

Zn Complex on Tryptophan-Functionalized MCM-41 as an Efficient and Promising Reusable Nanocatalyst in One-Pot Three-Component Synthesis of Amino Benzyl Quinolinols and Naphthols via a Betti Reaction

Shakila Hamedimehr, Khadijeh Ojaghi Aghbash, and Nader Noroozi Pesyan*

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ABSTRACT: In this research, a Zn transition metallic complex on functionalized mesopore silica MCM-41 has been designed and utilized as an effective, recoverable, and thermally stable heterogeneous nanocatalyst in the formation of the C–C bond. Also, the synthesis of amino benzyl quinolinols and amino benzyl naphthol derivatives was performed by the three-component reaction of Betti from aromatic aldehydes, type II amines, and 8-hydroxyquinoline and 1-naphthol in the presence of the MCM-41-tryptophan-Zn nanocatalyst under reflux conditions. The short reaction times, high efficiency of the products, the easy separation of catalysts due to their heterogeneity, and their reusability are the advantages of this method. Finally, the nature of catalysts was studied by using some techniques such as the Fourier transform infrared spectrum, energy-dispersive spectroscopy, hot filtration test, transmission electron microscopy, scanning electron microscopy, X-ray diffraction, inductively coupled plasma optical emission spectrometry, and N₂ adsorption-desorption (BET). The synthesized heterogeneous catalysts showed perfect catalytic activity and also good recyclability in the Betti reaction. They could be reused at least for five consecutive cycles without significant loss of their catalytic activities.



1. INTRODUCTION

In recent decades, green chemistry has been expanding as an important branch of chemistry and has shown an alternative pathway to reduce the intense need for a hazardous response environment in academic and industrial chemistry. Multi-component reaction (MCR) is one of the best ways to develop green chemistry. In recent years, MCR has become a sophisticated tool for synthesizing complex structural components of drugs. In general, the MCR strategy reduces time and cost and leads to the synthesis of an important group of compounds by providing environmental pathways.¹

Betti base [1-(α -aminobenzyl)-2-naphthol, 7, (Figure 1)] is known as an important compound and its applications in the ring-closure reactions to give naphthalene-based heterocyclic derivatives have not been thoroughly investigated.² The few references that have been reported on this topic focused on the reaction of 5 with aldehydes.² More details on Betti base and its derivatives' (7 and 8) applications in organic synthesis are extensively discussed in the literature.³

C–C and C–N bonding reactions play an important role in organic synthesis.^{4,5} The establishment of new C–C and C–N bonds provides new opportunities for total synthesis in pharmaceutical chemistry, chemical biology, and nanotechnology. In particular, cross-coupling reactions between aromatic

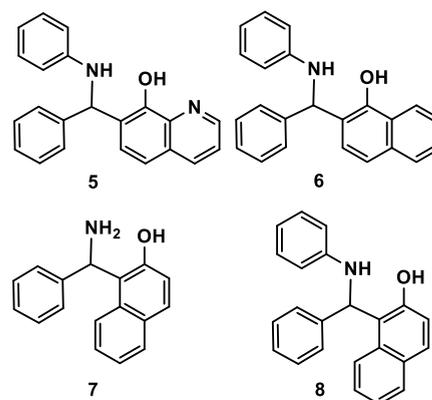


Figure 1. Molecular structures of Betti base and its derivatives.

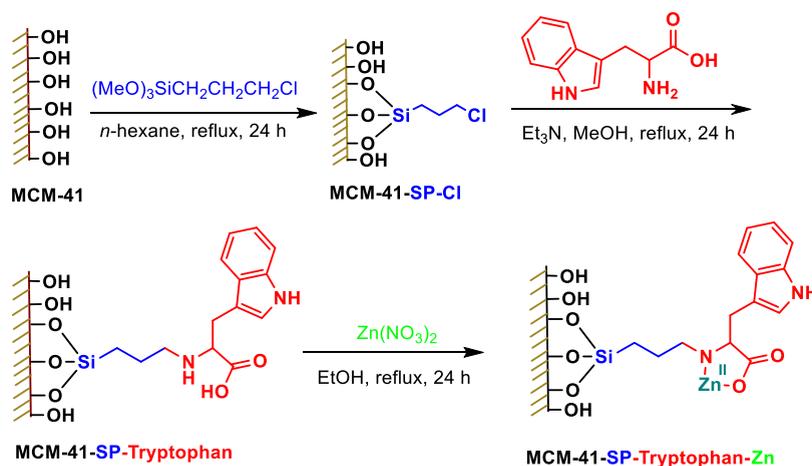
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Scheme 1. Synthesis of MCM-41-Trp-Zn



and aliphatic compounds have been extensively studied. This type of reaction usually occurs in the presence of a catalyst.⁶ Among the various reactions, Betti's basic synthesis (modified Mannich reaction or Mannich amino alkylation) is found to be of great importance because of the mild conditions of reaction for forming C–C and C–N bonds.^{7,8} These compounds are important due to their applications as chiral ligands^{9–12} and their essential medicinal properties including anti-inflammatory, anthelmintic, antifungal, antibacterial, analgesic, anticonvulsant, anti-microbial, antimalarial, anticancer, and antiviral properties.^{13–20} Three-component Betti reaction have been catalyzed with various zinc catalysts such as diisopropenyl zinc,²¹ arylzinc,^{22,23} ZnCl₂,²⁴ Zn(OAc)₂·2H₂O,²⁵ diethylzinc,²⁶ etc.

