Multi-component synthesis of spiro[diindeno[1,2-b:2′,1′-e]pyridine-11,3′-indoline]-triones using zinc terephthalate metal-organic frameworks

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ABSTRACT
The present article describes an efficient one-pot method for the preparation of spiro[diindeno[1,2-b:2′,1′-e]pyridine-11,3′-indoline]-trione derivatives from a three-component condensation reaction of 1,3-indandione, aromatic amines and isatins in the presence of a zinc terephthalate metal-organic framework Zn (BDC) MOF as the catalyst under solvent-free conditions. High yields, short reaction times, simple workup and environmentally benign procedure are advantages of this protocol. The Zn (BDC) MOF catalyst can be recovered and reused several times without loss of activity. The catalyst was characterized by scanning electron microscopy, energy-dispersive X-ray spectroscopy, Fourier transform infra red, X-ray powder diffraction and thermal gravimetric analysis.

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Introduction
Multi-component reactions (MCRs) described as one-pot reactions are those in which at least three functional groups combine through covalent bonds. These have gained significance in synthetic organic chemistry. Commonly, filtration of products from MCRs is simple because all the organic reagents utilized are consumed and inserted into the target compound. The benefits of MCRs include a shorter reaction time, energy savings, ease of performance, lower cost, high atom-economy and lack of time consuming, and costly purification processes (1–4).

In this field, heterocycles containing an indenone moiety demonstrate interesting attributes that make them attractive for use in MCRs. The indole moiety is a well-known heterocycle, with the customary and significant properties of different natural products and pharmacological agents (5). It has been suggested that the sharing of the 3-carbon indole atom during the production of spiroindoline derivatives can greatly increase biological activity (6). Many efficient and applied methods have been reported for the synthesis of versatile spiroindole-fused heterocycles (7–9).

Indenone-fused heterocycles represent significant biological and medicinal scaffolds. Indenopyridines display biological activities such as phosphodiesterase inhibitors (10), cytotoxicity (11), adenosine receptor antagonists (12), anti-inflammatory/anti-allergic properties (13) calcium modulation (14) and coronary dilation (15). These compounds have also been studied as a cure for arteriosclerosis, hyper lipoproteinemia (16) and neurodegenerative disease (17). Methods are reported in the literature for the synthesis of indenone-fused pyridines in the presence of various catalysts, including p-TSA (18), PEG-OSO3H/[NMP]H2PO4 (19), Brensted acid ionic liquid (BAIL) (20) and oxalic acid dihydrate: proline LTTM (21).

Metal-organic frameworks (MOFs) are a class of crystalline porous compounds made by the linkage of metal ions or clusters by means of organic ligands (22). MOF-based structures offer benefits such as simplicity of processing, tunable pore size, high surface area, adjustability and stable alternative materials (23,24).
Recently, MOFs have been shown to have applications such as for drug delivery (25), luminescence (26), heterogeneous catalysis (27) and gas storage or separation (28). MOFs have been used as solid catalysts in Suzuki (29), alkene epoxidation (30), Biginelli (31), aldol (32),aza-Michael (33) Sonogashira (34), Friedel–Crafts alkylation and acylation (35), and condensation or reactions. Compared to customary heterogeneous catalysts, MOFs offer a range of chemical compositions, structural and topological properties. MOFs can be prepared with different metal ions and in various coordination environments, making them appropriate for use as catalytic active sites in organic transformations (36).

In order to perform chemical processes with lower waste generation, the solvent-free synthesis is a valuable tools for the researchers. Solvent-free synthesis has emerged as an important path of investigation. It is often claimed that the best solvent is no solvent (37).

In continuation of our research on sustainable approaches for the preparation of organic compounds using nanocatalysts (38–42), the current article reports on the efficient synthesis of functionalized spiro[diindeno[1,2-b:2’,1’-e]pyridine-11,3'-indoline]-triones by MCRs using zinc terephthalate metal-organic frameworks Zn (BDC) MOFs. It is innocuous to the environment, mild, inexpensive, economical, non-toxic and recyclable under solvent-free conditions (Scheme 1, Figure 1).

