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Carboxymethyl Cellulose (CMC) as a Recyclable Green Catalyst Promoted Eco-Friendly Protocol for the Solvent-Free Synthesis of 1*H*-Pyrzolo[1,2-*b*]Phthalazine-5,10-Dione Derivatives

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ABSTRACT

An eco-safe synthetic route for convenient one-pot synthesizing 1*H*-pyrazolo[1,2-*b*] phthalazine-5,10-dione derivatives *through* Knoevenagel-Michael cyclocondensation is reported in carboxymethyl cellulose (CMC) based on green chemistry principles. The prominent benefits include use of recyclable green catalyst, commercially accessible inexpensive starting materials, operational simplicity, non-hazardous reaction circumstances, high atom-economy, solvent-free, short reaction times and high yields.

ARTICLE HISTORY

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KEYWORDS

1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives; carboxymethyl cellulose (CMC); green procedure; recyclable and biodegradable catalyst

Introduction

Pyrazolophthalazines with various pharmacological features^{1,2} like anticancer,³ anti-inflammatory,⁴ anti microbiological⁵ and they have been reported to possess vasorelaxant,⁶ cardiotonic,⁷ anticonvulsant⁸ and antifungal.⁹ There are numerous approaches for synthesizing these compounds using various catalysts such as Ce(SO₄)₂·4H₂O,¹⁰ SBA-Pr-SO₃H,¹¹ InCl₃,¹² NiCl₂·6H₂O,¹³ [Bmim] OH,¹⁴ Ultrasound-assisted,¹⁵ *P*-TSA,¹⁶ STA,¹⁷ CuI nanoparticles,¹⁸ PTSA/[Bmim]Br,¹⁹ TBBAD,²⁰ Cu(OAc)₂·H₂O,²¹ K₂CO₃,²² β-Cyclodextrin,²³ [Bu₃NH][HSO₄],²⁴ CuO nanoparticles,²⁵ NZF@HAP-Cs,²⁶ theophylline²⁷ and STA-Amine-Si-Magnetite.²⁸ Each of these methods has its own merits but some of these methods are limited in terms of the use of expensive catalysts, long reaction periods, low yields, harsh reaction conditions, tedious work-up and needing additional quantities of catalysts or reagents and hazardous or toxic catalysts with column chromatographic separation. Hence, finding the environmentally friendly and appropriate approaches for synthesizing this kind of compounds is vital. Since we partly aimed to develop green synthetic processes^{29–33} and due to the above considerations, the search for eco-safe, simple and effective strategies capable of promoting organic reactions under green circumstances has attracted a huge deal of interest in producing 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives. Hence, here solvent-free environmentally friendly synthesis of 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives are reported *via* carboxymethyl cellulose (CMC) (Figure 1) as a green, recyclable, and biodegradable catalyst³⁴ *via* tandem Knoevenagel-Michael cyclocondensation provided the anticipated products in outstanding yields and short reaction times which might solve some cost problems in industry. Subsequently, we studied the recyclability of the green CMC for the above reaction. However, the CMC can be recycled at least five times with no considerable reduction in activity making it greatly advantageous in addressing the industrial requirements and environmental worries.

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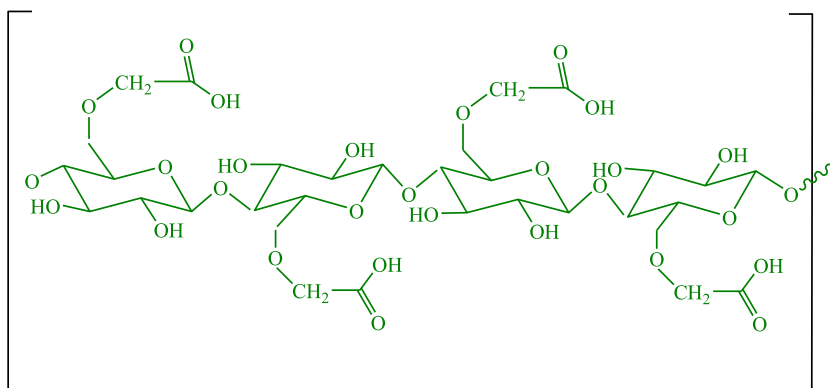


Figure 1. Chemical structure of CMC.