Tryptophan, an α -amino acid, is used as a ligand. It contains a side chain indole, an α -carboxylic acid group, and an α -amino group. The hydroxyl group and indole nitrogen play a key role in the formation of the chelate ring of metal complexes.^{27,28} Recently, studies have been carried out on the immobilization of amino acids on the surfaces of solid supports to provide a heterogeneous catalyst for the synthesis of heterocycle compounds.^{29–33}

A heterogeneous catalyst has emerged as a useful method to reduce waste production due to the simplicity of the process, less contamination of the product with active catalytic species, and less use of toxic solvents. The metallic nanostructure has excellent promise owing to its unique properties like a great surface area–volume ratio, great surface area, and separation.³⁴

Mesoporous silicate has been used as one of the four most important discoveries in materials science over the past decade. Silica material between the cavity includes SBA and MCM. MCM is a group of porous meso silicate compounds that are widely used due to their high surface area and high OH groups. They are utilized as surface adsorbents ion exchangers and various catalysts.^{35–38}

Nanoparticle (NP) catalytic activity is a rich source for chemical processes used in both industry and academia.^{39–42} High interest in catalysis using nanomaterials has led to the synthesis and study of a variety of highly functional NPs including nanocarbon catalyst-integrated nanocatalysts as well as various metal nanostructures.^{43,44}

In this study, we would like to prepare a new zinc complex deposited on a tryptophan-functionalized nanostructured

mesoporous surface (MCM-41) as an ideal green heterogeneous catalyst with easy catalyst preparation, high stability, and simple separation of the reaction mixture without significant loss of its catalytic activity to synthesize 7-(phenyl(phenylamino)methyl)quinolin-8-ol and 2-(phenyl(phenylamino)methyl)naphthalen-1-ol from aromatic aldehydes, type II amines, and 8-hydroxyquinoline and also with α -naphthol catalyzed by a new effective nanocatalyst (MCM-41-Trp-Zn) under reflux conditions in CH₃COOH solvent at 120 °C.

2. RESULTS AND DISCUSSION

In our investigations into the use of new heterogeneous and reusable catalysts in organic converting,^{36,38,45–47} our decision was made to develop a new heterogeneous nanocatalyst (MCM-41-tryptophan-Zn) and apply it in the synthesis of 7-(phenyl(phenylamino)methyl)quinolin-8-ol. The details of the method of preparing the catalyst are documented and presented by the short path given in Scheme 1.

2.1. Characterization of the Nanocatalyst. The Zn complex is supported over mesoporous MCM-41, and their properties are determined through technical BET, XRD, energy-dispersive spectroscopy, scanning electron microscopy (SEM), and Fourier transform infrared (FT-IR). At first, the FT-IR spectra were separately documented at different steps of preparing the nanocatalyst as shown in the a–d curves in Figure 2. In the a curve for the MCM-41, the symmetric–asymmetric stretching modes of the Si–O–Si mesoporous scaffolds appeared as 496, 803, 1094, and 1615 cm^{−1} absorption bands. The 3434 cm^{−1} absorption is for the stretching mode of OH bands. The b curve showed the stretching vibration of C–H at 2929 and 2728 cm^{−1} for the MCM-41-*n*Pr. The presence of the tryptophan ligand was confirmed by stretching vibrations at 1405 cm^{−1} (C–N), carbonyl of amino acid at 1602 cm^{−1} (C=O), and peak in the region at 3405 cm^{−1} (O–H, N–H) (curve c). After loading Zn metal over MCM-41-tryptophan, an adsorption band is observed at 1385 cm^{−1} in the spectrum, which is related to the stretching vibration of N–O in NO₃[−] and indicates that zinc ion is coordinated on MCM-41-tryptophan (curve d).

In Figure 3, SEM graphs for MCM-41 (a and b) and MCM-41-tryptophan-Zn (c) are depicted. Morphological changes in the synthesized nanostructures were examined by SEM. SEM images confirm that the samples have a regular,

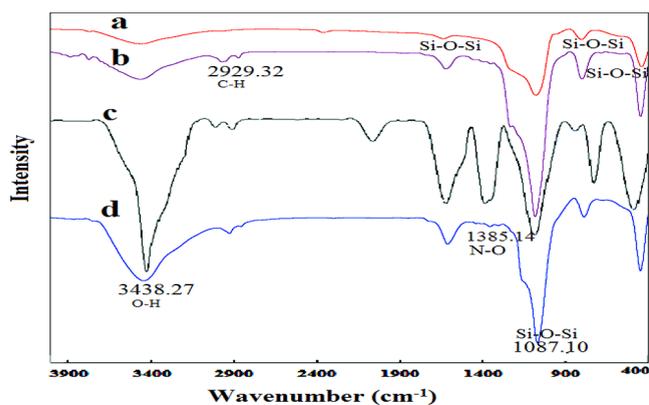


Figure 2. FT-IR spectra of MCM-41 (a), MCM-41-(CH₂)₃Cl (b), MCM-41-Trp (c), and MCM-41-Trp-Zn (d).

