ORIGINAL PAPER

Anchoring of Cu–dimethylglyoxime complex in MCM-41 matrix: a new, recyclable, and highly efficient nanocatalyst for the green preparation of 2,3-dihydroquinazolin-4(1H)-ones

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Abstract

A new heterogeneous nanocatalyst containing Cu–dimethylglyoxime complex grafted to an MCM-41 matrix was synthesized. This recoverable nanocomposite was applied as a highly effective, green, thermally stable catalyst in the one-pot reductive cyclization of aromatic aldehydes with 2-aminobenzamide. 2,3-Dihydroquinazolin-4(1H)-ones were obtained in 80–98% yield. The MCM-41-silylcyclopropyl-dimethylglyoxime-Cu heterogeneous nanocomposite was well identified using X-ray diffraction, field emission scanning electron microscopy, Fourier transform infrared spectroscopy, energy-dispersive X-ray spectroscopy, Brunauer–Emmett–Teller technique, and thermogravimetric analysis. The eco-friendly nanocatalyst demonstrated excellent recyclability: it could be reused for at least six successive cycles without any notable decrease in its catalytic function.

Graphic abstract
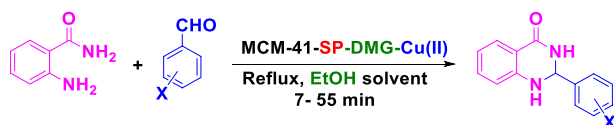
Keywords MCM-41 · Cu–dimethylglyoxime complex · 2,3-Dihydroquinazolin-4(1H)-one · Nanocatalyst

Introduction

Recently, nanoporous materials have a superior role in green chemistry and have received a lot of attention amongst the industrial and academic scientific society, owing to facile recovery, high surface area, and their fundamental influence on the human life quality and economic advance [1, 2]. Also, these nanostructures have versatile applications in materials science owing to shape and size-selective different usages. Among them, mesoporous silica materials like MCM-41 as the typical representative of the M41S material family has attracted much attention because of their several unique properties including large pore volume (normally over 0.8 cm^3/g), unique channel structure, well thermal stability, large surface area (~1000 m^2/g), and good biocompatibility as well as ready surface functionalization [3, 4]. Due to unique properties these mesoporous creating suitable properties such as controllable, morphologies, narrow pore size distribution, structural simplicity, and ease synthesis with insignificant pore obstruction and pore networking [5–8]. These properties have made MCM-41 frequent support for heteropoly acids [9], immobilizing acids [10], complexes [11, 12], metal oxides [13], etc. Also, these mesoporous have been used for several applications such as adsorption [14], support of various catalysts [15], environmental science [16], sensors [17], drug delivery [18], etc. Finally, there is also the possibility of presentation transition metals including Cr [19], Pd [20], Mn [21], Cu [22], VO [23], Ni [15,

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24], Mo [25], V [26], Co [27], and Fe [28] in the MCM-41 matrix to preparing different redox heterogeneous catalysts.

From a long time ago, copper-dimethylglyoxime complexes have been examined due to their possible utilization in organic and catalysis reactions [29]. Its catalytic property increases by grafting of the copper-dimethylglyoxime complex onto the MCM-41 matrix. 2,3-Dihydroquinazolinones, six-membered heterocyclic compounds, are of interest to many chemists owing to extensive range of different biological and pharmacological activities, anticancer, antifertility, antihistaminic, antibiotic, cytotoxicity, antibacterial, antitumor, painkiller, angiotensin II AT1 receptor antagonists, and mono amine oxidase inhibition [30–32]. Generally, various methods have been presented for the synthesis of 2,3-dihydroquinazolinones [33–36]. Among them, 2,3-dihydroquinazolin-4(1*H*)-one derivatives were synthesized via reductive cyclization of 2-aminobenzamide with various ketones or aldehydes in the presence of acid catalysts [32, 37]. In this synthetic process, many methods have been developed using different catalytic systems under different conditions [38–41]. Nevertheless, most of these catalytic processes have unfavorable reaction conditions such as harsh reaction conditions, longer reaction time, use of a major amount of poisonous and costly catalysts, unpleasant yields, and frustrating workup. So one of the goals of organic chemists is to develop better reaction conditions for the synthesis of these compounds using novel catalysts.

Herein, we synthesized a complex of copper-dimethylglyoxime based on MCM-41 nanoparticles and utilized it for the preparation of 2,3-dihydroquinazolin-4(1*H*)-one derivatives using cyclocondensation of aromatic aldehydes with 2-aminobenzamide under conditions of reflux in ethanol solvent with excellent yields, low reaction time, and easy workup.

Results and discussion

In our examinations on the use of heterogeneous nanocatalysts in organic reactions [42], herein, we synthesized a novel copper-dimethylglyoxime complex supported on MCM-41. We also checked its function as an effective heterogeneous and recoverable nanocatalyst in the preparation of 2,3-dihydroquinazolin-4(1*H*)-one derivatives. The details of the nanocatalyst preparation method were presented in Scheme 1.