Experimental

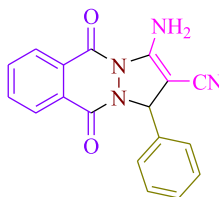
General

Utilizing an Electro thermal 9100 device, all compounds' melting points were found. Moreover, recording nuclear magnetic resonance, ¹H NMR spectra was carried out on a Bruker DRX-400 and Bruker DRX-300 Avance tool with DMSO-d₆ as solvent. All solvents and reagents were bought from Acros, Merck, and Fluka chemical companies and were utilized with no additional purification.

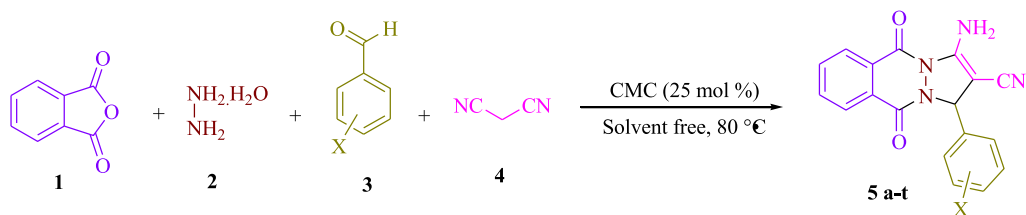
Overall process of preparing (5a-t)

A combination of phthalic anhydride (**1**, 1.0 mmol), hydrazine monohydrate (**2**, 1.0 mmol) and CMC (25 mol%) was heated for 2 h at 80 °C. Then, adding aromatic aldehyde (**3**, 1.0 mmol) and malononitrile (**4**, 1.0 mmol), the reaction was heated for apposite period (Scheme 1). The reaction progress was monitored by TLC utilizing *n*-hexane/EtOAc (4:1) as an eluent. The reaction mass was chilled to room temperature after completing the reaction and then poured on hot water. The achieved solid was filtered, rinsed with water and the crude solid was recrystallized from ethanol to provide the pure material without requiring more purification. The aqueous filtrate was refined at 100 °C to eliminate water to give CMC as white powder. Then powder washed with ethyl acetate and filtered, air dried and reused which was used for the next run under similar reaction conditions. The CMC was improved and reused with no activity loss. Comparing the spectroscopic information, the products were categorized (¹HNMR). Supporting Information associated with this article can be found, in the online version.

3-Amino-1-(phenyl)-5,10-dihydro-5,10-dioxo-1H-pyrazolo[1,2-b]phthalazine-2-carbonitrile (5d)



Yield: 91%; M.p. 271-273 °C; ¹H NMR (300 MHz, DMSO-d₆): 6.14 (1H, s, CHAr), 7.33-7.48 (3H, m, ArH), 7.46 (2H, d, *J* = 8.4 Hz, ArH), 7.97-8.29 (6H, m, NH₂ and ArH).

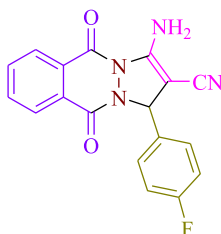


Scheme 1. Synthesis of 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives.

Table 1. Optimizing the reaction circumstance in the existence of various quantities of CMC.

Isolated yields (%)	Time (min)	Temperature (°C)	CMC (mol%)	Entry
No product	420	80	Catalyst free	1
23	240	80	5	2
37	180	80	10	3
54	115	80	15	4
76	85	80	20	5
91	75	80	25	6
No product	420	rt	25	7
29	210	40	25	8
47	155	50	25	9
62	120	60	25	10
73	90	70	25	11
91	75	90	25	12
92	75	80	30	13

3-Amino-1-(4-fluorophenyl)-5,10-dihydro-5,10-dioxo-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carbonitrile (5o)



Yield: 94%; M.p. 261-263 °C; ^1H NMR (400 MHz, DMSO- d_6): 6.17 (1H, s, CHAr), 7.20 (2H, t, $J = 8.8$ Hz, ArH), 7.53-7.57 (2H, m, ArH), 7.96-8.26 (6H, m, NH_2 and ArH).

