Received: 19 February 2021 | Revised: 26 May 2021 | Accepted: 9 June 2021

DOI: 10.1002/aoc.6346

FULL PAPER

Applied Organometallic Chemistry WILEY

Sustainable design and novel synthesis of highly recyclable magnetic carbon containing aromatic sulfonic acid: Fe_3O_4@C/Ph—SO_3H as green solid acid promoted regioselective synthesis of tetrazoloquinazolines

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Abstract

A green and stable magnetic Fe_3O_4 core encapsulated in porous carbon shell was prepared. Then, the surface of as-synthesized Fe_3O_4@meso-C immobilized with activated 4-aminobenzenesulfonic acid to achieve Fe_3O_4@C/Ph—SO_3H. Structural, physico-chemical, and magnetic properties of synthesized Fe_3O_4@C/Ph—SO_3H catalyst using various analyses such as FT-IR spectroscopy, TGA, XRD, SEM, TEM. EDX, and VSM were investigated. Fe_3O_4@C/Ph—SO_3H was used as a stable and recoverable acid catalyst for regioselective three-component synthesis of tetrazoloquinazolines under mild conditions. This catalyst offers a green protocol of one pot due to having active SO_3H groups, reacting in mild conditions with high yield and ability to recover and reuse without significant loss in activity.

KEYWORDS

Fe_3O_4@C/Ph—SO_3H, magnetic nanoparticle, mesoporous carbon, tetrazoloquinazolines, three-component reaction

Funding information
Iran National foundation of Science (INSF)

1 | INTRODUCTION

Multi-component reactions (MCRs) have emerged because of their valuable features such as atom economy, straightway reaction design, and the opportunity to manufacture target compounds by introducing several variation elements in a single chemical event as efficient and powerful tools in modern synthetic organic chemistry.[1–3] Typically, purification of products derived from MCRs is also simple, since all the organic reagents used are consumed and combined into the target compound.[2,3] On the other hand, a MCR domino and solid catalysts strategies are a powerful tool in which reactants simultaneously lead to the rapid synthesis of various complex organic molecular structures which prevented the need for separation and purification of intermediates, resulting in decrease of waste and reaction time and as a result improves the overall performance.[4] Tetrazolopyrimidines cover activities with a wide range of purposes because they are a common structural motif in pharmacologically important molecule.[5,6] The use of these compounds have been reported in the treatment of obesity, atherosclerosis, diabetes, hypertension, hypercholesterolemia, coronary heart disease, hyperlipidemia, depression, hypothyroidism, cardiac arrhythmias, thyroid cancer, glaucoma, and congestive heart failure.[7–9] Amino tetrazole is also known as part of certain drugs such as cefazolin, Corazol, and Cefoperazone.[6]

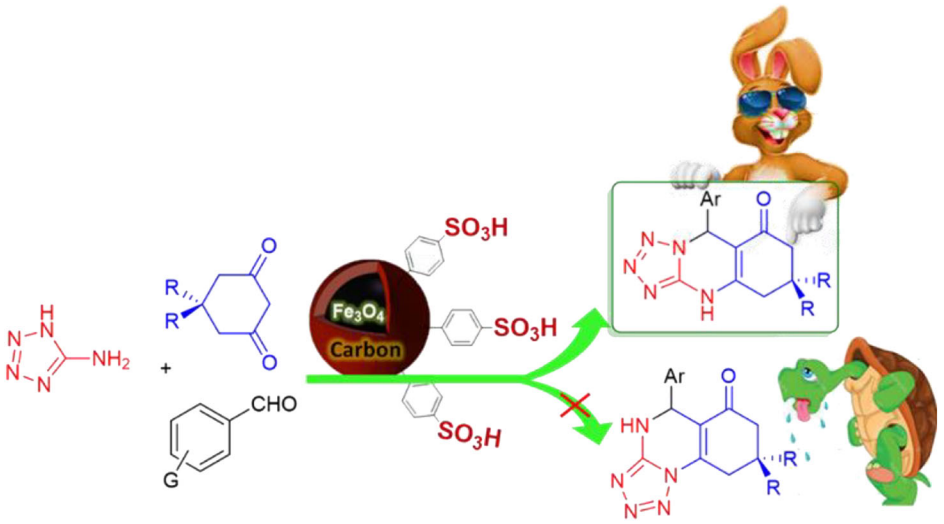
In recent years, due to waste minimization and atom economy in the use of raw materials, traditional homogeneous acid catalysts have been replaced by environmentally friendly, sustainable sources, and reusable catalysts.[10] Solid acids can be attributed to an easy separation of catalyst from the liquid phase of reaction mixture with minimum corrosion, easy reaction handling, and good recycling for catalyst.[11,12] On the other hand, green chemistry has led to increased research toward synthesis of sustainable and green heterogeneous catalysts with easy separation, especially magnetic nanoparticles.[13]

