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A new approach for the synthesis of fluorescent pyrido[1,2-a]benzimidazoles

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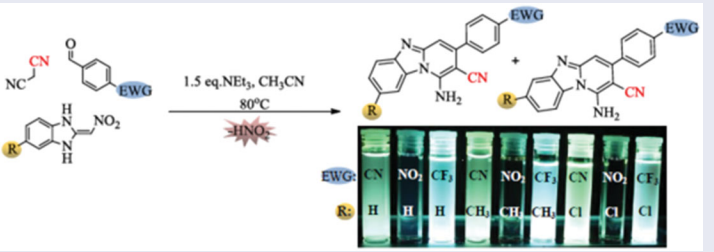
ABSTRACT

A facile synthesis of new pyrido[1,2-a]benzimidazoles through a one-pot multicomponent reaction of malononitrile, electron-deficient aryl aldehydes and heterocyclic enamines followed by a nitrous acid release under mild condition, is reported. Depending on the substituents on heterocyclic enamine, newly synthesized products have been obtained either as an inseparable mixture of regioisomers or as a single regioisomer. A plausible mechanism can be proposed for the formation of pyrido[1,2-a]benzimidazoles via this unfamiliar transformation with the HNO_2 extrusion. The structures of all title compounds were elucidated using spectroscopic methods and physical characteristics involving single crystal X-ray diffraction and TOF-MS measurements. In addition, among all the products, CN- and CF_3-substituted ones showed promising absorption and fluorescent properties.

GRAPHICAL ABSTRACT



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Heterocyclic enamine; multicomponent reaction; pyrido[1,2-a]benzimidazole

Introduction

Benzimidazoles are of great importance in organic synthesis due to their diverse biological activities and they are known to be used as a precursor intermediate, in particular, to provide ready access to the variety of fused benzimidazole or benzo [4,5]imidazo[1,2-a]pyridines.[1–3] Among them, pyrido[1,2-a]benzimidazoles have been extensively studied and gained increasing interest over the past decades.[4–7] Compounds bearing these heterocyclic units not only display a variety of biological

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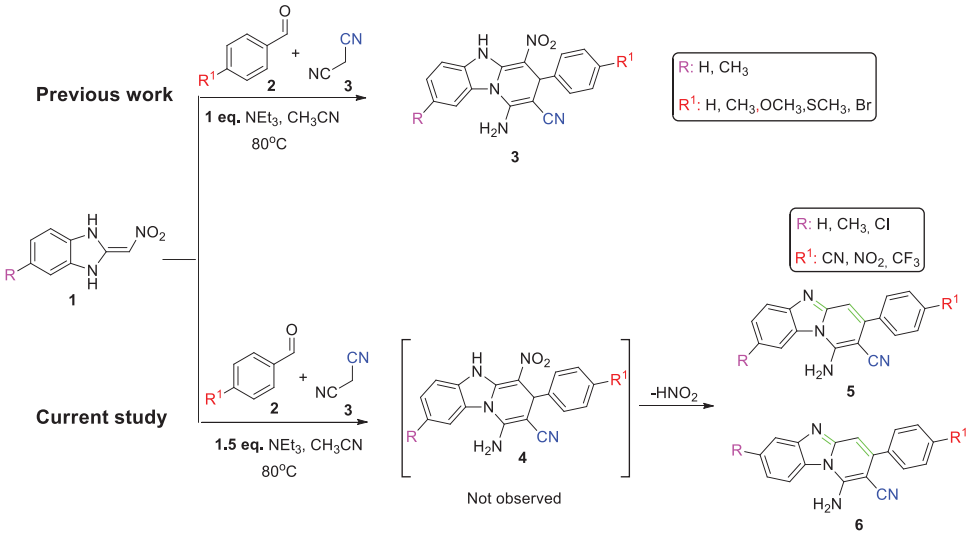


Figure 1. Multicomponent reaction of heterocyclic enamines **1** with malononitrile **3** and electron-rich and electron-deficient aldehydes **2**.

activities such as antimalarial,[8] antitumor,[9] and antifungal [10] but also show some promising photophysical and fluorescent properties.[11,12]