Results and discussion

In the preliminary experiments, Zn (BDC) MOF was prepared and characterized by scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), Fourier transform infra red (FT-IR), X-ray powder diffraction (XRD) and thermal gravimetric analysis (TGA).

SEM is a useful tool for determining the size distribution, particles shape, and porosity. The morphology and particle size of Zn (BDC) MOF were determined by SEM, Zn (BDC) MOF exhibits the shape of a random slab (Figure S1, see Supplementary Information).

The chemical purity of the samples as well as their stoichiometry was tested by using EDX. Figure S2 (see Supplementary Information) shows that the elemental compositions are the C, O, N and Zn of Zn (BDC) MOF.

Figure S3 (see Supplementary Information) shows the FT-IR spectrum of Zn (BDC) MOF. The spectra of the materials exhibited bands assigned to aromatic groups at 1628 and 823 cm$^{-1}$ and C9O stretching vibration at 1688 cm$^{-1}$ (43). The two sharp peaks at 1388 and 1504 cm$^{-1}$ correspond to the symmetrical and asymmetrical vibrations of COO$^-$, respectively, reflecting the presence of the dicarboxylate linker. The peaks centered at 1628 cm$^{-1}$ were ascribed to C9C stretching vibration of the aromatic.

Figure S4 (see Supplementary Information) shows the X-ray diffraction pattern of Zn (BDC) MOF. The strong XRD intensity of the peak at 9.8° demonstrates that many crystal faces at (220) were formed in the Zn (BDC) MOF. The weak XRD intensity of the three theta peaks at 15.5°, 16.8° and 20.8° reveals a few
crystal faces at (400), (420), and (531), respectively, in Zn (BDC) MOF. These results are consistent with the SEM characterization shown in Figure S1. The lack of some crystal faces indicates that the morphology of Zn (BDC) MOF is a random slab of a certain thickness rather than a cubic crystal. The Zn (BDC) MOF is probably an impure crystal phase with an irregular shape as shown in the SEM photo. This may be caused by the multiple coordination modes in the Zn-MOF crystals that formed the multiple structures.

Figure S5 (see Supplementary Information) shows the TGA curve of Zn (BDC) MOF. The Zn (BDC) MOF exhibited two-stage weight loss in the TGA curve. The first weight loss of the Zn (BDC) MOF occurred below 250°C, indicating the removal of the occluded DMF and/or water from the coordinating polymer frameworks (44). Below 250°C, Zn (BDC) MOF exhibited weight loss of 23%–25% (wt) %. After the first weight loss, the TGA curve for the Zn (BDC) MOF reached a plateau at temperatures of 230–360°C. This illustrates the stability of the framework of the Zn (BDC) MOF structure at the temperature of guest desorption and framework decomposition. The onset temperature of the second weight loss corresponded to the decomposition of organic species in the framework. After this process, the residual weights were only 30–40% of their original weights and mainly consisted of zinc oxide as calculated by their weight loss in the TGA curves. The onset decomposition temperature at 430°C was for Zn (BDC) MOF in the second stage.

In order to determine the optimized reaction conditions, compound 4a (Table 3, Entry 1) was synthesized by the reaction of 1,3-indandione, aniline and isatin as a model study. Next, the reaction conditions were optimized on the basis of the solvent, catalyst and temperature.

To study the effect of the catalyst content on the yield of the reaction, the model reaction was carried out using different amounts of Zn (BDC) MOF under solvent-free conditions. No product could be detected in the absence of the catalyst even after 4 h (Table 1, Entry 1). Good yields were obtained in the presence of the catalyst. The best results were obtained using 0.005 g of the catalyst (Table 1, Entry 4) and increasing the catalyst content did not increase the yield products (Table 1, Entry 5), whereas decreasing the catalyst content leads to a decrease in the product yield.

