Heterocyclic Letters
Vol. 8| No.4|773-781|Aug-Oct|2018
ISSN : (print) 2231–3087 / (online) 2230-9632
CODEN: HLEEAI
http://heteroletters.org

AN EFFICIENT ONE-POT NEAT SYNTHESIS OF PYRAZOLO[1,2-*b*]PHTHALAZINES USING CELLULOSE SULFURIC ACID AS A BIODEGRADABLE AND RECOVERABLE HETEROGENEOUS CATALYST

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Abstract: An efficient and environmentally friendly procedure for the synthesis of 3-amino-1-aryl-5,10-dioxo-5,10-dihydro-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carbonitriles through the one-pot, three-component reaction of phthalhydrazide, an aromatic aldehyde, and malononitrile in the presence of cellulose sulfuric acid (cellulose-SO_3H) is described. The reactions occur under thermal solvent-free conditions and the process is operative with various aromatic aldehydes, giving the corresponding products in high yields. Other beneficial features of this protocol include inexpensive, biodegradable and easily obtained catalyst, avoiding the use of harmful organic solvents, simple work-up, and the recyclability and reusability of the catalyst for up to five consecutive runs.

Keywords: Cellulose sulfuric acid, Pyrazolo[1,2-*b*]phthalazines, Solvent-free conditions

Introduction

In the last few decades, the synthesis of nitrogen heterocyclic compounds has received considerable attention due to their presence in a large number of natural products and pharmacologically active compounds. Among them, structures containing pyrazole and phthalazine scaffolds have been studied intensively due to their broad spectrum of activities. Earlier reports had already established pyrazoles as analgesics[i], antimalarial[ii], antibacterial[iii], antiviral[iv], and antitumor[v] agents. These compounds have also been considered as fungicides[vi], pesticides[vii], and as the chelating and extracting reagents for different metal ions[viii]. Furthermore, some of pyrazoles are included in many commercialized drugs for brain ischemia[ix] and myocardial ischemia[x]. Moreover, the success of COX-2 inhibitors containing a pyrazole moiety[xi] has highlighted the importance of this motif in medicinal chemistry. A number of these compounds are also used as potential inhibitors of arginine methyltransferases[xii], P38 kinase[xiii], and TNF-α[xiv]. On the other hand, the phthalazine is the base of many bioactive molecules having important biological properties such as

antimicrobial[xv], analgesic[xvi], antifungal[xvii], antiproliferative[xviii], antitumor[xix], and anticonvulsant[xx] activities. It has also been known that certain phthalazines are employed as selective inhibitors of p38 MAP kinase[xxi], VEGF receptors I and II[xxii], and phosphodiesterase 5[xxiii].

There are five pyrazolophthalazine scaffolds, including pyrazolo[1,2-*b*]phthalazine **I**, pyrazolo[5,1-*a*]phthalazine **II**, pyrazolo[3,4-*g*]phthalazine **III**, pyrazolo[4,3-*f*]phthalazine **IV**, and pyrazolo[3,4-*f*]phthalazine **V** (Figure 1), which are constructed from two fused pyrazole and phthalazine rings. Among them, pyrazolo[1,2-*b*]phthalazine core (**I**), has been relatively of more interest because of reported interesting biological properties including anti-inflammatory, analgesic, antimicrobial, antituberculosis and antioxidant activities[xxiv,xxv]. Recently, they have also been known as tacrine-like AChE inhibitors with potential use in Alzheimer's disease[xxvi]. A literature survey reveals a few methods for the synthesis of pyrazolo[1,2-*b*]phthalazines using various promoting agents[xxvii-xxx]. Synthesis of these compounds under microwave or ultrasonic irradiation have been also reported[xxxi,xxxii]. Hence, the discovery of a new method for the synthesis of these compounds is highly desirable.

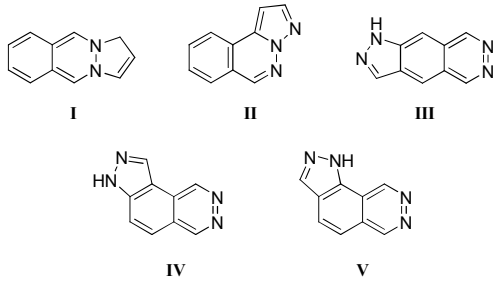


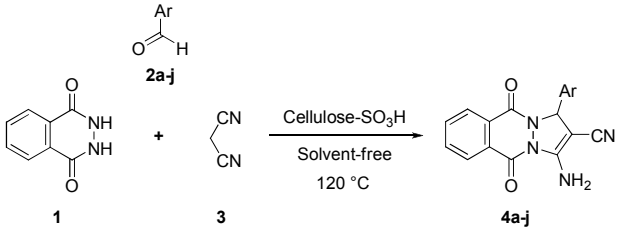
Figure 1. Structures of various pyrazolophthalazines