In this work, we designed and synthesized a magnetic porous carbon (MPC) containing sulfonic acid (MPC/SO_3H or Fe_3O_4@C/Ph—SO_3H) as a high-active, environmentally sustainable, and recyclable catalyst by immobilization of 4-aminobenzenesulfonic acid onto the surface of Fe_3O_4@C for the synthesis of tetrazoloquinazolines (Scheme 1).

2 | EXPERIMENTAL

2.1 | Materials and apparatus

4-aminobenzenesulfonic acid was obtained from Merck. The other chemicals were also purchased from Sigma-Aldrich, Merck (Germany) and Fluka (Switzerland) and were used without further purification. Infrared Raman (IR) spectra were obtained by a Shimadzu IR-460 spectrometer. ^1H and ^{13}C NMR spectra were measured on a Bruker DRX-500 AVANCE spectrometer at 500.13 and 125.7 MHz, respectively. Scanning Electron Microscopy (SEM) images were recorded by a Zeiss-DSM 960A



SCHEME 1 Domino 3-CRs synthesis of tetrazoloquinazolines using Fe_3O_4@C/Ph—SO_3H

microscope. Transition Electron Microscopy (TEM) images were recorded on a Zeiss EM 900 TEM. The crystalline structure of the nanoparticles was examined by X-Ray Diffraction (XRD) instrument (Philips-PW1800 diffractometer).

2.2 | Synthesis of Fe_3O_4 nanoparticles

Fe_3O_4 nanoparticles were synthesized according to our previous method.[14] Initially, after dissolving $FeCl_3$ (0.65 g, 4.0 mmol), sodium acetate (1.20 g), and trisodium citrate (0.20 g, 0.68 mmol) in 20 ml pure ethylene glycol, the mixture was stirred vigorously for 45 min under Ar atmosphere (small glove box-absence of air). The resulting homogenate mixture was transferred to an autoclave of Teflon stainless steel (100 ml capacity), sealed, and heated for 12 h at 200°C (degree rate: 2°C/min under Ar electrical Furnace). After cooling autoclave at room temperature (degree rate: 2°C/min), the resulting black precipitate was separated magnetically. The obtained Fe_3O_4 nanoparticles were washed several times with deionized water and ethanol and then dried at 60°C under vacuum for 12 h.

2.3 | Synthesis of Fe_3O_4@C

The Fe_3O_4@C core-shell were prepared by our previous method.[14,15] Briefly, 0.1 g of Fe_3O_4 as synthesized was dispersed in 25 ml distilled water containing 2.0 g glucose under ultrasonication for 30 min. The resulting mixture was transferred into autoclave of 50 ml stainless steel and heated at 200°C for 12 h with a 2°C/min (note: temperature rating is important for carbonization of glucose in exact morphology; sonication of Fe_3O_4 is important for core-shell achievement). After cooling to room temperature, the black solid precipitate was collected from the solution by an external magnet and washed several times with distilled water and ethanol. Finally, the black product was dried under vacuum at 60°C for 12 h.

