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Efficient Synthesis of Naphthopyranopyrimidine Derivatives Using Copper(II)/Polyimide Linked Covalent Organic Frameworks: A Solvent-Free and Microwave Assisted Approach

Nasimeh Mahmoudinasab-Manoujan, Farid Moeinpour*, Fatemeh S. Mohseni-Shahri

Department of Chemistry, Bandar Abbas Branch, Islamic Azad University, Bandar Abbas 7915893144, Iran.
E-mail: f.moeinpour@gmail.com

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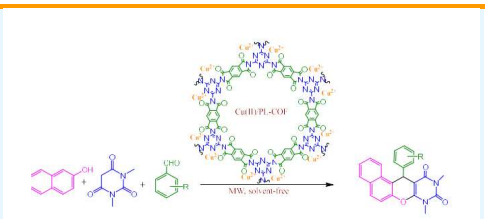
Abstract: Efficient synthesis of naphthopyranopyrimidine derivatives using copper (II)/polyimide linked covalent organic frameworks (Cu (II)/PL-COF) has been achieved through a solvent-free and microwave-assisted method. This one-pot protocol combines *β*-naphthol, aromatic aldehydes, and *N,N*-dimethylbarbituric acid. The process offers various advantages, including safe operation, minimal pollution, fast product formation, and easy setup due to microwave irradiation and a lack of solvents. Additionally, the catalyst demonstrates high reusability, allowing for multiple repetitions of the reaction without experiencing any significant loss in activity.



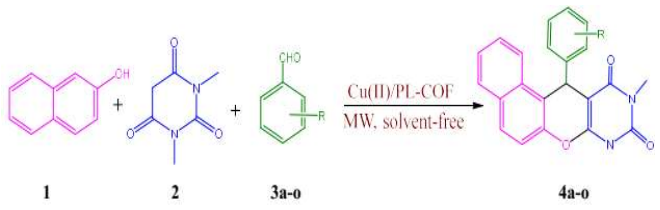
Keywords: Covalent organic frameworks, Naphthopyranopyrimidine, Solvent-free. Microwave

1. Introduction

There has been promising research demonstrating the potential of covalent organic frameworks (COFs) to serve as a diverse ligand in immobilizing transition metal ions for various organic reactions.[1-4]. In recent times, the imidization reaction has been utilized to create covalent organic polyimide frameworks (PI-COFs) with remarkable attributes such as excellent thermal resistance, outstanding mechanical properties, significant pore sizes, and strong chemical stability. Although there have been numerous studies exploring the potential of PI-COFs in areas such as drug delivery[5], chemo and biosensors[6] and organic dye removal[7], little attention has been given to investigating their capability as carriers of transition metals.

Multicomponent reactions (MCRs) have become extensively utilized in diverse fields, particularly in organic synthesis and medicinal chemistry. These reactions, widely acknowledged for their efficiency in compound creation, are instrumental in producing complex compounds. One such reaction frequently employed in the synthesis of pyranopyrimidines is included within this category.[8] The various biological properties of pyranopyrimidines have been attracting a great deal of attention in recent years. These compounds show a wide range of medicinal possessions such as antitumor, cardiotonic, antimalarial, antibronchitis, antihypertensive, analgesic, antiviral, antimicrobial and antifungal activities.[9] The usual method for preparing pyranopyrimidine is the reaction of arylidene malononitriles with barbituric acid under heat conditions or microwave radiation. Recently, these compounds are prepared from the direct condensation of aldehydes, malononitrile and barbituric acid in the presence of various catalysts such as diammonium hydrogen phosphate. Despite the various advantages of several described techniques, there are some drawbacks associated with them. These include challenging reaction conditions, extended reaction duration, limited product output, costly and hazardous catalysts, and elevated temperature requirements. Hence, it remains necessary to offer an uncomplicated, mild, and effective approach to address this matter.[10] In the biomedical and biological fields, nitrogen-containing heterosystems, specifically naphthopyranopyrimidines, have gained significant recognition and importance in recent years, elevating their status to a privileged position. These promising compounds are physiologically anticonvulsant,11 antihypertensive, [12] analgesic, [13], fungicidal[11] and antitumor.[14] In the presence of catalysts such as I_2[15], $InCl_3$, P_2O_5 [16], lactic acid[17], $ZrOCl_2$/nano TiO_2[18], Triazine bis(pyridinium) hydrogen sulfate ionic liquid[19], $Al(H_2PO_4)_3$[20], L-Proline[20] and $LaMo_xFe_{1-x}O_3$ nanosheets[21], a three- component reaction of 2-naphthol, aldehyde and 1,3-dimethyl barbituric acid can be used to synthesize naphthopyranopyrimidines. Due to the importance of naphthopyranopyrimidine derivatives and the objective of developing an affordable, non-toxic, efficient, reusable, and benign catalyst for their synthesis, an improved catalyst is needed. In our investigation on nano-catalysts in the field of nano-catalysts[22-29], we present in this report the utilization of polyimide linked covalent organic frameworks (PL-COFs) as nano-catalysts for the synthesis of

