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ORIGINAL PAPER

Phthalimide-*N*-sulfonic acid, an efficient catalyst for the synthesis of various isoindoline-1,3-dione derivatives

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Abstract An environmentally friendly method is described for the synthesis of various isoindoline-1,3-dione derivatives from the reaction of phthalic anhydride with aromatic/aliphatic amines in ethanol at 80 °C by phthalimide-*N*-sulfonic acid as an efficient heterogeneous acid catalyst. Some advantages include the metal-free and environmentally friendly protocol, simple operation and reusable processes, easy recovery, short reaction times, and high yields.

Keywords Phthalic anhydride · Amines · Phthalimide-*N*-sulfonic acid · Isoindoline-1,3-diones · Heterogeneous acid catalyst

Introduction

Many organic reactions such as acylation (Anderson and Tepe 2002), isomerization (McCubbin et al. 2011), alkylation (Jervis et al. 2006), nitration (Aridoss and Laali 2011), and rearrangements like Beckman (Hashimoto et al. 2008) were catalyzed by acid catalysts. Therefore, acid catalysts play important roles in organic transformations. Most of these reactions were carried out by employing the conventional mineral acids like HCl, HNO_3, and H_2SO_4 or Lewis acids such as $AlCl_3$ and BF_3, but the conventional catalysts are replaced with the newer acid catalysts because of the environmental and economical reasons (Gu et al. 2003).

Organocatalytic reactions were found to be an efficient synthetic tool for creating various new carbon–carbon and carbon–heteroatom bonds (Kiyani and Ghiasi 2015) and are good alternative to other catalyzed processes because of their lower costs and benign environmental impact (Diez-Gonzalez et al. 2007). Other advantages of the organocatalysts are their availability, relatively low toxicity, simple functionality, and non-sensitivity to air and moisture. They bring a metal-free environment to promote a wide range of chemical transformations via the various activation modes (Nyce et al. 2002; Grasa et al. 2004, 2003; Wu et al. 2006; Reynolds and Rovis 2005; Suzuki et al. 2003; Nair et al. 2006; Tachibana et al. 2004; Ciganek 1995; Zhou et al. 2001).

N-Substituted cyclic imides have successfully been used in syntheses (Bouissane et al. 2009; Easwar and Argade 2006; Mangaleswaran and Argade 2004), medicine (Janda et al. 1990), biology (Sortino et al. 2011), and material science (Pontrello et al. 2005) by application of different reagents such as $[Bmim]PF_6$ (Le et al. 2004), Ac_2O/NaOAc (Mehta et al. 1960), and others (Peterson and Eggleston 2007). There are many methods for the synthesis of cyclic imides using acetyl chloride (Dhivare et al. 2016), acetic acid (Pan et al. 2016), $SOCl_2$ (Rajput and Rajput 2007), and $ZnBr_2$/HMDS (Reddy et al. 1997) catalysts. In addition, several methods were reported for preparation of phthalimides, especially *N*-phenyl phthalimide by the use of $[Hmim]HSO_4$ (Dabiri et al. 2007), $[bmim]PF_6$ (Zhou et al. 2003), choline chloride + $ZnCl_2$ (Xie et al. 2009), TFA (Shinde et al. 2011), sulphamic acid (Langade 2011), Fe_3O_4@SiO_2–SO_3H (Thale et al. 2014), and DABCO (Heravi et al. 2005) catalysts.

Since cyclic imides are well known to be very important antitumor agents such as mitonafide and amonafide,

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Sharma and coworkers synthesized two series of cyclic imide derivatives containing two cyclic imide moiety in their structures (one series of bis-cyclic imide derivatives synthesized by condensation of acid anhydrides with diamines and another series synthesized by condensation of various diamines with diacids), and screened them for in vitro anticancer activity against five human cancer cell lines, i.e., breast (T47D), lung (NCl H-522), colon (HCT-15), ovary (PA-1), and liver (Hep G2) (Kumar et al. 2016). Guo and coworkers reported an efficient synthesis of *N*-substituted phthalimides from *o*-phthalic acids or anhydrides with amines using SiO_2-tpy-Nb as heterogeneous and reusable catalyst (Wan et al. 2016). Shimizu and coworkers reported direct synthesis of cyclic imides from carboxylic anhydrides and amines by Nb_2O_5 as a water-tolerant Lewis acid catalyst (Ali et al. 2016). Kumar and coworkers used *p*-toluenesulfonic acid as catalyst for the three steps synthesis of *N*-substituted cyclic imides from the reaction of phthalic, maleic, and succinic anhydride with various Ar-NH_2 in solid phase (Kumar et al. 2014). Langade reported an efficient one-pot synthesis of *N*-alkyl and *N*-aryl phthalimides and succinimides using 10 mol % sulphamic acid catalyst (Langade 2011).

