial attention in recent years [19, 20]. The catalytic activity of chitosan was increased when chitosan was functionalized by triacid imide. Herein, we report the use of chitosan functionalized by triacid imide as an effective catalyst for the synthesis of diazepines by one-pot reactions of 1,2-phenylenediamine with dimedone and aryl aldehydes or isocyanides and Meldrum's acid at room temperature (Scheme 1). We found that chitosan functionalized by triacid imide produces our desired compounds in high yields (83–93%) with excellent recovery and simple work-up method. In addition, chitosan functionalized by triacid imide has a good recycling property and this advantage is significant from economic point of view.



Scheme 1 Synthesis of benzodiazepines catalyzed by chitosan functionalized by triacid imide



Scheme 2 Preparation of triacid imide



Scheme 3 Chitosan functionalized by triacid imide

Results and discussion

We prepared triacid imide according to Scheme 2. Chitosan was functionalized using triacid imide (Scheme 3).

XRD pattern of chitosan and chitosan functionalized by triacid imide is indicated in Fig. 1. The XRD pattern of chitosan and chitosan functionalized by triacid imide displayed two sharp peaks at 20.2 and 11.4 (2 Theta).

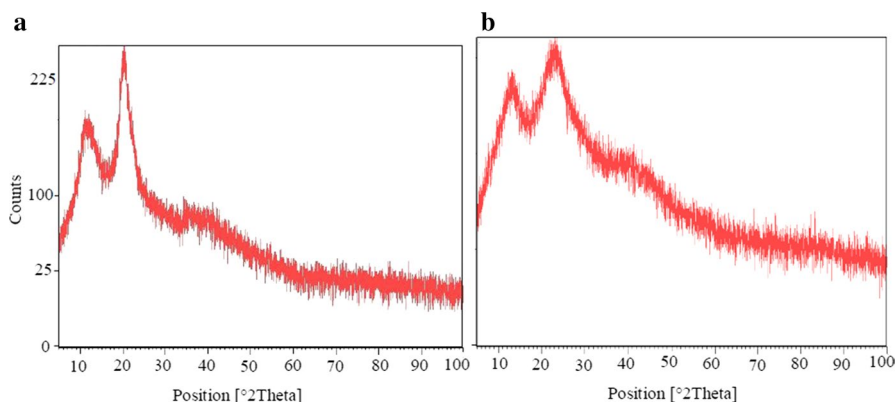


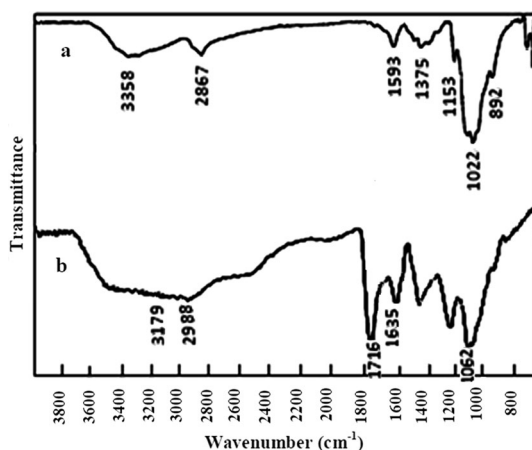
Fig. 1 XRD pattern of (a) chitosan and (b) chitosan functionalized by triacid imide

FT-IR spectra of chitosan and chitosan functionalized by triacid imide are indicated in Fig. 2. The peak at $2600\text{--}3400\text{ cm}^{-1}$ (Fig. 2b) is ascribed to the stretching vibrational absorptions of OH (COOH). The peak at 1715 cm^{-1} is ascribed to the stretching vibrational absorptions of carbonyl of acidic groups. The peaks at about $1615\text{--}1640\text{ cm}^{-1}$ are ascribed to the absorptions of the carbonyl of amide groups which are formed from the reaction of carboxyl group of the triacid imide with amino group of chitosan [21–23].