Catalyst characterization

The nanocatalyst was fully characterized by XRD (X-ray diffraction), FESEM (field emission scanning electron microscopy), FT-IR (Fourier transform infrared spectroscopy), EDS (energy-dispersive X-ray spectroscopy), BET (Brunauer–Emmett–Teller technique), and TGA (thermogravimetric analysis). The FT-IR spectra of MCM-41-dimethylglyoxime-Cu were shown in Fig. 1. As is depicted in Fig. 1, for the MCM-41, the absorption peaks at 1652, 1085, 805, and 459 cm^{-1} are related to stretching vibrations (symmetric and asymmetric) of Si–O–Si. Also, the peak at 3409 cm^{-1} indicates the presence of O─H groups stretching vibration. Stretching vibrations at 2953 and 3305 cm^{-1} attributed to the C─H of the chloropropylsilyl group. The existence of the grafted complex of Cu–dimethylglyoxime to functionalized MCM-41 can be confirmed by the presence of
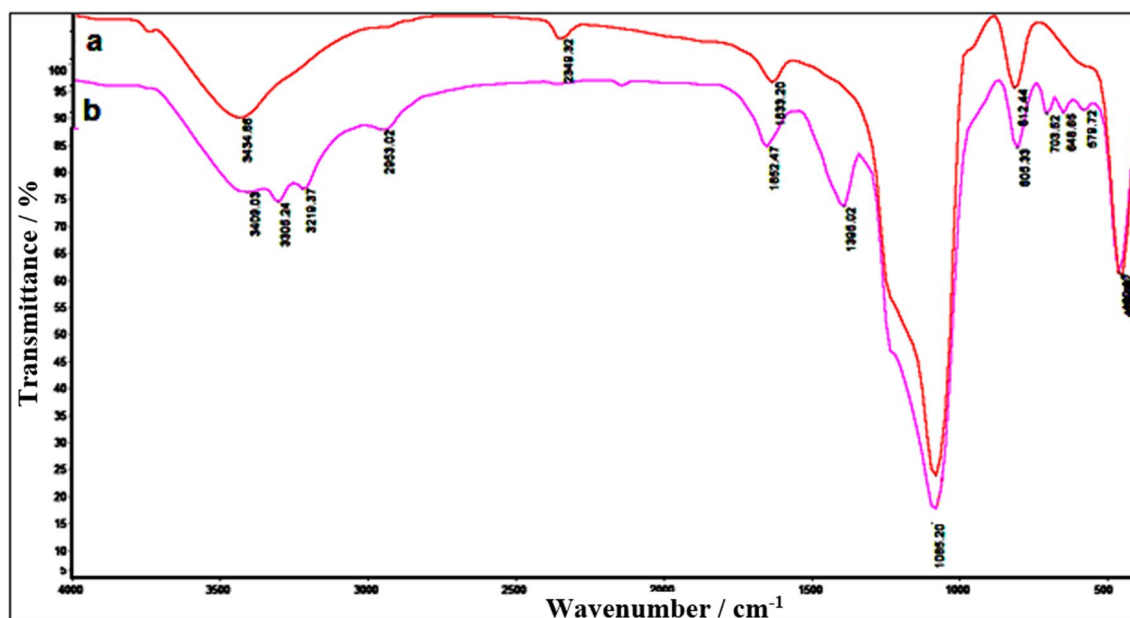
Scheme 1
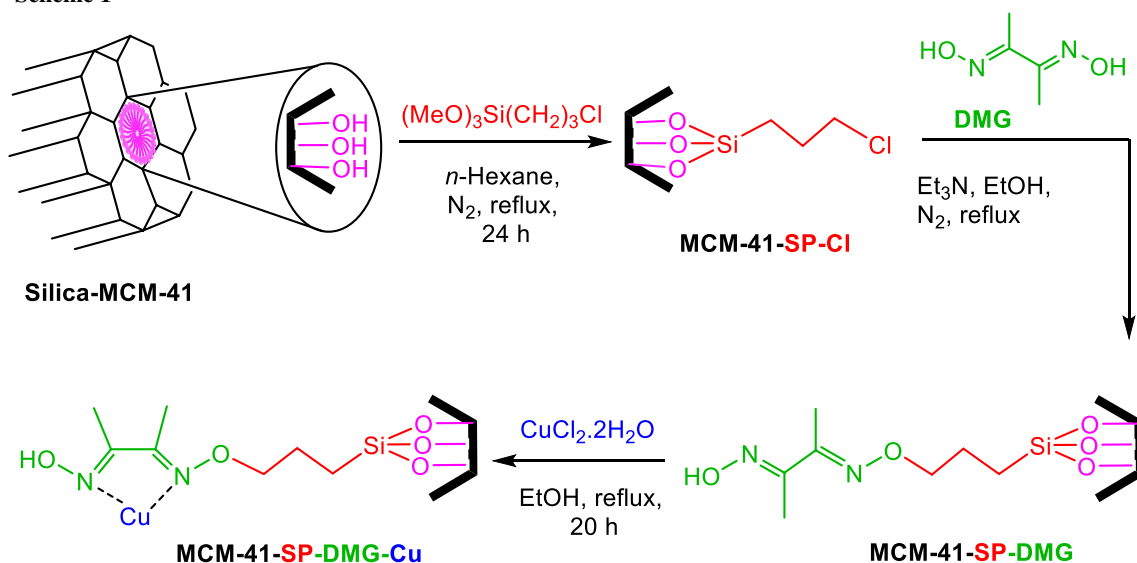
Fig. 1 FT-IR spectra of **a** MCM-41 **b** Cu(II)-dimethylglyoxime-MCM-41

peaks at 1395 and 579 cm^{-1} that are allocated to stretching vibrations of C—N and Cu—N (Fig. 1) [43, 44].

The spherical morphology of MCM-41 and Cu(II)-dimethylglyoxime-MCM-41 was studied using FESEM images (Fig. 2). The FESEM images confirm that MCM-41 has almost spherical and regular morphology. The size distribution histogram for MCM-41 and Cu(II)-dimethylglyoxime-MCM-41 nanoparticles (Fig. 2) was shown a mean of 89.8 ± 2.4 nm and 86.8 ± 2.0 nm, respectively. It confirmed that these structures maintain their high monodispersity profile even after immobilization on the support.

