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A Regioselective Approach to Synthesize Indolyl Diketone Derivatives via Magnetic Polymeric Copper-Catalyst

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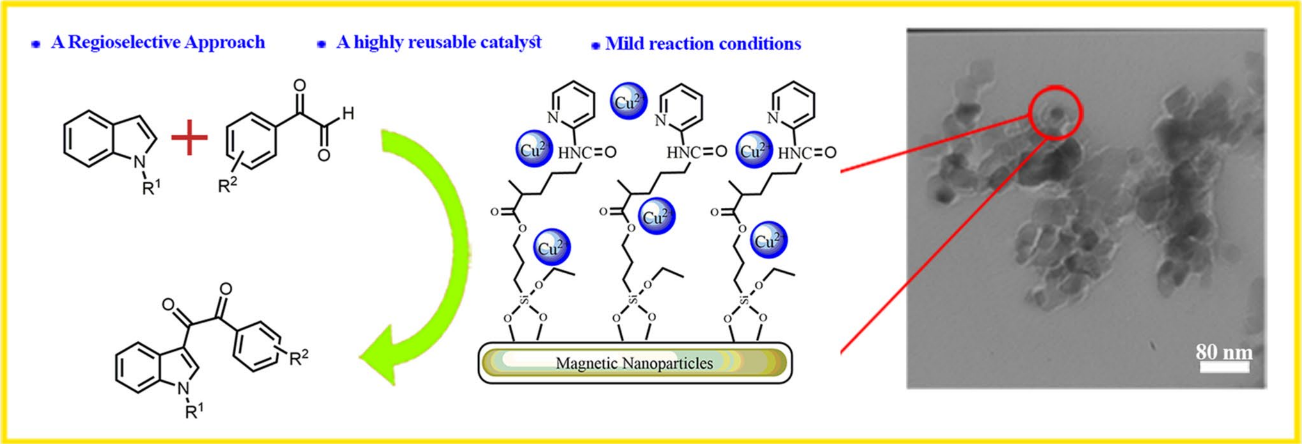
Abstract

In the present paper, an efficient Cu-catalyzed regioselective acylation of indoles with phenylglyoxals was developed which is the first example of indolyl diketones synthesis by a heterogeneous catalyst. The magnetic polyacrylonitrile was synthesized through anchoring acrylonitrile monomers on Fe_3O_4 nanoparticles surface and then modified with 2-aminopyridine. At the final step, copper nanoparticles were immobilized onto the polymeric support containing stable ligands from functionalized nitrile groups of polyacrylonitrile. The different techniques such as Fourier transmission infrared spectroscopy, X-ray diffraction, Field emission scanning electron microscopy, Transmission electron microscopy, and Thermogravimetric analysis were used to prove the structure of the polymeric catalyst. Finally, the activity of the magnetic PAN Cu-catalyst was investigated as an efficient magnetic heterogeneous catalytic system to synthesize 3-acylated indoles with good to high yields. The synthesis of a variety of 3-acylated indoles derivatives was illustrated ready access to this important building blocks class.

Graphic Abstract



Keywords Acylation · C–H functionalization · Heterogeneous catalysis · Indole moieties · Nitrogen heterocycles

1 Introduction

Indole moieties have been known as important building blocks in biologically active compounds, natural products, pharmaceuticals and ubiquitous intermediates in organic synthesis [1–3]. For instance, these heterocyclic compounds are fundamental scaffolds in a large number of anti-cancer, anti-HIV and type 2 diabetes' treatment drugs [4].

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3-Acylindoles are among the most value-added structures because they are not only privileged structures in medicinal chemistry and drug design but also ideal intermediates for the preparation of versatile indole derivatives (Fig. 1) [5–7]. Owing to their carbonyl groups, acylindoles have attracted synthetic organic chemists' interest for versatile transformations such as carbon–carbon and carbon-heteroatom coupling reactions and also polyheterocyclic compounds fabrication as efficient probes for Hg^{2+} and Fe^{3+} [1–3, 8]. The 3-acylindole has proven successful for the heteroannulated 3-(2-aminophenyl)-pyridines synthesis, a significant structure in drug discovery and drug design, using electron-donating aminoheterocycles with 3-acylindoles [9]. It is also used for the synthesis of SB-216763, selective glycogen synthase kinase-3 inhibitor, containing condensation of (aryl or indolyl)glyoxyl esters with (aryl or indolyl)acetamides [10].

Thereupon, the new preparation methods for the synthesis of 3-acylindoles have received intense attention and to date, the carbo-acylation of indoles synthesis with transition-metal-catalyst were disclosed in the literature reports [11–14]. For instance, Xiang and Li's group illustrated a new method to synthesize 3-acylation indole derivatives by oxidative cross coupling of indoles and α-amino carbonyl compounds with aim of Pd-catalyst (Scheme 1a) [1]. Yang and Ji's group reported copper-catalyzed the construction of 3-acylated indoles using α-carbonyl aldehydes in good to excellent yields (Scheme 1b) [15]. In addition, Zhang and Duan et al. provided an expedient synthetic method for 3-acylindoles with the aid of α-carbonyl aldehydes and $Cu(OAc)_2$ as a homogeneous catalyst. A broad substrate scope and moderate to good yields were some advantages of their method [16]. In 2017, the synthesis of 3-acylindoles was developed by Yi et al., they applied iron as a catalyst and indoles with α-amino carbonyl compounds as easily available starting materials (Scheme 1c) [17]. The high atom economy synthesis of dicarbonyl indoles investigated by Guo and his co-workers. They applied styrenes and indoles as starting materials under metal-free conditions [18].

Despite of such efforts, there are some restrictions, such as the need for expensive palladium, side reactions, reusability and separation problems of catalysts, and the more important poor selectivity.