On the other hand, numerous synthetic approaches have been developed for the construction of pyrido[1,2-a]benzimidazoles and its derivatives either by reacting the benzimidazole-2-acetonitrile with β-ketoesters[13] and 3-substituted chromones,[14] or by transition metal-catalyzed,[15] acid-catalyzed,[16] base-catalyzed[17] and photoinduced radical cyclization[18] of modified 2-aminopyridines, but many of them require harsh reaction conditions and unwanted side products. Therefore, it is always desired and challenging to design an easy and efficient route to reach the corresponding heterocycles. The multicomponent reaction is one of the most utilized methods for the synthesis of structurally diverse and complex heterocycles in terms of simplicity and efficiency.[19] For this purpose, heterocyclic ketene aminals seem to be attractive intermediates for the preparation of fluorescent pyrido[1,2-a]benzimidazoles due to their bisnucleophilic nature that allow the cyclization reaction with olefins bearing an electron-withdrawing groups in a one-pot process.

Very recently, we reported the synthesis of a series of 3,5-dihydropyrido[1,2-a]benzimidazoles under mild conditions in high yield via a multicomponent reaction between cyclic enamines **1**, aryl aldehydes **2** -bearing electron-donating groups and malononitrile **3** (Fig. 1).[20] As a continuation of our work on extending the scope of the aforementioned reaction: electron-deficient aryl aldehydes have been subjected to three-component reaction which unexpectedly afforded new pyrido[1,2-a]benzimidazoles via the extrusion of HNO_2, and many of the products exhibited promising fluorescent properties. To the best of our knowledge, there are few studies related to the optical properties of pyrido[1,2-a]benzimidazoles, so, this progressively driven us to develop and examine the fluorescent properties of this new heterocycles in detail.

Table 1. Optimization of reaction conditions affording product **5a**.

1a 2a 3 5a

Entry	Solvent	Reaction time	Base	Eq.	Temperature (°C)	Yield (%)[c]
1[a]	CH_3CN	6h	K_2CO_3	1.0	80	55
2[a]	CH_3CN	6h	Piperidine	1.0	80	60
3[a]	CH_3CN	12h	KO^{t}Bu	1.0	80	62
4[a]	CH_3CN	3h	NEt_3	1.0	80	68
5[a]	CH_3CN	3h	NEt_3	1.0	25	–
6[a]	**CH_3CN**	**3h**	**NEt_3**	**1.5**	**80**	**72**
7[b]	CH_3CN	3h	NEt_3	1.5	80	52
8[a]	CH_3CN	3h	NEt_3	2.0	80	72
9[a]	Ethanol	3h	NEt_3	1.5	80	55
10[a]	Dioxane	3h	NEt_3	1.5	80	70

[a]The reaction was carried out by conventional heating.
[b]Microwave irradiation, 300 W, 80°C.
[c]Yield of isolated product.

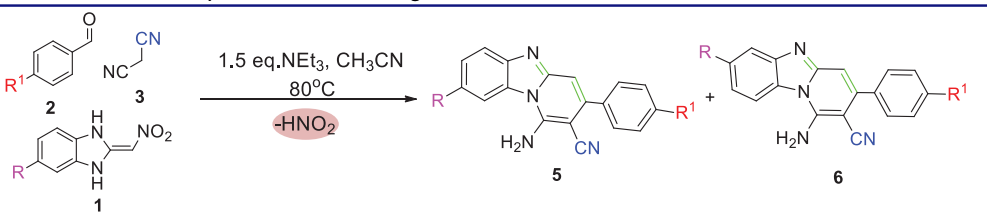
Results and discussion

Our initial effort was to expand the scope of the multicomponent reaction reported in our previous study. For this purpose, heterocyclic enamine **1a**, p-cyanobenzaldehyde **2a**, and malononitrile **3** were tested by following the previously described method using of 1 equiv. of NEt_3 under reflux temperature of CH_3CN to afford new parent 3,5-dihydro-pyrido[1,2-a]benzimidazoles. Unexpectedly, this model reaction gave rise to the formation of fully conjugated pyrido[1,2-a]benzimidazole **5a** with the liberation of HNO_2 instead of expected- product **4a** (Fig. 1). In order to better understanding the limitation of this unfamiliar reaction, we utilized some aryl aldehydes bearing electron-withdrawing groups (**2a–c**) with malononitrile **3** and various heterocyclic enamines (**1a–c**) in the multicomponent reaction.