naphthopyranopyrimidine derivatives. The process is detailed in Scheme 1.



R = a: H, **b:** 4-Cl, **c:** 4-NO_2, **d:** 4-OCH_3, **e:** 4-CH_3, **f:** 3-OCH_3-4-OH, **g:** 2-Cl, **h:** 3-Cl, **i:** 2,4-di Cl, **j:** 4-F, **k:** 2-NO_2, **l:** 3-NO_2, **m:** 3-OH, **n:** 4-CN, **o:** 2-Naphthyl

Scheme 1. Cu(II)/PL-COF catalyst-mediated synthesis of naphthopyranopyrimidines

2. Experimental

Synthesis of PL-COF

The synthesis of PI-COF involved a reaction between MEL (melamine) and BTCD (benzene-1,2,4,5-tetracarboxylic dianhydride), as previously described by Han et al. in a publication[30]. For more information, please refer to the Supporting Information.

Generic method for the preparation of naphthopyranopyrimidines

The experiment involved mixing *β*-naphthol **1**, *N,N*-dimethyl barbituric acid **2**, aromatic aldehyde **3a-o**, and of the COFs-catalyst (0.01 g) in a Pyrex test tube. The mixture was then exposed to microwave radiation at a power of 180 W for a convenient reaction time of 1-4 minutes without the presence of a solvent. The progress of the reaction was monitored using TLC. Once the reaction was complete, the temperature of the mixture was reduced to room temperature and hot ethanol was added. The Cu(II)/PL-COF catalyst was separated from the mixture by filtration, washed with acetone, and dried overnight for the next run. The resulting catalyst-free reaction mixture was transferred to another beaker. The separated colloidal solution was treated with hot ethanol to obtain the pure product, which was a yellow precipitate. Further purification of the naphthopyranopyrimidine products was done through recrystallization. The structures of the products were confirmed using ^{1}H-NMR, and their melting points were compared with previously reported values in the literature.

Characterizations

The FT-IR spectra were recorded using Shimadzu spectrometers (model 8400s) from Kyoto, Japan. These spectra were collected in the range of 400-4000 cm^{-1} by employing KBr pellets. Crystallographic structure analysis was conducted using X-ray diffractometers (Philips) with Cu K_α radiation (λ = 1.54 Å). The structure and morphology of the nano powders were examined using field emission scanning electron microscopy (FESEM, TESCAN model MIRA III) and transmission electron microscopy (TEM, Zeiss model LEO 912AB, 120 kV) from Germany. The Brunauer-Emmett-Teller porosity and surface area at 77 K were determined using the Autosorb-1 Quantachrome Sorptometer (USA). ^{1}H NMR spectra at ambient temperature in $CDCl_3$ were recorded using a 400 MHz spectrometer. An ICP-AES analysis (inductively coupled plasma atomic emission spectroscopy) was performed on a Varian VISTA-PRO instrument. The thermo gravimetric analyses (TGA) were carried out using a thermogravimetric/differential thermal analyzer (Netzsch- TGA 209 F1) by gradually heating to 800 °C at a rate of 10 °C per minute.

Selected spectral data

(Table 2, Entry 1) **12-Phenyl-8,12-dihydro-8,10-dimethyl-9H-naphtho[1′,2′:5,6]pyrano[2,3-d]pyrimidine-9,11-(10H)-dione:**

^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 3.35 (3H, s), 3.56 (3H, s), 5.87 (s, 1H), 7.26-7.15 (3H, m), 7.40-7.43 (5H, m), 7.78 (2H, m), 7.94 (1H, d, J = 7.7 Hz).

