

Layered double hydroxides@poly(*p*-phenylenediamine)@ Cu as a novel catalyst for the synthesis of pyrrole derivatives: preparation and characterization

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Abstract

In this research, the synthesis route of ZnCr-layered double hydroxides functionalized with poly(*p*-phenylenediamine) and Cu(II) was described. LDHs@PpPDA@Cu was synthesized as a novel, green, and recyclable catalyst and characterized by various techniques including Fourier transform infrared spectroscopy, scanning electron microscopy, thermogravimetry analysis, X-ray diffraction, and energy dispersive X-ray analysis. The catalytic activity of the LDHs@PpPDA@Cu was investigated in the synthesis of pyrrole derivatives via one-pot four-component coupling of amine, aldehyde, nitromethane and acetylacetone. The results of analyses demonstrated that the reactions proceeded to completion which has led to the successful synthesis of high purity compounds.

Graphic abstract



Keywords Synthesis · Novel · Green · Recyclable · Catalyst · One-pot · Coupling

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Fig. 1 Examples of pyrroles with biological activities

Introduction

The synthesis of novel heterocyclic compounds is of paramount importance among research studies, owing to their abundance, and extensive pharmaceutical and industrial application domains [1]. Nitrogen-containing heterocycles as one of the most substantial categories of heterocyclic compounds are of eclectic pharmacological and biological performances [2]. Pyrrole and its derivatives are widely circulated organic molecules having prime significance in natural products, synthetic chemistry, drugs, and the food industry [3]. Some instances of Pyrrole are known to perform as antitumor [4], anti-inflammatory [5], antibacterial [6], antioxidant [7], and antifungal agents (Fig. 1) [8]. Besides, these compounds have been systematically employed in material science [9]. Thus far, a good number of synthetic routes have been proposed and developed for the preparation of pyrroles and their derivatives. The classical methods utilized for the synthesis of pyrroles include Hantzsch [10], Knorr [11], Paal–Knorr [12], and Clauson-Kaas condensation [13] reactions. However, despite their utility, such methods frequently suffer from some drawbacks such as multistep synthetic operations, severe reaction conditions, scarcity of the initiate materials, functional group compatibility, and region-specificity. To overcome these challenges, various efficient strategies such as transition metal catalysis [14] and multicomponent [15] reactions (MCRs) have been developed. MCRs include reactions in which three or more starting materials react in a one-pot process to form a new product. The advantages of MCRs extend from high atom economy, the ability to build complex molecules, avoiding the necessity of isolation and purifications

of the intermediates, consistency with the principles of green chemistry, operational simplicity, and minimizing waste, labor, and cost [16]. Given the mentioned significance and applications of pyrrole and its derivatives, researchers worldwide have put tremendous efforts to devise and develop the most facile route for synthesizing these compounds. Heretofore, a variety of methods have been reported for the synthesis of pyrrole in the literature. Among which the routinely applied, one involves one-pot four-component coupling of amine, aldehyde, nitromethane and acetylacetone. A lot of research studies using variant catalysts and reaction settings have been conducted. Maiti et al. [17], in 2010, reported the preparation of novel derivatives of pyrrole derivatives were synthesized by Rajeshwar Reddy et al. using I_2 as a catalyst at 90–95 °C. In another research work, Gupta et al. [18], in 2014, reported the new derivatives of these compounds prepared at 90–95 °C in the presence of [bmim]HSO₄ [19]. In 2015, pyrroles were synthesized by Tang et al. [20] using FeMIL-101 as a catalyst in toluene.

Layered double hydroxides (LDHs) have been recognized for over 150 years since the discovery of the mineral hydrotalcite. Hydrotalcite is a layered double hydroxide of general formula of $Mg_6Al_2CO_3(OH)_{16}$.⁴(H₂O), which was first studied by Manasse in 1915 [21]. LDHs are reputed as hydrotalcite-like clays of the general chemical formula:

$$\left[M^{2+}_{(1-x)}M^{3+}_{x}(OH)_{2}\right](A^{n-})_{x/n}\cdot zH_{2}O$$

In which M^{2+} and M^{3+} are divalent and tri-valent metal cations, A^{n-} is the replaceable interlayer anion and *x* has usually values between 0.20 and 0.33 (Fig. 2) [22].