First of all, we began the study by optimizing the reaction conditions using different bases and solvents (Table 1). First, the effect of different bases on the reaction conditions was investigated by using **1a**, **2a**, and **3** as substrates in the model reaction. Among the bases (K_2CO_3, piperidine, KO*t-Bu*, Et_3N) tried on the reaction, triethylamine afforded the desired product **5a** in the highest yield (Table 1, entries 1-4). Even no product was observed at room temperature in CH_3CN (Table 1, entry 5), using of 1.5 equiv. of triethylamine at the same reaction conditions gave the best reaction yield (72%) (Table 1, entry 6). However, the trial with MW energy caused a slight decrease in the reaction yield (52%) (Table 1, entry 7) and also using of 2.0 equiv. of Et_3N had no positive contribution in the yields (72%) of model reaction (Table 1, entry 8). In last optimization trials, the effects of different solvents (ethanol, dioxane) under optimized reaction conditions (1.5 eq. NEt_3, 80 °C) have been investigated and this time, slight decreases were observed in terms of reaction yields (Table 1, entries 9–10).

Thus, under the optimal conditions, we extended the scope of the reaction by using variety of heterocyclic enamines **1a–c**, malononitrile **3** and electron-deficient aryl aldehydes **2a–c** to provide nine new pyrido[1,2-a]benzimidazoles (**5** and **6**) as either single

Table 2. Formation of product **5** and its regioisomer **6**.



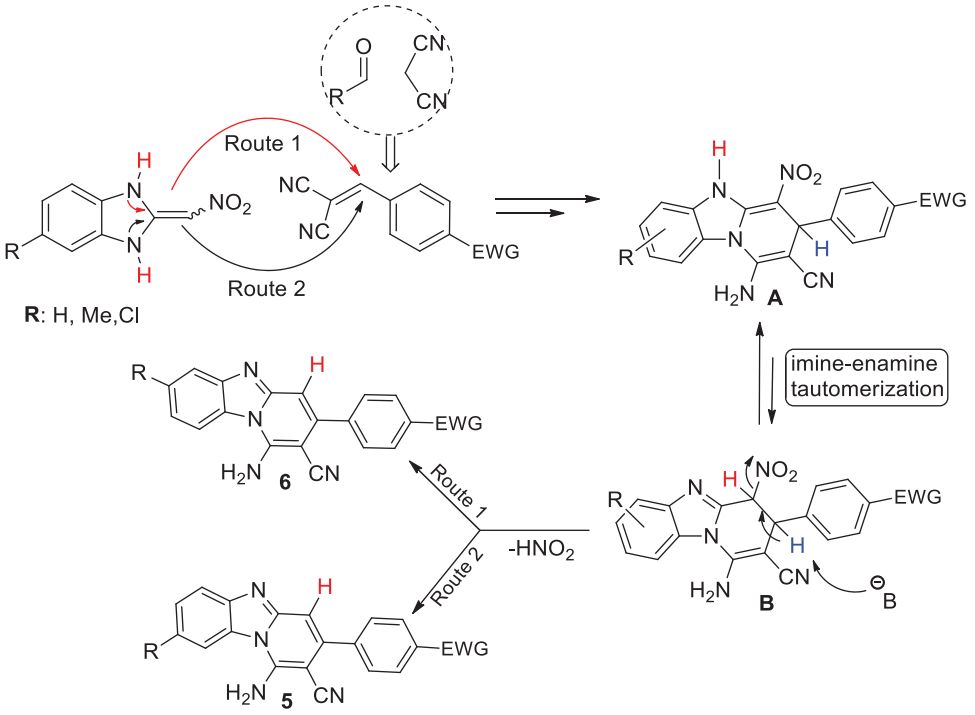
Entry	*R*	R^1	Product 5	Product 6	Regioisomer ratio (5:6)[b]	Yield (%)
1	**1a**: R=H	**2a**: R=CN	**5a**	–	–	72
2	**1a**: R=H	**2b**: R=NO_2	**5b**	–	–	52
3	**1a**: R=H	**2c**: R=CF_3	**5c**	–	–	65
4	**1b**: R=Me	**2a**: R=CN	**5d**	**6d**	50:50	60[a]
5	**1b**: R=Me	**2b**: R=NO_2	**5e**	**6e**	49:51	48[a]
6	**1b**: R=Me	**2c**: R=CF_3	**5f**	**6f**	69:31	56[a]
7	**1c**: R=Cl	**2a**: R=CN	**5g**	**6g**	52:48	64[a]
8	**1c**: R=Cl	**2b**: R=NO_2	**5h**	**6h**	53:47	50[a]
9	**1c**: R=Cl	**2c**: R=CF_3	**5i**	**6i**	66:34	62[a]