(Table 2, Entry 5) **12-(4-Methylphenyl)-8,12-dihydro-8,10-dimethyl-9H-naphtho[1′,2′:5,6]pyrano[2,3-d]pyrimidine-9,11-(10H)-dione:**

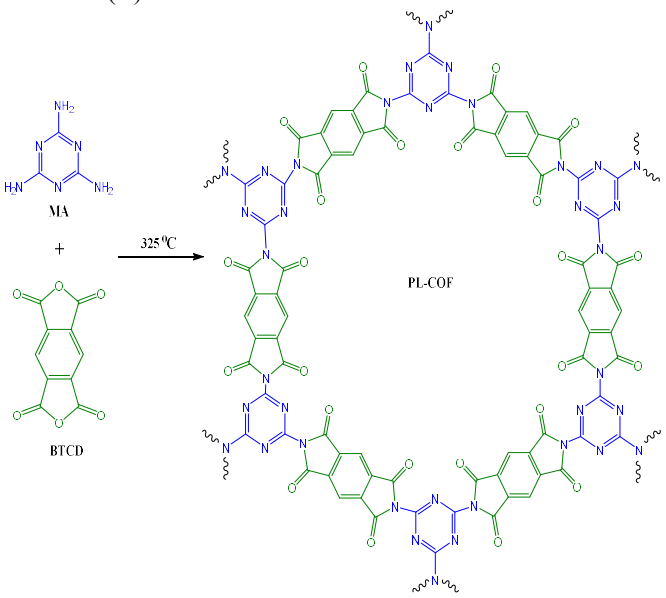
^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.97 (1H, d, J = 8.2 Hz), 7.83-7.81 (2H, m), 7.43-7.34 (3H, m), 7.32 (2H, d, J = 7.4 Hz), 7.03 (2H, d, J = 7.4 Hz), 5.78 (1H, s), 3.67 (3H, s), 3.26 (3H, s), 2.25 (3H, s).

(Table 2, Entry 8) **12-(3-Chlorophenyl)-8,12-dihydro-8,10-dimethyl-9H-naphtho[1′,2′:5,6]pyrano[2,3-d]pyrimidine-9,11-(10H)-dione:**

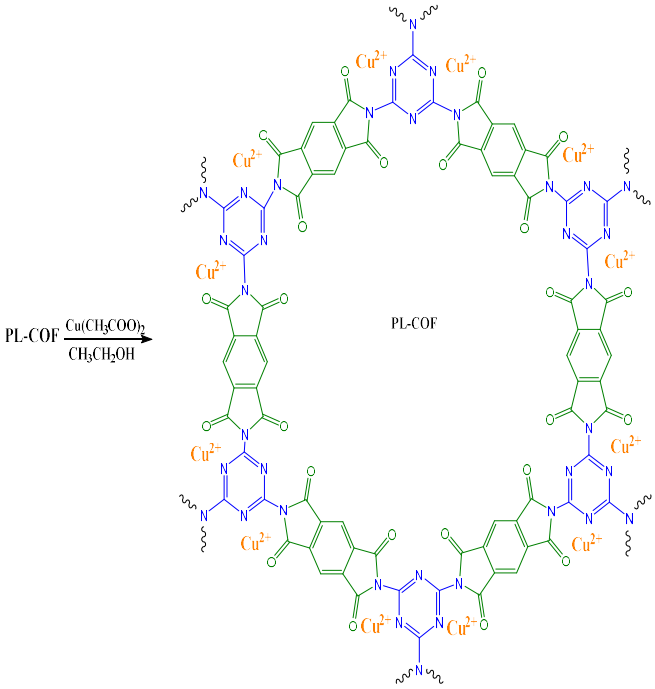
^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 3.36 (3H, s), 3.65 (3H, s), 5.85 (1H, s), 7.07-7.53 (m, 7H, ArH), 7.82-7.80 (m, 3H, ArH).

3. Results and Discussion

Schemes 2 and 3 demonstrate the procedure followed to obtain Cu(II)/PL-COF.



Scheme 2. Synthesis of PL-COF



Scheme 3. An illustration of a Cu(II)/PL-COF catalysis platform

The stability of the catalyst is an important aspect for its practical use. The thermal analysis (TGA) indicated that Cu(II)/PL-COF remained undamaged at temperatures exceeding 380 °C, indicating excellent resistance to heat (refer to Figure 1).