The development of heterogeneous acidic catalysts and how they affect specific transformations in chemical synthesis has become a major area of research. The potential advantages of these materials over homogeneous systems such as $AlCl_3$, $FeCl_3$, HF, and H_2SO_4, in terms of their simplified recovery and reusability, could potentially allow for the development of environmentally benign chemical procedures in both academic and industrial settings. Catalysts of this type have the potential to make the processes in which they are applied cleaner, safer, higher-yielding, and relatively inexpensive[xxxiii-xxxv].

The immobilization of SO_3H-functional group on a solid support is one of the important routes for developing novel heterogeneous acidic catalysts. Cellulose is the most abundant natural polymer and has been widely studied during the past several decades because it is biodegradable material and a renewable resource[xxxvi]. Its unique properties make it an attractive alternative to conventional organic or inorganic supports in catalytic applications. Very recently, cellulose sulfuric acid (cellulose-SO_3H) has been introduced as a biodegradable heterogeneous solid acid catalyst for the synthesis of a number of organic transformations[xxxvii-xli]. However, to the authors' knowledge, there is no report in the literature on the use of cellulose-SO_3H for the preparation of the titled compounds, pyrazolo[1,2-*b*]phthalazines.

Considering the above facts and due to our interest in heterocycles[xlii-xlviii], and also in extension of our previous studies on the development of new environmental friendly methodologies in the synthesis of organic compounds using reusable catalysts[xlix-xlxvi], we report here our results on the efficient and high yielding synthesis of 3-amino-1-aryl-5,10-dioxo-5,10-dihydro-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carbonitriles **4a-j** by one-pot reaction

of phthalhydrazide **1**, an aromatic aldehyde **2a-j**, and malononitrile **3** in the presence of cellulose-SO_3H as a biodegradable heterogeneous catalyst (Scheme 1).



Scheme 1. Synthesis of 3-amino-1-aryl-5,10-dioxo-5,10-dihydro-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carbonitriles catalyzed by cellulose-SO_3H

Experimental

All chemicals were purchased from Merck and Aldrich and used without purification. Melting points were measured on a Stuart SMP3 melting point apparatus. The ^1H spectra were measured on a Bruker 300 FT spectrometer using TMS as the internal standard. IR spectra were recorded on a Tensor 27 Bruker spectrophotometer in KBr disks.

Preparation of cellulose-SO_3H

To a magnetically stirred mixture of cellulose (5.0 g) in *n*-hexane (20 ml), chlorosulfonic acid (1.0 g, 9 mmol) was added dropwise at 0 °C during a 2 h period. After the completion of the addition, the mixture was stirred for another 2 h at room temperature until HCl gas was completely removed. Then, the mixture was filtered and washed with acetonitrile and dried at room temperature to obtain cellulose-SO_3H as a white powder[xlxvii].

General procedure for the synthesis of 3-amino-1-aryl-5,10-dioxo-5,10-dihydro-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carbonitriles 4a-j catalyzed by cellulose-SO_3H

A mixture of phthalhydrazide **1** (1 mmol), an aromatic aldehyde **2a-j** (1 mmol), malononitrile **3** (1 mmol), and cellulose-SO_3H (0.06 g) was heated in an oil bath at 120 °C. Upon completion of the transformation, monitored by TLC, the mixture was cooled to room temperature and hot ethyl acetate was added. The catalyst was filtered off and the filtrate was concentrated and allowed to stand at room temperature until precipitation occurred. The crude product was recrystallized from ethanol to give compounds **4a-j** in high yields. All the products were known and characterized by comparison of their melting points with those of authentic samples and for some cases using ^1H NMR and IR spectral data.

3-Amino-1-(4-chlorophenyl)-5,10-dioxo-5,10-dihydro-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carbonitrile (4a): ^1H NMR (300 MHz, DMSO-d_6, δ, ppm): 6.16 (s, 1H, pyrazole CH), 7.43 (d, *J* = 8.5 Hz, 2H, arom-H), 7.53 (d, *J* = 8.5 Hz, 2H, arom-H), 7.95-8.01 (m, 2H, arom-H), 8.06-8.12 (m, 1H, arom-H), 8.14 (s, 2H, NH_2), 8.25-8.30 (m, 1H, arom-H); FT-IR (KBr disk, *υ*, cm^{-1}): 3375 and 3262 (NH_2), 2199 (CN), 1661 (C=O).