LDHs demonstrate exceptional physical and chemical behaviors such as high anion exchange capacity, swelling property, memory effect, and high specific surface area [23]. Moreover, these materials are cost-effective and stable at a high elevated temperature (500–600 °C) and harsh pH conditions [24]. The remarkable properties of these materials presented them as an excellent candidate for such broad fields of applications including pharmaceutics, electrocatalysts, electrochemistry, photocatalysts, and photochemistry [25]. In addition, their utilization in catalytic fields as well as catalyst supports has been investigated [26].

In this article, an efficient and highly active reusable heterogeneous catalyst was designed by combining the LDHs, poly-para (phenylenediamine) (PpPDA), and $Cu(OAc)_2$. Thereafter, its catalytic activity for one-pot synthesis of pyrrole compounds was examined (Scheme 1).



Fig. 2 Layered double hydroxides structure



Scheme 1 Synthesis of substituted pyrroles using LDHs@PpPDA@Cu

Experimental

Chemicals and instruments

All commercial materials were purchased from Merck and Fluka Companies 1 H NMR and 13 C NMR spectra were measured on a spectrometer at 400 MHz and 100 MHz, respectively, in DMSO-d₆. Fourier transform infrared (FT-IR) spectra were obtained using the Shimadzu 435-U-04 FT spectrophotometer from KBr

pellets. Melting points were recorded with a BUCHI 510 apparatus in open capillary tubes.

General procedure for preparation of LDHs@PpPDA@Cu catalyst

Preparation of layered double hydroxides (LDHs)

LDHs can be synthesized in base solutions using metal salts containing M^{2+} and M^{3+} . Initially, an aqueous solution containing Zn^{2+} and Cr^{3+} metals were prepared using precursor salts, i.e., $Zn(NO_3)_2 \cdot 6H_2O$ and $Cr(NO_3)_3 \cdot 9H_2O$ in a ratio of 2:1 in deionized water. Then, NaOH aqueous solution of 2 M was dropped into the solution and was added dropwise till the pH value of the solution reached 11.5. Afterward, the mixture was allowed to react for 18 h without stirring at the 65 °C. Finally, a green slurry was obtained and washed several times with distilled water and dried in a 60 °C oven for 24 h [27].

Preparation of LDHs@PpPDA

LDHs@PpPDA were synthesized at room temperature through the emulsion polymerization method as follows. In a typical reaction, 0.75 g of LDHs were poured into a 250 mL flask and then 20 mL chloroform and 30 mL of distilled water were added to those. In the next step, flask contents were well-dispersed for about 30 min by an ultrasonic bath in the ambient temperature and then the solution containing PpPDA monomer (1 g) in 30 mL of HCl (1 M) was added to the aqueous solution. The oxidizing solution was prepared via 1.5 g (6.5 mmol) of APS in 20 mL of deionized water and was added to the reaction mixture. After 24 h, the resultant precipitate was washed several times with deionized water. Finally, the precipitate was dried in vacuum oven at 50 °C for 24 h [28].

Preparation of LDHs@PpPDA@Cu

At first, 1 g from the LDHs@PpPDA was dispersed in 50 mL EtOH using an ultrasonic bath (15 min). Then, 0.5 g of $Cu(OAc)_2$ was dissolved in 5 mL EtOH and added to the previous mixture. After refluxing the mixture for 24 h, the resulting precipitate was separated and washed with 50 mL EtOH and dried in an oven.

General procedure for synthesis of pyrrole derivatives

A mixture of aromatic aldehyde (1 mmol), amine (1 mmol), acetylacetone (1 mmol), nitromethane (1 mL), and LDHs@PpPDA@Cu (0.05 g) was stirred at 100 °C for an appropriate time. The reaction was monitored by TLC (*n*-hexane:acetone, 7:1). After completion of the reaction, the reaction mixture was poured into a centrifuge tube and the catalyst was separated by centrifugation. The resulting solution was poured into a petri dish, and the excess nitromethane was removed under vacuum. Then, resulting precipitates were collected and washed with cold diethyl ether. The

products were identified using physical and spectroscopic data (FT-IR, NMR and CHN).