[a]Total yield of inseparable regioisomers.
[b]Ratio of diastereomers in each mixture were determined according to olefinic=CH peaks in proton NMR spectra of the products.

product or inseparable regioisomers in moderate to high yields, (Table 2, entries 4–9). It is well understood that this regioisomeric product distribution (from 50:50 to 69:31) was due to two reactive amine functionalities, which both can be tautomerized on heterocyclic enamine to enable the multicomponent reaction (Table 2, entries 4–9). When unsubstituted cyclic enamine **1a** was used, no matter which amine group reacts with *in situ* formed arylidenemalononitrile to afford the expected product without any regioisomer. However, the use of $-CH_3$ or –Cl substituted heterocyclic enamines (**1b** or **1c**) bearing two different amine groups which individually act as a nucleophilic reagent in reaction leading to the formation of regioisomers. Interestingly, we did not observe any sign for the formation of regioisomeric mixture (**5** or **6**) in our earlier report when **1b** was used in the multicomponent reaction with malononitrile and electron-rich aryl aldehydes **2**.[20]

We believe the reason behind such a transformation is the presence of electron-withdrawing group ataryl aldehydes. During the reaction progress, after the formation of expected product **4**, the methinic proton in the dihydropyridine structure **4** becomes acidic by the resonance effect of electron-withdrawing groups on para-position and this allows the formation of more stable product **5** or **6** by a release of HNO_2 molecule. Presumably, the reaction initiates by nucleophilic attack of enamine at the benzylic position of *in situ* formed arylidenemalononitriles over two possible directions, thus affording an intermediate cyclization product A which gives intermediate B by imine-enamine tautomerization. Finally, the acidic methinic proton next to nitro group on pyridine ring is abstracted by the action of base simultaneous cleavage of NO_2 group ensures the fully aromatic regioisomeric products **5** or **6** over two possible separate routes, respectively (Scheme 1).

The structures of title compounds were identified on the basis of IR, NMR, MS. ^{1}H-NMR measurements showed that the most confirmative proton signal for the



Scheme 1. Plausible mechanism for formation of regioisomers **5** and **6**

formation of products (**5** or **6**) was olefinic C-H of pyridine ring. It was a distinct singlet resonated at around 7.00 ppm for all products. Besides, ratio of diastereomers in each mixture were determined according to olefinic = CH peaks in ^{1}H NMR spectra of the products (Table 2, entries 4–9). The NH_2 protons resonated at around 7.90 ppm as singlet and in some of the products; it was observed as a doublet signal. Moreover, different aromatic proton signals due to the isomerization were mostly observed on the benzene ring of benzimidazole, when this was substituted with Cl- or CH_3- groups. Also, HRMS measurements disclosed that both ESI [M-H] and APCl [M + H] values accurately coincide with the molecular formulas of the expected products. Moreover, a fine crystal of **6g** from the regioisomeric mixture, the structure of **6g** was further established by single crystal X-ray analysis and full data have been deposited at the Cambridge X-ray database with number 1995474 (Fig. 2).

Once a new approach for the preparation of functionalized pyrido[1,2-a]benzimidazole was achieved, main aim was to evaluate the influence of different substituents on absorption and fluorescence properties. Figure 3 shows the UV-Vis absorption and fluorescence (FL) spectra of the molecules in dimethylformamide (DMF), which are measured at room temperature. As it is seen in Figure 3a, the absorption for the molecules with *p*-NO_2 substitution on phenyl ring (i.e. **5b**, **5e–6e**, and **5h–6h**) starts at about 600 nm, and a peak maximum appears between 350 and 400 nm, assigned to $\pi \rightarrow \pi^*$ transition. With the change of substituents (e.g. CH_3 and Cl) on the benzimidazole phenyl moiety, no significant changes in the absorption features have been observed. However, when the -NO_2 group was substituted with –CN and –CF_3 substituents at