spherical morphology. **Figure 3c** shows that the catalyst does not make any noteworthy change in the morphology of the surface for the duration of surface modification and also the solid framework is maintained. **Figure 3d,e** shows the TEM pictures for the constructed MCM-41 and MCM-41-tryptophan-Zn. TEM images of synthesized MCM-41 by the sol-gel method (**Figure 3d**) and of the MCM-41-tryptophan-Zn catalyst (**Figure 3e**) clearly show the high order of two-dimensional channels and the hexagonal structure of equal and uniform pores. The micrographs are in good agreement

with the patterns of XRD, which show the structural order of the channels and pores for the synthesized nanostructure.

X-ray energy diffraction (EDX) spectroscopy analysis was performed on the synthesized MCM-41-tryptophan-Zn catalyst to show the zinc metal deposited on the substrate surface (**Figure 4**). The result confirmed the existence of N,

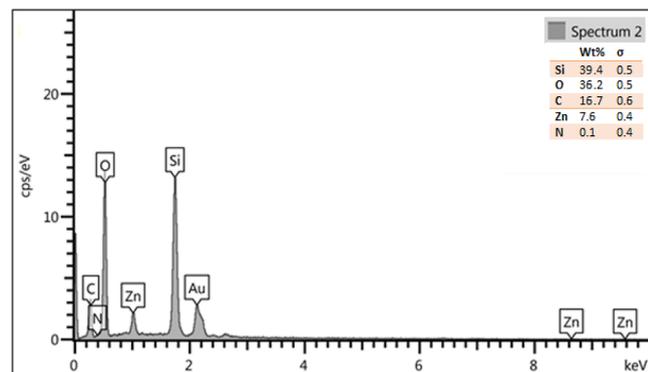


Figure 4. EDX spectrum MCM-41-tryptophan-Zn.

Zn, C, O, and Si in MCM-41-tryptophan-Zn, and in addition, the mass percentages were obtained as 0.1, 7.6, 16.7, 36.2, and 39.4 respectively. Moreover, based on the inductively coupled plasma optical emission spectrometry (ICP-OES) measure-

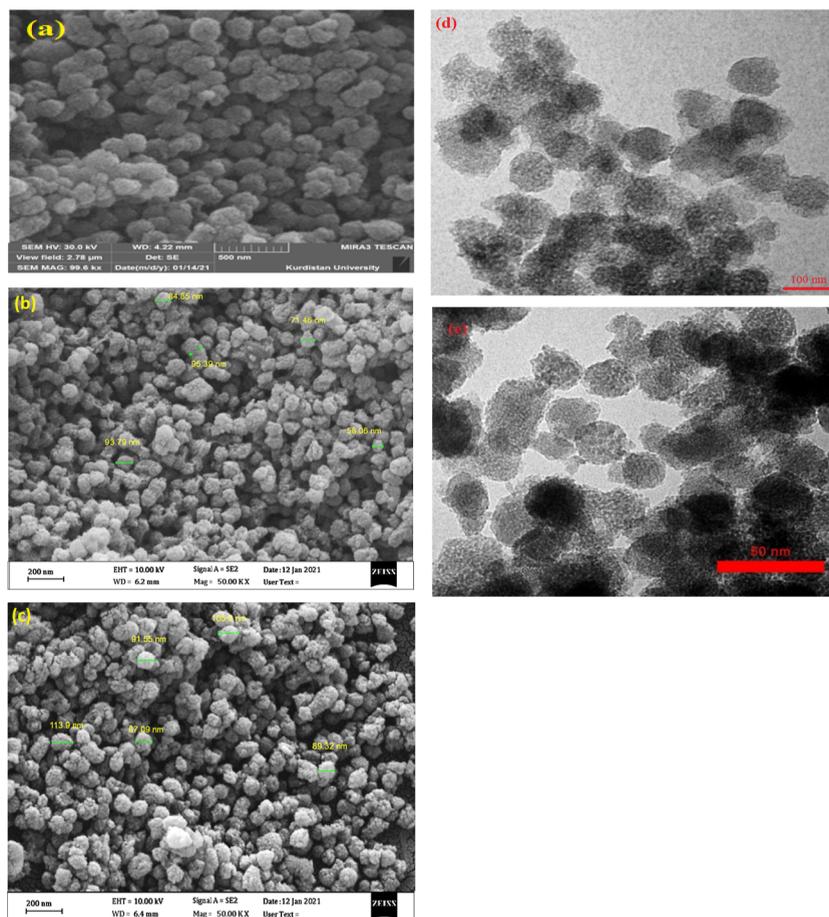


Figure 3. SEM micrographs: (a,b) for MCM-41 and (c) for MCM-41-Trp-Zn. Micrographs of TEM: (d) for MCM-41 and (e) for MCM-41-Trp-Zn.

ment, the exact amount of Zn in this nanocatalyst has been determined to be $1.5 \times 10^{-3} \text{ mol g}^{-1}$.