Analytical data of selected products

1-(4-(2,3-dichlorophenyl)-2-methyl-1-(*p***-tolyl)-1***H***-pyrrol-3-yl)ethan-1-one (5e) Mp. 90–92 °C. FT-IR (KBr, \nu, cm⁻¹): 3010, 2968, 1607, 1501, 1246. ¹H NMR (400 MHz, DMSO-d_6, \delta, ppm): 7.74 (s, 1H), 7.43–7.15 (m, 6H), 6.55 (s, 1H), 4.02 (s, 3H), 3.82 (s, 3H), 3.46 (s, 3H). ¹³C NMR (100 MHz, DMSO-d_6, \delta, ppm): 26.17, 31.57, 48.29, 108.09, 113.95, 120.64, 126.06, 130.26, 134.41, 144.02, 150.73, 188.35. Anal. Calcd. For C₂₀H₁₇Cl₂NO: C, 67.05; H, 4.78; N, 3.91. Found: C, 66.95; H, 4.83; N, 3.85.**

1-(4-(4-chloro-3-nitrophenyl)-2-methyl-1-phenyl-1*H***-pyrrol-3-yl)ethan-1-one** (5f) Mp. 106–108 °C. FT-IR (KBr, ν , cm⁻¹): 3072, 2936, 1604, 1509, 1346. ¹H NMR (400 MHz, DMSO- d_6 , δ , ppm): 8.87 (s, 1H), 8.49 (s, 1H), 8.17 (s, 1H), 7.48–7.04 (m, 5H), 6.84 (s, 1H), 3.45 (s, 3H), 3.23 (s, 3H). ¹³C NMR (100 MHz, DMSO- d_6 , δ , ppm): 29.92, 48.75, 104.33, 112.27, 116.47, 118.95, 124.39, 128.98, 131.47, 134.43, 139.44, 150.32, 180.82. Anal. Calcd. For C₁₉H₁₅ClN₂O₃: C, 64.32; H, 4.26; N, 7.9. Found: C, 64.28; H, 4.28; N, 7.83.

1-(4-(2-bromophenyl)-2-methyl-1-(*p***-tolyl)-1***H***-pyrrol-3-yl)ethan-1-one (5** g) Mp. 100–103 °C. FT-IR (KBr, ν , cm⁻¹): 3001, 2930, 1606, 1508, 1245. ¹H NMR (400 MHz, DMSO-*d*₆, δ, ppm): 8.51 (s, 1H), 7.75–7.15 (m, 7H), 6.48 (s, 1H), 4.02 (s, 3H), 3.82 (s, 3H), 3.35 (s, 3H). ¹³C NMR (100 MHz, DMSO-*d*₆, δ, ppm): 20.72, 28.26, 44.98, 101.81, 107.66, 114.36, 119.80, 123.13, 126.06, 129.84, 131.92, 138.61, 199.60. Anal. Calcd. For C₂₀H₁₈BrNO: C, 65.23; H, 4.93; N, 3.8. Found: C, 65.15; H, 4.98; N, 3.75.

1-(4-(1*H***-indol-3-yl)-2-methyl-1-(***p***-tolyl)-1***H***-pyrrol-3-yl)ethan-1-one (5 h) Mp. 120–122 °C. FT-IR (KBr, \nu, cm⁻¹): 3308, 3067, 2835, 1606, 1509, 1248. ¹H NMR (400 MHz, DMSO-***d***₆, \delta, ppm): 11.96 (s, 1H), 8.8 (s, 1H), 8.04–6.61 (m, 9H), 3.68 (s, 3H), 3.44 (s, 3H), 3.15 (s, 3H). ¹³C NMR (100 MHz, DMSO-***d***₆, \delta, ppm): 25.88, 33.36, 50.45, 86.89, 94.83, 97.88, 101.82, 108.87, 112.81, 117.21, 125.14, 129.49, 135.66, 146.62, 151.04, 205.89. Anal. Calcd. For C₂₂H₂₀N₂O: C, 80.46; H,6.14; N, 8.53. Found: C, 80.38; H, 6.2; N, 8.47.**