As shown in Fig. 3, the EDX has confirmed the presence of every anticipated element in the produced Cu(II)-dimethylglyoxime-MCM-41, including O, C, Si, N, and Cu with the relative weight percents 30.0, 20.5, 39.30, 1.3, and 8.9%, respectively. Moreover, the presence of the element of nitrogen in elemental analysis demonstrates dimethylglyoxime ligand has fully bonded onto MCM-41, and the metal of Cu has coordinated successfully to the ligand of dimethylglyoxime. These results indicate that the new nanocatalyst was constructed successfully.

Figure 4 demonstrates the low-angle powder XRD patterns of Cu(II)-dimethylglyoxime/MCM-41 and MCM-41 samples. The XRD pattern of MCM-41 illustrates the presence of three reflection peaks corresponding to (200), (110), (100) and planes at $2\theta = 4.33°$, $3.87°$, and $2.41°$, respectively. These patterns confirm the presence of ordered hexagonal mesoporous structure of MCM-41 (Fig. 4a). As shown in Fig. 4b, during the functionalization steps, a remarkable decline in the severity of XRD peaks was seen. It is due to decrease of dispersion contrast between the channel wall of

complex and framework of silicate, or may be because of the alterations in the thickness of the wall. Thus, it can be found out that the preparation of the nanocatalyst has been accomplished on the internal pore channels of the MCM-41/Si.

Thermal stability of Cu(II)-dimethylglyoxime/MCM-41 mesoporous was determined using the TGA and TGA/DTA analysis. TGA (Fig. 5, top) and TGA/DTA (Fig. 5, bottom) curves showed primary weight decrease at temperatures under 200 °C owing to desorption of organic solvent and water molecules. Furthermore, raising the temperature from 200 to 800 °C, the major weight decrease was observed due to the thermal decomposition of organic functional groups located inside the framework.

The N$_2$ adsorption–desorption isotherm of the Cu(II)-dimethylglyoxime/MCM-41 nanocomposite was shown in Fig. 6. The pore features of the prepared nanocomposite were investigated using the Brunauer–Emmett–Teller (BET) technique. The BET entire cavity capacity and surface areas for the nano composites were 0.1819 m^3 g^{-1} and 1.400 m^2 g^{-1}, respectively. By using the technique of Barrett-Joyner-Halenda (BJH), the corresponding pores diameter distributions of Cu(II)-dimethylglyoxime/MCM-41 were specified as 1.21 nm (Fig. 7). The structure of the porous medium contains pores more than 2 nm ($D_v > 2$ nm) which demonstrates a mesoporous structure.

Catalytic activity of the Cu(II)-dimethylglyoxime/ MCM-41

We examined the catalytic activity of Cu(II)-dimethylglyoxime/MCM-41 as a novel, stable, and recyclable
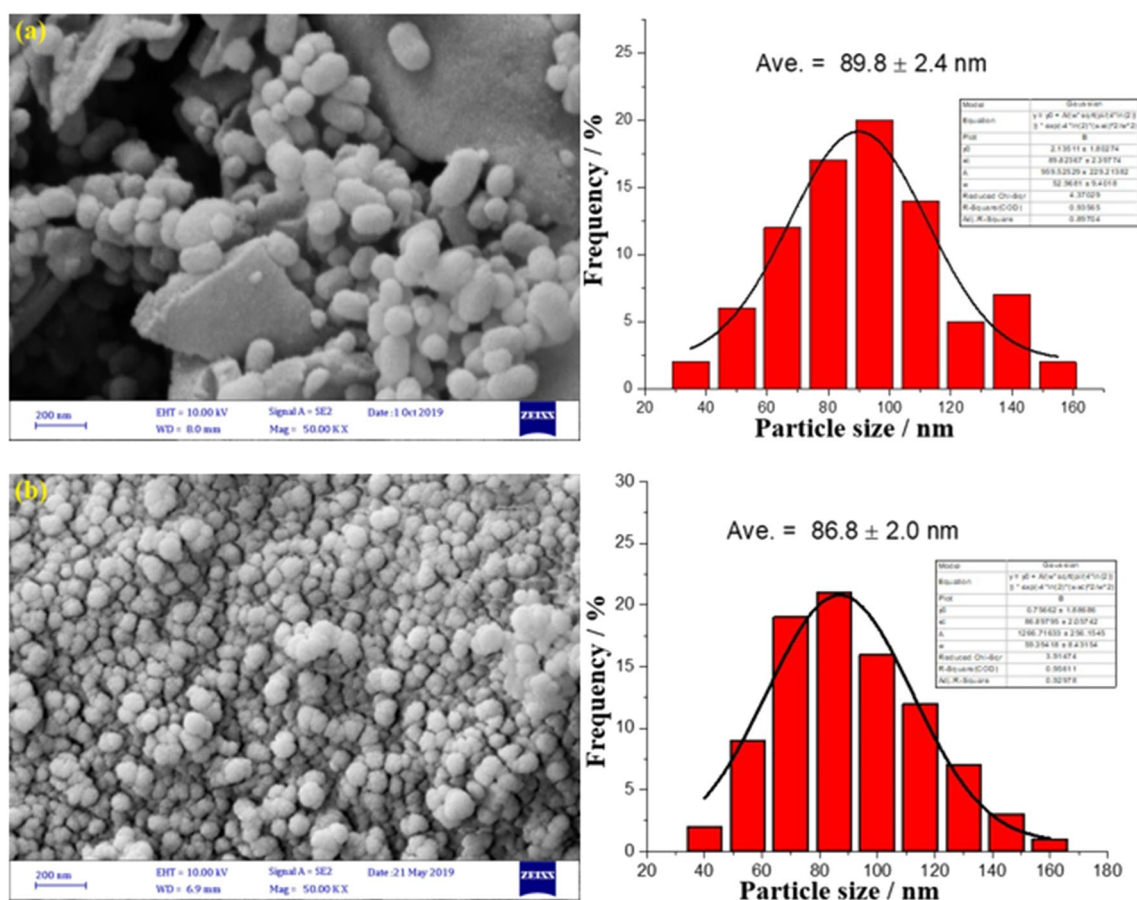
Fig. 2 SEM micrographs and size distribution histograms of **a** MCM-41 and **b** Cu(II)-dimethylglyoxime-MCM-41
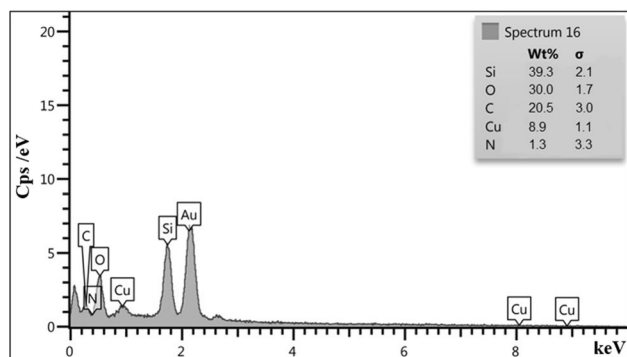