To the best of our knowledge, no examples of heterogeneous catalysts have been disclosed to synthesize indolyl diketone derivatives before. Inspired by our aforementioned background, we decided to apply a heterogeneous catalyst involving stable ligands for the immobilization of copper to improve the reaction conditions [19]. Among different catalyst supports, polyacrylonitrile (PAN) has been attractive with broad properties such as superior catalytic activity, superior thermal stability, low cost, and accessibility [20–22]. Since, PAN has plentiful cyano groups, it is the main precursor for a series of functional groups such as tetrazoles, amidines, amines, aldehydes, amides and

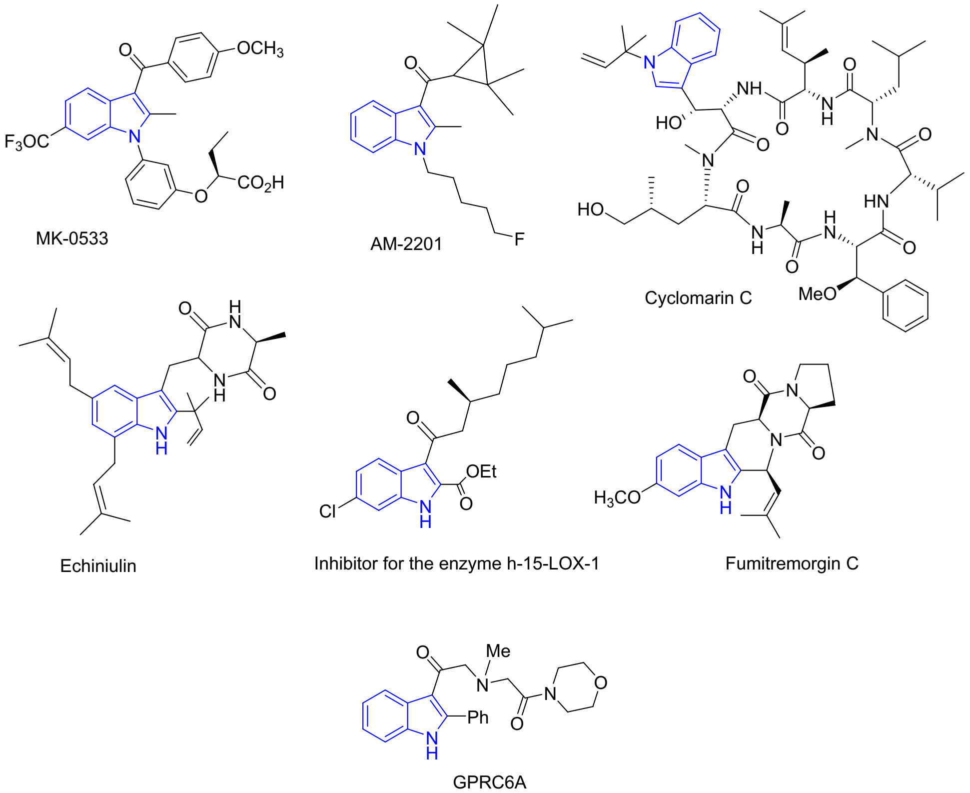
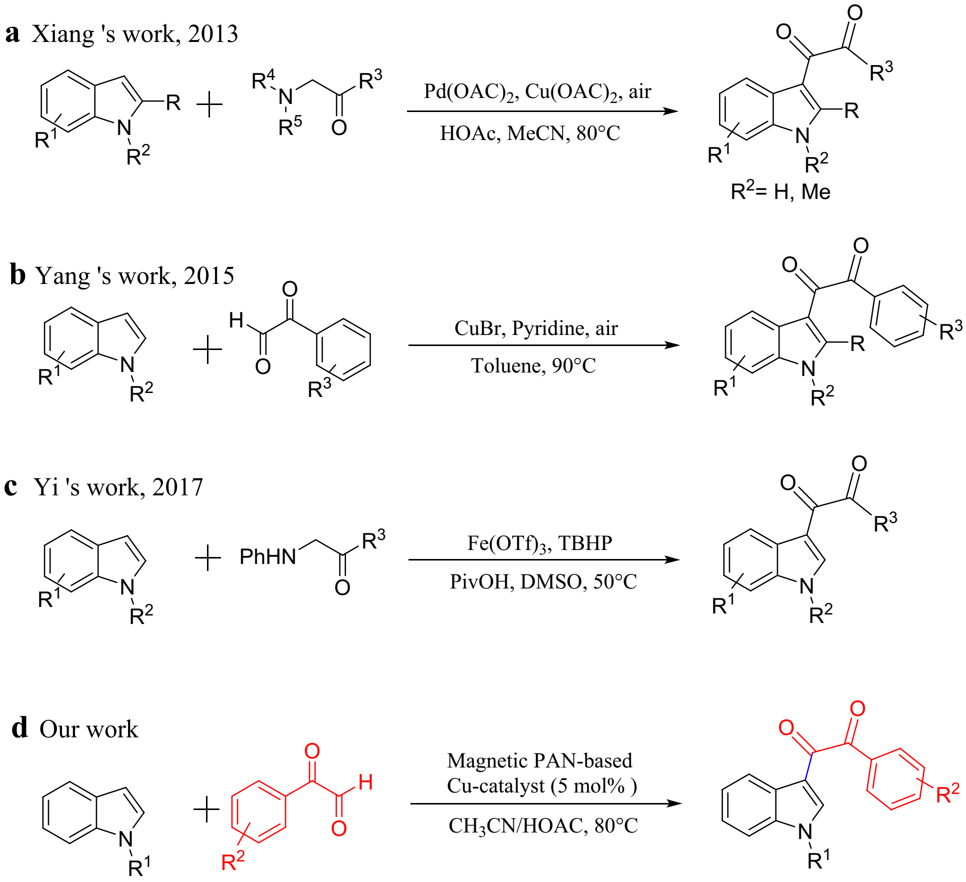


Fig. 1 Examples of biologically active compounds based on 3-acylated indoles

Scheme 1 Synthesis of 3-acylindoles



carboxyl derivatives which makes it an excellent candidate to immobilize various transition metals [23, 24].

On the other hand, incorporation of Fe_3O_4 nanoparticles with the polymeric chains can enhance nanocomposite properties like its chemical, thermal, and mechanical stability compared to the pure polymer. In addition, homogeneous dispersibility, fast and easy separation, excellent stability, and high surface area are some advantages of magnetic catalysts under different conditions [25].