3-Amino-1-(4-nitrophenyl)-5,10-dioxo-5,10-dihydro-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carbonitrile (4b): ^1H NMR (300 MHz, DMSO-d_6, δ, ppm): 6.32 (s, 1H, pyrazole CH), 7.82 (d, *J* = 8.8 Hz, 2H, arom-H), 7.98-8.02 (m, 2H, arom-H), 8.10-8.17 (m, 1H, arom-H), 8.21 (s, 2H, NH_2), 8.24 (d, *J* = 8.8 Hz, 2H, arom-H), 8.26-8.30 (m, 1H, arom-H); FT-IR (KBr disk, *υ*, cm^{-1}): 3434 and 3325 (NH_2), 2200 (CN), 1665 (C=O).

3-Amino-1-(4-methylphenyl)-5,10-dioxo-5,10-dihydro-1H-pyrazolo[1,2-*b*]phthalazine-2-carbonitrile (4c). ^1H NMR (300 MHz, DMSO-d_6, δ, ppm): 2.31 (s, 3H, CH_3), 6.10 (s, 1H, CH), 7.18 (d, *J* = 7.8 Hz, 2H, arom-H), 7.35 (d, *J* = 7.8 Hz, 2H, arom-H), 7.95-8.00 (m, 2H, arom-H), 8.05-8.15 (m, 3H, overlapped arom-H and NH_2), 8.24-8.29 (m, 1H, arom-H); FT-IR (KBr disk, *υ*, cm^{-1}): 3366 and 3266 (NH_2), 2202 (CN), 1662 (C=O).

3-Amino-1-(3-bromophenyl)-5,10-dioxo-5,10-dihydro-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carbonitrile (4f): ^{1}H NMR (300 MHz, DMSO-d_6, δ, ppm): 6.17 (s, 1H, pyrazole CH), 7.35 (t, *J* = 8.4 Hz, 1H, arom-H), 7.53 (d, *J* = 7.6 Hz, 2H, arom-H), 7.80 (s, 1H, arom-H), 7.94-8.10 (m, 3H, arom-H), 8.17 (s, 2H, NH_2), 8.25-8.28 (m, 1H, arom-H); FT-IR (KBr disk, *υ*, cm^{-1}): 3364 and 3255 (NH_2), 2191 (CN), 1655 (C=O).

3-Amino-1-(3-nitrophenyl)-5,10-dioxo-5,10-dihydro-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carbonitrile (4g): ^{1}H NMR (300 MHz, DMSO-d_6, δ, ppm): 6.37 (s, 1H, pyrazole CH), 7.70 (t, *J* = 7.9 Hz, 1H, arom-H), 7.95-8.12 (m, 5H, arom-H), 8.23 (s, 2H, NH_2), 8.25-8.30 (m, 1H, arom-H), 8.46 (s, 1H, arom-H); FT-IR (KBr disk, *υ*, cm^{-1}): 3363 and 3253 (NH_2), 2191 (CN), 1654 (C=O).

Results and discussion

To begin our study, cellulose-SO_3H was prepared according to method cited in the literature[xlxvii]. First, we directed our studies toward examination of the effect of various parameters like catalyst composition, effect of solvent, and influence of temperature on the reaction of phthalhydrazide **1** (1.0 mmol),, 4-chlorobenzaldehyde **2a** (1.0 mmol),, and malononitrile **3** (1.0 mmol) for the synthesis of compound **4a** as the model reaction in the absence or presence of cellulose-SO_3H as catalyst. The results are summarized in Table 1. When the reaction was carried out without catalyst (Table 1, entry 1) under solvent-free conditions at high temperature, only a trace amount of the product was formed even after 180 min. Next, several reactions were scrutinized under solvent-free conditions and also using various solvents such as H_2O, MeOH, EtOH, CH_3CN, and CH_2Cl_2, in the presence of cellulose-SO_3H as catalyst. As shown in Table 1, the trial reaction gives the best result in the presence of 0.06 g of cellulose-SO_3H under solvent-free conditions and proceeds at 120°C to afford the desired product **4a** in 100 min (entry 10). The higher amount of the catalyst or temperature had no significant effect on the yield and reaction time. Therefore, 0.06 g of the catalyst cellulose-SO_3H under solvent-free condition at 120 °C was found to be the optimized conditions and used in all subsequent reactions.