Fig. 3 The EDX spectrum of Cu(II)-dimethylglyoxime/MCM-41

heterogeneous nanocatalyst in preparation of 2,3-dihydro-quinazolin-4(1*H*)-one derivative (Scheme 2). In this regard, the one-pot reaction of benzaldehyde and 2-aminobenza-mide was performed to find the optimal conditions. The optimized conditions were shown in Table 1 for preparing the quinazoline in the presence of different catalytic quantities of Cu(II)-dimethylglyoxime/MCM-41.

The various parameters such as the reaction temperature, the amount of catalyst, time, and various solvents were studied, and the results were summarized in Table 1. The best results were obtained with 1.0 mmol benzaldehyde and 1.0 mmol 2-aminobenzamide in the presence of 5 mg Cu(II) dimethylglyoxime/MCM-41 and 5 cm^3 ethanol as solvent at 80 ℃ (Table 1, entry 12). Furthermore, with the utilization of further quantities of 8 mg catalyst and fewer quantities of 4 mg catalyst, the favored results and desired yields did not obtain (Table 1, entries 13 and 2, respectively).

After optimization experiments, the optimized conditions (ethanol solvent, 5 mg catalyst, and reflux conditions) were applied for the preparation of 2,3-dihydroquinazolin-4(1*H*)-ones (Table 2). The reaction of 2-aminobenzamide (**1**) with various aromatic aldehydes derivatives was carried out. It can be observed that a broad range of aldehyde derivatives **2** react with 2-aminobenzamide readily and give compounds **3** in suitable yields. It is also notable that only small condensation rate differences occur between aldehydes substituted with electron-donating and electron-attracting groups.