Thus, the preparation of viable catalytic system including magnetic PAN as a catalyst support and copper nanoparticles can be useful to construct the 3-acylated indoles regioselectively in mild reaction conditions with excellent yields (Scheme 1d).

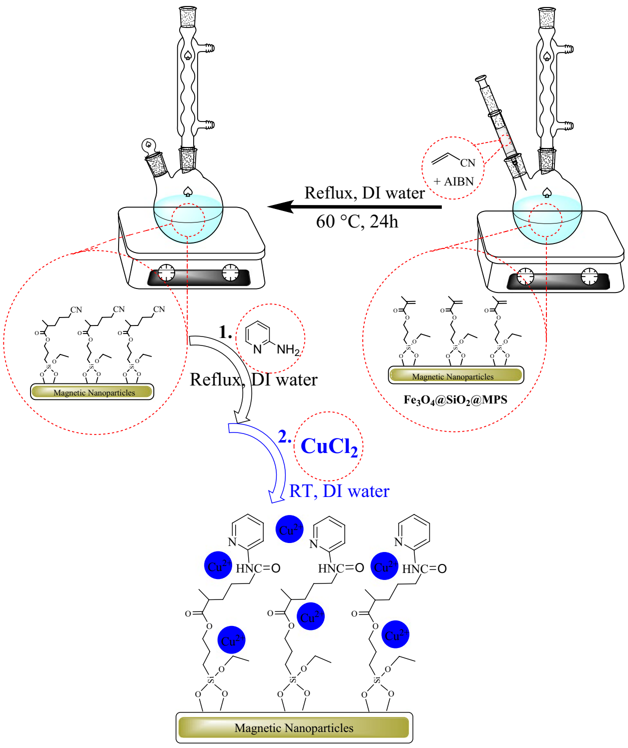
2 Results and Discussion

2.1 Synthesis of the Catalyst

The magnetic PAN support was synthesized through a several-step procedure, including the synthesis of Fe_3O_4 nanoparticles modified with 3-(trimethoxysilyl)propylmethacrylate (MPS) to prepare the magnetite surface for polymerization. Afterwards, the acrylonitrile monomers and initiators were added to the flask equipped with a condenser and heated which led to complete the reaction. In the following step, the magnetic PAN support was modified with 2-aminopyridine (2-AP). Finally, the immobilization of copper nanoparticles on the magnetic PAN/2-AP support was performed by dispersion of the magnetic PAN/2-AP support in a $CuCl_2$ solution. In this step, the copper ions were coordinated with various functional groups in the prepared support (Scheme 2). After proving the catalyst structure, our investigation was initiated using indole and arylglyoxal as coupling partners in the presence of 5 mol% of the magnetic PAN-based Cu catalyst to provide a variety of corresponding products. The activity of the catalyst was examined through C_3 functionalized indoles which suffers from competing reactions between N_1, C_2 and double acylated at N_1 and C_3 positions because the aromatic system has intrinsic reactivity [26, 27].

2.2 Characterization of the Catalyst

According to FT-IR spectroscopy, all spectra with demonstrated bands at 576, 1100, 1412 and 1718 cm^{-1} have been identified as Fe–O, Si–O, C=C (for MPS) and C=O (for



Scheme 2 Preparation procedure of the magnetic PAN Cu-catalyst

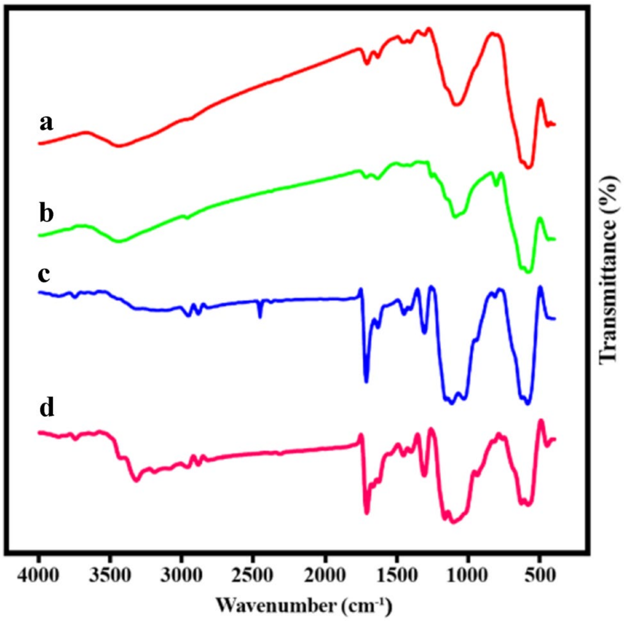


Fig. 2 FT-IR spectra of **a** $Fe_3O_4@SiO_2$, **b** $Fe_3O_4@SiO_2@MPS$, **c** $Fe_3O_4@PAN$, **d** Final Cu-catalyst

MPS) stretching vibrations which ensures the successful coating of MPS on the Fe_3O_4 surface (Fig. 2b) [28]. The presence of PAN on the Fe_3O_4 core–shell was proved by the characteristic peak at 2375 cm^{-1} assigned to cyano stretching vibrations, which its intensity decreased after modification by 2-AP in the next step (Fig. 2c). As can be seen in Fig. 2d, the final catalyst showed pyridine-related peaks at 1671 cm^{-1} (N-sp^2 stretching vibration), 1176 cm^{-1} (N-sp^3 C bond), and 641 cm^{-1} (C−H bending vibration of pyridine) [29, 30].

The XRD pattern indicates that the crystalline nature of Fe_3O_4 nanoparticles has preserved during catalyst synthesis; based on comparing data with JCPDS Card (No.98-011-1284) [19]. Also, no sharp diffraction peaks are detected for PAN which shows the amorphous structure of this polymer [31].