2.4 | Synthesis of Fe_3O_4@C/Ph—SO_3H

For the synthesis of Fe_3O_4@C/Ph—SO_3H, the synthesized Fe_3O_4@C was grafted with sulfophenyl group by reduction of diazonium salt. Initially, sulfophenyl diazonium salt was synthesized from mixing compounds containing aminobenzenesulfonic acid (0.5 mmol), $NaNO_2$ (0.5 mmol), and 1 M HCl (1 ml) in a cooling bath of 3°C. The obtained diazonium salt was slowly poured into the toluene-dispersed Fe_3O_4@C. After stirrer the mixture for 30 min at room temperature, the reaction mixture was refluxed (open vessel reflux system) overnight to graft sulfonyl group onto porous carbon surface of Fe_3O_4@C. Finally, the Fe_3O_4@C/Ph—SO_3H was separated from the mixture by an external magnet, and it was washed with ethanol and then dried under vacuum at 60°C for 6 h. CHNS analysis indicates the presence of sulfur in 3.96% in Fe_3O_4@C/Ph—SO_3H.

2.5 | Synthesis of tetrazoloquinazolines

A mixture of benzaldehyde (1 mmol), 2-aminotetrazol (1 mmol), and dimedone (1 mmol) was added to 3 ml ethanol in the presence of 0.1 g of the Fe_3O_4@C/Ph—SO_3H catalyst and stirred at 80°C for 1 h. After completion of the reaction for precipitate the synthesized product, the reaction temperature was downed to 25°C. Fe_3O_4@C/Ph—SO_3H separation using an external magnet and then the obtained precipitate was washed with water and dried. Finally, to achieve the final product, the crude product was recrystallized with ethanol. All compounds (supporting information) are known and are previously reported in the literatures.[6,16,17]

3 | RESULTS AND DISCUSSION

In line with our continuing efforts to synthesize solid heterogeneous catalysts and organic compounds,[18–22] herein, we describe the regioselective 3-CRs synthesis of tetrazoloquinazolines via the condensation reaction of aldehydes, 5-aminotetrazole, and dimedone or 1.3-cyclohexanedione in the presence of a novel magnetic mesoporous carbon bonded aromatic sulfonic acid of Fe_3O_4@C/Ph—SO_3H (Figure 1).

FTIR spectra were used to detect the changes after encapsulation and immobilization of the —SO_3H groups onto surface of Fe_3O_4@C. The strong peak at 579 cm^{-1} is characteristic of Fe—O vibrations, and the peak at 1,581 cm^{-1} is related to C—C bond vibrations. The peak at 3,449 cm^{-1} indicates the residual O—H groups (Figure 2a).[23–25] After sulfonation, the vibration bands at 1,068 cm^{-1}1 (S═O symmetric stretching), 1,136 cm^{-1} (S═O asymmetric stretching), and 1,413 cm^{-1} (asymmetric O═S═O stretching) were related to the —SO_3H groups[26] (Figure 2a). The components of Fe_3O_4@C/Ph—SO_3H synthesized were analyzed by the SEM electron dispersive spectroscopy (SEM-EDS), also which results are indicated in Figure 2b,d. The characteristic lines of C, N, O, Fe, and S elements indicate that the Fe_3O_4 nanoparticles are coated by the carbon layer and