The proposed mechanism for the preparation of quinazolines derivatives was presented in Scheme 3. According
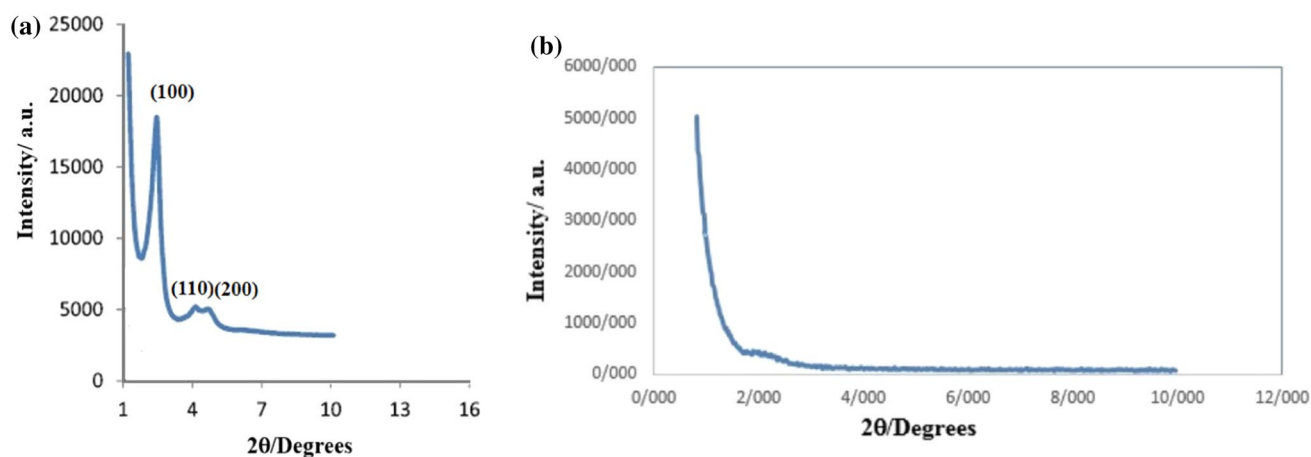
Fig. 4 XRD patterns of MCM-41 **a** and Cu(II)-dimethylglyoxime/MCM-41 (**b**)
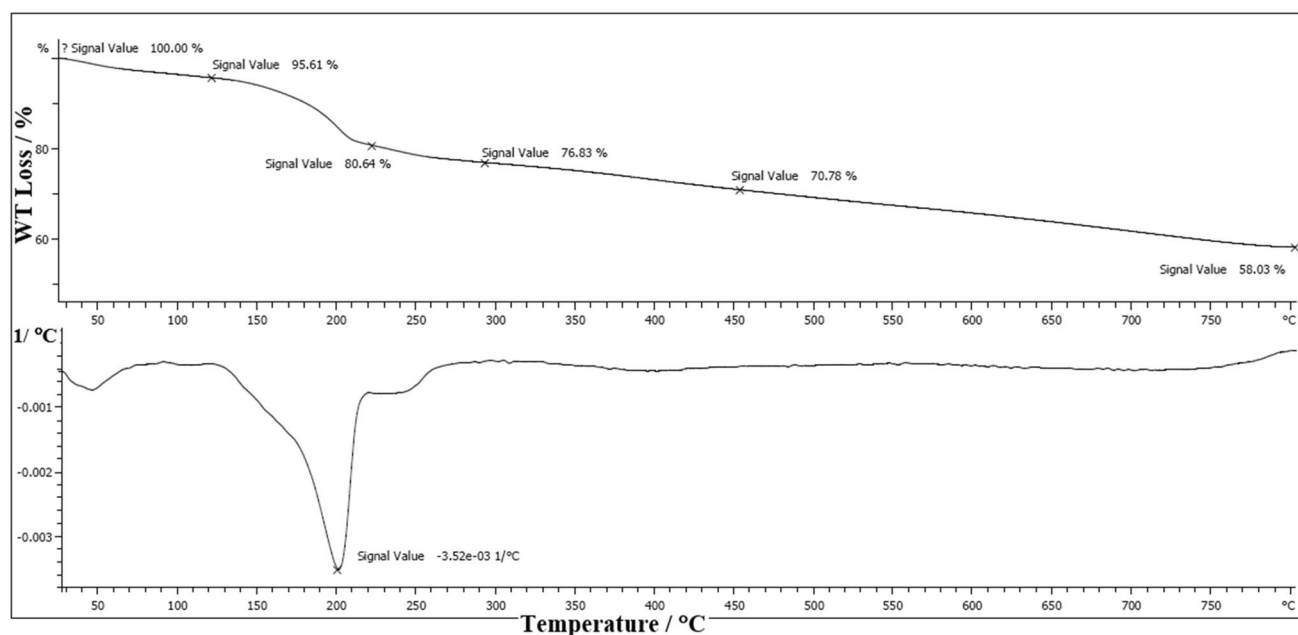

Fig. 5 TGA thermogram of Cu(II)-dimethylglyoxime/MCM-41 (top) and TGA/DTA analysis of Cu(II)-dimethylglyoxime/MCM-41 (bottom)

to this mechanism, Cu(II)-dimethylglyoxime/MCM-41 is an active catalyst for the production of 2,3-dihydroquinazoline-4(1*H*) ones. First, the proposed catalyst coordinated to aldehyde oxygen to produce intermediate **a**, then imine **b** generated by nucleophilic attack of primary amine to intermediate **a** with loss of water. Afterward, under an intramolecular cyclization by the attack of amide nitrogen on the activated imine carbon, the desired 2,3-dihydroquinazoline-4(1*H*) one **3** is formed. It is noteworthy that the mechanism for the reaction has not been studied. We suggest the following possibility. We have not determined whether or not the catalyst functions in both steps as shown.

Further, the efficiency of the prepared catalyst was compared with other nanoparticle-based metal catalysts for the synthesis of the model product. To have a more precise comparison, the reaction condition, time, temperature, and solvent for each protocol were considered in Table 3. As obvious, our procedure is comparable with those of previously reported catalytic systems in terms of yields of the products, reaction temperature, and time. In addition to this, the present catalyst is more effective than the others due to some important benefits such as its eco-friendly, stability, recyclability, etc.
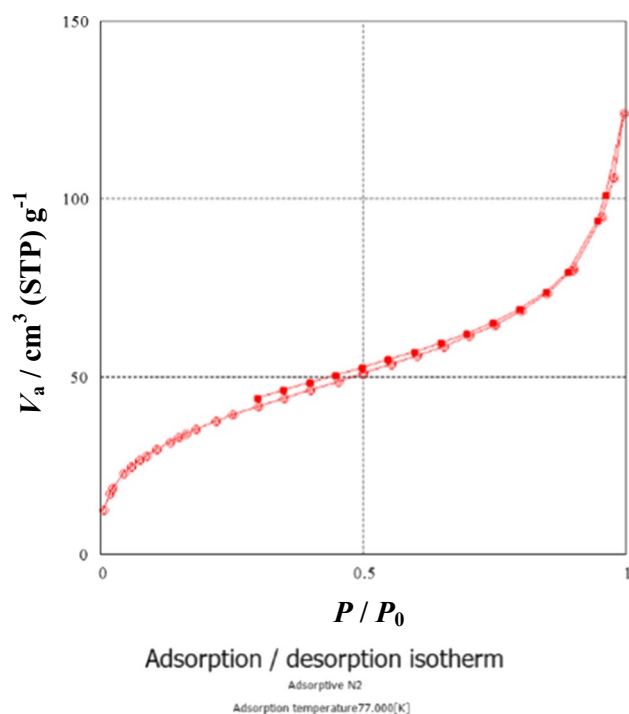
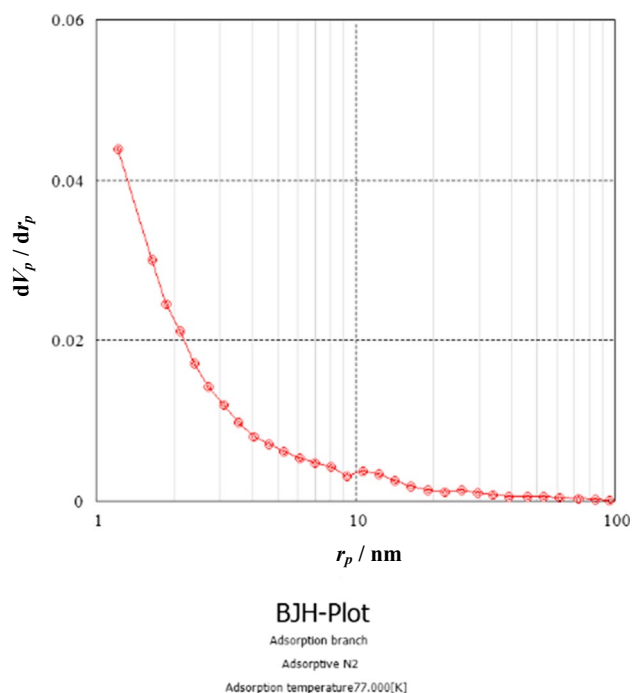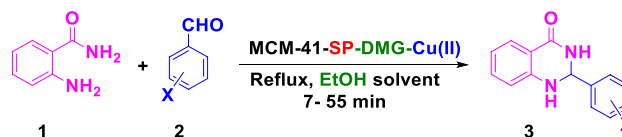**Fig. 6** Nitrogen adsorption–desorption isotherm of Cu(II)-dimethylglyoxime/MCM-41

