FULL PAPER



Silver phosphate supported on metal–organic framework $(Ag_3PO_4@MOF-5)$ as a novel heterogeneous catalyst for green synthesis of indenoquinolinediones

Ryhan Abdullah Rather | Zeba N. Siddiqui 回

Department of Chemistry, Aligarh Muslim University, Aligarh 202002, India

Correspondence

Zeba N. Siddiqui, Department of Chemistry, Aligarh Muslim University, Aligarh, 202002, India. Email: siddiqui_zeba@yahoo.co.in

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Council of Science and Technology, Lucknow, U.P., Grant/Award Number: CST/SERPD/D-283 (Dated, 14 May 2015) A new Ag₃PO₄@MOF-5 catalyst has been synthesized and successfully applied in the green synthesis of a library of indenoquinolinediones via one-pot, multi-component reaction of aldehyde, indanedione and enaminone under solvent-free conditions at room temperature. The catalyst was characterized using various spectroscopic techniques and elemental analysis. The catalyst can be easily separated and exhibits significant recyclability, with insignificant loss of activity after four consecutive runs. The protocol presented has advantages like green reaction conditions, high yield of products, no need for column chromatography and good recyclability.

KEYWORDS

Ag₃PO₄, grinding, indenoquinolinediones, MOF-5, solvent-free

1 | INTRODUCTION

In recent years there have been remarkable developments in the synthesis of porous hybrid materials.^[1,2] One such interesting category of porous material is metal-organic frameworks (MOFs).^[3] They find budding applications in gas storage and separation, nonlinear optics, photoluminescence, biomedicine, magnetic and electronic materials, etc.^[4–9] The striking features of MOFs are their crystalline nature, high specific surface area (up to 4000 m² g⁻¹),^[10] large pore aperture $(98^{\circ}\text{\AA})^{[11]}$ and low density (0.13 g cm⁻³).^[12] The most substantial feature of MOFs is their potential inner porosity so that guest molecules can access the pores which are important for catalytic purposes.^[13] A variety of heterogeneous MOF-based catalysts has been reported over the past few decades obtained by hosting various types of catalytic sites.^[9,14] The active sites, both metal centres and organic linkers, contribute to catalytic activities.^[15] Especially, organic bridging linkers may be used as frameworks with which various catalytic complexes, biomolecules and homogeneous catalysts can be immobilized or

encapsulated. Furthermore, the synthetic flexibility of MOFs gives considerable control over the size and environment of the pores, permitting selectivity to be tuned more effectively.^[16] MOF pores can serve as hosts for small guest molecules (active homogeneous catalysts) or as supports for metal or metal oxide nanoparticles.^[17] These properties distinguish MOFs from other porous materials such as zeolites and activated carbons, and hence they have found increasing applications as heterogeneous solid catalysts for the synthesis of organic compounds.^[18]

A variety of metal-based MOFs have been designed and screened in heterogeneous catalysis for various organic transformations.^[19–22] In this context particular attention has been paid to silver salts due to high carbophilicity of silver compounds.^[23] Silver-mediated reactions have emerged as a frontier area in organic chemistry. The excellent reactivity and high selectivity exhibited by silver salts in the activation of various organic substrates place them among the most prominent reagents in the organic field. Various silver salts have been used in a variety organic reactions like Mannich,



SCHEME 1 Schematic representation of synthesis of catalyst

cycloaddition, aldol, coupling, Michael addition, alkynylation, allylation, domino and tandem reactions.^[24,25] Silver-catalysed organic transformations feature high functional group tolerance, excellent regio-, diasterio- or enantioselectivity and high product turnover numbers under mild reaction conditions.^[24] However, Ag₃PO₄ catalysts always suffer from stability concerns because they can be photochemically decomposed if no sacrificial reagent is involved, which leads to their limited practical application. Thus, much research has focused on ways of increasing Ag₃PO₄ stability through surface modification.^[26,27]

Indenoquinoline derivatives have received considerable attention because of their broad range of applications in medicinal, bioorganic and industrial chemistry as well as in the field of synthetic organic chemistry. Their derivatives have been found to exhibit various biological activities like 5-HT-receptor binding^[28] and antiinflammatory activities,^[29] and are antitumor agents,^[30] steroid reductase inhibitors,^[31] acetylcholinesterase inhibitors,^[32] antimalarials,^[33] new potential topo I/II inhibitors^[34] and antituberculosis agents.^[35] Because of the biological activities they exhibit, these compounds have distinguished themselves as heterocycles of



FIGURE 1 FT-IR spectral analysis of (a) of MOF-5, (b) Ag₃PO₄@MOF-5and (c) recycled Ag₃PO₄@MOF-5

profound chemical and biological significance. Thus the development of efficient methodologies for the synthesis of such compounds has attracted considerable attention.^[36]

With the increasing application of MOFs as solid catalysts for the synthesis of nitrogen-containing heterocycles and in continuation of our ongoing research,^[37] herein we report the synthesis of MOF-5-immobilized Ag_3PO_4 as a novel solid catalyst. The catalyst efficiently catalysed multicomponent reactions resulting in the formation of indenoquinolines in excellent yield under solvent-free conditions.