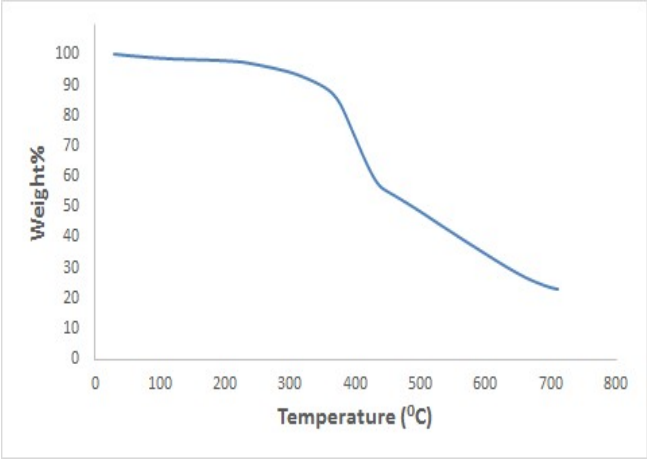


Figure 1. TGA plot of Cu(II)/PL-COF.

Figure S4 (in Supporting Information) provides visual representations of the N_2 adsorption and desorption isotherms of both the PI-COF and Cu(II)/PL-COF composites. The pure PL-COFs have a specific surface area (SSA) of 43.90 m^2/g and pore volumes of 0.035 cm^3/g. The adsorption quantities of nitrogen gradually decline after the active ingredient is loaded. In contrast, the Cu(II)/PL-COF has an SSA of 30.73 m^2/g and a pore volume of 0.029 cm^3/g. Additionally, the pore sizes have been measured to be 3.7 nm for PL-COF and 3.3 nm for Cu(II)/PL-COF (as shown in Fig. S5 in Supporting Information). Consequently, it is possible that a pore-blocking effect may occur due to the presence of Cu(II) in the pores of the PL-COF material[31, 32].

The catalytic activity of Cu(II)/PL-COF was examined in the synthesis of naphthopyranopyrimidines. Following the characterization of Cu(II)/PL-COF, the focus shifted towards improving the reaction parameters for the preparation of naphthopyranopyrimidines. Hence, an investigation was conducted to analyze the effectiveness of the catalyst in the production of naphthopyranopyrimidines. The experiment involved different reaction conditions such as varying microwave strength, catalyst dosage, and time. The objective was accomplished by conducting a three-component reaction using β-naphthol **1**, *N,N*-dimethylbarbituric acid **2**, and 4-chlorobenzaldehyde **3b**, as illustrated in Scheme 1, under solvent-free conditions. The results obtained from this experiment were documented in Table 1. Based on the results presented in Table 1, it was observed that the reaction did not progress even after 20 minutes when the Cu(II)/PL-COF was not present, indicating the effect of catalyst in this reaction. The experiments further demonstrated that the most favorable conditions were achieved when the reaction was conducted in the absence of solvent and with the presence of 0.01 g Cu(II)/PL-COF, using microwave irradiation. The time of reaction and the amount of catalyst was optimized, using various amount of catalyst in different times was carried out (Table 1). The best yield was acquired for a 1 min period in presence of 0.01 g of catalyst (Table 1, Entry 5). Surprisingly, there was no significant change in the reaction yield when increasing the amount of catalyst up to 0.1 g. However, reducing the catalyst dosage to 0.001 g also led to a decrease in yield. The effect of microwave power was tested by varying it from 100 to 300 W (Table 1, Entries 2-4). Initially, an increase in yield was observed when the microwave power was increased because the high microwave power led to effective collisions between the reactants and the active sites of the catalyst[33]. The highest yield of 97% was obtained at 180 W microwave power, however, increasing the microwave power to 300 W led to a decrease in efficiency, because in parallel, the temperature of the reaction mixture immediately increased, leading to a strong collision between the reactants and the active sites of the catalyst[34]. To demonstrate the role of Cu(II) in the reaction, the performance of PL-COF under optimal conditions was examined. The use of PL-COF resulted in no progress in the reaction, indicating that Cu(II) is necessary for catalyzing the reaction. Furthermore, the reaction was conducted with $Cu(CH_3COO)_2$ for 1 minute under optimal conditions, yielding a 35% yield of naphthopyranopyrimidine **4b**. The findings indicate that $Cu(CH_3COO)_2$ has significantly lower performance compared to Cu(II)/PL-COF under optimized conditions. Solvents were not taken into account due to the environmentally friendly design of the experiment.