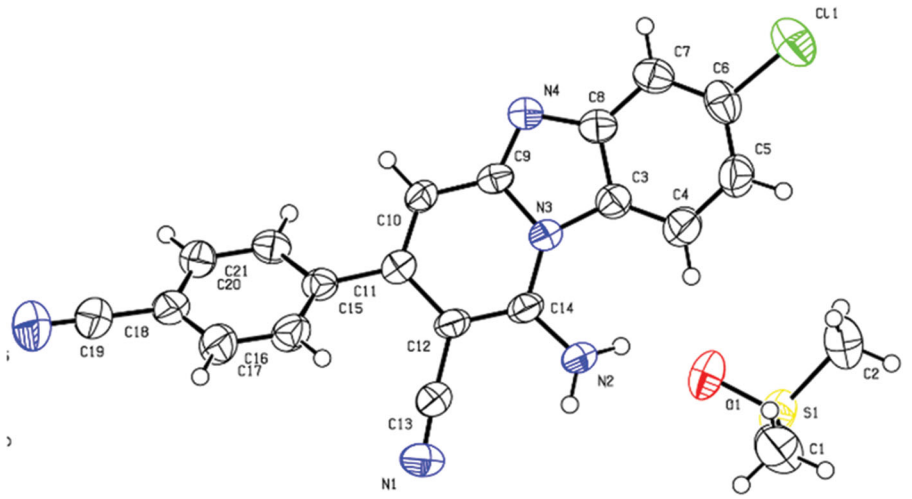


Figure 2. X-ray crystal structure of **6 g**.

para position of phenyl ring, the absorption features were dramatically changed. Figure 3b and c represents the absorption curves of the molecules bearing *p*-CN (i.e. **5a**, **5d–6d**, and **5 g–6g**) and *p*-CF_3 (i.e. **5c**, **5f–6f**, and **5i–6i**) groups, respectively. It is clearly seen that the starting point of the absorption shifts to about 500 nm and a new main peak emerged in the absorption bands. This new peak between 300 and 350 nm might be attributed to internal charge transfer (ICT), which is typically observed in organic dyes having donor-acceptor structure.[21,22] Due to the CT transitions which are one of the possible mechanisms responsible for the fluorescence emission in these materials,[23] the fluorimetric measurements were also performed for all samples. Figure 3d shows the images of all samples illuminated under ultraviolet light (365 nm). The samples bearing *p*-NO_2 substituent on the phenyl ring as an acceptor group (i.e. **5b**, **5e–6e**, and **5 h–6h**) did not exhibited fluorescence properties. However, it was found that the samples showed FL emission when the phenyl ring was para-substituted with CN and CF_3 moieties. Figure 3e and f depicts the FL emission spectra of the samples with *p*-CN and *p*-CF_3 substitutions on the phenyl ring (i.e. **5a**, **5d–6d**, **5g–6g**, and **5c**, **5f–6f**, **5i–6i**), respectively. The fluorescence measurements were performed at room temperature and under 400 nm excitation and the concentration of the samples in DMF was kept at 1.0×10^{-5} M during the measurements to avoid the self-absorption effect. The pyrido[1,2-a]benzimidazoles bearing electron-accepting –CN and –CF_3 groups exhibited bluish and greenish emissions, respectively, and the maximum peak positions of the FL emissions were summarized in Table 3.

In the compounds with the electron-donor groups –CH_3 and –Cl, it was observed that nearly symmetric emission profile was gradually disappeared and a new shoulder started to appear in the longer wavelengths side of the emission band. This new shoulder at longer wavelength has become more apparent in the form of dual emission profile in the case of the sample bearing –CF_3 and –Cl as electron-acceptor and -donor system (i.e. **5i–6i**), respectively (Fig. 3f). Furthermore, full-width half-maximum (fwhm)