To investigate the degree of substitution of carboxylic acid groups of chitosan functionalized by triacid imide, a certain amount of the chitosan functionalized by triacid imide was soaked in 5 mL of 0.1 N NaOH solution. The excess of NaOH solution was titrated with 0.1 N HCl in the presence of phenolphthalein. Acidic capacity of this catalyst was estimated 2.1 mmol/gr.

The thermal properties of chitosan and chitosan functionalized by triacid imide were investigated by TGA/DTG (Fig. 3) and DSC (Fig. 4) at a heating rate of $10\text{ }^{\circ}\text{C/}$

Fig. 2 FT-IR spectra of (a) chitosan and (b) chitosan functionalized by triacid imide



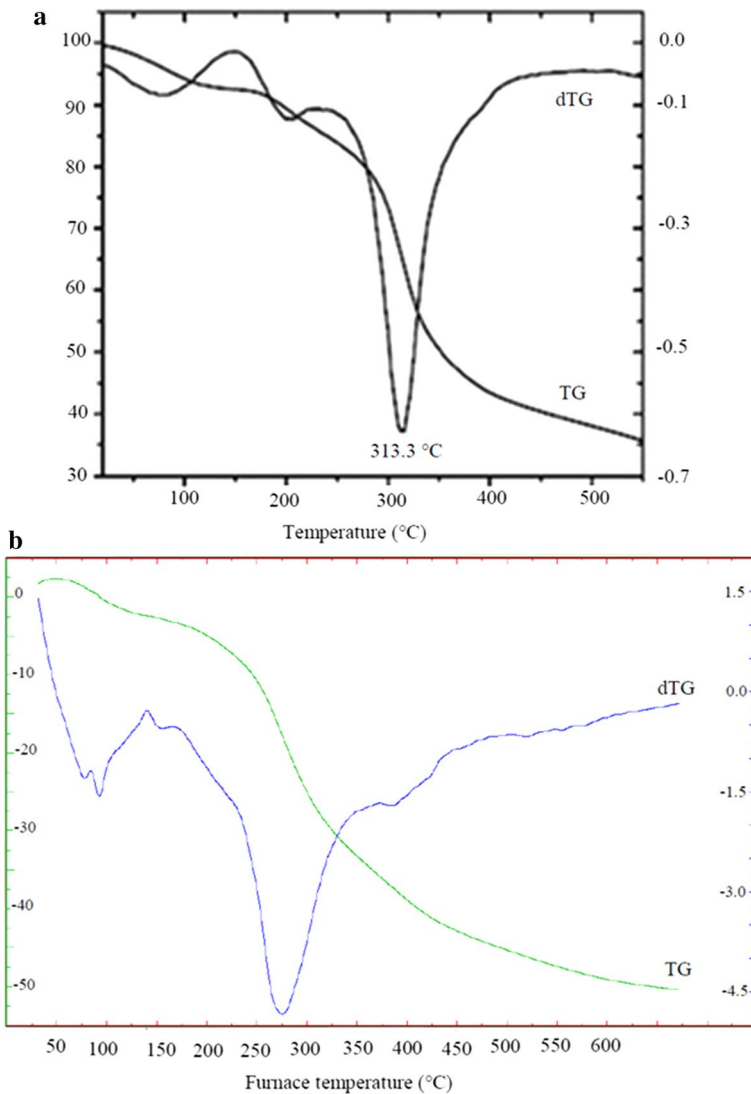


Fig. 3 TGA/DTG patterns of chitosan (a) and chitosan functionalized by triacid imide (b)

min under N_2 atmosphere. The weight loss at 40–250 °C for chitosan is owing to the moisture vaporization. The weight loss at 250–400 °C is owing to the degradation of chitosan. The weight loss at 40–250 °C for chitosan functionalized by triacid imide is owing to the removal of adsorbed solvent and surface hydroxyl groups. The weight loss at 250–550 °C is due to degradation of chitosan and triacid imide.