3-Amino-1-(4-methylphenyl)-5,10-dihydro-5,10-dioxo-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carbonitrile (5q)

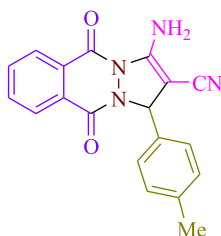
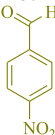
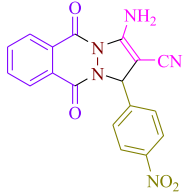
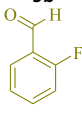
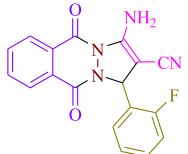

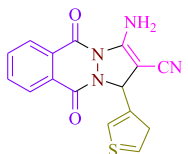
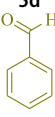
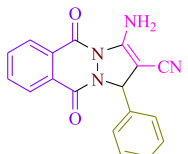
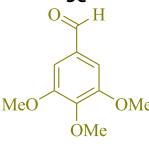
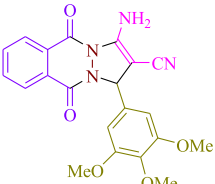
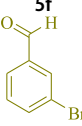
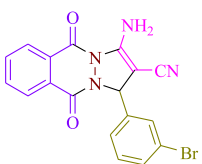
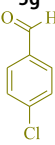
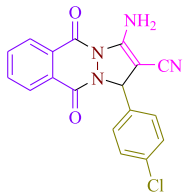
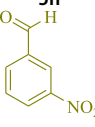
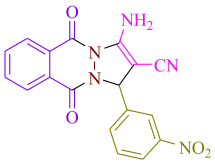
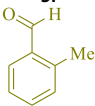
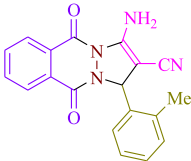
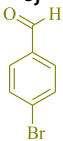
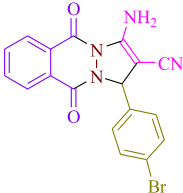
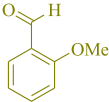
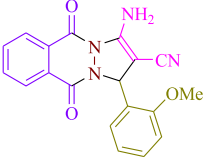
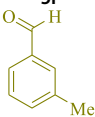
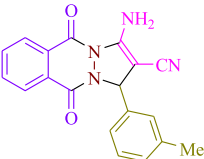
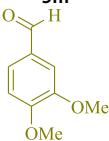
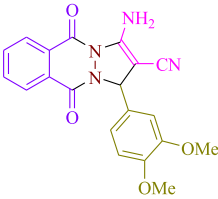
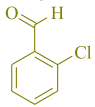
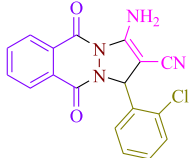
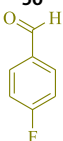
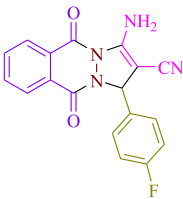
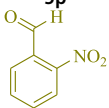
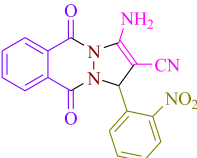


Table 2. CMC catalyzed synthesizing 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives.

Lit. M.p. °C	M.p. °C	Isolated yields (%)	Time (min)	Product	Ar	Entry
228–229 ¹⁷	227–229	89	70			1
268–270 ¹²	270–272	93	60			2
244–246 ¹⁸	245–247	87	75			3
270–272 ¹⁸	271–273	91	75			4
253–255 ¹²	253–255	86	90			5
270–272 ¹¹	268–270	82	95			6
270–272 ¹⁹	272–274	81	85			7
269–271 ¹⁹	267–269	86	70			8

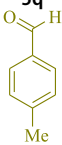
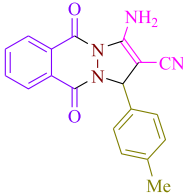
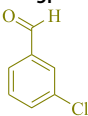
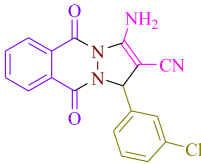
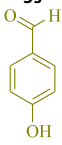
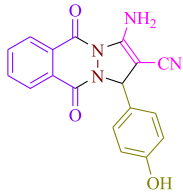
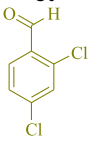
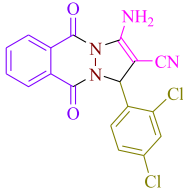
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Table 2. Continued.