One of the technical tools utilized to determine the structural properties and structural investigation of the channels and pores in synthesized mesoporous materials is the XRD pattern. Figure 5 shows the XRD pattern for the

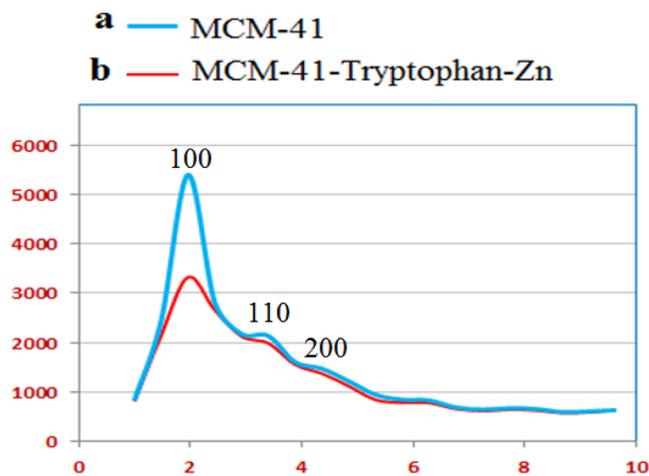


Figure 5. XRD pattern of MCM-41 (a) and MCM-41-tryptophan-Zn (b).

MCM-41 and Zn-tryptophan-MCM-41 samples. The XRD pattern for the MCM-41 nanopores appears to be a sharp peak at an angle of $2\theta = 2.38^\circ$ for diffraction at the d100 plane and two lower peaks at $2\theta = 4.18^\circ$ and $2\theta = 4.78^\circ$ for diffraction at d110 and d200. It can be seen that this pattern is established for all steps of functionalization. This pattern shows that the structural order of the substrate has not changed after functionalization. In addition, using this pattern, useful information can be obtained about the filling of cavities by functional groups in such a way that the XRD before and after being functionalized by the functional groups changes the intensity of the peaks. The peak intensity decreases when the pores are filled by the functional groups but does not change at the peak intensity when the functional groups are placed on an external surface. Therefore, the observation of decreasing peak intensity indicates the filling of the pores with

functional groups in pattern (b) XRD MCM-41-tryptophan-Zn.

In order to determine weight changes of functionalized MCM-41, thermogravimetric analysis (TGA) was carried out. Figure 6 shows the TGA curves for MCM-41 (a), MCM-41-(CH_2)₃Cl (b), MCM-41-tryptophan (c), and MCM-41-tryptophan-Zn (d). According to these TGA curves, the initial weight loss at temperatures below 200 °C is because of the removal of chemically and physically adsorbed H₂O and organic solvents such as EtOH inside the channels of the pores and surface hydroxyl (–OH) groups, and the large weight loss by increasing the temperature from 200 to 800 °C is mainly related to the decomposition of covalently bonded organics (200–500 °C) and the silanol (Si–OH) groups (500–800 °C), respectively.

In Figure 7, the isotherms of nitrogen adsorption–desorption for samples of MCM-41 and Zn-tryptophan-

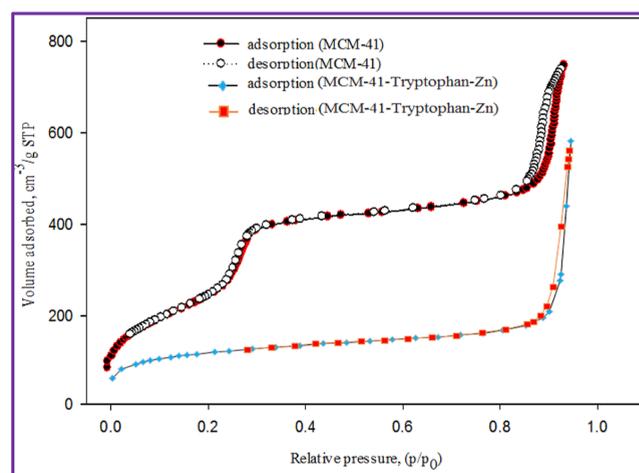


Figure 7. Isotherms of BET for MCM-41 and MCM-41-tryptophan-Zn.

MCM-41 are depicted. In Table 1, surface features like wall thickness of samples, total pore volume (V_{total}), BET surface (S_{BET}), and pore diameter (D_{BJH}) are summarized. The results showed that the area of the surface and volume of the pore for the MCM-41-tryptophan-Zn catalyst are decreased compared