DSC of chitosan indicated two peaks, an endothermic broad peak at about 90–100 °C owing to dehydration of chitosan and another exothermic at about 305–310 °C peak due to exothermic decomposition of chitosan. The DSC of

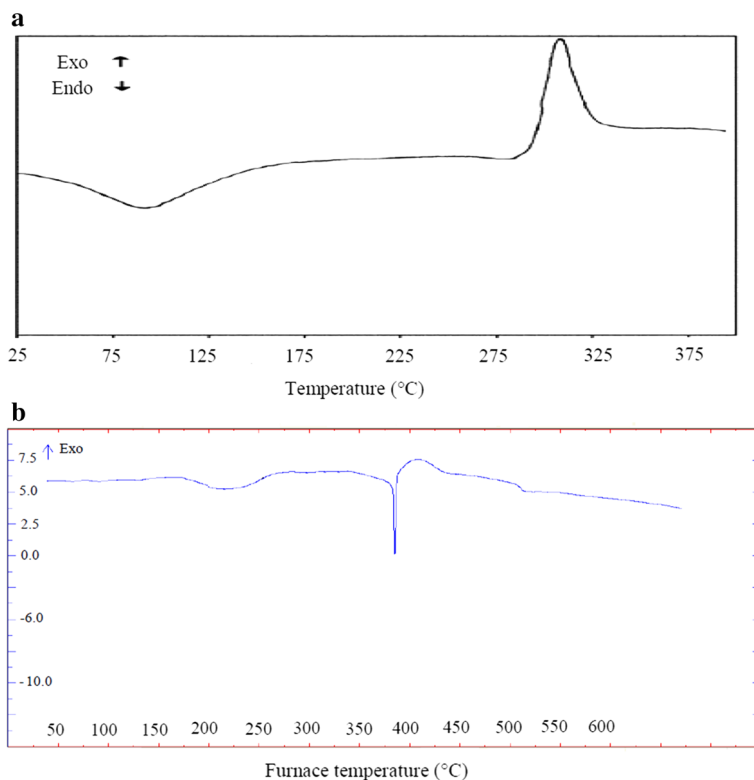


Fig. 4 DSC patterns of chitosan (**a**) and chitosan functionalized by triacid imide (**b**)

chitosan functionalized by triacid imide displayed a broad endothermic peak at about 200 °C, a couple of sharp endothermic peaks at about 390 °C and an exothermic peak at 420 °C. These results indicated that heat stability of chitosan functionalized by triacid imide is satisfactory.

We used the reaction of 1,2-phenylenediamine, 4-chlorobenzaldehyde and dime-done as a model reaction. Meanwhile, we used the reaction of 1,2-phenylenediamine, *tert*-butyl isocyanide and Meldrum's acid as a model reaction. The model reactions were performed using different catalysts including nano-MgO, Et₃N, *p*-TSA, chitosan, triacid imide, chitosan functionalized by triacid imide (Table 1). We found that the reaction gave convincing results in the presence of chitosan functionalized by triacid imide (7 mg) at room temperature. In further studies on the catalyst amount, we noticed that yields of compounds **4a** and **5a** remained almost the same when 9 mg of chitosan functionalized by triacid imide was used (Table 1). Use of lower catalyst amount (5 mg) afforded **4a** in 85% yield and **5a** in 82% yield. The catalyst showed the best activity for the synthesis of **4a** in ethanol and for the synthesis of **5a** in CH₂Cl₂ compared to other solvents such as CH₃CN, H₂O, DMF and CHCl₃. Further to this, we also applied different aryl aldehydes and isocyanides as substrates and found uniformly good results (Table 2).