Table 1. Screening of the reaction conditions for the synthesis of **4b**[a]

Entry	Catalyst (g)	Power (W)	Time min)	Yield (%)
1	-	180	20	-
2	Cu(II)/COF (0.01)	180	5	98
3	Cu(II)/COF (0.01)	300	5	88
4	Cu(II)/COF (0.01)	100	5	85
5	**Cu(II)/COF (0.01)**	**180**	**1**	**97**
6	Cu(II)/COF (0.02)	180	1	98
7	Cu(II)/COF (0.05)	180	1	98
8	Cu(II)/COF (0.1)	180	1	98
9	Cu(II)/COF (0.001)	180	1	28
10	Cu(II)/COF (0.002)	180	1	40
11	Cu(II)/COF (0.005)	180	1	56
12	Cu(II)/COF (0.008)	180	1	75
13	COF (0.01 g)	180	1	-
14	$Cu(CH_3COO)_2$ (0.01 g)	180	1	35

[a]Reaction condition: β-naphthol (1.0 mmol), N,N-dimethylbarbituric acid (1 mmol), 4-chlorobenzaldehyde (1 mmol) and Cu(II)/PL-COF as catalyst under microwave irradiation in solvent-free condition. Yields refer to isolated product.

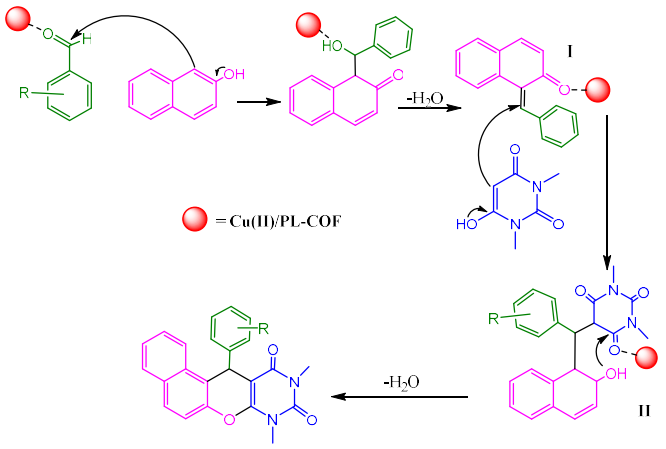
After determining the most appropriate reaction conditions, we needed to evaluate the range and effectiveness of the reaction. To accomplish this, we chose *β*-naphthol, *N,N*-dimethylbarbituric acid, and aromatic aldehydes to undergo the reaction and produce the corresponding naphthopyranopyrimidine derivatives (**4a-o**). The results from these reactions are displayed in Table 2. In terms of the aromatic aldehydes that include substituents with both electron-withdrawing and electron-donating properties, they can be successfully converted into naphthopyranopyrimidine derivatives (**4a-o**) with high yields, as indicated in Table 2. This procedure, as indicated in Table 2, demonstrates its applicability to all aromatic aldehydes. Employing microwave irradiation noticeably reduced the reaction time while achieving the highest synthesis efficiency without generating by-products and necessitating column or flash chromatography purification. By comparing the obtained products with authentic samples, their identification was accomplished relying on their melting points and ^{1}HNMR spectra.

Table 2. Synthesis of benzodiazepine derivatives **4a-o** in the presence of Cu(II)/PL-COF[a]

Entry	R	Product	Time (min)	Yield (%)[a]	Mp (°C)	
					Observed	Literature
1	H	**4a**	1	99	226-228	223-225 [21]
2	4-Cl	**4b**	1	97	272-274	274-275 [35]
3	4-NO_2	**4c**	2	96	294-296	291-293 [18]
4	4-OCH_3	**4d**	2	97	254-258	257-258 [8]
5	4-CH_3	**4e**	2	98	200-203	200-202 [8]
6	3-OCH_3-4-OH	**4f**	4	96	251-252	250-253 [8]
7	2-Cl	**4g**	3	93	270-272	270-272 [16]
8	3-Cl	**4h**	2	96	226-227	226-228 [15]
9	2,4-di Cl	**4i**	3	93	221-223	219-221 [15]
10	4-F	**4j**	1	95	300-303	303-305 [16]
11	2-NO_2	**4k**	4	96	286-288	288-290 [16]
12	3-NO_2	**4l**	4	98	308-310	310-312 [36]
13	3-OH	**4m**	3	98	293-296	293-294 [37]
14	4-CN	**4n**	3	98	277-281	276-280 [15]
15	2-Naphthyl	**4o**	4	94	195-198	193-195 [15]

[a]General reaction conditions: β-naphthol (1.0 mmol), N,N-dimethylbarbituric acid (1 mmol), aldehyde (1 mmol) and Cu(II)/PL-COF (0.01 g) as catalyst under microwave irradiation in solvent-free condition. Yields refer to isolated product.