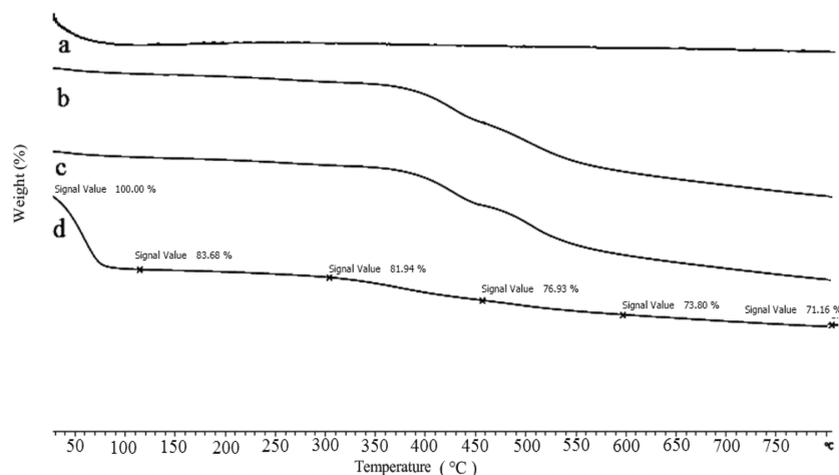


Figure 6. TGA thermograms of MCM-41 (a), MCM-41-(CH_2)₃Cl (b), MCM-41-Tpr (c), and MCM-41-tryptophan-Zn (d).

Table 1. Deduced Textural Factors from Sorption Isotherms of Nitrogen

sample	pore volume (cm ³ /g)	pore diameter by the BJH method (nm)	S _{BET} (m ² /g)
MCM-41	1.1	3.2	990
MCM-41-tryptophan-Zn	0.8	1.6	321

to that of the MCM-41. Also, the thickness of the wall for MCM-41-tryptophan-Zn increased. Decreased effective surface area and pore volume and increased wall thickness in the MCM-41-tryptophan-Zn catalyst indicate stabilization of organic groups and the zinc complex on the mesoporous wall.

2.2. Synthesis of 7-(Phenyl(phenylamino)methyl)quinolin-8-ol) via MCM-41-Tryptophan-Zn Catalytic Amounts. In the wake of characterizing and confirming the structure of the nanocatalyst, we tried to develop an efficient and environmentally friendly method for the synthesis of 7-(phenyl(phenylamino)methyl)quinolin-8-ol) (**5**) and 2-(phenyl(phenylamino)methyl)naphthalen-1-ol (**6**) derivatives through the MCM-41-tryptophan-Zn catalyst as a fine recyclable and stable nanocatalyst. Representatively, for this purpose, selecting the reaction of benzaldehyde (**1**), aniline (**2**), and 8-hydroxyquinoline (**3**) was as a simple model reaction for the one-pot synthesis of **5**. **Table 2** shows the results. The reaction conditions were optimized by applying the solvent effect, the amount of catalyst, and the temperature. The best results were yielded via benzaldehyde (0.09 g), aniline (0.12 g), and 8-hydroxyquinoline (0.14 g) in the presence of a catalyst (20 mg, 3 mol %) and solvent of CH₃COOH (5 mL) at 120 °C (**Table 2**).

After obtaining the optimal conditions, the reaction of benzaldehyde derivatives with aniline and 8-hydroxyquinoline **3** and also with α -naphthol (**4**) was used to expand the application of this catalyst in the synthesis of **5** and **6**. The reaction of different types of benzaldehyde derivatives with electron-withdrawing and electron-donating groups was investigated and the results are presented in a table (**Table 3**). Summarized results in **Table 3** indicated that in general a wide range of aldehydes could perform the Betti reaction easily and give amino benzyl quinolinols and naphthols in high yields and moderate turnover frequency number and turnover number. As the results show, the reaction has high yields for benzaldehyde with electron-withdrawing groups.

Based on studies on the synthesis of 7-(phenyl(phenylamino)methyl)quinolin-8-ol compounds from the reaction of benzaldehyde derivatives with aniline and 8-hydroxyquinoline in the presence of a metal catalyst, the following mechanism can be studied for the catalytic system. First, the carbonyl group of benzaldehyde is activated by a metal nanocatalyst, and then, an intermediate **I** is formed by the nucleophilic attack of the aniline amine group on the activated carbonyl of benzaldehyde. Next, intermediate **I** is converted to imine intermediate **II** via the loss of a water molecule. Eventually, the desired product is subsequently formed through the intermolecular attack of 8-hydroxyquinoline α -carbon on the activated imine by a metal catalyst and then with the transfer of a proton (**Scheme 2**).

To evaluate the advantages of the used method in this study with the reported methods in the literature for synthesizing 7-(phenyl(phenylamino)methyl)quinolin-8-ol compounds, a comparison was performed between the obtained results and the reported results in the literature (**Table 4**).

As can be seen, many catalysts such as nano MgO, NS-PCS, and GO-MnO-Au have been reported for the synthesis of these compounds. Most of these methods have limitations like toxic catalysts and homogeneous catalysts, and the use of strong acidic conditions are very problematic to separate from the reaction mixture as well as the long reaction times and toxic solvents. However, the MCM-41-tryptophan-Zn heterogeneous catalyst has been used in this study, which is easily prepared, recycled, and reused. In addition, zinc metal which is an available metal is used in this reaction, and also, high catalytic activity is exhibited for the synthesis of 7-(phenyl(phenylamino)methyl)quinolin-8-ol compounds.