Table 1 Optimization of reactions conditions using different catalysts

Entry	Catalyst	Solvent 4a/5a	Time (min) 4a/5a	Yield (%) 4a^a/5a^b
1	–	–	500/400	12/trace
2	MgO NPs (10 mg)	EtOH/CH ₂ Cl ₂	250/300	33/44
3	Triethylamine (15 mol%)	EtOH/CH ₂ Cl ₂	300/400	25/30
4	<i>p</i> -TSA (10 mol%)	EtOH/CH ₂ Cl ₂	200/250	49/58
5	Chitosan (10 mg)	EtOH/CH ₂ Cl ₂	180/250	53/62
6	Triacid imide (10 mg)	EtOH/CH ₂ Cl ₂	100/150	70/63
7	Chitosan functionalized by triacid imide (5 mg)	EtOH/CH ₂ Cl ₂	40/60	85/82
8	Chitosan functionalized by triacid imide (7 mg)	H ₂ O/DMF	100/120	64/60
9	Chitosan functionalized by triacid imide (7 mg)	CH ₃ CN/ CHCl ₃	60/90	70/75
10	Chitosan functionalized by triacid imide (7 mg)	EtOH/CH ₂ Cl ₂	40/60	93/90
11	Chitosan functionalized by triacid imide (9 mg)	EtOH/CH ₂ Cl ₂	40/60	93/90

^aIsolated yield (reaction conditions: 1,2-phenylenediamine, dimedone and 4-chlorobenzaldehyde at room temperature);

^bIsolated yield (reaction conditions: 1,2-phenylenediamine, Meldrum's acid and *tert*-butyl isocyanide at room temperature)

After completion of the reaction, the catalyst was separated from the mixture by filtration. Then, the catalyst was washed with ethanol and dried in air. The reusability of the chitosan functionalized by triacid imide catalyst was tested for the preparation of **4a** and it was found that product yields lessened only to a very small extent on each reuse (run 1, 93%; run 2, 93%; run 3, 92%; run 4, 92%; run 5, 91%, run 6, 91%). This confirms that the catalyst is stable and can be reused multiple times.

To compare the efficiency of chitosan functionalized by triacid imide with the reported catalysts for the synthesis of diazepines, we have tabulated the results in Table 3. Our study has some advantages in comparison with other mentioned studies including high yield of synthetic compound, reasonable time reaction and easy catalyst recovery.

Scheme 4 shows a plausible mechanism for the synthesis of diazepines (**4a–e**). At first, intermolecular imine formation from dimedone and 1,2-phenylenediamine promoted by the chitosan functionalized by triacid imide occurs. The amino group of 1,2-phenylenediamine attacks the C=O of dimedone with elimination of H₂O to form imine intermediate **I**. A 1,3-hydrogen shift results in isomeric (tautomerized) to give enamine **II**. The other amino group of the 1,2-phenylenediamine part of enamine **II** attacks the carbonyl group of aldehyde, which is itself activated by the chitosan functionalized by triacid imide to form intermediate **III**. The seven-membered ring products **4a–e** are formed via intramolecular cyclization of **III** [24].

Scheme 5 shows a plausible mechanism for the synthesis of diazepines (**5a–e**). Intermediate **II** was formed from condensation reaction between

Table 2 Synthesis of benzodiazepines catalyzed by chitosan functionalized by triacid imide (7 mg)

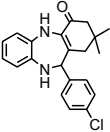
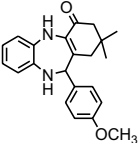
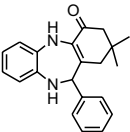
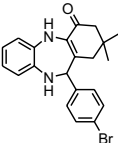
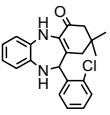
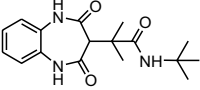
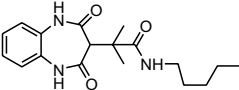
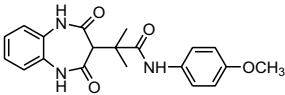
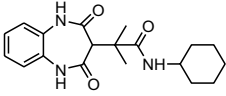
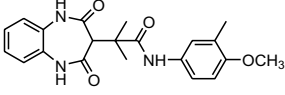
Entry	Product	Time, min	Yield (%) ^a	M. p. (°C)	M. p. (°C) [references]
1	 4a	40	93	233–235	237–238 [24]
2	 4b	60	83	224–226	229–231 [25]
3	 4c	50	87	240–242	246–248 [25]
4	 4d	40	93	235–237	241–242 [25]
5	 4e	40	91	230–232	232–233 [24]
6	 5a	60	90	> 300	> 300 [26]
7	 5b	70	86	286–288	286–288 [27]

Table 2 (continued)