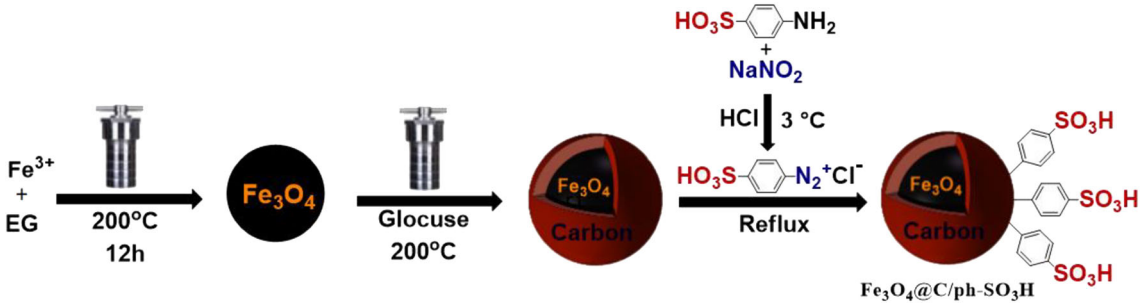


FIGURE 1 Synthetic pathway of sulfonated Fe_3O_4@C/Ph—SO_3H

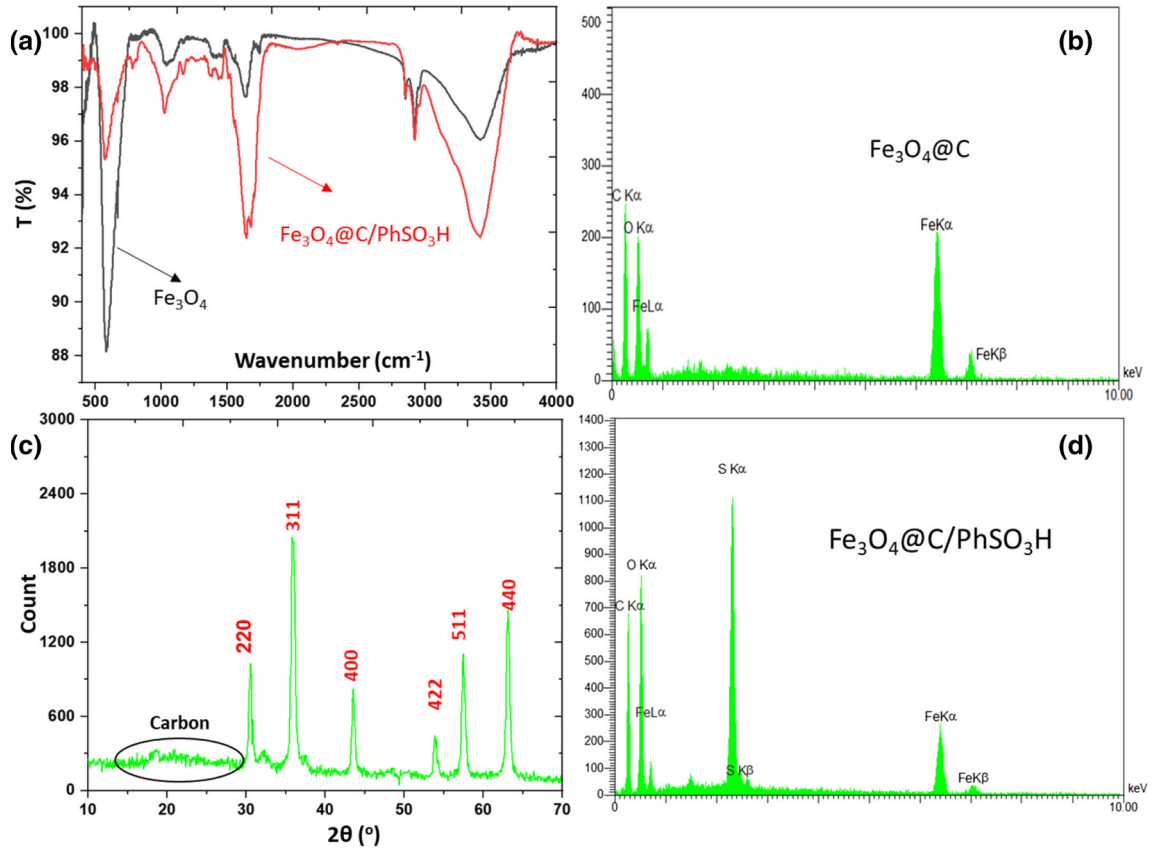


FIGURE 2 (a) FTIR spectra of Fe_3O_4, Fe_3O_4@C/Ph—SO_3H, (b) SEM-EDS analysis of Fe_3O_4@C, (c) XRD patterns of Fe_3O_4@C/Ph—SO_3H, and (d) SEM-EDS analysis of Fe_3O_4@C/Ph—SO_3H

successfully sulfonated with aromatic —SO_3H group. The result of this analysis could be another confirmation of the catalyst's successful synthesis. The XRD patterns of the as-synthesized Fe_3O_4@C/Ph—SO_3H are shown in (Figure 2c). Six characteristic peaks matching with the (220), (311), (400), (422), (511), and (440) were observed for all three samples corresponding to cubic Fe_3O_4 phase (JCPDSICDD copyright 1938, file number 01-1111) with the Fd-3 m Space Group.[27] As can be seen, the relative position and intensity of all peaks are in accordance with the standard Fe_3O_4 pattern, indicating that although the peak intensities decreased slightly during carbon encapsulation and subsequent functionalization of MNPs but the crystal structure did not change.