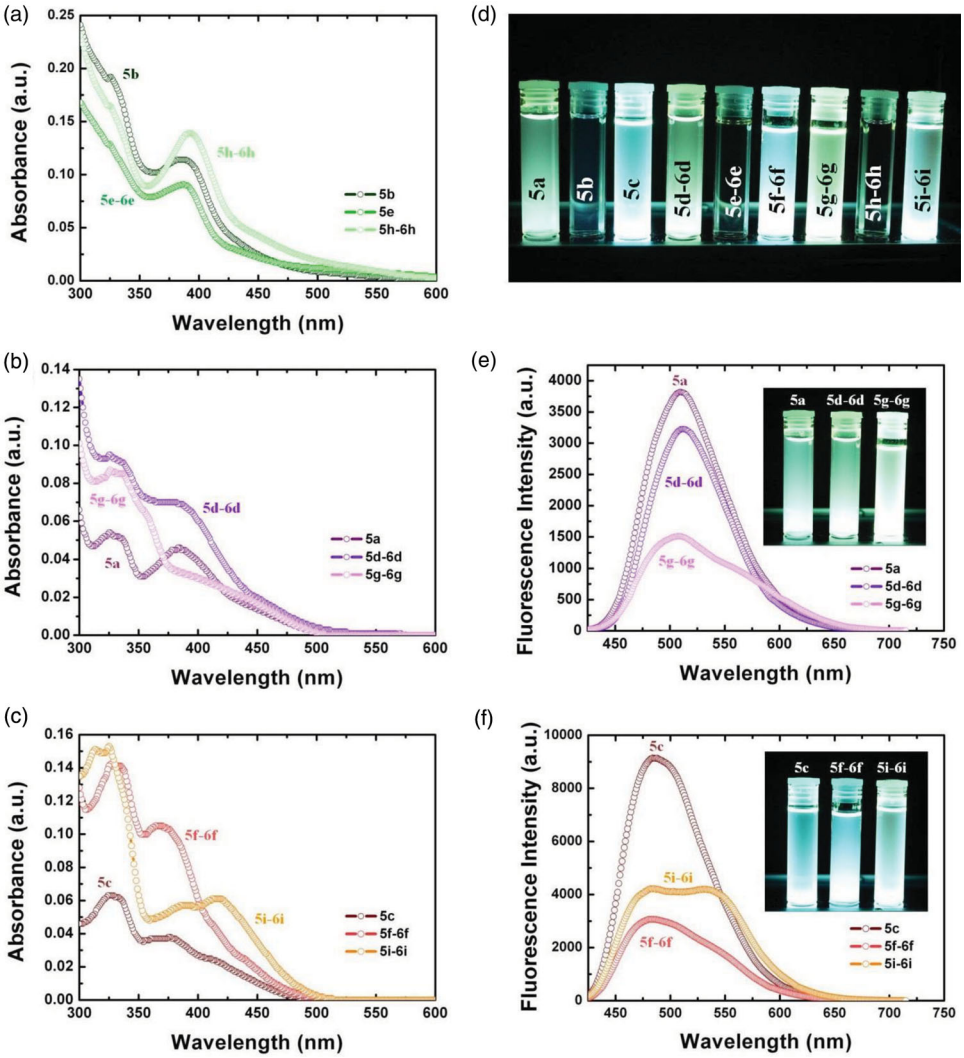


Figure 3. UV-Vis absorption spectra of (a) **5 b**, **5e–6e**, **5 h–6h**, (b) **5a**, **5d–6d**, **5 g–6g** and (c) **5c**, **5f–6f**, **5i–6i**. (d) Photograph of solutions of the samples in DMF under UV-excitation. Fluorescence emission spectra of (e) **5a**, **5d–6d**, **5 g–6g** and (f) **5c**, **5f–6f**, **5i–6i**.

Table 3. Absorption and fluorescence properties of the title compounds.

			Absorption (nm)		FL Emission (nm)		fwhm (nm)
Sample	Donor group	Acceptor group	Peak-1	Peak-2	Peak-1	Peak-2	
5a	H	CN	327	384	510	–	85
5d–6d	CH_3	CN	327	385	512	–	83
5g–6g	Cl	CN	327	385	508	–	114
5b	H	NO_2	–	385	–	–	–
5e–6e	CH_3	NO_2	–	386	–	–	–
5h–6h	Cl	NO_2	–	392	–	–	–
5c	H	CF_3	327	370	483	–	87
5f–6f	CH_3	CF_3	331	370	484	–	105
5i–6i	Cl	CF_3	325	390	483	532	127

of the FL emission peak were found to be increased with introduction of the electron-donor moiety by –H, $–CH_3$, and –Cl, respectively (Table 3). The isomer formation in the case of the $–CH_3$ and –Cl substitutions on benzimidazole for both samples having *p*-CN and *p*-CF_3 as an electron-accepting groups can be responsible for both of the new peaks and the broadening at fwhm in the FL emission profile. When the Cl is positioned instead of CH_3 moiety on the benzimidazole ring, it is observed that the intensity of the newly appeared FL emission peak increases (i.e. **5i–6i**). Also, this changing in electron-donor position give rise to the broadening in fwhm of the FL emission profile. These findings can be an indication of a change in the number of isomer molecules in solution.