FE-SEM and TEM analyses of the catalyst are given in two magnifications that confirm the catalyst morphology. The FE-SEM images show that the nanoparticles are small and well-dispersed within the polymeric support material (Fig. 3a and b). Based on the TEM image, the average nanoparticles size of the catalyst is 37 nm with a core–shell structure which is related to surface modification of magnetite with PAN polymer and other organic layers. Therefore, they can confirm the proposed structure of the resulting catalyst (Fig. 4a and b).

In addition, the EDX analysis was taken to show the contents of various components in the catalyst (Fig. 5).

TGA spectrum for the final catalyst is provided as a reference for the investigation of ligand amounts anchored to the inorganic core at a temperature range of 25–750 °C. It can confirm the presence of PAN and 2-AP and the thermogram shows good thermal stability of the catalyst, which is an important property. The first step (100 °C region) is assigned to water molecules [32] and the whole organic phase is burned in the next steps (Fig. 6).

2.3 Catalytic Activity

With the new magnetic PAN-based Cu-catalyst in hand, a series of experiments were performed to obtain optimized conditions for the acylation of indoles. It commenced with indole **1** and arylglyoxal **2** to investigate different solvents such as toluene, 1,2-dichloroethane (DCE), dimethyl sulfoxide (DMSO), acetonitrile (MeCN) and 1,4-dioxane under an air atmosphere in which the yield was higher in MeCN (Table 1, entry 1–5). After solvent optimization, we found that, reducing the amount of catalyst from 5 to 3 mol% decreased the yield of the product from 92 to 87% (Table 1, entry 4 and 6) and increasing the catalyst amount from 5 to 10 mol% did not drastically improve the product yield (Table 1, entry 4 and 7). No product was produced without the catalyst even though time and temperature of reaction were increased (Table 1, entry 8 and 9). Also, the use of higher temperature did not enhance the yield (Table 1, entry 10). In the next step, the treatment of indole **1** with arylglyoxal hydrate **2** at room temperature provided only 20% of the

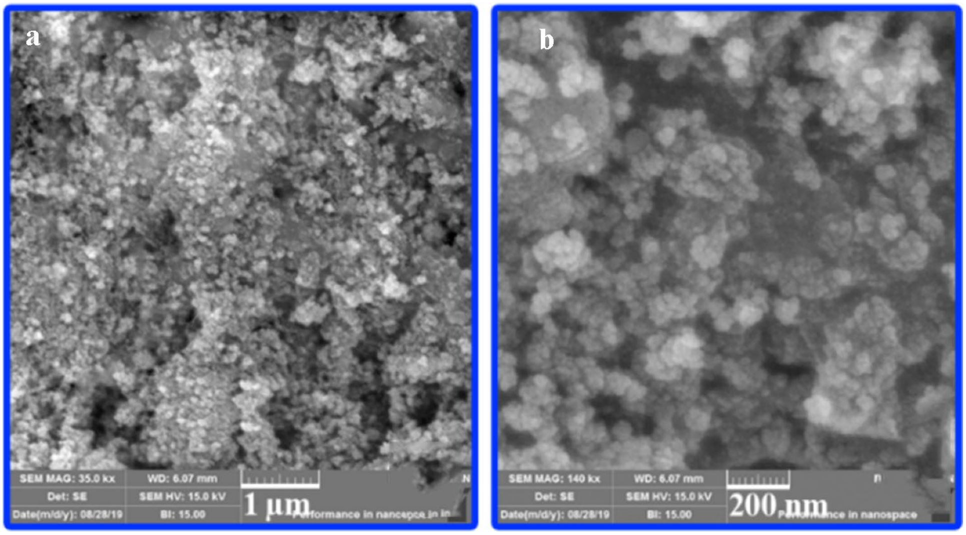


Fig. 3 FE-SEM images of the magnetic PAN Cu-catalyst in two magnifications **a** 1 µm and **b** 200 nm

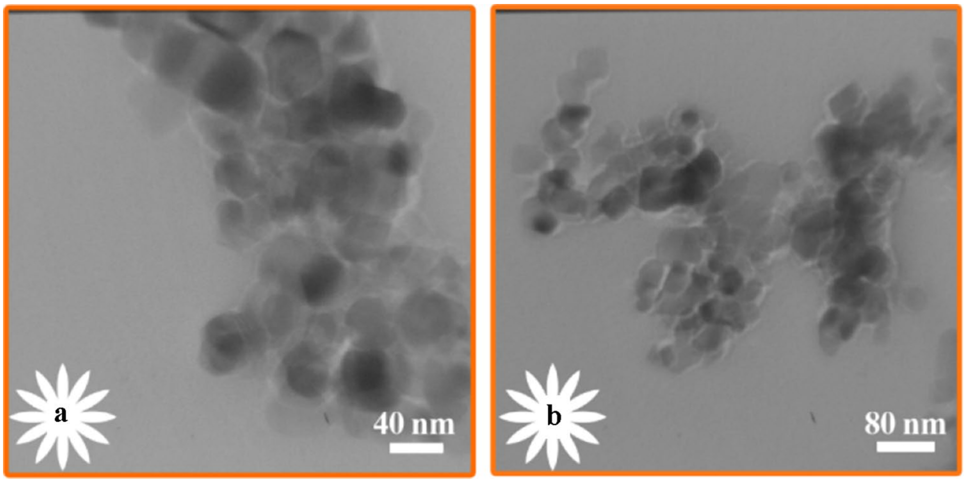


Fig. 4 TEM images of the magnetic PAN Cu-catalyst in two magnifications **a** 40 nm and **b** 80 nm