FE-SEM and TEM images were used to investigate the size and surface morphology of the Fe_3O_4@C and aromatic sulfonic acid functionalized magnetic carbon of Fe_3O_4@C/Ph—SO_3H as shown in Figure 3. Figure 3a,b shows FE-SEM images of Fe_3O_4@C after encapsulation of magnetic into the carbon, which show that the morphology of the nanoparticles is spherical and that the size of magnetic nanoparticles is about 150–200 nm. In the TEM images (Figures 3c,d), the core and shell structure of Fe_3O_4@C are clearly defined, indicating that the Fe_3O_4

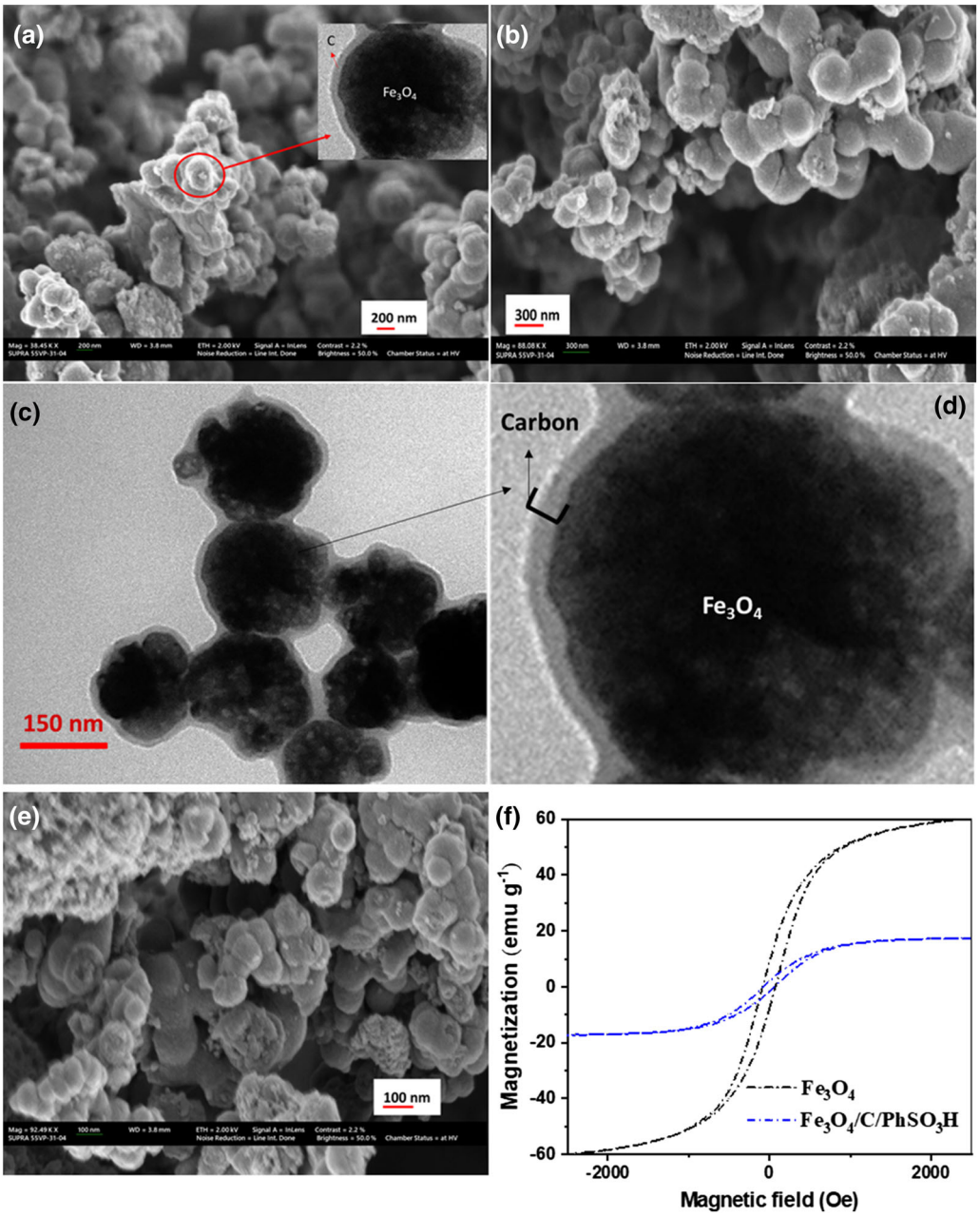
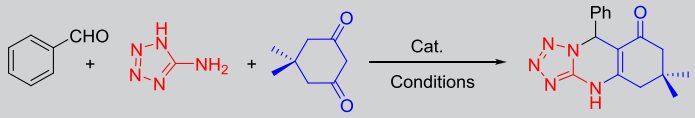