Table 1. Synthesis of compound **4a** in the presence of cellulose-SO_3H under different reaction conditions [a]

Entry	Catalyst (g)	Solvent	T (°C)	Time (min)	Isolated Yield (%)
1	-----	-----	120	180	Trace
2	0.02	-----	80	150	70
3	0.02	-----	100	150	75
4	0.02	-----	120	140	75
5	0.04	-----	80	150	75
6	0.04	-----	100	120	76
7	0.04	-----	120	110	80
8	0.06	-----	80	120	77
9	0.06	-----	100	110	78
10	0.06	-----	120	100	85
11	0.06	-----	140	100	84
12	0.08	-----	120	110	86
13	0.06	H_2O	Reflux	100	70
14	0.06	MeOH	Reflux	120	70
15	0.06	EtOH	Reflux	120	80
16	0.06	CH_3CN	Reflux	120	60
17	0.06	$CHCl_3$	Reflux	120	60

[a]Reaction conditions: phthalhydrazide **1** (1 mmol), 4-chlorobenzaldehyde **2a** (1 mmol), and malononitrile **3** (1 mmol).

Thereafter, to evaluate the applicability of the method, a variety of other aromatic aldehydes were used for the synthesis of a range of 3-amino-1-aryl-5,10-dioxo-5,10-dihydro-1*H*-

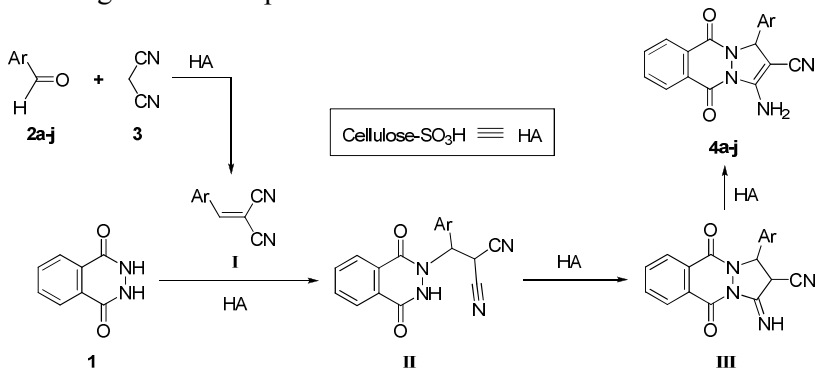
pyrazolo[1,2-*b*]phthalazine-2-carbonitriles under optimized conditions. As shown in Table 2, all electron-rich as well as electron-poor aromatic aldehydes reacted successfully and gave the products in high yields. The type of substituent on the aromatic aldehydes had no significant effect on the reaction time and yield.

Table 2. Cellulose-SO_3H catalyzed synthesis of compounds **4a-j**[a]

Entry	Ar	Product	Time (min)	Isolated Yield (%)
1	4-ClC_6H_4	**4a**	100	85
2	4-$O_2NC_6H_4$	**4b**	110	95
3	4-MeC_6H_4	**4c**	110	92
4	4-$MeOC_6H_4$	**4d**	110	89
5	3-ClC_6H_4	**4e**	120	88
6	3-BrC_6H_4	**4f**	110	89
7	3-$O_2NC_6H_4$	**4g**	120	88
8	2-ClC_6H_4	**4h**	110	91
9	2-$O_2NC_6H_4$	**4i**	110	92
10	C_6H_5	**4j**	120	89

[a]Reaction conditions: phthalhydrazide **1** (1 mmol), an aromatic aldehyde **2a-j** (1 mmol), malononitrile **3** (1 mmol), cellulose-SO_3H (0.06 g), 120 ºC, solvent-free.

A plausible pathway for the formation of compounds **4a-j** is proposed as depicted in Scheme 2. It is proposed that dicyano olefin **I** can be readily formed *in situ* by Knoevenagel condensation of aldehydes **2a-j** and malononitrile **3**. Nucleophilic conjugate addition of phthalhydrazide **1** to the dicyano olefin **I** produces the intermediate **II** which subsequently undergoes cyclization reaction to afford the intermediate **III**. Finally, the products **4a-j** are obtained from the latter intermediate after tautomerization. As shown in Scheme 2, it is reasonable to assume that several accessible SO_3H groups in cellulose-$SO_3H \equiv$ HA could act as Brønsted acid centres, and therefore activate the aldehyde and the intermediates in this reaction by increasing their electrophilic character.