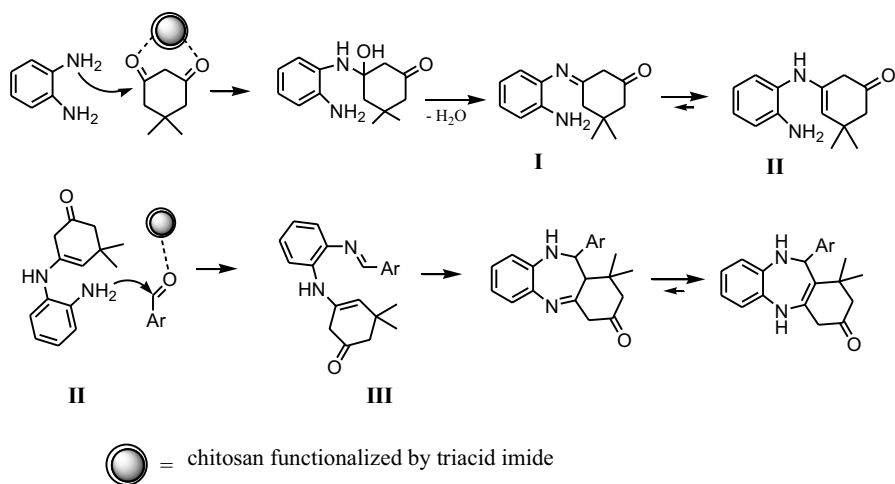
Entry	Product	Time, min	Yield (%) ^a	M. p. (°C)	M. p. (°C) [references]
8	 5c	60	90	305–307	306–308 [27]
9	 5d	70	87	> 300	> 300 [26]
10	 5e	60	90	280–282	–

^aIsolated yield**Table 3** Comparison of catalytic activity of chitosan functionalized by triacid imide (7 mg) with other reported catalysts

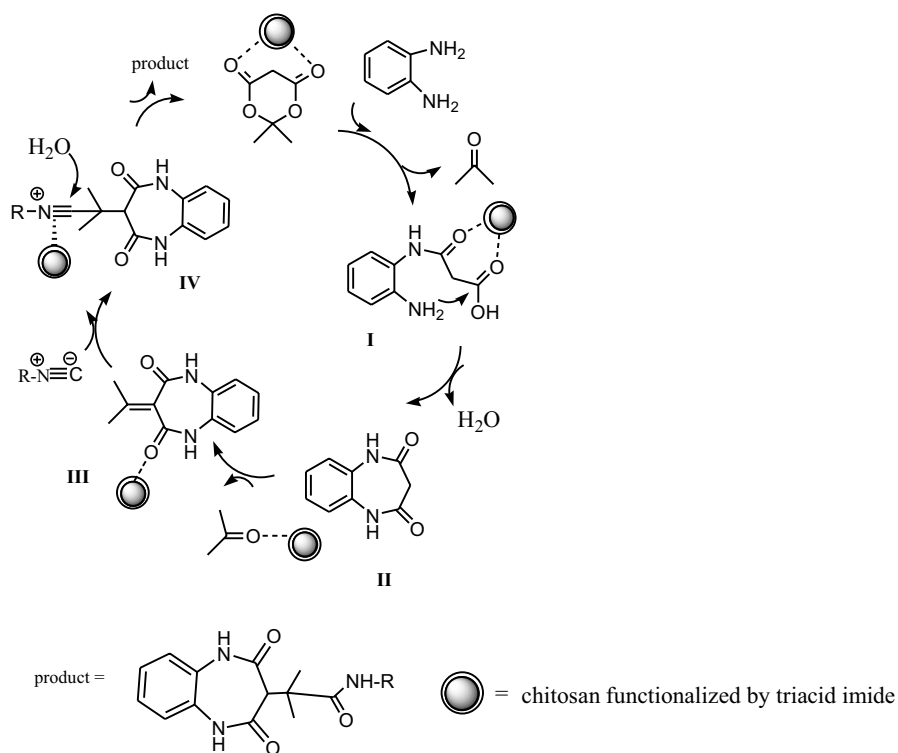
Entry	Catalyst (condition)	Time (min)	Yield ^a , %	[References]
1	Fe ₃ O ₄ @chitosan (EtOH, 0.03 g)	120	84	[24]
2	Acetic acid (10 mol%, reflux, ethanol)	60	61	[28]
3	H ₂ SO ₄ (EtOH, 15 mol%)	180	70	[29]
4	Oxalic acid (H ₂ O, reflux, 40 mol%)	120	88	[30]
5	Chitosan functionalized by triacid imide (EtOH, 7 mg)	40	93	This work