FIGURE 3 (a, b) FE-SEM image of Fe_3O_4@C; (c) TEM image of Fe_3O_4@C and its magnified TEM (d); (e) FE-SEM image of Fe_3O_4@C/Ph—SO_3H; and (f) comparative VSM analysis of Fe_3O_4@C and Fe_3O_4@C/Ph—SO_3H

nanoparticles are encapsulated by the carbon layer. The average size of Fe_3O_4 nanoparticles is about 140–200 nm, and the carbon shell thickness is about 14–15 nm, which is in full agreement with SEM images. Figure 3e shows FE-SEM images of Fe_3O_4@C/Ph—SO_3H after functionalization of the magnetic carbon by aromatic sulfonic acid group, which show that the morphology of the nanoparticles is yet spherical and no changed in particles size. As shown in Figure 3f, VSM analysis shows super-magnetic behavior for Fe_3O_4 and Fe_3O_4@C/Ph—SO_3H. For pure Fe_3O_4, the magnetization shifts from −60 to +60 emu/g, while after Ph—SO_3H functionalization, a great decrease in magnetization (−18 to +18 emu. g) is observed.

After using a three-step synthesis route for the preparation of Fe_3O_4@C/Ph—SO_3H, as a Bronsted sulfonic acid functionalized core-shell Fe_3O_4@C, its application to the synthesis of tetraculoquinazoline using benzaldehyde (1 mmol), 2-aminotrazole (1 mmol) in presence of the dimedone (1 mmol) was evaluated. Initially, the reaction progress was evaluated in water at 80°C in the absence of the catalyst, and after 24 h, no product was observed

TABLE 1 Reaction optimization for the synthesis of tetrazoloquinazolines[a]



Entry	Catalyst	Solvent	T (°C)	Time (h)	Yield[a] (%)
1	-	H_2O	80	24	[b]
2	$AlCl_3$ (5 mol %)	H_2O	80	4	trace
3	$FeCl_3$ (5 mol %)	H_2O	80	4	30
4	Cu_2O (5 mol %)	H_2O	80	4	30
5	Fe_3O_4@C/Ph—SO_3H (0.05 g)[c]	H_2O	80	1	70
6	**Fe_3O_4@C/Ph—SO_3H (0.1 g)[c]**	**H_2O**	**80**	**1**	**96**
7	Fe_3O_4@C/Ph—SO_3H (0.15 g)[c]	H_2O	80	1	96
8	Fe_3O_4@C/Ph—SO_3H (0.1 g)[c]	H_2O	r.t	1	35
9	Fe_3O_4@C/Ph—SO_3H (0.1 g)[c]	H_2O	60	1	75
10	Fe_3O_4@C/Ph—SO_3H (0.1 g)[c]	H_2O	100	1	97
11	Fe_3O_4@C/Ph—SO_3H (0.1 g)	EtOH	80	1	80
12	Fe_3O_4@C/Ph—SO_3H (0.1 g)	CH_3CN	80	1	65
13	Fe_3O_4@C/Ph—SO_3H (0.1 g)	DMF	80	1	70
14	Fe_3O_4@C/ph—SO_3H (0.1 g)	DMSO	80	1	50
15	Fe_3O_4@C/Ph—SO_3H (0.1 g)	Toluene	80	1	25
16	Fe_3O_4@C/Ph—SO_3H (0.1 g)	Solvent-free	130	1	95