For many years, synthesis of the cyclic imides, especially isoindoline-1,3-dione and its derivatives, has been of remarkable interest in organic and medicinal chemistry, because these heterocyclic nuclei are in a large number of drugs and natural products and also their broad range of applications as analgesics, anticonvulsants, anti-inflammatories, immuno-modulatory, herbicidal, insecticidal agents, and hyperlipidemic activities (Ahuja et al. 2014; Dunn 2013; Al-Azzawi and Al-Razzak 2013; Sharma et al. 2010).

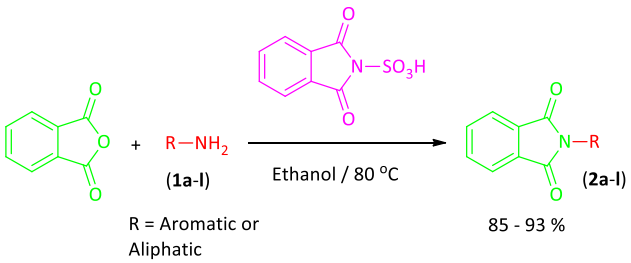
Former methods suffer from some drawbacks such as the use of expensive and hazardous reagents, application of non-green reagents, low yields, long reaction times, harsh reaction conditions, and tedious work-up (Braish and Fox 1992).

Herein, we would like to report the application of phthalimide-*N*-sulfonic acid as a highly efficient and reusable heterogeneous acid catalyst for the synthesis of various isoindoline-1,3-dione compounds **(2a–l)** via the condensation reaction of phthalic anhydride with various aliphatic and aromatic amines **(1a–l)** in ethanol at 80 °C (Scheme 1).

Experimental

General procedure for preparation of the phthalimide-*N*-sulfonic acid catalyst

Phthalimide-*N*-sulfonic acid catalyst was prepared according to the literature (Kiyani and Ghiasi 2015). Briefly, a flask was charged with a solution of potassium phthalimide (3.704 g, 20 mmol) in anhydrous CH_2Cl_2 (15 mL), and then, chlorosulfonic acid (2.33 g, 20 mmol) was added dropwise over a period of 20 min at 0 °C. After completion, the reaction mixture was filtered off; the solid residue washed with water and diethyl ether and dried under vacuum; the yield was about 92%.



Scheme 1 Phthalimide-*N*-sulfonic acid catalyzed synthesis of isoindolinediones

General procedure for the synthesis of isoindoline-1,3-dione derivatives

A mixture of phthalic anhydride (0.296 g, 2 mmol), amine (2.2 mmol), and phthalimide-*N*-sulfonic acid (0.091 g, 20 mol%) in ethanol (5 ml) was stirred for 3 h at 80 °C. After completion (monitored by TLC), the insoluble catalyst was separated by filtration, washed with acetone, and dried (weight: 0.090 g, 99% recovered). The organic layer was concentrated under reduced pressure to give the desired product, washed with water, and recrystallized in ethanol (each product was dissolved in a minimum amount of hot ethanol and allowed to reach gradually to room temperature). In addition, some products needed extra purification step by small column chromatography using the CH_2Cl_2/*n*-hexane solvent mixture (50:50).

Isoindoline-1,3-dione derivatives were characterized by comparing their IR and ^{1}H-NMR spectra with those of corresponding authentic samples.

Results and discussion

In the synthesis of various isoindoline-1,3-dione derivatives, the role of solvents on the reaction rate as well as the product yield was initially investigated by screening different solvents (DMF, DMSO, toluene, and ethanol) in model reaction [synthesis of 2-phenyl-isoindoline-1,3-dione (**2a**) from the reaction of phthalic anhydride with aniline in ethanol at 80 °C by phthalimide-*N*-sulfonic acid]. Results showed that the reaction rate is highly efficient in ethanol with excellent yield (3 h, 92%) (Table 1).