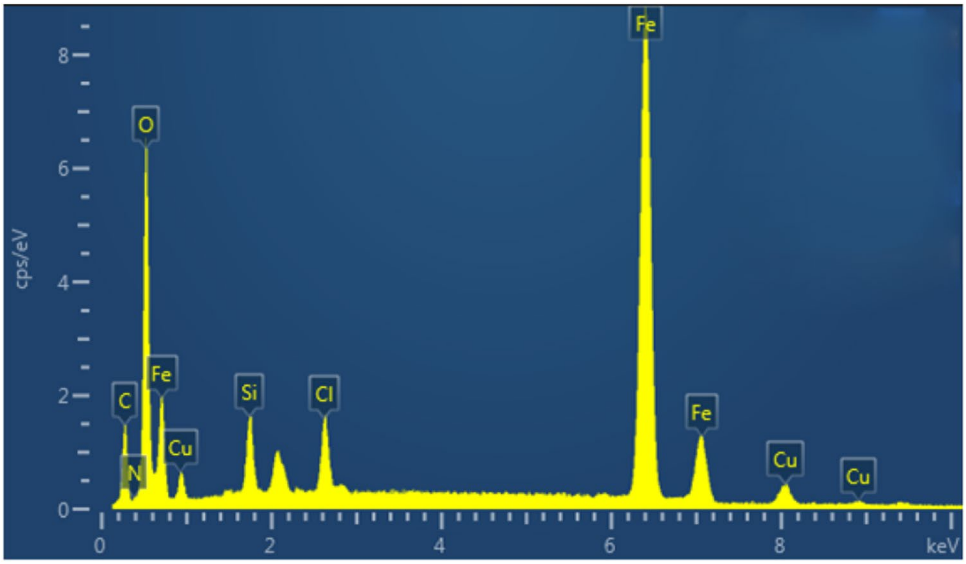


Fig. 5 EDX of the magnetic PAN Cu-catalyst

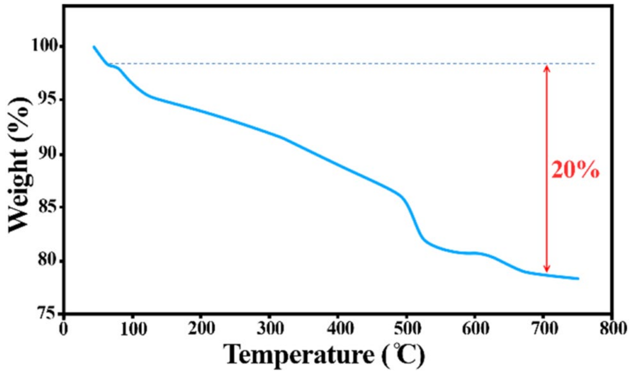
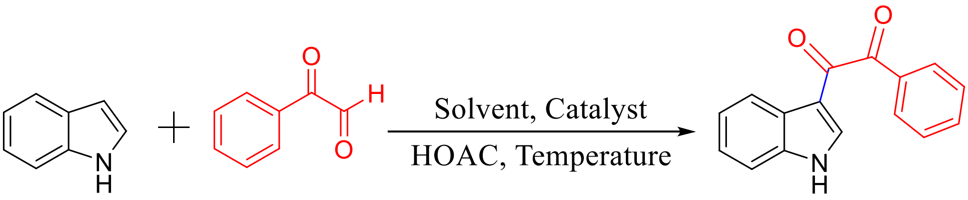


Fig. 6 TGA thermogram of the catalyst

desired product **3** whereas the amount of catalyst and time of reaction were evaluated (Table 1, entry 11). when the reaction was done under a nitrogen atmosphere, the lower yield was observed that is confirmed the reaction atmosphere plays an important role in this reaction (Table 1, entry 12). In the following, Fe_3O_4 nanoparticles (5 mol%) and Fe_3O_4@Cu nanoparticles (5 mol%) were applied as the catalyst (Table 1, entry 13 and 14; respectively). The trace amount of desired product was provided by 5 mol% of Fe_3O_4 nanoparticles after 12 h and Cu nanoparticles decorated Fe_3O_4 could produce only 38% of the product after 24 h. It is worth to note; the present polymeric catalyst was prepared as a nitrogen-rich support to immobilize high amounts of Cu nanoparticles into it. Also, such polymeric support could reduce the leaching of Cu nanoparticles by trapping them into the polymeric chains [25]. This property led to high loading of Cu nanoparticles without significant leaching and therefore, the lower amounts of the catalyst provided more active sites to complete the reaction. Hence, the present catalyst provided higher activity than the Cu/Fe_3O_4 catalyst with similar catalyst loading.

After screening the reaction parameters and inspired by the results, diverse products were synthesized under an air atmosphere at 80 °C for 4 h using 5 mol% of Cu-catalyst, and combination of MeCN/acetic acid (HOAC) as the solvent (2 mL), then the results were collected in Table 2. A series of arylglyoxal hydrates with *para*-position substituted were compatible under the reaction conditions, and the desired products were isolated in good yields. All methoxy,

Table 1 Screening of reaction condition for Copper@Magnetic PAN/2-AP catalyst on 3-acylated indoles



1a **2a** **3aa**

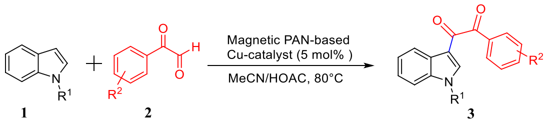
Entry	Solvent	Catalyst (mol%)	T (°C)	Time (h)	Yield (%)[a]
1	Toluene	5	80	4	59
2	DCE	5	80	4	47
3	DMSO	5	80	4	Trace
4	**MeCN**	**5**	**80**	**4**	**92**
5	1,4-Dioxane	5	80	4	71
6	MeCN	3	80	4	87
7	MeCN	10	80	4	92
8	MeCN	–	80	12	Trace
9	MeCN	–	120	12	Trace
10	MeCN	5	100	4	92
11	MeCN	10	rt	24	20
12[b]	MeCN	5	80	4	77
13	MeCN	Fe_3O_4 (5 mol%)	80	12	Trace
14	MeCN	Fe_3O_4@Cu NPs (5 mol%)	80	24	38