2 | EXPERIMENTAL

2.1 | Preparation of MOF-5

MOF-5 was synthesized according to a reported procedure with slight modification.^[38] In a typical procedure, terephthalic acid (5.065 g, 30.5 mmol) and trimethylamine (8.5 ml) were dissolved in 400 ml of dimethylformamide (DMF). Zn $(OAc)_2 \cdot 2H_2O$ (16.99 g,



FIGURE 2 XRD analysis of (a) MOF-5, (b) fresh Ag₃PO₄@MOF-5 and (c) recycled Ag₃PO₄@MOF-5



FIGURE 3 Solid-state ¹³C MAS NMR spectrum of Ag₃PO₄@MOF-5

77.4 mmol) was dissolved in DMF (500 ml) and gradually added to the organic solution with constant stirring over 15 min, forming a precipitate. After the formation of the precipitate, the mixture was stirred further for 2.5 h. The precipitate was filtered and immersed in DMF (250 ml) overnight. It was then filtered again and immersed in CHCl₃ (350 ml, HPLC grade). The solvent was exchanged three times over 7 days: after 2 days, 3 days and 7 days. The bulk of the solvent was decanted and the product evacuated overnight to a pressure of 10 mTorr. The product was activated at 120°C for 6 h.

2.2 | Preparation of Ag₃PO₄@MOF-5

The prepared MOF-5 (2 g) was suspended in 50 ml of water in a 100 ml round-bottom flask and stirred for 30 min at room temperature. To this was added disodium phosphate (Na₂HPO₄; 0.1 g) and again stirred for 30 min at room temperature. Then in a separate 50 ml beaker, AgNO₃ (1 g) was dissolved in water (20 ml) and added to the round-bottom flask containing the solution of MOF-5 and Na₂HPO₄. The reaction mixture was stirred for 6 h at room temperature. The resultant precipitate was filtered, washed repeatedly with ethanol and dried at 120°C under vacuum for 24 h. The amount of silver incorporated into MOF-5 was found to be 4.4% using inductively coupled plasma atomic emission spectroscopy (ICP-AES).

2.3 | General procedure for synthesis of indenoquinolinediones (4a-n)

A mixture of aromatic aldehyde **1a-k** (1 mmol), active methylene compound 1,3-indanedione (**2**; 1 mmol),

β-aminoketone **3a–k** (1 mmol) and a catalytic amount of Ag₃PO₄@MOF-5 (200 mg) was ground together in a mortar with a pestle at room temperature for a specified period. On completion of reaction (as monitored by TLC), methanol (20 ml) was added and the reaction mixture filtered. The catalyst was washed with methanol several times. The solvent was evaporated under reduced pressure to afford the product in almost pure form, which was further purified by crystallization from suitable solvents (Supporting information).

3 | **RESULTS AND DISCUSSION**

Scheme 1 illustrates the synthesis of the catalyst.

The catalyst was characterized thoroughly using Fourier transform infrared (FT-IR) spectroscopy, powder X-ray diffraction (XRD), solid-state NMR spectroscopy, scanning electron microscopy (SEM), energy-dispersive



FIGURE 4 Raman spectra of (a) MOF-5 and (b) Ag₃PO₄@MOF-5



FIGURE 5 SEM images of (a, b) MOF-5, (c) Ag₃PO₄@MOF-5 and (d) recycled Ag₃PO₄@MOF-5

X-ray spectrometry (EDX), elemental mapping, transmission electron microscopy (TEM), Brunauer–Emmett– Teller (BET) measurements, thermogravimetric analysis (TGA), differential thermal analysis and ICP-AES.