^aIsolated yield

1,2-phenylenediamine and Meldrum's acid. The acid groups distributed on the surface of chitosan functionalized by triacid imide activate the carbonyl groups of the Meldrum's acid and intermediate **I**. Afterward, the intermediate **II** under a condensation reaction with in situ liberated acetone produces the intermediate **III**. Afterward, isocyanides react with α,β -unsaturated carbonyl compound (intermediate **III**) to create intermediate **IV** (Michael-type addition reaction), followed by nucleophilic attack of a H₂O molecule on the nitrilium moiety to produce the diazepines (5a-e) [26]. Here, chitosan functionalized by triacid imide activates the $C\equiv N^+$ and $C=O$ groups for better reaction with nucleophiles.



Scheme 4 Proposed reaction pathway for the synthesis of **4a–e**



Scheme 5 Proposed reaction pathway for the synthesis of **5a–e**

Conclusions

In conclusion, we have reported as an effective method for the synthesis of benzodiazepines by one-pot reactions of 1,2-phenylenediamine with dimedone and aryl aldehydes or isocyanides and Meldrum's acid at room temperature. Chitosan functionalized by triacid imide was confirmed by FT-IR (infrared spectroscopy), XRD (X-ray diffraction), DSC (differential scanning calorimetry) and TGA (thermogravimetric analysis). The current method provides obvious positive points containing easy work-up, extensive range of products, retrieval of the catalyst and low catalyst loading.

Experimental section

Chitosan with average molecular weight of 100,000–300,000 and 70–85% degree of acetylation was prepared from Sigma–Aldrich. NMR spectra were obtained on Bruker Avance-400 MHz spectrometer. IR spectra were performed on a Tensor 27 instrument. TGA was obtained on a Mettler TA4000 instrument with a heating rate of 10 °C min⁻¹ under N₂ atmosphere. DSC was done on a Shimadzu DSC-50 instrument under N₂ atmosphere. Wide-angle X-ray diffraction measurements were recorded on a Bruker-D8 X-ray diffractometer.

Preparation of triacid imide

Into a 25-mL round-bottom flask, 1.92 g of 1,3-dihydro-1,3-dioxoisobenzofuran-5-carboxylic acid, 1.33 g of *L*-aspartic acid and 10 mL mixture of acetic acid/pyridine (3:2) were placed. The mixture was stirred at room temperature for 2 h, and it was refluxed for 6 h. The solvent was evaporated under reduced pressure. Five milliliters of concentrated HCl was added to mixture. A white precipitate was formed, filtered off and dried, to give triacid imide [21, 22].

2-(1,2-dicarboxyethyl)-1,3-dioxoisindoline-5-carboxylic acid: m.p. 270–272 °C. FT-IR (KBr, ν_{max} /cm⁻¹): 3600–2400 (COOH), 1728–1700 (C=O), 1388, 1299, 1200, 1106, 991, 728. ¹H NMR (400 MHz, DMSO-*d*₆): δ 8.62–8.95 (m, 3H), 5.21 (CH, 1H), 4.79 (3 OH, exchanged with water of DMSO-*d*₆), 2.54–2.72 (m, 2H) ppm. Anal. Calcd for C₁₃H₉NO₈: C, 50.82; H, 2.95; N, 4.56; Found: C, 50.74; H, 2.87; N, 4.48.