Next, the effect of temperature was evaluated for the model reaction. Table 2 shows that no product was obtained at room temperature (Entry 1). The reaction was investigated at various temperatures and a high yield of the product was obtained at 80°C within 25 min (Table 2, Entry 4). A further increase in temperature from 80°C to 100°C in this reaction had no significant effect on the time required or product yield.

To investigate this procedure, a series of spiro[diindenopyridine-11,3′-indoline]-triones 4a were synthesized by a three-component

| Entry | Zn (BDC) MOF (g) | Time (min) | Product | Isolated yields (%) |
|-------|------------------|------------|---------|---------------------|
| 1     | –                | 240        | 4a      | –                   |
| 2     | 0.001            | 120        | 4a      | 53                  |
| 3     | 0.002            | 50         | 4a      | 79                  |
| 4     | 0.005            | 25         | 4a      | 96                  |
| 5     | 0.01             | 25         | 4a      | 96                  |

Note: Reaction condition: isatin, 1,3-indandione; aniline and Zn (BDC) MOF were heated at 80°C for the appropriate time.

| Entry | Temperature (°C) | Time (min) | Isolated yields (%) |
|-------|------------------|------------|---------------------|
| 1     | r.t              | 240        | –                   |
| 2     | 40               | 150        | 51                  |
| 3     | 55               | 60         | 73                  |
| 4     | 80               | 25         | 96                  |
| 5     | 100              | 25         | 96                  |

Note: Reaction condition: isatin, 1,3-indandione; aniline (1:2:1) with Zn (BDC) MOF (0.005 g) under solvent-free conditions.
condensation reaction of isatins (1.0 mmol), 1,3-indandione (2.0 mmol) and aromatic amines (1.0 mmol) in the presence of Zn (BDC) MOF (0.005 g) as a robust catalyst under solvent-free conditions (Table 3). The Zn (BDC) MOF was found to be an effective catalytic system which gave the desired products with excellent yields in short reaction times.

Next, to show the merits of the present work, the catalytic efficiency of Zn (BDC) MOF compared with other catalysts was examined in a model study. As shown in Table 4 Zn (BDC) MOF exhibited high activity in comparison with other catalysts and the corresponding product 4a produced an excellent yield and short reaction time (25 min for 4a 96% yield) under solvent-free conditions at 80°C.

**Proposed mechanism**

A plausible mechanism for the synthesis of spiro[diindeno[1,2-b:2′,1′-e]pyridine-11,3′-indoline]-triones (4a–l) using Zn(BDC) MOF as the catalyst is depicted in Scheme 2. The experimental results were examined together with those in the literature (19,20). It is thought that product 4 results from the initial addition of 1,3 indanedione 1 to the isatin 3 to yield intermediate 5, which reacted further with another molecule of 1. Finally, the addition of substitute aniline 2 to intermediate 6, followed by cyclization afforded product 4.

**Experimental**

**General**

Chemicals were purchased from Sigma-Aldrich and Merck in high purity. All of the materials were of commercial reagent grade and were used without further purification. All melting points are uncorrected and were determined in a capillary tube using a Boetius melting point microscope. 1H NMR and 13C NMR spectra were obtained on a Bruker 400 MHz spectrometer with DMSO-d6 as the solvent using TMS as an internal standard. FT-IR spectrum was recorded on the Magna-IR, spectrometer 550. The elemental analyses (C, H, N) were obtained from a Carlo ERBA Model EA 1108 analyzer. The compositional analysis was done by energy-dispersive analysis of X-ray (EDX, Kevex, Delta Class I). Microscopic morphology of the products was visualized by SEM (LEO 1455VP).