Scheme 3 presents a potential explanation for the mechanism of the reaction. The combination of *β*-naphthol and aldehyde, with the presence of Cu(II)/PL-COF, leads to the formation of an α,β-unsaturated carbonyl compound called intermediate **I**. Following this, intermediate **II** is developed through a Michael addition reaction involving compound I and 1,3-dimethyl barbituric acid. Finally, a cyclodehydration reaction occurs, resulting in the formation of the intended naphthopyranopyrimidine.[16,18,19]



Scheme 3. Plausible mechanism for Cu(II)/PL-COF catalyzed synthesis of naphthopyranopyrimidines

The effectiveness and ability to be reused of the Cu(II)/PL-COF were confirmed through a model reaction involving the production of naphthopyranopyrimidine **4b**. Following the completion of the reaction, ethanol was utilized to extract and isolate the Cu(II)/PL-COF via filtration. The isolated catalyst was then washed with ethanol, dried, and used again for the subsequent experiment without any significant loss in its catalytic performance. Figure 2 demonstrates the catalyst's reusability through its successful application in five consecutive cycles. ICP-MS analysis revealed a low concentration (0.5 ppm) of Cu(II) in the reaction mixture. TEM and SEM observations revealed that the catalyst's morphology underwent minimal alterations, regardless of whether it was fresh or reused (Figures 3a and 3b).

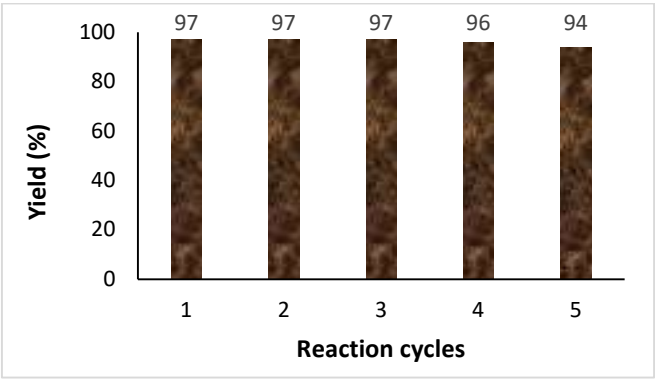


Figure 2. Recyclability of Cu(II)/PL-COF in the model reaction.