Fig. 7 Pore size distribution curves of Cu(II)-dimethylglyoxime/MCM-41

Scheme 2

Recyclability of the catalyst

Finally, the stability and reusability of the catalyst were significant parameters in the potential application of organic transformation. To survey this trait, the reusability of the Cu(II)-DMG@MCM-41 was examined in the reaction of aromatic aldehydes with 2-aminobenzamide as a model reaction according to optimal conditions (Fig. 8).

At the end of each reaction cycle, the catalyst was separated using centrifugation and dried after washing with distilled water and ethanol. Then the catalyst is utilized for the next run. The results in Fig. 8 indicate that the proposed catalyst is recycled up to six successive runs. Furthermore, after six runs, the nature of the recycled nanocomposite was examined by SEM (Fig. 9). The conclusions of the analyses showed that the recycled nanocomposite after six periods had no significant decrease in catalytic activity that the structure of the mesoporous silica did not change significantly.

Conclusion

In summary, a new complex of Cu–dimethylglyoxime anchored to MCM-41 nanostructure was synthesized using the sol–gel method. It developed as an innovative promise catalyst and effectively green recyclable catalytic system in the synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones. Advantages of the catalytic system are simple preparation, high reaction speed, green conditions, simple workup, excellent yields of products, excellent stability, and reusability of the catalyst.

Experimental

The used solvents and chemicals were obtained from Fluka, Aldrich, and Merck and used without further purification. The particle morphology and size of nanoparticles were checked using FESEM (Day Petronic Company, Tehran, Iran) with a TESCAN MIRA3, also EDS analysis was accomplished by a TESCAN MIRA3 (Day Petronic Company, Tehran, Iran). The FT-IR spectra were taken by KBr pellets utilizing a Nexus 670 FT-IR spectrometer (Urmia University, Urmia, Iran). Thin-layer chromatography was used for monitoring reaction over silica gel 60 F_{254} aluminum sheet. ^{1}H NMR spectrum (300 MHz) and ^{13}C NMR spectrum (75 MHz) were recorded by a Bruker NMR

Table 1 Optimization of reaction parameters for the synthesis of 2-phenyl-2,3-dihydroquinazolin-4(1H)-one (**3a**)
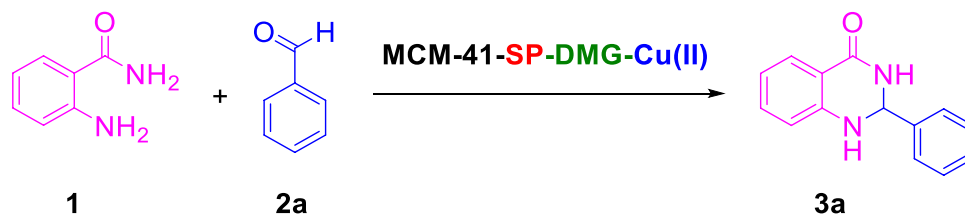
Entry	Cat./mg	Solvent	Temp /°C	Time/min	Yield/%[a, b]
1	0	EtOH	Reflux	60	Trace
2	4	EtOH	Reflux	60	90
3	5	Solvent free	100	60	No reaction
4	5	THF	Reflux	50	60
5	5	CH_2Cl_2	rt	50	55
6	5	CH_2Cl_2	Reflux	50	75
7	5	CH_3CN	Reflux	40	80
8	5	EtOAC	Reflux	45	78
9	5	PEG	100	50	45
10	5	EtOH	rt	50	80
11	3	EtOH	Reflux	40	92
12	**5**	**EtOH**	**Reflux**	**25**	**97**
13	8	EtOH	Reflux	25	97
14	5	H_2O	Reflux	60	68

[a]Reaction conditions: 2-aminobenzamide (1 mmol), aldehyde (1 mmol), 5 mg catalyst

[b]Isolated yields

spectrometer (Urmia University, Urmia, Iran). The crystalline structures were checked by powder XRD measurements with a wavelength of 1.54 Å and several templates of diffraction were entered in the 2θ confine (1–80°) (Day Petronic Company, Tehran, Iran). Melting points were recorded using an IA9200 melting point device. Curves of TGA were collected using an STA 1500 appliance from Scientific American Rheometric (Day Petronic Company, Tehran, Iran).