Optimization of the catalyst amount was the next step, so different amounts of phthalimide-*N*-sulfonic acid (0.0, 5.0, 10.0, 15.0, 20.0, and 25 mol%) were used in the model

Table 1 Effect of different solvents for the synthesis of isoindoline-1,3-diones

Solvent	DMF	DMSO	Toluene	Ethanol
Rate %	61	55	75	92
Time (hour)	4	4	4	3

Table 2 Effect of different catalyst amount on the synthesis of isoindoline-1,3-diones

Catalyst (mol %)	0.0	5.0	10.0	15.0	20.0	25.0
Yield %	29	46	70	88	92	92
Time (hour)	5	5	5	4	3	3

reaction to find the best condition. According to the obtained results, in terms of both time and yield, a 20 mol% of the catalyst was found to be the most effective (3 h, 92%), although the same result was obtained with more loading of the catalyst (Table 2).

To assess the efficiency of the applied methodology in the synthesis of various isoindoline-1,3-diones, several aliphatic and aromatic amines (aromatic amines with electron releasing and electron-withdrawing substituents) were tested with phthalic anhydride in the optimal reaction conditions to give the corresponding products in high yields and in short reaction times (Table 3).

Aliphatic amines showed high yields and short reaction times in comparison with the aromatic ones (Entries 2, 6, and 12). This observation can lead us that the aliphatic amines have more nucleophilic power, since the resonance of the nitrogen atom with the benzene ring reduces the nucleophilicity of the aromatic amines.

In addition, we investigated the reactivity of some bulky amines such as 2-methylaniline, 2,4-dimethylaniline, and 2,6-dimethylaniline (Entries 5, 10, and 11), and interestingly, all desired products were obtained in good-to-excellent yields and times. 4-Aminomethylpyridine also gave the good result (Entry 12).

Catalyst recyclability

In addition, the recyclability of the phthalimide-*N*-sulfonic acid catalyst was examined. Therefore, after completion of a model reaction [aniline (0.186 g, 2.2 mmol), phthalic anhydride (0.296 g, 2 mmol) and phthalimide-*N*-sulfonic acid catalyst (20 mol%) in ethanol at 80 °C], the catalyst was collected and directly reused for the next reaction runs. Consequently, we found that phthalimide-*N*-sulfonic acid is a capable and reusable catalyst even after five runs; and the catalytic activity was almost as same as the fresh one (92, 91, 89, 88, and 86%, respectively).

Mechanism of the reaction

The plausible mechanism for the synthesis of isoindoline-1,3-diones is shown below (Scheme 2). At first, carbonyl groups of phthalic anhydride will be activated by phthalimide-*N*-sulfonic acid, and then, nucleophilic attack of NH_2 group of amine to this activated carbonyl groups takes place to give the relative intermediate. By dehydration of the intermediate and another nucleophilic attack of the NH group, ring closing performs and the desired isoindoline-1,3-dione obtained (Reddy et al. 1997).

Comparison of the catalytic activities of different catalysts

A number of synthetic methods have been reported for preparation of various isoindoline-1,3-dione derivatives (Table 4), and application of the phthalimide-*N*-sulfonic acid catalyst (Entry 9) compared with those methods reported in the literature to show its advantages.

Selected spectral data of the products

N-(Phenyl)isoindoline-1,3-dione (**2a**): IR (in cm^{-1}): 3052, 1781, 1732, 1701, 1594, 1503, 1382, 1111; ^1H NMR (δ in ppm, $CDCl_3$) (90 MHz): 7.83–7.92 (4H, m, aromatic CH); 7.27–7.46 (5H, m, aromatic CH).

(2-Benzyl)isoindoline-1,3-dione (**2b**): IR (in cm^{-1}): 3061, 2951, 1765, 1715, 1432, 1391; ^1H NMR (δ in ppm, $CDCl_3$) (90 MHz): 7.53–7.80 (4H, m, aromatic CH); 7.16–7.35 (5H, m, aromatic CH), 4.83 (2H, s).

(4-Methoxyphenyl)isoindoline-1,3-dione (**2c**): IR (in cm^{-1}): 2926, 2851, 1717, 1389, 1258, 1176, 1024; ^1H NMR (δ in ppm, $CDCl_3$) (90 MHz): 7.82–7.90 (4H, m, aromatic CH); 6.97–7.37 (4H, m, aromatic CH), 3.85 (3H, s).