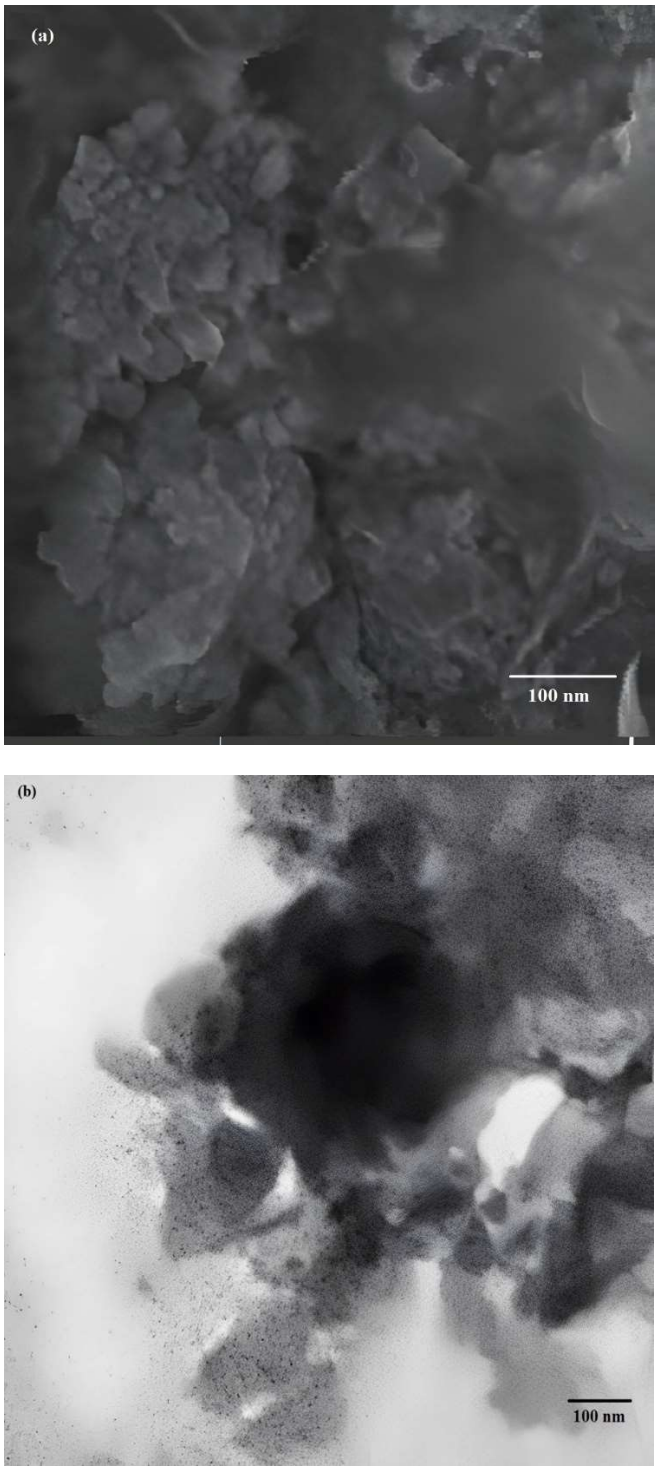


Figure 3. SEM (a) and TEM (b) images of recycled Cu(II)/PL-COF catalyst.

The efficiency of the current approach for synthesizing naphthopyranopyrimidine derivatives is compared to other relevant studies in Table 3. In this comparison, it is evident that the Cu(II)/PL-COF catalyst outperforms other catalysts in terms of both efficiency and time required for the reaction.

Table 3. Comparison of Cu(II)/PL-COF catalyst with other catalysts in the synthesis of naphthopyranopyrimidine **4a**

Entry	Catalyst/conditions	Time (min)	Yield (%)[a]	Ref.
1	$InCl_3$ /120 °C, solvent free	25	80	[37]
2	Heteropolyacid /100 °C/neat	20	90	[36]
3	$Al(H_2PO_4)_3$ /120 °C, solvent free	40	85	[20]
4	L-proline /100 °C, solvent free	20	96	[35]
5	Lactic acid/100 °C, solvent free	20	88	[8]
6	$(PATDBP)(HSO_4)_2$@HNT [a]/100 °C, solvent free	45	98	[19]
7	Nano-$ZnAl_2O_4$/MW (500 W, DMF)	10	88	[38]
7	Cu(II)/PL-COF/MW (180 W, solvent free)	1	96	This study

[a]$(PATDBP)(HSO_4)_2$@HNT = 1,10-(6-(propyl amino)-1,3,5-triazine-2,4-diyl)bis(pyridinium) hydrogen sulfate immobillized on functionalized halloysite nanotube

4. Conclusions

In summary, the Cu(II)/PL-COF catalyst, when combined with microwave irradiation, offers a convenient and efficient method for synthesizing naphthopyranopyrimidine derivatives. This method is notable for its simplicity and effectiveness in generating derivatives of this compound group. It boasts several advantages, including the use of microwaves, high product yield, mild reaction conditions, short reaction times, ease of operation, the ability to reuse nanocatalysts, straightforward workup, and high atom economy. The combination of microwave irradiation and heterogeneous catalysis holds promise for the development of improved, rapid, and environmentally friendly synthetic methods. Additionally, the Cu(II)/PL-COF catalyst can be reused multiple times without significant loss of activity. Furthermore, we anticipate that this synthetic approach will offer expanded possibilities for preparing naphthopyranopyrimidine analogs, making it a viable alternative to existing protocols.

Declaration of Interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Author Contributions

Nasimeh Mahmoudinasab-Manoujan: Investigation, Methodology, Data curation, Writing-Original draft preparation. Farid Moeinpour: Supervision, Conceptualization, Writing-Reviewing and Editing. Fatemeh S. Mohseni-Shahri: Writing-Reviewing and Editing, Visualization.

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Supporting Information

The Supporting Information is available free of charge at http://www.org.chem.res./doi: XXXX

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