Experimental

General procedure for the synthesis of 5-substituted-2-(nitromethylene)-2,3-dihydro-1H-benzo[d]imidazole (1)[24]

A mixture of 1,1-bis(methylthio)-2-nitroethylene (10 mmol, 1650 mg) and *o*-phenylenediamine derivatives (10 mmol) were refluxed in EtOH (25 ml) for 8–12 h. The reaction progress was followed by TLC and upon completion, the precipitated solid was filtered, and washed with cold EtOH (25 mL) to afford the title compound **1** in a pure state.

5-Methyl-2-(nitromethylene)-2,3-dihydro-1H-benzo[d]imidazole (1b)

Yield (72%) L.brown solid. mp 188 °C decomp. IR (KBr): $\nu = 3294$, (NH), 1615 (CN), 1581, 1498, 1429, 1325, 1159, 1127, 1035, 988, 802, 751 cm^{-1}. ^1H NMR (400 MHz, DMSO-d_6) δ 7.44 (*s*, 1H, NH), 7.32 (d, $J = 8.1$ Hz, 1H), 7.24 (*s*, 1H), 7.03 (d, $J = 8.1$ Hz, 1H), 6.79 (*s*, 1H, olefinic CH), 5.97 (*s*, 1H, NH), 2.34 (*s*, 3H, CH_3). ^{13}C NMR (101 MHz, DMSO-d_6) δ δ 147.68, 133.54, 131.26, 129.06, 125.28, 124.73, 112.48, 96.57, 21.61. HRMS (+APCl-TOF) calcd for $C_9H_{10}N_3O_2$ $[M + H]^+$ 192.0773, found 192.0766.

General procedure for the synthesis of 1-amino-3-(4-substitutedphenyl)-methylpyrido[1,2-a]benzimidazole-2-carbonitrile (5,6)

Aromatic aldehyde **2** (0.6 mmol) , heterocyclic enamines **1a–c** (0.5 mmol) and malononitrile **3** (0.6 mmol, 39.6 mg) were mixed in acetonitrile (15 mL) in a round-bottomed flask, and the reaction mixture was stirred and heated for 30 min. Triethylamine (0.75 mmol, 0.105 mL) was added and the reaction mixture was heated under reflux for 3 h. After completion of the reaction, solvent was removed by vacuum evaporation. The resultant crude product was purified by column chromatography on silica gel eluted by using a mixture of hexane:ethyl acetate as eluent to afford single product **5** and inseperable regiosomer mixture **5, 6** of new pyrido[1,2-a] benzimidazoles.

1-Amino-3-(4-cyanophenyl)benzo[4,5]imidazo[1,2-a]pyridine-2-carbonitrile (5a)

Yield (72%, 111 mg) yellow solid. mp 313 °C decomp. IR (KBr): $\nu = 3467$, 3310 (NH_2), 2202 (CN), 1654, 1624, 1593, 1509, 1408, 1251, 838, 722, 549 cm^{-1}. ^1H NMR (400 MHz,

DMSO-d_6) δ 8.48 (d, $J = 8.2$ Hz, 1H), 7.95 (d, $J = 7.8$ Hz, 2H), 7.80 (d, $J = 8.0$ Hz, 3H), 7.75 (d, $J = 7.6$ Hz, 2H, NH_2), 7.52 (*t*, J = 7.4 Hz, 1H), 7.36 (*t*, J = 7.5 Hz, 1H), 6.97 (*s*, 1H, olefinic CH). ^{13}C NMR (101 MHz, DMSO-d_6) δ 152.32, 148.99, 145.87, 142.54, 132.96, 129.98, 128.51, 126.55, 121.92, 119.36, 118.98, 117.62, 115.64, 112.24, 105.38, 105.37, 74.39. HRMS (-ESI-TOF) calcd for $C_{19}H_{10}N_5$ [M-H]- 308.0936, found 308.0915.

Experimental details, characterization data and copies of IR and NMR spectra for all synthesized compounds can be found in publisher's website along with the published article.

Conclusion

We have developed a new and efficient three-component approach for the synthesis of new benzimidazoles substituted at the C-3 position via elimination of a HNO_2 molecule under mild conditions in one-pot manner where one of the components is an electron-deficient aldehyde (**2**). Also, the absorption and fluorescence characteristics of title compounds display their high potential for using in chemical sensors and light-generation optoelectronic applications as optical materials.

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