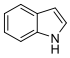
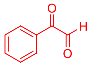
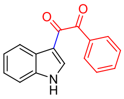
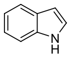
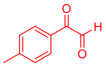
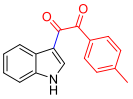
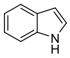
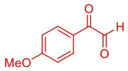
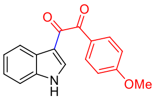
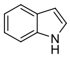
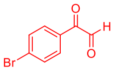
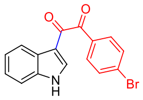
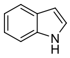
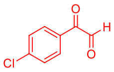
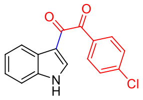
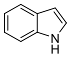
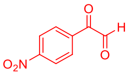
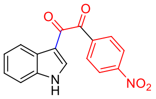
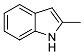
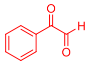
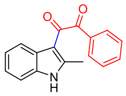
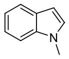
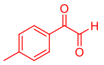
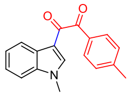
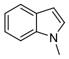
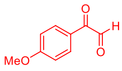
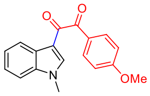
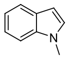
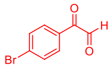
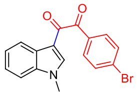
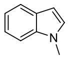
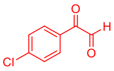
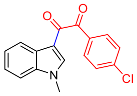
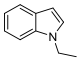
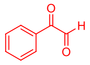
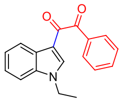
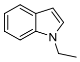
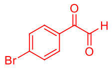
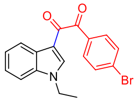
Indole (0.25 mmol), arylglyoxal hydrate (0.3 mmol), catalyst, solvent (2 mL) under an air atmosphere

[a] Isolated yields are given

[b] Under N_2 atmosphere

Table 2 Scope of Indoles and arylglyoxals toward 3-acylated indole synthesis



Entry	Indoles	Arylglyoxal hydrates	Products/Yield (%)a
1			 3aa, 93%
2			 3ab, 89%
3			 3ac, 91%
4			 3ad, 86%
5			 3ae, 87%
6			 3af, Trace
7			 3ba, Trace
8			 3cb, 82%
9			 3cc, 80%
10			 3cd, 81%
11			 3ce, 84%
12			 3da, 83%
13			 3dd,81%

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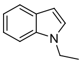
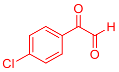
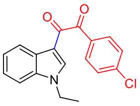
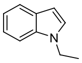
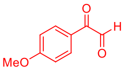
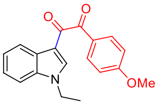
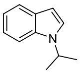
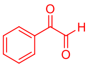
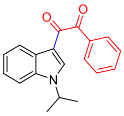
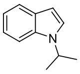
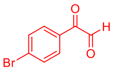
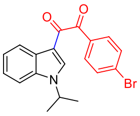
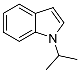
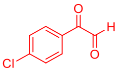
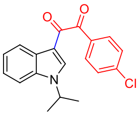
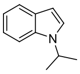
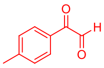
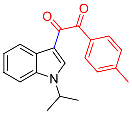
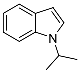
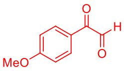
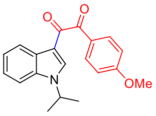
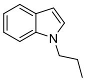
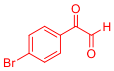
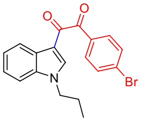
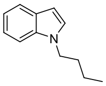
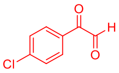
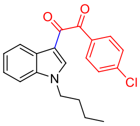
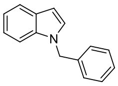
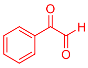
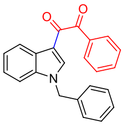
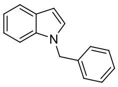
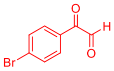
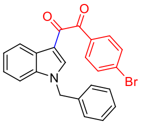
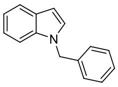
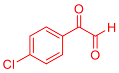
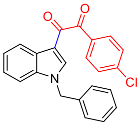
14			 **3de, 83%**
15			 **3dc, 82%**
16			 **3ea, 82%**
17			 **3ed, 80%**
18			 **3ee, 81%**
19			 **3eb, 81%**
20			 **3ec, 82%**
21			 **3fd, 82%**
22			 **3ge, 81%**
23			 **3ha, 80%**
24			 **3hd, 76%**
25			 **3he, 79%**

Indole (0.25 mmol), arylglyoxal hydrates (0.3 mmol), 5 mol% of catalyst, MeCN (2 mL) under an air atmosphere

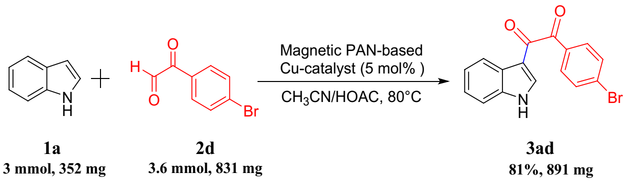
[a] Isolated yields are given

methyl, bromo- and chloro-substituted arylglyoxals exhibited the desired products in moderate to good yields. The free (N–H) indoles provided high reactivities and gave the corresponding products in good yields (**3aa-ae**). The arylglyoxals with electron-donating groups such as *p*-CH_3 and *p*-OCH_3 showed the high efficiency and the desired products were generated in excellent yields (**3ab:89% and 3ac:91%**). The arylglyoxals with electron-deficient substitutions (*p*-Cl and *p*-Br) reacted with free (N–H) indole and the yields of reaction were obtained in 87% and 86%, respectively (**3ad and 3ae**). However, the reaction of strong electron-deficient nitro-substituted arylglyoxal did not give the dicarbonyl indole product because the electron density was decreased (**3af**). And also, 2-methyl indole could not react with arylglyoaxal and it furnishes the favorite product **3ba** only in trace amount.

Subsequently, the diacylation of a series *N*-substituted indoles were tested with arylglyoxals bearing *p*–H, *p*-CH_3, *p*-OCH_3, *p*-Cl, and *p*-Br groups. The results showed they worked well under the optimal reaction conditions to provide the favorite products. As presented in Table 2, *N*-methyl (**3cb-ce**), *N*-ethyl (**3da-dc**), *N*-isopropyl (**3ea-ec**), *N*-propyl (**3fd**), *N*-butyl (**3ga**), and *N*-benzyl (**3 ha-he**) indole substrates could proceed to give the corresponding products in good yields. But, the indoles with bulkier *N*-substitutions such as *N*-benzyl group produced the products in lower yields. According to the above results, it is clear that the reaction could tolerate different functional groups with various electronic properties (^{1}H and ^{13}C NMR spectral data of compounds can be found in Supplementary data).