**Preparation of (Zn (BDC) MOF)**

Zn (BDC) MOF was prepared according to the method reported in the literature with some modifications (45). The equimolar quantities were for zinc nitrate hexahydrate (2 mmol) and terephthalic acid (2 mmol) in 30 mL of DMF. This solution was placed in a closed flask in an

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**Table 3.** Zn (BDC) MOF catalyzed synthesis of spiro[diindeno[1,2-b:2′,1′-e]pyridine-11,3′-indoline]-triones.

| Entry | Ar   | R   | Product | Time (min) | Yield %a | M.p. °C | M.p. °C Ref. |
|-------|------|-----|---------|------------|----------|---------|-------------|
| 1     | C6H6 | H   | 4a      | 25         | 96       | >300°C  | >300°C (19) |
| 2     | 4-Me-C6H5 | H   | 4b      | 20         | 90       | >300°C  | >300°C (19) |
| 3     | 4-OMe-C6H4 | H   | 4c      | 20         | 90       | >300°C  | >300°C (19) |
| 4     | 4-NO2-C6H4 | H   | 4d      | 25         | 97       | >300°C  | >300°C (19) |
| 5     | 4-Br-C6H5 | H   | 4e      | 30         | 95       | >300°C  | >300°C (19) |
| 6     | C6H6 | Br  | 4f      | 20         | 93       | >300°C  | >300°C (19) |
| 7     | 4-Me-C6H5 | Br  | 4g      | 20         | 95       | >300°C  | >300°C (19) |
| 8     | 4-OMe-C6H4 | Br  | 4h      | 15         | 92       | >300°C  | >300°C (19) |
| 9     | 4-NO2-C6H4 | Br  | 4i      | 25         | 89       | >300°C  | >300°C (19) |
| 10    | 4-Cl-C6H4 | Br  | 4j      | 25         | 94       | >300°C  | >300°C (19) |
| 11    | 4-OMe-C6H4 | Cl  | 4k      | 15         | 98       | >300°C  | >300°C (21) |
| 12    | 4-Cl-C6H4 | Cl  | 4l      | 20         | 95       | >300°C  | >300°C (21) |
| 13    | 4-NO2-C6H4 | Cl  | 4m      | 25         | 90       | >300°C  | >300°C (21) |
| 14    | 4-Cl-C6H4 | NO2 | 4n      | 30         | 89       | >300°C  | >300°C (21) |
| 15    | C6H6 | Cl  | 4o      | 20         | 95       | >300°C  | >300°C (21) |

*a*Isolated yield.

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**Table 4.** Comparison of the catalytic ability of some catalysts reported in the literature for the synthesis of spiro[diindeno[1,2-b:2′,1′-e]pyridine-11,3′-indoline]-trione (4a)a.

| Entry | Catalyst | Conditions | Time/yield (%) | References |
|-------|----------|------------|---------------|------------|
| 1     | p-TSA    | CH3CN/ reflux | 1 h/ 82       | (18)       |
| 2     | [(CH3)3SO2H][HSO4] | H2O/ reflux | 1.45 h/ 82    | (20)       |
| 3     | [NMP]H2PO4 | Ultrasonic/ 40°C | 5–15 min/ 94  | (19)       |
| 4     | PEG-OSO3H | Ultrasonic/ 40°C | 5–15 min/ 92  | (19)       |
| 5     | Oxalic acid; proline LTTM | LTTM/80°C | 20 min/ 94   | (21)       |
| 6     | Zn (BDC) MOF | Solvent-free, 80°C | 25 min/96    | This work  |

*a*Based on the three-component reaction of isatin, 1,3-indandione, aniline.
oven at 100°C for 12 h. The colorless cubic crystals were filtered and washed with 100 mL DMF solution. The filtered material was immersed in chloroform after an exchange with DMF solution for 24 h.

**General procedure for the Zn (BDC) MOF catalyzed synthesis of spiro[diindeno[1,2-b:2′,1′-e]pyridine-11,3′-indoline]-trione derivatives (4a–l)**

A mixture of isatin (1.0 mmol), 1,3-indandione (2.0 mmol) and aniline (1.0 mmol) in the presence of Zn (BDC) MOF (0.005 g) as a catalyst was stirred under solvent-free conditions at 80°C for appropriate time. After completion of the reaction as determined by thin layer

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**Scheme 2.** Proposed mechanism for the synthesis of spiro[diindeno[1,2-b:2′,1′-e]pyridine-11,3′-indoline]-trione 4.