FT-IR spectra (Figure 1) confirmed the presence of all functional groups present in MOF-5. Two peaks obtained at 1601 and 1388 cm⁻¹ (Figure 1a) were due to symmetric and asymmetric stretching of C—O bonded to Zn.^[39] Several small peaks occurring in the range 1135 to 952 cm⁻¹ corresponded to in-plane bending of C—H bond present in the benzene ring of the BDC linker. Similarly, the small peaks occurring between 900 and 670 cm⁻¹ were assigned to out-of-plane bending of C—H bond of the benzene ring of the BDC linker.^[40] The peaks occurring



FIGURE 6 TEM image of Ag₃PO₄@MOF-5

at *ca* 500 cm⁻¹ were characteristic of Zn–O stretching. The sharp peak at 550 cm⁻¹ (Figure 1b) in the spectrum of the Ag₃PO₄@MOF-5 catalyst corresponded to bending vibrations of O=P–O. Two other peaks at 821 and 1027 cm⁻¹ corresponded to symmetric and asymmetric stretching vibrations of P–O–P bond whereas that at 1386 cm⁻¹ were due to stretching vibrations of P=O bond.^[41] The broad peak at 3342 cm⁻¹ indicated the minor quantity of adsorbed O–H groups on the MOF-5 structure.

The XRD pattern of MOF-5 (Figure 2a) displayed characteristic peaks at $2\theta = 6.1^{\circ}$, 9.1° , 17.2° and 27.1° .^[42] The peaks at $2\theta = 21.1^{\circ}$, 29.6° , 33.1° , 36.5° , 47.7° , 52.3° , 57.4° , 61.5° and 71.4° were due to Ag₃PO₄ (Fig. 2b) in addition to peaks due to MOF-5.^[43]

The solid-state ${}^{13}C$ MAS NMR spectrum of Ag₃PO₄@MOF-5 is depicted in Figure 3. The signals



FIGURE 7 EDX analysis of Ag₃PO₄@MOF-5



FIGURE 8 Elemental mapping of (a) Ag₃PO₄@MOF-5, (b) carbon, (c) oxygen, (d) zinc, (e) silver and (f) phosphorus

observed in the spectrum corresponded to 28.32, 34.92, 126.34, 134.02 ppm (4C, CH), 138.87 and 144.08 ppm (2C) and 168.30 and 171.48 ppm (2C, 2C=O) supporting the structure of MOF-5 in the catalyst. The signals at 29.32 and 34.92 ppm were due to trapped DMF in $Ag_3PO_4@MOF-5$.^[44]

In the Raman spectra of MOF-5 and Ag₃PO₄@MOF-5 (Figure 4), a noticeable peak at 1640 cm⁻¹ was attributed to C=O stretching of carboxylate group^[45] whereas the peaks at 1460 and 1164 cm⁻¹ were due to in-plane vibrations of benzene ring. Another peak at 892 cm⁻¹ appeared as caused by out-of-plane deformation modes of C-H bond. The peaks at 575 and 715 cm⁻¹(Figure 4b) were attributed to P-O bending stretching vibration of

 Ag_3PO_4 .^[46] The peak at 254 cm⁻¹ may be attributed to H–O–H.

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SEM images (Figure 5a,b) illustrated the preservation of characteristic cubic symmetry of MOF-5. It was evident from the image that Ag_3PO_4 had efficiently doped on the surface of MOF-5 (Figure 5c). TEM observations also clearly showed the deposition of Ag_3PO_4 (black dots) on the surface of MOF-5 (Figure 6). Furthermore EDX confirmed the presence of C, O, P, Ag and Zn elements in the structure of the material (Figure 7), while the elemental mapping of individual elements C (Figure 8b), O (Figure 8c), Zn (Figure 8d), Ag (Figure 8e) and P (Figure 8f) showed their uniform distribution on the surfaces of MOF-5. ICP-AES analysis of the material showed that the amount of Ag in the material was



FIGURE 9 BET analysis of Ag₃PO₄@MOF-5



FIGURE 10 TGA of Ag₃PO₄@MOF-5



SCHEME 2 Effect of catalyst on the model reaction

TABLE 1 Effect of various catalysts on reaction under solventfree conditions^a

Entry	Catalyst	Time (h) ^b	Yield (%) ^c
1	AgNO ₃	4	62
2	Ag ₂ SO ₄	5	60
3	Ag ₂ CO ₃	6	58
4	Ag ₂ WO ₄	6.5	58
5	Ag ₃ PO ₄	2	68
6	MOF-5	1.5	72
7	Ag ₃ PO ₄ @MOF-5(200 mg)	7 min	96

^aReaction of *para*-chlorobenzaldehyde (**1a**; 1 mmol), indane-1,3-dione (**2**; 1 mmol) and β -aminoketone (**3a**; 1 mmol) in presence of different catalysts (10 mol%) under solvent-free grinding conditions at room temperature.

^bReaction progress monitored by TLC.

^cIsolated yield.