To confirm the potential of this catalytic system, the product **3ad** was successfully synthesized from free (N–H) indole and *para*-bromoarylglyoxal under the optimal conditions in a gram scale. It is noteworthy, the reaction time increased slightly and the desired product was detected in 81% yield (Scheme 3).

On the basis of the experimental results and previous literature reports [16, 18, 33], a plausible reaction mechanism is demonstrated in Scheme 4. Initially, arylglyoxal **2** was activated by the Cu-catalyst system to provide intermediate I; Subsequently, indole **1** attacked by the C_3 position through Friedel–Crafts type reaction and gave intermediate II. At the final step, the product **3** was formed by aerobic oxidation of intermediate II leading to the release of the Cu-catalyst.



Scheme 3 Reaction carried out on a large scale of 1-(4-bromophenyl)-2-(1*H*-indol-3-yl) ethane-1,2-dione

To demonstrate the reusability of the prepared Cu-catalyst, the catalyst was applied to several model reactions and the reactions were monitored by thin layer choromatograghy (TLC). When each reaction was completed, it was separated, washed with methanol (5×5 mL), dried at 50 °C to 24 h and used for the next run to confirm the recyclability of the prepared Cu-catalyst. It is worth mentioning that no significant metal leaching and reduction product yield was detected and the recovered catalyst was reused in nine cycles with no significant loss of activity (Table 3).

Based on the ICP-OES results, the loading amounts of Cu^{2+} were calculated 0.77 and 0.71 mmol g^{-1} for fresh catalyst and the recovered catalyst after ninth cycle; respectively, which is high loading of copper ions per 1 g of the catalyst.

Compared to the previous literature, the reported works suffer from some restrictions such as the use of expensive palladium nanoparticles, stoichiometric amounts of iodine and copper, and complex ligands as well as by-products generation, long reaction time, and low yield (Table 4, entry 1–6). To overcome these limitations, the magnetic PAN Cu-catalyst was synthesized and used in the synthesis of indolyl diketones (Table 4, entry 7). The worthwhile characteristics of the presented method are summarized below: (a) The use of an inexpensive catalyst with high-loading copper through stable ligands; (b) Mild reaction conditions (higher yield versus lower reaction time); (c) The use of low catalyst weight percentages for acylation of indoles due to its high catalytic efficiency; (d) Having high catalyst reusability because the prepared catalyst can be easily separated via an external magnet and applied to several runs, making it useful for industrial applications.

3 Conclusion

In summary, we have demonstrated a simple, scalable and regioselective Cu-catalyzed acylation of indoles with phenylglyoxals. In this work, indole moieties were selected because they are important scaffolds for natural products, biologically active and pharmaceutical compounds. On the other hand, it is a new method to construct magnetic PAN Cu-catalyst as an efficient magnetic heterogeneous catalyst with a several-step procedure. The high ability of the polymeric support to hold Cu ions which is resulted from its different functional groups accompanied by its stability and easy recyclability plays a crucial role in improving the catalyst activity. Finally, a wide range of starting materials were studied, and the desired indolyl diketone derivatives were synthesized in excellent yields as common intermediates. Moreover, a gram scale experiment was performed in good yield suited for further derivatization.

Scheme 4 Possible reaction mechanism

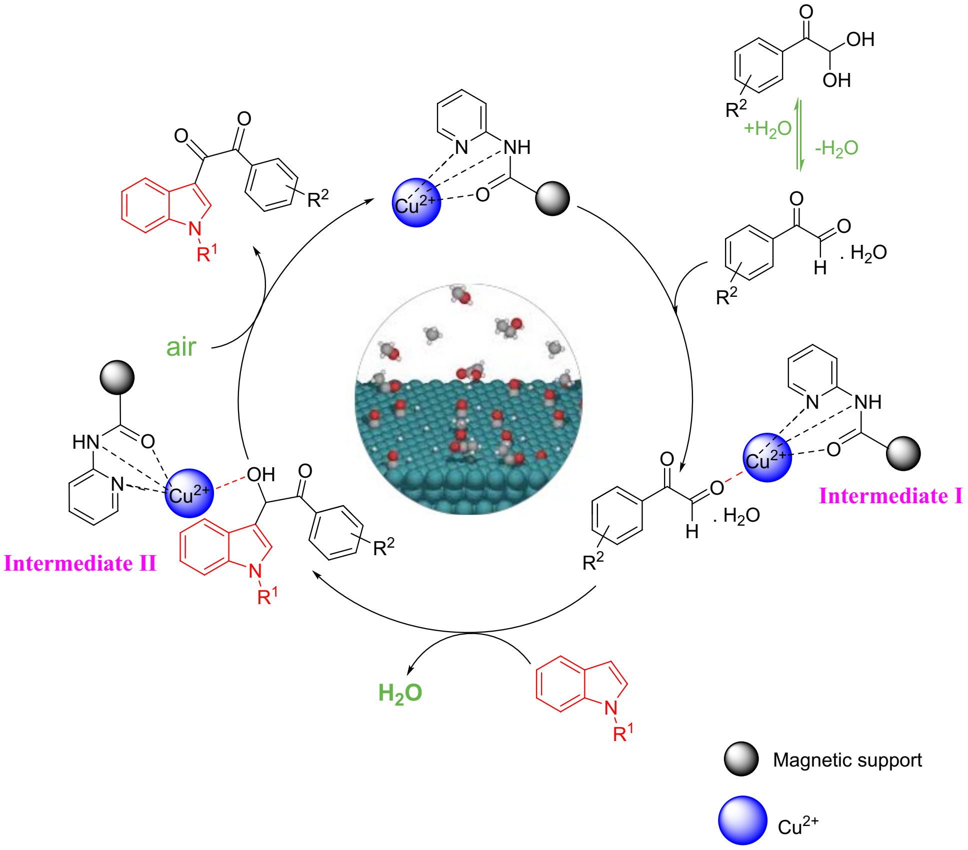
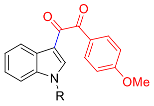