**Figure 2.** Reusability of the Zn (BDC) MOF.
chromatography (TLC), the reaction mixture was dissolved in dichloromethane. The catalyst that was insoluble in CH$_2$Cl$_2$ was separated by simple filtration. The solvent was evaporated and the residue was recrystallized from ethanol to afford the pure product.

All the products were characterized and identified with m.p., $^1$H NMR, $^{13}$C NMR and FT-IR spectroscopy techniques.

Spectral data of some products are listed in Supplementary Information.

5-(4-Chlorophenyl)-5′-Bromo-5H-spiro[diindenol[1,2-b:2′,1′-e]pyridine-11,3′-indoline]-2′,10,12-trione (4j)
red powder, mp: >300°C.$^1$H NMR (400 MHz, DMSO-d$_6$) δ: 5.56 (2H, d, ArH), 6.67–7.92 (13H, m, ArH), 10.45 (1H, s, NH); $^{13}$C NMR (100 MHz, DMSO-d$_6$) δ: 46.1, 61.0, 5, 5.9, 110.9, 111.9, 113.9, 114.4, 117.9, 119.7, 122.2, 125.6, 127.7, 127.9, 128.7, 131.1, 134.4, 143.3, 146.7, 148.9, 165.6, 175.8, 190.0; FT-IR (KBr): 3,404, 3,087, 2,910, 1,705, 1,622, 1,475 cm$^{-1}$; MS (EI) (m/z): 590.00 (M$^+$); Anal. Calcd for: C$_{32}$H$_{16}$BrCIN$_2$O$_3$ (Mr = 591.85) are: C 64.94, H 2.73, N 4.73. Found: C 65.12, H 2.50, N 4.70.

5′-Chloro-5-(4-Methoxyphenyl)-5H-spiro[diindenol[1,2-b:2′,1′-e]pyridine-11,3′-indoline]-2′,10,12-trione (4k)
red powder, mp: >300°C.$^1$H NMR (400 MHz, DMSO-d$_6$) δ: 3.96 (3H, s, OMe), 5.60 (2H, d, ArH), 6.70–3.13 (13H, m, ArH), 10.67 (1H, s, NH); $^{13}$C NMR (100 MHz, DMSO-d$_6$) δ: 46.1, 56.5, 109.3, 113.5, 114.0, 114.5, 118.1, 119.7, 122.9, 125.2, 127.3, 127.7, 128.7, 131.2, 134.4, 144.6, 146.6, 148.8, 165.5, 174.1, 190.1; FT-IR (KBr): 3,404, 3,087, 2,910, 1,705, 1,593, 1,475 cm$^{-1}$; MS (EI) (m/z): 542.10 (M$^+$); Anal. Calcd for: C$_{32}$H$_{16}$ClCIN$_2$O$_3$ (Mr = 512.95) are: C 73.09, H 3.50, N 5.11. Found: C 74.99, H 3.30, N 5.39.

### Reuse of catalyst

The reusability of the catalyst was examined by repeating the model reaction using Zn (BDC) MOF under optimized reactions. After completion of the reactions (as determined by TLC), the residue was dissolved in hot ethanol or CH$_2$Cl$_2$, and the catalyst was filtered and reused six times with only a slight decrease in activity as indicated in Figure 2.

### Conclusions

An efficient, easy and economical method for the synthesis of indenone-fused pyridine heterocycles has been developed using Zn (BDC) MOF as a catalyst under solvent-free conditions. The catalyst can act as both Brønsted and Lewis acid active sites and can be recycled without a significant decrease in activity. This method offers several advantages, including short reaction time, simple workup procedure and excellent yield.

### Disclosure statement

No potential conflict of interest was reported by the authors.

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