2.3. Leaching Study of Nanocatalysts. To investigate the heterogeneity of the nanocatalyst, a hot filtration experiment was performed. For this purpose, benzaldehyde, aniline, and 8-hydroxyquinoline were utilized as model substrates. In the half-time of the reaction (60 min), the reaction was stopped and the nanocatalyst was filtered. In this step, 52% of the product was obtained. After hot filtering the nanocatalyst, possible catalytic activity in the resulted solution was followed in the absence of the catalyst for a further 60 min. When the reaction was completed, no considerable progress was observed and only 54% of the product was obtained. This test confirmed that negligible Zn leaching occurred.

Table 2. Effect of Different Parameters on the Synthesis of 5^a

entry	cat. (mg)	solvent	temp (°C)	time (min)	yield (%) ^b
1	8	PEG	rt	200	83
2	10	CH ₃ COOH	120	120	85
3	20	CH ₃ COOH	120	120	90
4	5	EtOH	100	50	80
5	5		120	110	70
6	5	Toluene	110	40	88
7	3	H ₂ O	rt	60	75

^aConditions of reaction: benzaldehyde (1.2 mmol), aniline (1.0 mmol), and 8-hydroxy quinolin (1.0 mmol). ^bIsolated yields.

Table 3. MCM-41-Tryptophan-Zn Catalyzed the One-Pot Synthesizing 5 and 6 in CH₃COOH

Entry	Product (5 and 6)	Time (min)	Yield (%) ^{a,b}	TON	TOF (min ⁻¹)	Mp (°C)
1		120	90	30	0.25	140-142
2		100	90	30	0.3	120-122
3		80	92	30.6	0.38	116-118
4		250	92	30.6	0.12	121-123
5		45	94	31.3	0.69	129-131
6		260	91	30.3	0.11	149-151

^aThe structure of all produced derivatives was confirmed utilizing a comparison of their spectral and physical data with the ones of authentic samples. ^bIsolated yield.

2.4. Recyclability of the Nanocatalyst. To investigate this, recycling the MCM-41-tryptophan-Zn catalyst was studied in the Betti condensation reaction of benzaldehyde, aniline, and 8-hydroxyquinoline as a reaction of the model under optimized conditions in acetic acid. After the completion of the reaction, the catalyst was separated from the reaction mixture by centrifugation, washed several times with water and ethanol, and after drying, it was used in the next cycle. As shown in Figure 8, the MCM-41-tryptophan-Zn catalyst was reused at least 5 times without any reduction in activity. Reproducible and high catalytic activity and significantly low leaching of Zn (during catalytic reactions) from MCM-41 functionalized with the tryptophan group were observed, the amount of Zn in the five recoveries determined by ICP-OES. According to the ICP-OES results, the amount of Zn was determined for the unreacted catalyst (1.2%) and

after 5 runs (1.1%). The nature of the catalyst was investigated after five reuses by IR (Figure 9), SEM, and TEM (Figure 10) techniques. The SEM and TEM analyses confirmed that the catalyst may be reused at least 5 times without any change in its the mesoporous scaffold.

3. CONCLUSIONS

In this study, a new zinc complex deposited on the mesoporous surface was prepared by the sol-gel process, and its structure was identified by various analysis techniques. The catalyst showed high activity in the Betti reaction for the synthesis of 7-(phenyl(phenylamino)methyl)quinolin-8-ols. Also, easy catalyst preparation, high stability, and easy separation from the mixture of the reaction without loss of catalytic activity make MCM-41-tryptophan-Zn an ideal and green heterogeneous catalyst.

Scheme 2. Representatively, the Proposed Mechanism for the Synthesis of 7-(Phenyl (phenylamino)methyl)quinolin-8-ol (5) Compounds in the Presence of the MCM-41-Tryptophan-Zn Catalyst

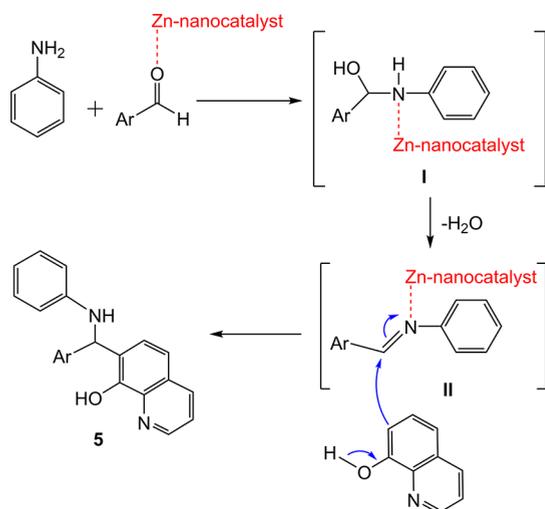


Table 4. Comparing the Efficiency of Several Catalysts to the Synthesis of 7-(Phenyl(phenylamino)methyl)quinolin-8-ol in the Literature

entry	catalyst	time (min)	temp (°C)	yield (%)	lit.
1	MCM-41-tryptophan-Zn	120	120	90	this work
2	nano MgO	120	rt	88	48
3	GO-MnO ₂ -Au	10	rt	96	49
4	NS-PCS	240	rt	98	1
5	FHS	300	rt	97	1
6	MCM-41-SO ₃ H	70	rt	97	50