Table 3 The recyclability of the magnetic PAN Cu-catalyst in the synthesis of indolyl diketone under optimal conditions

Run	1	2	3	4	5	6	7	8	9
Yield%	93	93	93	91	90	89	86	86	85

Table 4 Comparison of catalysts activity in 3-acylated indole synthesis

Entry	Catalyst(Loading)	Reaction conditions	Time (h)	Yield (%) 	References
1	Pd(OAC)$_2$ (5 mol%)	Indoles, α-amino carbonyl compounds, Cu(OAc)$_2$ (2 mol%), CH$_3$CN/HOAc, air, 80℃	12	64 (R = H)	[1]
2	CuBr (10 mol %)	*N*-methylindoles, α-carbonyl aldehydes, pyridine (50 mol %), toluene, 90 ℃	6	70 (R = Me)	[15]
3	Fe(OTf)$_3$ (10 mol%)	Indoles, α-amino carbonyl compounds, TBHP, PivOH, DMSO, 50℃	15	70 (R = H)	[17]
4	CuCl$_2$ (10 mol%)	Indoles, α-amino carbonyl compounds, TBHP, CH$_2$Cl$_2$, air, RT	18	76 (R = H)	[11]
5	CuTC (10 mol %)	*N*-methylindoles, α-Hydroxy Ketones, Toluene, O$_2$, 80℃	12	81 (R = Me)	[13]
6	Pyrrolidine (25 mol%)	Indoles, acetophenone, I$_2$, DMSO, 90℃	12	84 (R = H)	[12]
7	Copper@Magnetic PAN/2-AP (5 mol%)	Indoles, α-carbonyl aldehydes, CH$_3$CN/HOAc, air, 80℃	4	91 (R = H) 80 (R = Me)	This work

4 Experimental Section

4.1 General Procedure for the Synthesis of Polyacrylonitrile-Based Copper Catalyst

4.1.1 Immobilization of Acrylonitrile on Fe_3O_4@SiO_2 Microspheres

The co-precipitation method was applied to synthesize Fe_3O_4@SiO_2 nanoparticles and then coated with MPS as mentioned in the previous literature [34, 35]. In order to polymerize polyacrylonitrile on the surface of Fe_3O_4@SiO_2@MPS, 3 mL of acrylonitrile was added to 0.30 g of Fe_3O_4@SiO_2@MPS in 20 mL of deionized water and completely degassed under N_2 atmosphere. Then, 10 mg of azobisisobutyronitrile (AIBN) was added and refluxed for 24 h. After that, an external magnet was used to collect the resulting magnetite nanoparticles and washed with deionized water (DI water) and methanol three times, and finally dried in a vacuum oven at 50 °C for 12 h to provide the magnetic PAN [19].

4.1.2 Preparation of the Magnetic PAN/2-Aminopyridine (2-AP) Cu-Catalyst

In a round bottom flask, 0.50 g of magnetic PAN in DI (30 mL) and 10 mmol of 2-aminopyridine were mixed and refluxed. After 4 h, the magnetic PAN/2-AP was collected, washed with DI water and methanol three times, and dried in a vacuum oven at 50 °C for 12 h to provide the magnetic PAN/2-AP. Finally, the saturated $CuCl_2$ salt was added to the mixture and stirred at room temperature for 24 h. The catalyst was collected by an external magnet, washed with water three times (3 × 20 mL) and dried under reduced pressure.

4.1.3 Preparation of the Fe_3O_4@Cu Nanoparticles Catalyst

In a round bottom flask, the saturated $CuCl_2$ salt was added to 0.20 g of Fe_3O_4@SiO_2 in DI water (10 mL) and stirred at room temperature for 24 h. Then, the catalyst was collected by an external magnet, washed with water three times (3 × 10 mL) and dried under reduced pressure.

4.2 General Procedure for the Synthesis of Indolyl Diketone (3)

A reaction tube was charged with indoles and *N*-substituted indoles 1 (0.25 mmol), arylglyoxal 2 (0.3 mmol), the magnetic PAN/2-AP Cu-catalyst (5 mol%) in MeCN (2 mL) and HOAC as an additive under an air atmosphere. The reaction mixture was stirred at 80 °C until the reaction was finished (monitored by TLC), then the mixture diluted with ethyl acetate and water and the layers were separated. The ethyl acetate was used to extract aqueous layer (3 × 10 ml). The combined organic layers were washed with saturated brine, dried with Na_2SO_4 and after filtration, evaporated under reduced pressure. The crude products were purified by column chromatography on silica gel (n-hexan/ethyl acetate, 10/3, v/v) to provide the desired products 3.

4.3 Spectroscopic Characterization of the Product 3ad

1-(4-bromophenyl)-2-(1*H*-indol-yl)ethane 1,2-dione: Analytical TLC on silica gel, (n-hexan/ethyl acetate, 10/3, v/v); Yellow solid; 891 mg, yield 81%; mp 210–212 °C; ^{1}H NMR (500 MHz, DMSO-d_6): δ 12.15 (s, 1H), 8.22 (s, 1H), 8.18 (s, 1H), 7.97 (d, J = 7.1 Hz, 2H), 7.61 (d, J = 6.9 Hz, 2H), 7.56 (d, J = 6.0 Hz, 1H), 7.32 (d, J = 3.4, 2H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 194.43, 188.99, 138.39, 137.46, 135.11, 133.45, 132.02, 130.17, 129.58, 129.11, 125.47, 124.29, 123.27, 121.61, 113.22, 113.04; FT-IR (neat) 3120, 3050, 1663, 1625, 1584, 1525, 1468, 1396, 1338, 1305, 1277, 1209, 1249, 1173, 1140, 1125, 1091, 1068, 991, 865, 843, 815, 776, 751, 736, 700, 624, 618, 579, 541, 504, 465, 423 cm^{-1}.

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Declarations

Conflict of interest There are no conflict to declare.

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