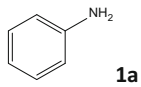
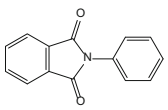
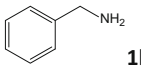
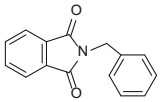
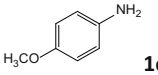
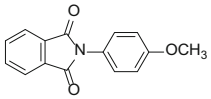
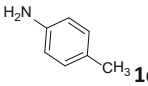
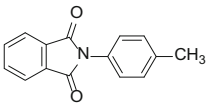
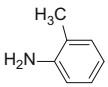
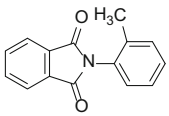
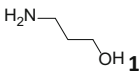
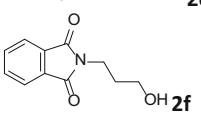
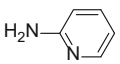
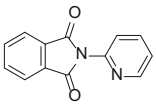
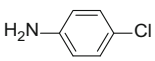
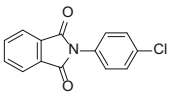
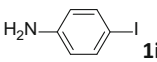
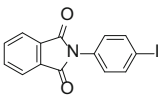
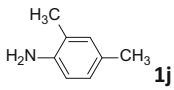
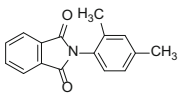
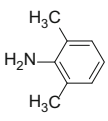
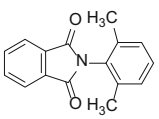
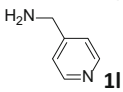
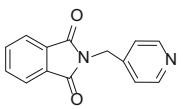
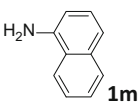
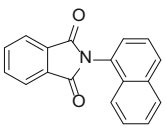
(*p*-Tolyl)isoindoline-1,3-dione (**2d**): IR (in cm^{-1}): 3460, 1717, 1516; 1384; 724, ^1H NMR (δ in ppm, $CDCl_3$) (90 MHz): 7.73–8.00 (4H, m, aromatic CH), 7.13–7.31 (4H, m, aromatic CH), and 2.42 (3H, s).

(*o*-Tolyl)isoindoline-1,3-dione (**2e**): IR (in cm^{-1}): 3050, 1745, 1717, 1516, 1384; ^1H NMR (δ in ppm, $CDCl_3$) (90 MHz): 7.74–8.02 (4H, m, aromatic CH), 7.25–7.39 (4H, m, aromatic CH), and 2.22 (3H, s).

N-(3-Hydroxypropan)isoindoline-1,3-dione (**2f**): IR (in cm^{-1}): 3035, 2949, 1774, 1739; 1714, 1242; ^1H NMR (δ in ppm, $CDCl_3$) (250 MHz): 7.68–7.84 (4H, m, aromatic CH), 4.27 (2H, $J = 10$ Hz, t), 3.92 (2H, $J = 10$ Hz, t), and 1.98 (2H, s).

N-(Pyridin-2-yl)isoindole-1,3-dione (**2g**): IR (in cm^{-1}): 3055, 1715, 1586, 1467, 1384; ^1H NMR (δ in ppm, $CDCl_3$) (250 MHz): 8.66–8.67 (1H, d, $J = 4$ Hz, aromatic CH), 7.9–7.95 (2H, m, aromatic CH), 7.83–7.87 (1H, m,

Table 3 Synthesis of various isoindoline-1,3-diones catalyzed by phthalimide-*N*-sulfonic acid

Entry	Amines (1a - m)	Isoindoline-1,3-diones (2a - m)	Time (h)	Yield (%)[a]
1	 **1a**	 **2a**	3	92
2	 **1b**	 **2b**	2	90
3	 **1c**	 **2c**	2	87
4	 **1d**	 **2d**	2	92
5	 **1e**	 **2e**	3	88
6	 **1f**	 **2f**	2	90
7	 **1g**	 **2g**	2	93
8	 **1h**	 **2h**	3	89
9	 **1i**	 **2i**	3	90
10	 **1j**	 **2j**	3	86
11	 **1k**	 **2k**	4	85
12	 **1l**	 **2l**	2	92
13	 **1m**	 **2m**	4	86

All reactions were performed by mixing of phthalic anhydride (0.296 g, 2 mmol), amine (2.2 mmol), and phthalimide-*N*-sulfonic acid (20 mol%) in ethanol at 80 °C

[a] Isolated yield

aromatic CH), 7.75–7.79 (2H, m, aromatic CH), and 7.34–7.44 (2H, m, aromatic CH).

N-(4-Chlorophenyl)isoindoline-1,3-dione (**2h**): IR (in cm^{-1}): 3061, 1708, 1497, 1392, 718 ^{1}H NMR (δ in ppm, $CDCl_3$) (90 MHz): 7.75–8.02 (4H, m, aromatic CH); 7.41–7.49 (4H, m, aromatic CH).