Lit. M.p. °C	M.p. °C	Isolated yields (%)	Time (min)	Product	Ar	Entry
248–250 ¹⁸	249–251	94	65	5i 		9
265–267 ¹¹	267–269	85	95	5j 		10
153–155 ²⁶	154–156	89	75	5k 		11
250–252 ¹⁸	249–251	92	65	5l 		12
150–152 ²⁰	151–153	88	85	5m 		13
257–259 ¹⁷	256–258	84	75	5n 		14
263–265 ¹¹	261–263	94	65	5o 		15
265–266 ¹¹	264–266	91	65	5p 		16

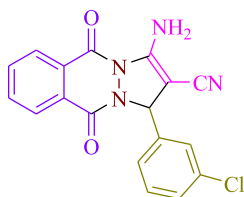
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Table 2. Continued.

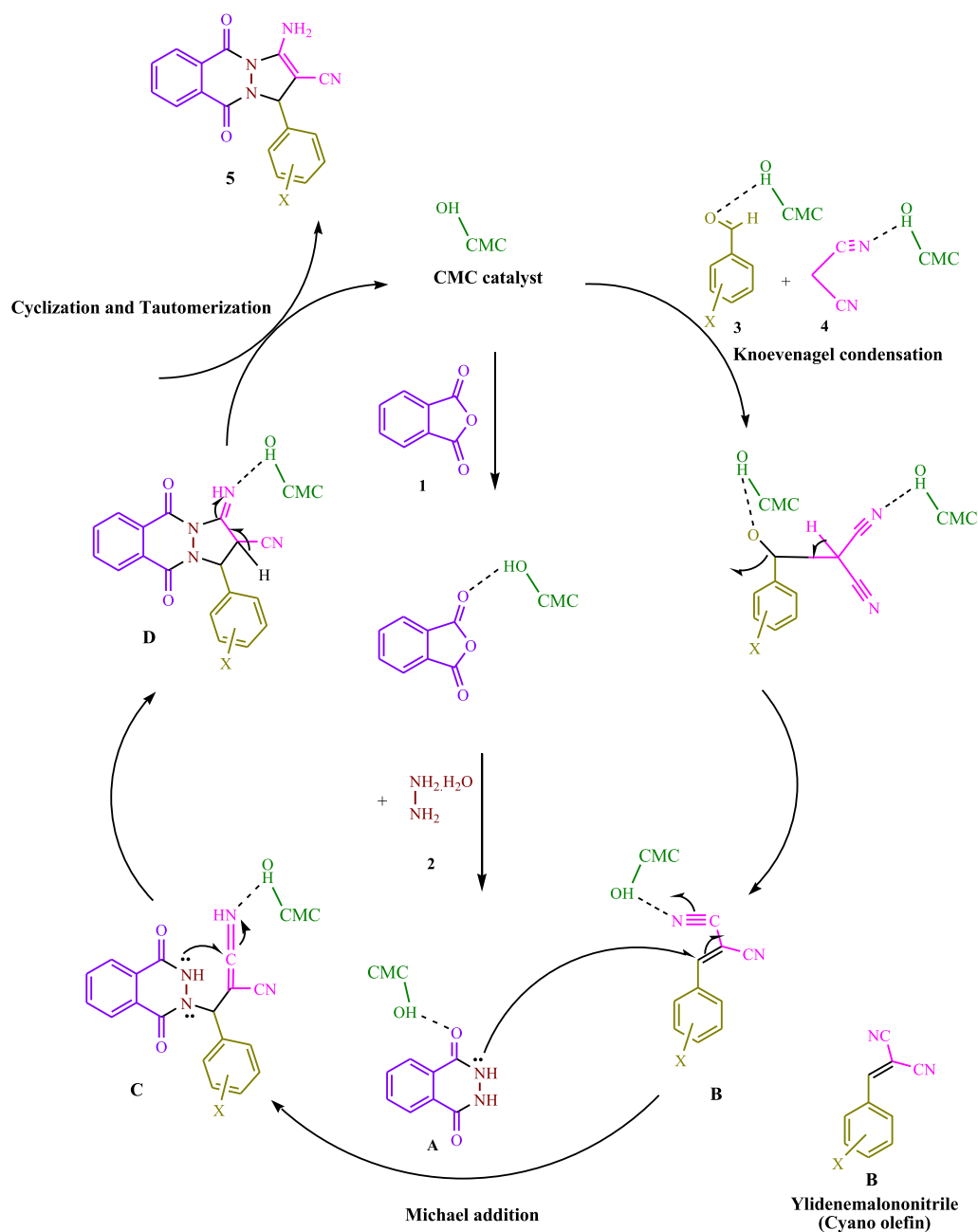
Lit. M.p. °C	M.p. °C	Isolated yields (%)	Time (min)	Product	Ar	Entry
253–255 ¹⁸	255–257	89	70			17
266–267 ¹¹	264–266	78	85			18
270–272 ¹²	272–274	80	95			19
230–232 ²⁵	231–233	77	90			20