Preparation of chitosan functionalized by triacid imide

0.85 g (2.38 mmol) of chitosan was dissolved in 100 mL of 2.0 wt% acetic acid solution. 0.365 g (1.19 mmol) of triacid imide was added to solution. The mixture was stirred at room temperature for 8 h, and it was placed for 10 h at 80 °C under

stirring. The mixture was washed several times with deionized water and ethanol and then dried in an oven.

General method for the synthesis of benzodiazepines (4a–e)

A mixture of 1,2-phenylenediamine (1 mmol) dimedone (1 mmol), aryl aldehyde (1 mmol) and chitosan functionalized by triacid imide (7 mg) was stirred in ethanol (10 mL) at room temperature. After completion of the reaction, (monitored by TLC), the catalyst was separated from the mixture by filtration. The solvent was removed under vacuum, and the solid obtained was rinsed with *n*-hexane and EtOAc (7:3) to afford pure benzodiazepine.

General procedure for the preparation of benzodiazepines (5a–e)

Chitosan functionalized by triacid imide (7 mg) was added to a mixture of 1,2-phenylenediamines (1 mmol), isocyanide (1 mmol) and Meldrum's acid (1 mmol) in 5 mL CH₂Cl₂ at room temperature. After completion of the reaction, (monitored by TLC), the catalyst was separated from the mixture by filtration. The solvent was evaporated under vacuum, and the solid obtained was washed several times with water and ethanol to afford pure benzodiazepine.

3-Dimethyl-2,3,4,5,10,11-hexahydro-11-[(4-chloro)phenyl]-1H-dibenzo[*b,e*][1,4]diazepin-1-one (4a)

Green solid, M. p. 233–235 °C; FT-IR (KBr): ν =3304, 3238, 3056, 2958, 1582, 1385, 1535, 1328, 1426, 1273 cm⁻¹; ¹H NMR (400 MHz, DMSO-*d*₆): δ (ppm)=1.03 (s, CH₃, 3H), 1.08 (s, CH₃, 3H), 2.15 (A.Bq, CH₂, *J*=16.0 Hz, 2H), 2.58 (s, CH₂C=O, 2H.), 4.97 (s, NH, 1H.), 6.08 (s, 1H, CH), 6.47 (d, *J*=8.2 Hz, Ar, 1H), 6.55 (m, Ar, 3H), 6.84 (d, *J*=8.2 Hz, Ar, 1H), 6.98 (d, *J*=8.4 Hz, Ar, 2H), 7.02 (d, *J*=8.4 Hz, Ar, 1H), 8.63 (s, NH, 1H.); ¹³C NMR (100 MHz, DMSO-*d*₆): δ (ppm)=28.17, 28.68, 32.24, 43.86, 49.92, 56.45, 108.35, 120.63, 120.74, 121.06, 122.12, 124.28, 127.35, 130.44, 137.08, 144.05, 150.03, 152.05, 192.83.—Analysis for C₂₁H₂₁ClN₂O: calcd. C 71.48, H 6.00, N 7.94; found C 71.56, H 6.08, N 7.99.

N-tert-butyl-2-(2,3,4,5-tetrahydro-2,4-dioxo-1H-benzo[*b*][1,4]diazepin-3-yl)-2-methyl propanamide (5a)

White powder, M. p.>300 °C; FT-IR (KBr): ν =3385, 2965, 2903, 1693, 1645, 1603, 1528 cm⁻¹; ¹H NMR (400 MHz, DMSO-*d*₆): δ (ppm)=1.21(9H, s, C(CH₃)₃), 1.37 (s, 2CH₃,6H), 3.17 (s, CH,1H), 6.15–7.15 (bs, 4H-Ar and -NH, 5H), 10.37 (bs, 2NH, 2H); ¹³C NMR (100 MHz, DMSO-*d*₆): δ (ppm)=23.14, 27.95, 42.64, 50.45, 55.12, 121.43, 125.42, 131.34, 168.15, 177.32.—Analysis for C₁₇H₂₃N₃O₃: C 64.33, H 7.30, N 13.24, Found: C 64.25, H 7.27, N, 13.15.

Supporting information

NMR spectra of products are presented in Supporting Information available online.

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