N-(4-Iodophenyl)isoindoline-1,3-dione (**2i**): IR (in cm^{-1}): 3055, 1709, 1486, 1385, 1119, 719; ^{1}H NMR (δ in

Scheme 2 Suggested mechanism for the synthesis of isoindoline-1,3-diones

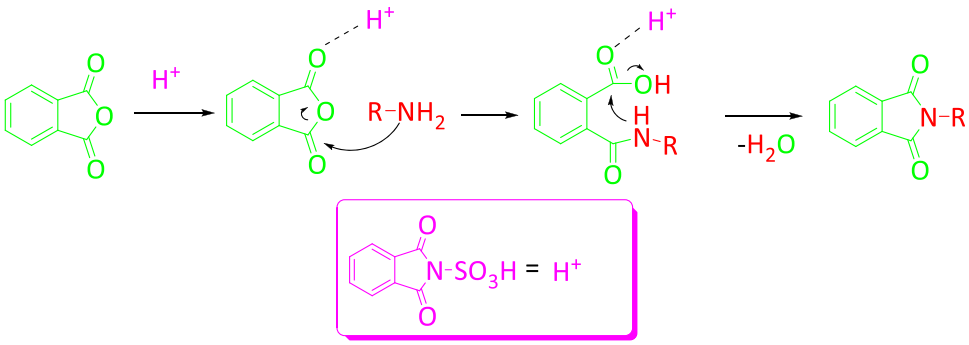


Table 4 Comparison of the catalytic activities of different catalysts

Entry	Catalyst	*T* (°C)	Time (min)	Yield %	Ref.
1	[Hmim]HSO_4	80	30–90	89	Dabiri et al. (2007)
2	[Bmim]PF_6	80	480	93	Zhou et al. (2003)
3	Choline chloride + $ZnCl_2$	60	60	87	Xie et al. (2009)
4	TFA	70	20–45	89	Shinde et al. (2011)
5	Sulphamic acid	Not reported	5–10	98	Langade (2011)
6	Fe_3O_4@SiO_2–SO_3H	80	45–90	89	Thale et al. (2014)
7	DABCO	Not reported	Immediately	79	Heravi et al. (2005)
8	*p*-Ts-OH	80	30–120	65-88	Nasr-Esfahani et al. (2012)
9	Our catalyst	80	2–4 h	92	Our catalyst

ppm, $CDCl_3$) (90 MHz): 7.94–7.97 (2H, m, aromatic CH), 7.78–7.85 (4H, m, aromatic CH); 7.21–7.26 (2H, m, aromatic CH).

N-(2,4-Dimethylphenyl)isoindoline-1,3-dione (**2j**): IR (in cm^{-1}): 3050, 1724, 1706, 1502, 1380; ^{1}H NMR (δ in ppm, $CDCl_3$) (90 MHz): 7.73-8.01 (4H, m, aromatic CH), 7.12–7.18 (3H, m, aromatic CH), 2.39 (3H, s), and 2.18 (3H, s).

N-(2,6-Dimethylphenyl)isoindoline-1,3-dione (**2k**): IR (in cm^{-1}): 3058, 1738, 1710, 1470, 1377; ^{1}H NMR (δ in ppm, $CDCl_3$) (90 MHz): 7.75-8.03 (4H, m, aromatic CH); 7.19–7.25 (3H, m, aromatic CH), 2.17 (6H, s).

N-(4-Picolinylamine)isoindoline-1,3-dione (**2l**): IR (in cm^{-1}): 3035, 2917, 1772, 1703, 1602, 1421, 1394; ^{1}H NMR (δ in ppm, $CDCl_3$) (250 MHz): 8.40–8.80 (2H, m, aromatic CH), 7.60–8.00 (4H, m, aromatic CH), 7.00-7.40 (2H, m, aromatic CH), and 4.80 (2H, s, CH_2).

N-(2-Naphtyl)isoindoline-1,3-dione (**2m**): IR (in cm^{-1}): 3058, 1738, 1710, 1470, 1377; ^{1}H NMR (δ in ppm, $CDCl_3$) (90 MHz): 7.93–8.03 (4H, m, aromatic CH), 7.82–7.85 (2H, m, aromatic CH), 7.58–7.64 (2H, m), and 7.46–7.54 (3H, m, aromatic CH).

Conclusions

We successfully developed and presented an efficient and easy catalytic protocol for preparation of various isoindoline-1,3-diones, using the catalytic amount of phthalimide-*N*-sulfonic acid in ethanol at 80 °C. The interesting features of the proposed methodology include the high efficiency, simplicity, and generality which lead to short reaction times, high yields, a cleaner reaction profile, and the catalyst recyclability.

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