1-(2-methyl-1-phenyl-4-(pyridin-2-yl)-1*H*-**pyrrol-3-yl)ethan-1-one** (5) Mp. 88–90 °C. FT-IR (KBr, ν , cm⁻¹): 3032, 2921, 1596, 1502, 1331. ¹H NMR (400 MHz, DMSO- d_6 , δ , ppm): 8.56 (s, 1H), 7.77 (s, 1H), 7.43–7.14 (m, 8H), 3.83 (s, 3H), 3.35 (s, 3H). ¹³C NMR (100 MHz, DMSO- d_6 , δ , ppm): 30.36, 49.15, 100.15, 102.23, 109.35, 111.41, 114.77, 119.38, 121.88, 130.68, 134.83, 138.19, 141.95, 147.79, 151.97, 197.12. Anal. Calcd. For C₁₈H₁₆N₂O: C, 78.24; H, 5.84; N, 10.14. Found: C, 78.16; H, 5.93; N, 10.05.

1-(2-methyl-4-(pyridin-2-yl)-1-(*p***-tolyl)-1***H***-pyrrol-3-yl)ethan-1-one (5i) Mp. 114–115 °C. FT-IR (KBr, \nu, cm⁻¹): 3028, 2860, 1577, 1410. ¹H NMR (400 MHz, DMSO-d_6, \delta, ppm): 8.87 (s, 1H), 8.17 (s, 1H), 7.48–6.6 (m, 7H), 3.66 (s, 3H), 3.43 (s, 3H), 3.21 (s, 3H). ¹³C NMR (100 MHz, DMSO-d_6, \delta, ppm): 21.16, 30.76, 43.72, 97.64, 103.06, 109.78, 112.28, 117.27, 121.05, 125.64, 131.51, 135.68, 141.15, 145.70, 181.64. Anal. Calcd. For C₁₉H₁₈N₂O: C, 78.59; H, 6.25; N, 9.65. Found: C, 78.53; H, 6.28; N, 9.58.**

Results and discussion

The LDHs@PpPDA@Cu was prepared according to the procedure depicted in Scheme 2. At first, the LDHs were prepared in accordance with the method previously reported. After which the LDHs were built, PpPDA was coated on its surface. Ultimately, the LDHs@PpPDA@Cu catalyst was synthesized utilizing Cu(OAc)₂ precursor and the characterization analyses were also performed using Fourier transform infrared (FT-IR) spectroscopy, scanning electron microscopy (SEM), thermogravimetry analysis (TGA), and X-ray diffraction (XRD). The concentration of copper in LDHs@PpPDA@Cu, which was 9.17 wt%, was determined by inductively coupled plasma optical emission spectrometry (ICP-OES).

Study of the FT-IR spectra

The FT-IR spectra of LDHs, LDHs@PpPDA, LDHs@PpPDA@Cu are represented in Fig. 3. Notably, the IR spectra of LDHs are illustrated in (a) section of Fig. 3 and the characteristic peaks of 3400 cm^{-1} , 1400 cm^{-1} , and 500 cm^{-1} were related to O–H, NO₃⁻ anion, and metal–oxygen stretching vibrations,



Scheme 2 Total procedure for synthesis of LDHs@PpPDA@Cu



Fig. 3 FT-IR spectra of LDHs (a), LDHs@PpPDA (b), LDHs@PpPDA@Cu (c)

respectively. The IR spectra of LDHs@PpPDA are shown in (b) section of Fig. 3 and the characteristic peaks of 3371 cm^{-1} , 1568 cm^{-1} , 1501 cm^{-1} , and 1285 cm^{-1} were attributed to N–H, C=N, C=C, and C–N stretching vibrations, respectively. As could be observed, the intensity as well as the geometry of O–H stretching vibration in Fig. 3a was dramatically changed as a result of bonding formation between PpPDA on LDHs which approves the successful synthesis of PpPDA on the LDHs. Besides, the existing of Cu–N absorption band in 520 cm⁻¹ in Fig. 3c along with the decrement of N–H peak intensity demonstrates that LDHs@ PpPDA@Cu catalyst was effectively prepared.