4.4 wt% which corresponds to a loading amount of 0.40 mmol g^{-1} of the catalyst.

The specific surface area of Ag_3PO_4 @MOF-5 was calculated using the BET method and was found to be 721 m² g⁻¹ (Figure 9) which was lower than that of MOF-5, i.e. 890 m² g⁻¹.^[47] The appreciable decrease in surface area may be due to deposition of Ag_3PO_4 on the surface of MOF-5.

The thermal stability of Ag_3PO_4 @MOF-5 was studied by carrying out TGA experiments. As shown in Figure 10, two main steps of weight loss were observed. The first step displayed a weight loss of 12.38%, which was caused by loss of adsorbed solvent or water molecules before 254.30°C. The second step showed a weight loss of about 1.58 wt% from 254.30 to 471.82°C, resulting from thermal decomposition of MOF-5.^[48]

3.1 | Optimization of reaction conditions

To optimize the catalytic reaction conditions for the synthesis of indenoquinoline derivatives, we chose 2, parachlorobenzaldehyde (1a) and β -aminoketone 3a as model substrates. Several silver salts were tested as source of silver to enhance the reaction under solvent-free conditions by grinding at room temperature (Scheme 2; Table 1). As evident from the table, most silver salts afforded moderate yield of product. The best result was obtained in terms of yield and reaction time with silver phosphate (Table 1, entry 5) as compared to silver nitrate, silver sulfate, silver carbonate and silver tungstate (Table 1, entries 1-4). The reaction with MOF-5 alone afforded good yield of indenoquinoline derivative 4a but took a longer time period (Table 1, entry 6). However, the yield of the product was enhanced markedly in a very short period of time when MOF-5-supported silver phosphate was tested as catalyst for the model reaction (Table 1, entry 7). To explore the viability of our solvent-free, grinding protocol at room temperature for the synthesis of indenoquinolinediones, various solvents like ethanol, methanol, water, acetonitrile, chloroform, acetic acid, acetone and isopropyl alcohol were screened. It was observed that these solvents could not produce satisfactory results in terms of yield of product and time period (Scheme 3; Table 2, entries 1-10). Solvents like poly(ethylene glycol) (PEG) also did not work (Table 2, entries 11-13). Finally when the reaction was carried out by grinding under solvent-free conditions at room temperature, product formation occurred within 7 min with 96% yield (Table 2, entry 14). In order to determine the optimized amount of the catalyst, the model reaction was carried out with varying amounts of the catalyst



SCHEME 3 Effect of solvent on the model reaction

TABLE 2 Comparative study of various solvents versus the solvent-free method^a

Entry	Solvent (5 ml)	Temperature (°C)	Time (h) ^b	Yield (%) ^c
1	Ethanol	Reflux	3	62
2	Ethanol	RT	5	55
3	Methanol	Reflux	4	60
4	Methanol	RT	6	48
5	Water	Reflux	5	50
6	Acetonitrile	Reflux	6	45
7	Chloroform	RT	7	44
8	Acetic acid	Reflux	2.5	76
9	Acetone	RT	7	42
10	Isopropyl alcohol	Reflux	7	40
11	PEG-200	Reflux	8	38
12	PEG-400	Reflux	8	38
13	PEG-600	Reflux	8	38
14	Solvent free	Grinding (RT)	7 min	96
15	Solvent free	Grinding (RT)	15 min	94
16	Solvent free	Grinding (RT)	20 min	90

^aReaction of *para*-chlorobenzaldehyde (**1a**; 1 mmol), indane-1,3-dione (**2**; 1 mmol) and β -aminoketone (**3a**; 1 mmol) in presence of Ag₃PO₄@MOF-5 (200 mg) under various conditions.

^bReaction progress monitored by TLC.

^cIsolated yield.

(Scheme 4; Table 3). It was found that the formation of indenoquinolinedione derivative increased linearly with an increase in the amount of catalyst from 60 to 200 mg.

3.2 | Catalytic reaction

After optimization of reaction conditions, the choice of substrate for the Ag₃PO₄@MOF-5-catalysed synthesis of indenoquinolinedione derivatives was examined. The results showed that the reaction tolerated both aldehydes and β -aminoketones with electron-withdrawing as well as electron-releasing groups efficiently (Scheme 5; Table 4). The reaction also significantly tolerated heteroaromatic aldehydes like 4-pyridinecarboxaldehyde,

TABLE 3 Effect of catalyst loading on reaction^a

Entry	Catalyst (mg)	Time (min) ^b	Yield (%) ^c
1	60	120	45
2	100	60	60
3	140	45	72
4	180	30	82
5	200	7	96
6	220	7	96

^aReaction of *para*-chlorobenzaldehyde (**1a**; 1 mmol), indane-1,3-dione (**2**; 1 mmol) and β -aminoketone (**3a**; 1 mmol) in presence of various catalyst loading under solvent-free grinding conditions at room temperature.