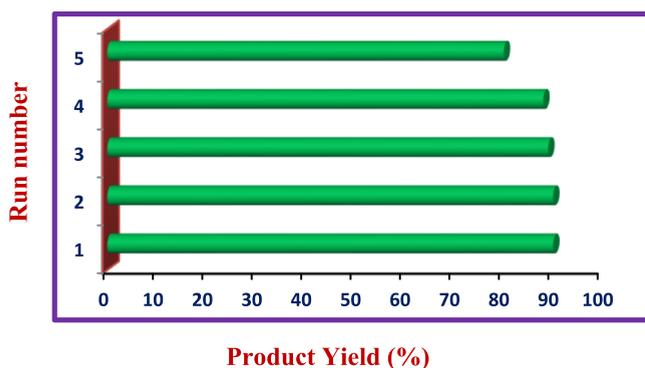


Figure 8. Reusability of catalysts MCM-41-tryptophan-Zn.

4. EXPERIMENTAL SECTION

3.1. Materials and Tools. All used chemicals and solvents are from Merck, Aldrich, and Fluke chemicals in this work. The spectra of FT-IR were performed with pellets of KBr using a Nexus 670 FT-IR spectrometer (Daypetronic Company, Tehran, Iran). TL-chromatography was used to monitor the reaction over silica gel plates. Particle morphology was investigated using a scanning electron microscope (Daypetronic Company, Iran-Tehran) through FESEM-TESCAN MIRA3. The 500 MHz spectra of ¹H NMR and ¹³C NMR were obtained using a spectrometer of

BRUKER NMR (Daypetronic Company, Iran-Tehran). Energy scattered X-rays of MNP_s were recorded using FESEM-TESCAN MIRA3 (Daypetronic Company, Tehran, Iran). XRD was performed using Co radiation with a 40 kV wavelength scattering P AD V X-ray (Beamgostar Taban Company, Tehran, Iran). The samples were scanned in the ranges of 2θ = 1–10° and 2θ = 10–80°; a BET isotherm of N₂ was recorded at 77 °K employing a standard gas manifold to explain the features of materials like average pore diameter, pore-volume, and catalyst surface (Daypetronic Company, Tehran, Iran). In addition, adsorption data are presented in Brunauer–Emmett–Teller (BET) method. In all reactions, CH₃COOH with a molecular weight of 400 was applied. The content of Zn was measured by ICP-OES analysis (Tarbiat Modares University, Tehran, Iran).

3.2. Silica MCM-41 Preparation. MCM-41 was obtained via the sol–gel process in accordance with the procedure found in the literature.⁵¹ The Si-MCM-41 construction method is as follows: in the conventional method, 1 g of surfactant cetyltrimethylammonium bromide was added to a solution of deionized H₂O (480 mL) and NaOH (2 M, 3.5 mL), which was stirred at 353.15 K. As soon as the solution was homogenized, tetraethyl orthosilicate (5 mL) was slowly added to form a white slurry. Then, the resultant mixture was refluxed for 2 h with continuous stirring at the same temperature. The produced solid was collected, filtered, washed with DI-H₂O, and dried at 343.15 K in an oven. For removing residual surfactants, it was then calcinated at 823.15 K for 5 h. In the end, mesoporous Si-MCM-41 was obtained.

3.3. MCM-41-(CH₂)₃Cl Preparation. The powder MCM-41 (4.8 g) was added to the mixture of 3-chloropropyl-(trimethoxy)-silane (CPTMS) (5 g) in 96 mL of *n*-hexane, and the mixture was stirred under a N₂ atmosphere and refluxed for 24 h. Collecting the obtained solid was done by utilizing filtration and then washed several times in *n*-hexane and next dried in a vacuum to give MCM-41-Cl.

3.4. MCM-41-Tryptophan Preparation. 1 g of MCM-41-Cl, 1 g of tryptophan, and 2 mL of triethylamine (Et₃N) with 50 mL of ethanol in a 100 mL reflux flask were stirred for 24 h under refluxing. Next, the obtained product was washed with methanol several times and dried at 333.15 K for 12 h in a vacuum oven.

3.5. MCM-41-Tryptophan-Zn Preparation. Surface-modified MCM-41 (tryptophan-MCM-41) (2 g) was mixed with Zn(NO₃)₂·6H₂O (1 g) in 35 mL of EtOH and stirred for 20 h under refluxing. For removing any metal ions without being anchored, the resulted white solid was filtered, washed with EtOH several times, and dried at 333.15 K for 12 h in a vacuum. Therefore, the produced catalyst was termed MCM-41-tryptophan-Zn.