Study of the X-ray diffraction

X-ray diffraction spectroscopy was applied to investigate the crystal structure of LDHs, LDHs@PpPDA and LDHs@PpPDA@Cu. Figure 4c shows the XRD pattern of LDHs and the series peaks including 2θ : 10°, 30°, 40° which is consistent with earlier reported research works [29]. The characteristic peaks (2θ : 10°, 30°) spotted in XRD pattern of LDHs@PpPDA (Fig. 4b) manifestly attests the coating of PpPDA on LDHs [30]. Moreover, the syntheses of LDHs@PpPDA@Cu are confirmed through taking a comparison among the XRD patterns for LDHs@PpPDA@Cu (Fig. 4a), LDHs and LDHs@PpPDA. The emerging of the peaks related to Cu in 2θ : 15°, 17°, 19° as well as a different pattern in 24° also shows the formation of Cu in LDHs@PpPDA. In the end, the average crystal size of LDHs@PpPDA@Cu is calculated using Debye–Scherrer's ($D = K\lambda/(\beta \cos \theta)$) formula.



Fig. 4 XRD pattern of LDHs (c), LDHs@PpPDA (b), LDHs@PpPDA@Cu (a)

Study of the thermal gravimetric (TGA) properties

In order to evaluate the thermal stability of LDHs@PpPDA@Cu catalyst, thermogravimetry analysis (TGA) was performed. As remarked in Fig. 5, three massloss regions were detected within the curves. In the first fracture of the diagram at 100–200 °C, a weight loss is taken place, which might be attributed to the loss of water, residual monomer and other solvent in the catalyst. In addition, the second mass-loss appeared at 200–300 °C, which could be due to the decomposition of PpPDA oligomers. In the last step, after 350 °C, the cleavage and decomposition of PpPDA polymeric chains and Cu/polymer complex in the catalyst have begun



Fig. 5 TGA curve of LDHs@PpPDA@Cu

to occur. As a result, the optimum temperature for employing LDHs@PpPDA@ Cu as a catalyst could be up to 300 °C.

Morphological study

The morphological evaluation as well as the average particle size examination of LDHs@PpPDA@Cu was performed via SEM technique. According to Fig. 6, the LDHs@PpPDA@Cu composite catalyst possess a consistent structure which indicates that the synthetic procedure resulted in uniformity of the samples. Besides, referring to the images, it could also be implied that the catalyst particles are of a layered–layered composite configuration.

Study of the energy dispersive X-ray (EDX) spectrum

Energy dispersive X-ray analysis of the LDHs@PpPDA@Cu is shown in Fig. 7. The presence of C, O, N, Cu, Cr, and Zn was validated through this spectrum. The presence of Cu signal indicates that the LDHs@PpPDA has suitably been covered with Cu.

Synthesis of pyrrole derivatives using LDHs@PpPDA@Cu as catalyst

In green chemistry, it is of fundamental importance to make use of a suitable catalyst for the synthesis of organic compounds. In light of this, the catalytic activity of LDHs@PpPDA@Cu was investigated in the one-pot four-component coupling of amine, aldehyde, nitromethane, and acetylacetone. At first, the catalytic activity of LDHs@PpPDA@Cu was compared with that of other catalysts reported in the literature and the results are tabulated in Table 1. Based on these data, the LDHs@PpPDA@Cu catalyst is one of the most effective catalysts in the synthesis of pyrroles. In this catalyst, owing to the presence of Cu, its free orbitals perform as a Lewis acid and activate the carbonyl groups through the interaction with oxygen. Thereafter, the reaction of benzaldehyde (1 mmol), 4-methyl aniline



Fig. 6 SEM images of LDHs@PpPDA@Cu



Fig. 7 EDX spectrum of LDHs@PpPDA@Cu

Entry	Catalyst	Temp. (°C)	Time (h)	Yield (%)	Ref.
1	[bmim]HSO ₄	90–95	3	87	[19]
2	Montmorillonite clay K10	60	10	70	[31]
3	FeCl ₃	Reflux	7	56	[17]
4	Fe-MIL-101	100	20	85	[13]
5	CoFe ₂ O ₄ @SiO ₂ -PrNH ₂ -Mo(acac) ₂	90	4	90	[32]
6	LDHs@PpPDA@Cu	100	3	98	This work