Yield: 89%; M.p. 255–257 °C; ¹H NMR (400 MHz, DMSO-d₆): 2.30 (3H, s, CH₃), 6.10 (1H, s, CHAr), 7.18 (2H, d, *J* = 8.0 Hz, ArH), 7.34 (2H, d, *J* = 8.0 Hz, ArH), 7.97–8.28 (6H, m, NH₂ and ArH).

3-Amino-1-(3-chlorophenyl)-5,10-dihydro-5,10-dioxo-1H-pyrazolo[1,2-b]phthalazine-2-carbonitrile (5r)



Yield: 78%; M.p. 264–266 °C; ¹H NMR (300 MHz, DMSO-d₆): 6.15 (1H, s, CHAr), 7.39–7.41 (2H, m, ArH), 7.44–7.48 (1H, m, ArH), 7.65 (1H, s, ArH), 7.88–8.29 (6H, m, NH₂ and ArH).



Scheme 2. Recommended mechanistic path for synthesizing 1H-pyrazolo[1,2-b]phthalazine-5,10-dione derivatives.

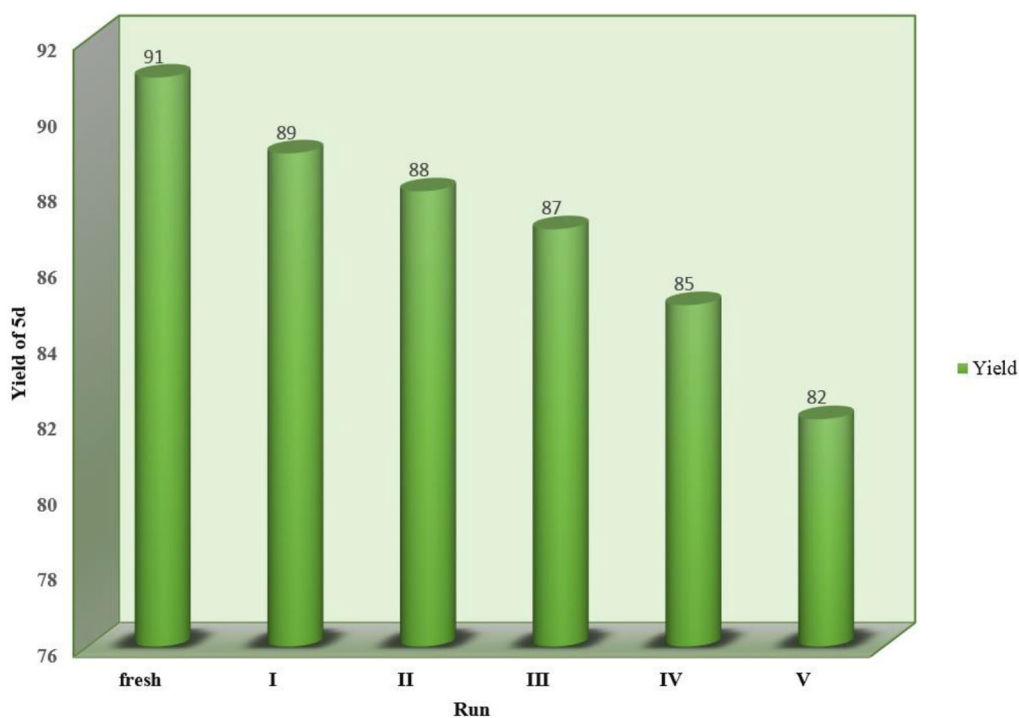
Results and discussion

The primarily, carboxymethyl cellulose's catalytic activity was examined in a model system in the four-element reaction between a combination of phthalic anhydride (1.0 mmol), hydrazine monohydrate (1.0 mmol), benzaldehyde (1.0 mmol) and malononitrile (1.0 mmol) under solvent-free conditions. The enhanced circumstances were defined by changing the number of the catalyst equivalents and different temperature factors. Lack of a catalyst, a product was not found at