3.6. Typical Procedure for Preparing 7-(Phenyl(phenylamino)methyl)quinolin-8-ol. In a 15 mL vacuum flask equipped with a magnetic stirrer, acetic acid (4 mL) and benzaldehyde (0.09 g) were added, and the flask was placed on a heater for 30 min. Then, after adding the MCM-41-tryptophan-Zn catalyst (0.02 g), it was subjected to reflux for 30 min. Subsequently, after adding aniline (0.12 g), it was refluxed for 1 h and next, 8-hydroxyquinoline (0.14 g) was added to the reaction mixture. The progress of the reaction was controlled by TLC using a 3:1 ratio of an *n*-hexane-ethyl acetate solvent mixture. After the reaction was complete, the nanocatalyst was separated by centrifugation. The product was

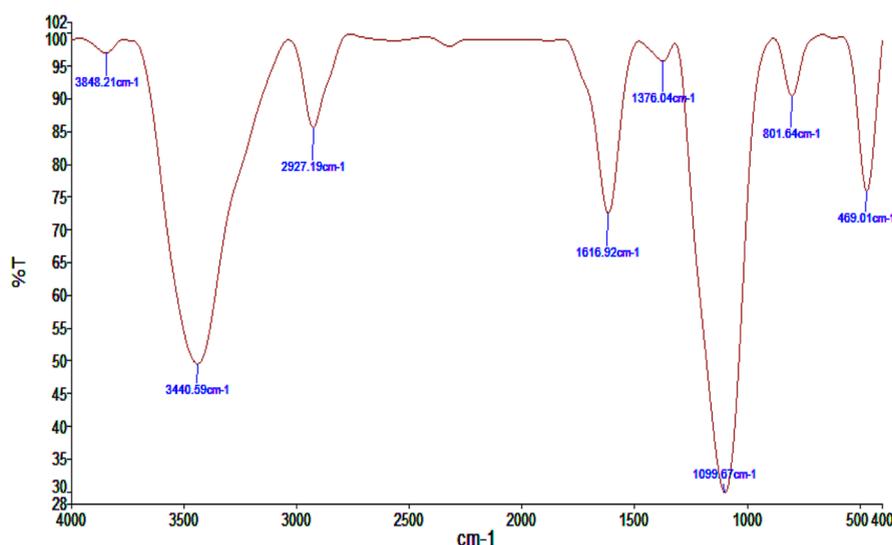


Figure 9. IR analysis of recycled MCM-41-tryptophan-Zn.

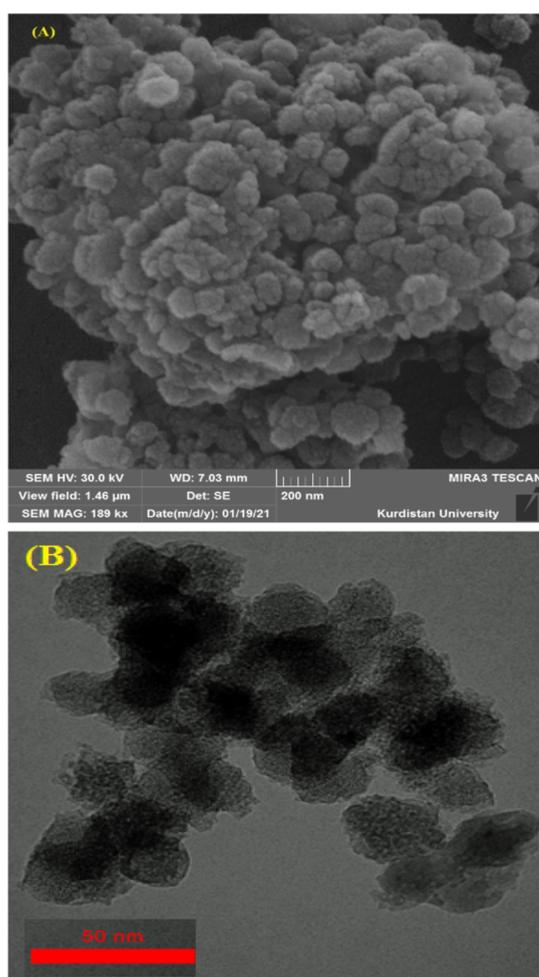


Figure 10. SEM (A) and TEM (B) techniques of recycled MCM-41-tryptophan-Zn.

extracted by evaporating the solvent. Finally, 7-(phenyl-(phenylamino)methyl)quinolin-8-ol products were obtained with high purity. The obtained products were identified and confirmed by spectroscopic techniques.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acsomega.2c05723>.

FTIR, ^{13}C , and ^1H NMR spectral data for derivatives 5a–5c and 6a (PDF)

■ AUTHOR INFORMATION

Corresponding Author

Nader Noroozi Pesyan – Department of Organic Chemistry, Faculty of Chemistry, Urmia University, 57159 Urmia, Iran; orcid.org/0000-0002-4257-434X; Email: n.noroozi@urmia.ac.ir

Authors

Shakila Hamedimehr – Department of Organic Chemistry, Faculty of Chemistry, Urmia University, 57159 Urmia, Iran
Khadijeh Ojaghi Aghbash – Department of Organic Chemistry, Faculty of Chemistry, Urmia University, 57159 Urmia, Iran

Complete contact information is available at: <https://pubs.acs.org/10.1021/acsomega.2c05723>

Notes

The authors declare no competing financial interest.

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