Table 1 Comparison of various catalysts for the synthesis of pyrrole derivatives

(1 mmol), acetylacetone (1 mmol) and nitromethane (1 mL) was selected as a model reaction in which diverse temperature and different catalyst amounts were utilized to find out the optimized condition for the reaction. The optimization of the reaction conditions was examined as shown in Table 2. According to these data, no reaction was observed in the absence of a catalyst. As regards the purity of the products, the reaction time, and the product yields, 0.05 g of the catalyst is an adequate amount for complementation of this reaction. Besides, reducing the amount of catalyst has decreased the reaction efficiency, whereas its further increment had no considerable impact in the progression of the reaction (Table 2, entries 2–4). Then, various temperatures were monitored within the model reaction. The highest yield of the product was achieved at a temperature of 100 °C. Therefore, the best result was achieved under the optimized conditions using 0.05 g of catalyst and a temperature of 100 °C (Table 2, entries 5).

In order to evaluate the effect of different substitution, this reaction was investigated under optimum condition employing various aldehydes and amines possessing different electron-withdrawing and electron-donating groups and the results are presented in Table 3. From the results obtained, it was concluded that

Table 2 Optimization of thereaction conditions for the	Entry	Catalyst	Temp. (°C)	Time (h)	Yield (%)
synthesis of pyrrole derivatives	1	-	100	24	Trace
catalyst	2	0.02	100	3	60
	3	0.04	100	3	88
	4	0.08	100	3	98
	5	0.05	100	3	98
	6	0.05	90	3	90
	7	0.05	80	3	78
	8	0.05	70	3	56
	9	0.05	60	3	45
	10	LDHs (0.05)	100	3	Trace
	11	LDHs@PpPDA (0.05)	100	3	40

Table 3 Synthesis of pyrrole derivatives using the LDHs@PpPDA@Cu catalyst



the reaction proceeds to a higher degree of completion when utilizing an aldehyde electron-withdrawing and an amine electron-donating group.

One of the most significant features of the catalyst of this study lies in its retrievability and reusability. For this purpose, the model reaction was investigated in the presence of 0.05 g of catalyst and temperature of 100 °C. After which the catalyst preparation was completed, it was separated from the reaction, washed several times with warm deionized water and ethanol and then was placed in an oven for drying. The results of the recycling for the catalyst are presented in Fig. 8. It was realized that the catalyst holds the potential to be reused for 5 successive cycles without any considerable loss of its initial catalytic activity. The catalyst activity was also examined after 5 times using EDX mapping technique. The mapping analysis of the recycled catalyst demonstrated the stability of the LDHs@PpPDA@Cu during the recycling procedure (Fig. 9).

A possible mechanism for the synthesis of pyrrole derivatives using LDHs@ PpPDA@Cu catalyst, via a four-component reaction is depicted in Scheme 3. As reported in the literature, pyrroles can be synthesized from Michael reaction of β -enamino ketones or esters and nitroalkenes followed by cyclization [33]. Nitroalkenes (intermediate I) from aldehyde and nitroalkane and β -enamino ketones or esters (intermediate II) from acetylacetone and amines could be generated in the presence of a catalyst. These two intermediates reacted via Michael addition which in turn formed a five-membered cyclic intermediate III, followed by intramolecular cyclization with the elimination of nitroxyl (HNO) and water to lead to the final product.

Conclusion

In this research, β -enamino ketones or esters and nitroalkenes could be generated simultaneously from available abundant starting materials in the presence of LDHs@PpPDA@Cu catalyst. Through Michael reaction between these two components followed by cyclization, functionalized pyrroles have been successfully synthesized. The utilization of procedure in this study offers several benefits



Fig. 8 Recyclability test of LDHs@PpPDA@Cu



Fig. 9 Results of EDX map patterns: LDHs@PpPDA@Cu



Scheme 3 Suggested reaction mechanism for the synthesis of pyrrole derivatives

including facile reaction work-up, short reaction duration, applicability, costeffectiveness, high yield, as well as green and mild reaction conditions. The main advantages of the catalyst are facile preparation route, easy separation from the reaction medium, recyclability and reusability. Finally, the chemical and physical structure of catalyst and the synthesized derivatives were analyzed and their structure was confirmed. Acknowledgements The authors would like to appreciate Bu-Ali Sina University, Center of Excellence in Development of Environmentally Friendly Methods for Chemical Synthesis (CEDEFMCS) for their financial supports of this study.

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