Table 3. Comparing catalytic capability.^a

References	Time/yield (%)	Conditions	Catalyst	Entry
12	1.5 h/85	Water, Reflux	InCl ₃	1
13	3 h/87	EtOH, Reflux	NiCl ₂ ·6H ₂ O	2
16	3 h/94	[Bmim]Br, 100 °C	<i>p</i> -TSA	3
17	20 min/94	Solvent-free, 70 °C	STA	4
18	27 min/91	MeCN, Reflux	CuI nanoparticles	5
20	15 min/89	Solvent-free, 80-100 °C	TBBAD	6
21	3 h/83	Solvent-free, 80 °C	Cu(OAc) ₂ ·H ₂ O	7
22	50 min/95	EtOH, Reflux	K ₂ CO ₃	8
23	2.5 h/86	H ₂ O/EtOH, 100 °C	β -Cyclodextrin	9
This work	75 min/91	Solvent-free, 80 °C	CMC	10

^aBased on synthesizing 3-Amino-1-(phenyl)-5,10-dihydro-5,10-dioxo-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carbonitrile.

**Figure 2.** The recyclability of the CMC in the preparation of **5d**.

80 °C within 420 min reaction period (Table 1, entry 1). Inserting 5 mol% of the catalyst, a considerable progress was found by the reaction completing in around 240 min (Table 1, entry 2). Using 25 mol% of catalyst, the competent advance and completion were found in the reaction in less reaction period (75 min) (Table 1, entry 6). No considerable enhancement in the product yield and reaction period was found by additional incrementing catalyst quantity (Table 1, entry 13). Further studies determined the effect of different temperature factors on the reaction rate and yields (Table 1). We studied temperature changes from rt to 90 °C. The results show that increasing the temperature to 80 °C results in an increase in reaction rate and product yield. It was also found that increasing the temperature to more than 80 °C had no effect on product yield (Table 1, entry 12). Finally, from the environmental and economic aspects, CMC (25 mol%) was chosen as catalyst at 80 °C under solvent-free conditions for all additional reactions (Table 1, entry 6). As observed in Table 2 and Scheme 1, it was indicated that this technique can work

with various substrates. It should be noted that for purifying the products (**5a-t**), a modest filtration, washed and recrystallizing with ethanol is needed.

Scheme 2 shows the suggested mechanism for synthesizing 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives. **Table 3** represents the comparison of the catalytic capability of some catalysts reported the literature for synthesis of 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives. Within this work, it is revealed that CMC possesses its amazing potential as a substitute green, recyclable, biodegradable and inexpensive catalyst for the one-pot synthesizing these naturally active heterocyclic compounds, along with outstanding yields and short reaction periods are the remarkable benefits of this current procedure.

Reusability of CMC

Recovery and reusability is very significant from both environmental and economic perspectives, the recovery and reusability of CMC was investigated in several subsequent runs. For this appeal, the reaction of phthalic anhydride (1.0 mmol), hydrazine monohydrate (1.0 mmol), benzaldehyde (1.0 mmol) and malononitrile (1.0 mmol) for synthesizing (**5d**) was examined in the existence of CMC (25 mol%) was heated at 80 °C for 75 min. After completing, the mixture was chilled to room temperature and poured on hot water. The created precipitate was filtered, rinsed with water and the crude product was purified by recrystallizing from ethanol to obtain the pure product **5d**. To eliminate water, the aqueous filtrate was distilled at 100 °C to give CMC as white powder. Then powder washed with ethyl acetate and filtered, air dried and reused which was used for the next run under similar reaction conditions. The recovered CMC was effectively utilized in consecutive runs (five runs) including the use of fresh medium with no further efficiency loss and with insignificant CMC loss (**Figure 2**). Slight decrease in the product yield was found in the first, second, third, fourth and fifth reaction runs (89%, 88%, 87%, 85% and 82%, respectively).

Conclusions

In conclusion, in the present work, it was demonstrated that a recyclable green and biodegradable catalyst, carboxymethyl cellulose (CMC), can be used as a greatly efficient and availability catalyst for solvent-free one-pot 4-component synthesizing 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives. Use of inexpensive initiating substances, solvent-free, time-saving aspects of the reaction, excellent yields, the application of non-hazardous reaction circumstances, direct work-up without column chromatographic separation, convenient and expedient procedure are the notable advantages of this green and simple protocol. However, the CMC can be recycled at least five times with no considerable reduction in activity making it greatly advantageous in addressing the industrial requirements and environmental worries.

Disclosure statement

There are no conflicts of interest.

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