Scheme 2. Plausible pathway for the formation of compounds **4a-j** catalyzed by cellulose-$SO_3H \equiv$ HA

The ^{1}H NMR spectra confirm that the isolated compounds are **4a-j** and not the corresponding structures **II** or **III**. For example, as shown in Figure 2, ^{1}H NMR spectrum of the isolated compound from the reaction of **1** with **2b** and **3** shows a singlet at 6.32 ppm belonging to the methine group in pyrazole ring that is in accordance with the structure **4b**. For the structures **II** or **III**, an AX splitting pattern for -CH-CH- group is expected. Furthermore, in the FT-IR spectrum, the absorption bands for NH_2 and cyano groups appeared at 3434, 3325 and 2200

cm^{-1}, respectively, confirming the structure **4b**. This compound is also melted at 262-263 °C that is in accordance with authentic sample[xxviii].

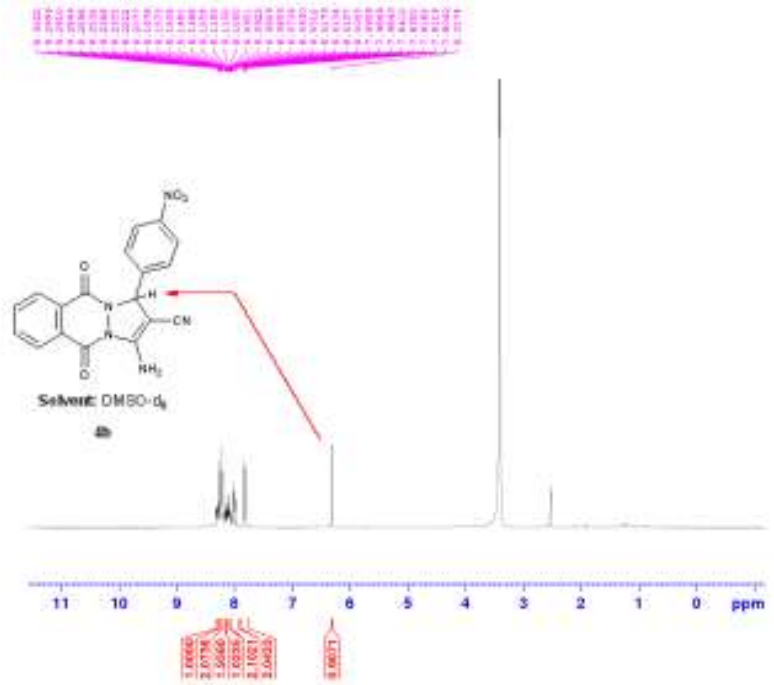


Figure 2. The ^{1}H NMR spectrum of compound **4b** in DMSO-d_6

The formation of compound **4b** was considered to evaluate the possibility of recycling and reusing of the cellulose-SO_3H. After cooling the reaction mixture to room temperature, at the end of first run, hot ethyl acetate was added and the catalyst was collected by simple filtration. The recycled catalyst was washed with ethyl acetate and dichloromethane, dried under vacuum at 70 °C for 1 h, and then used for the subsequent catalytic run. The catalyst could be used at least five times without substantial reduction of its activity (Figure 3).

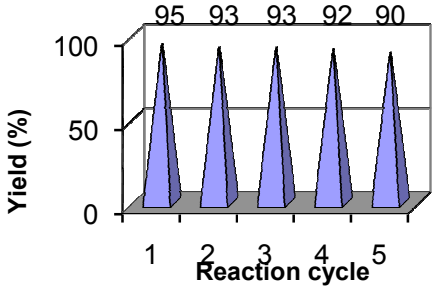


Figure 3. Reusability of cellulose-SO_3H in the formation of compound **4b**

Conclusion

In this paper, an efficient and eco-friendly method for the synthesis of 3-amino-1-aryl-5,10-dioxo-5,10-dihydro-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carbonitriles by the one-pot, three-component reaction of phthalhydrazide with a wide range of aromatic aldehydes and malononitrile using cellulose-SO_3H as a biodegradable heterogeneous solid acid catalyst has been successfully developed. The desired products were obtained in high yields under solvent-free conditions at 120 °C. The catalyst can be recycled after a simple work-up, and used at least five times without substantial reduction in its catalytic activity. The procedure is

also advantageous in the sense that it is a solvent-free reaction and therefore operates under environmentally friendly conditions.

Acknowledgement
This work was supported by Islamic Azad University, Mashhad Branch, Iran.

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Received on August 23, 2018.