Synthesis of siliceous MCM-41

Firstly, 1.0 g surfactant cetyltrimethylammonium bromide (CTAB, 2.74 mmol) was mixed with a solution containing 3.5 cm³ NaOH (2 M) and 480 cm³ deionized water under stirred at 80 °C. After homogenizing the mixture, 5 cm³ tetraethyl orthosilicate (TEOS) was slowly added dropwise into the solution, it gives a mixture of white suspension. The mixture of reaction was refluxed for 2 h at 80 °C. The product was filtered and dried at 70 °C after being washed with deionized water, then it was calcined at 550 °C for 5 h to eliminate the surfactants of remain. Eventually, Si-MCM-41 was prepared.

Synthesis of MCM-41-(CH₂)₃Cl

The prepared MCM-41 nanoparticles (4.8 g) with 5.0 g 3-chloropropyltrimethoxysilane (CPTMS) was mixed with 96 cm³ of *n*-hexane and the reaction mixture was stirred under N₂ atmosphere and reflux conditions for 24 h. The outcome sediments were gathered by filtration, after washing with ethanol. The resulting MCM-41-(CH₂)₃-Cl was dried under vacuum.

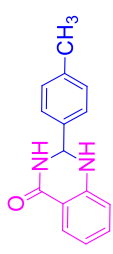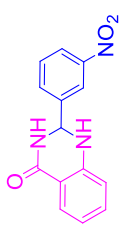### Synthesis of MCM-41-(CH₂)₃Cl functionalized with dimethylglyoxime (MCM-41-CPTMS-DMG)
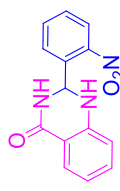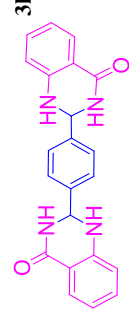
In a 50 cm³ round-bottomed flask equipped with a magnetic stirrer, 0.5 g dimethylglyoxime (DMG), 0.5 g MCM-41-(CH₂)₃Cl, 1.0 cm³ triethylamine (TEA), and 25 cm³ ethanol were mixed and refluxed for 24 h under N₂ atmosphere. After washing with ethanol, it was dried under vacuum at 60 °C for 12 h.

Table 2 MCM-41-DMG-Cu(II) catalyzed the one-pot synthesis of 2,3-dihydroquinazolin-4(1H) ones in EtOH
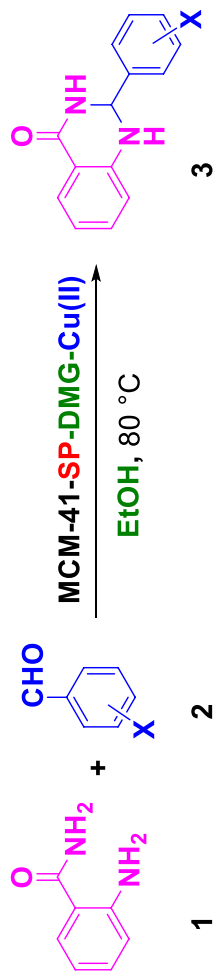
Entry	Product	Compound Numbers	Time/min	Yield/%[a,b]	M.p./°C [Ref]
1		3a	25	98	224–225 [32]
2		3b	15	95	198–200 [32]
3		3c	15	94	183–185 [34]
4		3d	10	92	202–204 [45]
5		3e	25	89	227–230 [46]
6		3f	45	85	190–193 [32]
7		3g	55	80	167–169 [34]
8		3h	30	91	210–213 [34]

Table 2 (continued)

Entry	Product	Compound Numbers	Time/min	Yield/%[a,b]	M.p./°C [Ref]
9		**3i**	35	96	225–227 [32]
10		**3j**	7	93	180–183 [46]
11		**3k**	15	90	190–192 [46]
12		**3l**	50	95	244–245 [34]

[a] All the products were identified and characterized by comparison of their physical and spectral data with those of authentic samples
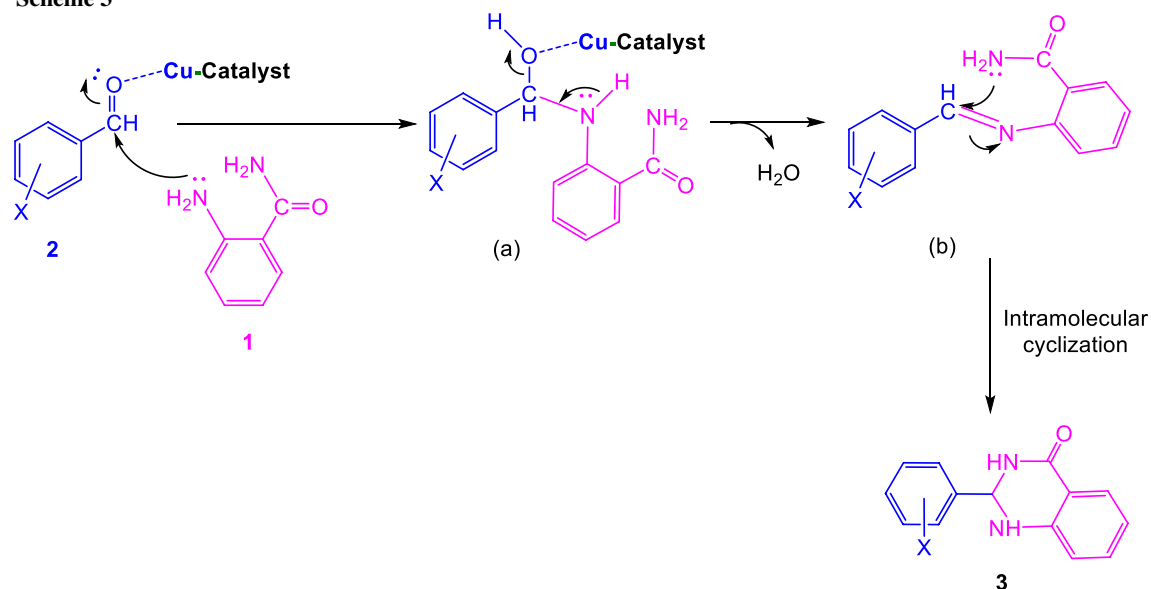
[b] Isolated yield