^bReaction progress monitored by TLC.

^cIsolated yield.

indole-3-carboxaldehye, 5-methyl-2-thiophenecarboxaldehyde and chloro-3-pyrazolecarboxaldehyde as substrates, giving products in excellent yields (Table 4).

In order to show the superiority of our protocol, a comparison with reported methods was done (Table 5). In comparison to reported procedures, our catalytic system was found to be more efficient in terms of time period and product yield.

3.3 | Reaction mechanism

The mechanism of the synthesis of indenoquinoline derivatives has been studied carefully by many chemists.^[52] Based on these reports, a mechanism (outlined in Scheme 6) can be selected as the most probable one. From a mechanistic point of view, first the aldehyde group of 1 is activated by the catalyst after which the first step is the formation of Knoevenagel product A. The second key intermediate is enamine 3, produced by the condensation of dimedone with aromatic amine. Condensation of these two fragments gives the acyclic Michael adduct intermediate B, which undergoes intramolecular cyclization with participation of the amino function and one of the indanedione carbonyl groups to form C, and subsequent loss of water molecule leads to the formation of indenoquinolone derivative 4. The catalyst could be regenerated in the last step and reused for further catalytic cycles.



SCHEME 4 Effect of catalyst loading on the model reaction



 $=3-CH_3C_6H_4, C_6H_5, 4-CH_3C_6H_4$







(Continues)

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TABLE 4 (Continued)



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TABLE 4 (Continued)



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^aReaction progress monitored by TLC.

^bIsolated yield.

 $^{c}TOF = TON/reaction time (h); TON = no. of moles of the starting materials being converted per mole of active site of the catalyst.$

TABLE 5 Comparison of efficiency of Ag₃PO₄@MOF-5 catalyst with that of other reported procedures

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Entry	Catalyst	Condition	Solvent	Yield (%)	Time	Ref.
1	Ag ₃ PO ₄ @MOF-5	Grinding, RT	_	96	7 min	Present work
2	_	Reflux	Ethanol	56	24 h	[49]
3	_	Microwave	Acetic acid	80	20 min-4 h	[50]
4	PTSA	Reflux	Ethanol	75	3–4 h	[51]



SCHEME 6 Plausible mechanism for synthesis of indenoquinoline derivatives

3.4 | Leaching study of catalyst

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The leaching of the catalyst before and after five catalytic cycles was studied using ICP-AES analysis. The analysis showed that the Ag concentration before (4.4 wt% Ag) and after recycling experiments (3.7 wt% Ag) was in fair agreement (within the experimental error). This indicates that Ag is tightly bound to the support and very little leaching of the catalyst occurs upon its reuse. To establish the heterogeneity of the catalyst, ICPAES analysis of the filtrate after extracting the product and filtering the catalyst was carried out, and the result revealed the absence of Ag in the filtrate. These results confirmed that the structure of the catalyst is stable and Ag is tightly bound to the support, which in turn facilitates efficient recycling.

3.5 | Catalyst recycling

Catalyst recycling experiments were performed in order to explore the extent of the recyclability of our catalytic system. The reaction between *para*-chlorobenzaldehyde, indane-1,3-dione and β -aminoketone under grinding conditions at room temperature using Ag₃PO₄@MOF-5 (200 mg) as a catalyst was chosen as model reaction. After completion of the reaction, the catalyst was recovered by extracting the mixture with ethyl acetate followed by



FIGURE 11 Recycling data for Ag₃PO₄@MOF-5 catalyst

filtration. The catalyst was then washed with ethyl acetate and reused for subsequent cycles. The catalyst was found to retain its activity for a minimum of four reaction cycles displaying a high catalytic performance with over 90% yield of the product (Figure 11).

4 | CONCLUSIONS

In summary, we have easily synthesized a new supported catalyst, Ag_3PO_4 @MOF-5, and used it for the green and sustainable synthesis of indenoquinoline derivatives. The catalyst was found to be highly efficient and could be reused for four catalytic cycles. No need for column chromatography for purification of compounds, solvent-free conditions, substrate tolerance and good yield of products are some of the clear achievements of this protocol. This newly developed energy-sustainable strategy provides a good alternative to reported methods.

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ORCID

Zeba N. Siddiqui D https://orcid.org/0000-0002-0952-8608

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