Scheme 3

Table 3 Comparison of the activity of diverse catalysts in the paper to preparation of 2-phenyl-2,3-dihydroquinazolin-4(1H)-one

Entry	Catalyst	Solvent	Temp/°C	Time/min	Yield /%	Lit
1	Cu(II)-DMG@MCM-41	EtOH	80	25	98	This work
2	Ga(OTf)$_3$	EtOH	70	55	87	[35]
3	Sc(OTf)$_3$	EtOH	70	25	92	[47]
4	Cyanuric chloride	CH$_3$CN	rt	10	96	[48]
5	TFE	TFE	78	90	90	[49]
6	PPA-SiO$_2$	Solvent free	70	90	91	[50]
7	Citric acid	Solvent free	rt	10	83	[51]
8	TABA	Solvent free	100	90	82	[52]
9	Fe$_3$O$_4$@MCM-41@EDTA@Ni	EtOH	78	25	92	[53]
10	NH$_2$SO$_3$H	H$_2$O	70	30	92	[54]
11	MCM41-Biurea-Ni	PEG	rt	30	90	[15]
12	CuCl$_2$/Fe$_3$O$_4$⁻TEDETA	EtOH	Reflux	30	97	[34]
13	Br$_3$-TBA-Fe$_3$O$_4$	H$_2$O	70	40	93	[55]

Synthesis of MCM-41-CPTMS-DMG-Cu

Finally, a mixture of 0.8 g MCM-41-CPTMS-DMG, 0.4 g CuCl$_2$·2H$_2$O, and 20 cm^3 ethanol was refluxed for 24 h under N$_2$ atmosphere. The purified resulting precipitate was washed with EtOH and dried at 60 °C for 12 h to product MCM-41-CPTMS-DMG-Cu.

General synthesis for the preparation of 2,3-dihydroquinazolin-4(1H)-ones

A mixture of 0.136 g 2-aminobenzamide (1 mmol), aldehyde (1 mmol), and 5 mg MCM-41-CPTMS-DMG-Cu was stirred in 5 cm^3 ethanol and heated at 80 °C. The reaction
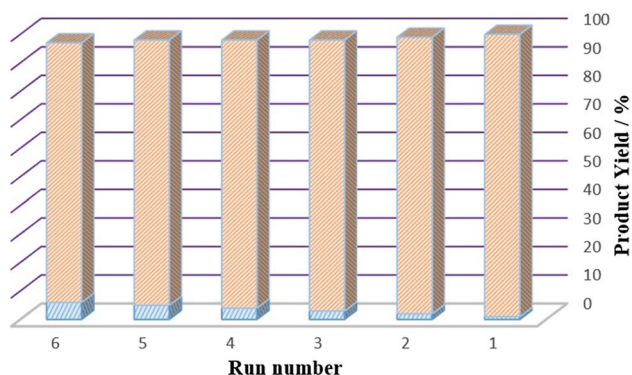
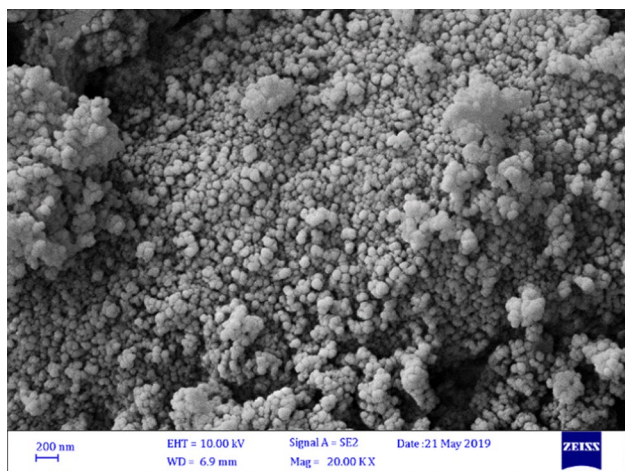**Fig. 8** Recycling of Cu(II)-DMG/MCM-in the preparation of 2-phenyl-2,3-dihydroquinazolin-4(1*H*)-one

Fig. 9 SEM image of recovered Cu(II)-DMG/MCM-41

progression was controlled using TLC (acetone:*n*-hexane, 2:8, V/V). After the completion of reaction, the mixture of reaction was cooled until room temperature, then nanocatalyst was filtered by centrifuge and reused for the subsequent reactions. The filtered solution was evaporated to remove the solvent, then obtained product was washed with ethanol, 80–98% yields.

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