[a]Isolated yield.
[b]No product detected.
[c]Reaction conditions: benzaldehyde (1 mmol), 2-aminotetrazole (1 mmol), and dimedone (1 mmol) in 3 ml of solvents.

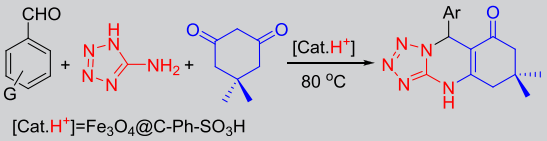
(Table 1, entry 1). Subsequently, various amounts of catalyst (0.05, 0.1, and 0.15) were chosen for the reaction, which the highest yield (96%) observed for both the 0.01 and 0.15 catalyst amounts. Therefore, 0.1 g was selected as the optimum amount of catalysis (Table 1, entries 5–7). In the following, to further optimize the reaction conditions, in addition to the 80°C, other temperatures such as room temperature, 60°C and 100°C, were also studied. It was found that the same yield was observed for 80°C and 100°C and, therefore, the 80°C was selected as the optimum temperature (Table 1, entries 6–10). In addition, the effect of different solvents was investigated, and it was found that water was more effective solvent (Table 1, entries 6, 11–15). On the other hand, reaction under solvent-free conditions requires a temperature of 130°C (Table 1, entry 16).

Based on the results of Table 1, the scope and generality of this protocol were investigated for various aromatic aldehydes in presence of the dimedone and 2-aminotetrazole in optimized conditions. According to the optimum conditions in Table 1, the use of aromatic aldehydes including electron-deficient groups such as 4-NO_2, 4-Cl, 4-Br, and 2,6-Cl_2 (Table 2, entries 2–8) and electron-rich such as 4-Me and 2,5-OMe (Table 2, entries 9 and 10) leads to synthesis of tetrazoloquinazolines at high yields, which indicates that Fe_3O_4@C/Ph—SO_3H is an effective catalyst for the synthesis of different tetraclucinazoline derivatives.

In the following, the use of 1.3-cyclohexanedione was also investigated to further increase the synthesis of tetraclucinazoline. Similarly, acceptable results with high yield were observed in line with the results from Table 2 for both the electron-deficient (Table 3, entries 2–8) and electron-rich (Table 3, entries 9 and 10) aromatic ring groups (Table 3).

The proposed mechanism for Fe_3O_4@C/Ph—SO_3H is illustrated in Scheme 2. This catalyst with having —SO_3H groups can act as acidic species (H^+). Initially, the aldehyde was protonated in the presence of Fe_3O_4@C/Ph—SO_3H, and in the next step, Knoevenagel

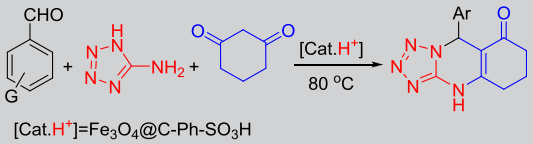
TABLE 2 Three-component coupling synthesis of tetrazoloquinazolines



Entry	Ar	β-diketone	Time (h)	Yield (%)
1	Ph	Dimedone	1	96
2	2-Cl—C_6H_4	Dimedone	1	90
3	4-Cl—C_6H_4	Dimedone	1	95
4	2,4-Cl_2—C_6H_4	Dimedone	1	95
5	4-Br—C_6H_3	Dimedone	1	90
6	4-O_2N—C_6H_4	Dimedone	1	95
7	2-O_2N—C_6H_4	Dimedone	1	90
8	3-O_2N—C_6H_4	Dimedone	1	95
9	4-CH_3—C_6H_4	Dimedone	1	95
10	4-OCH_3—C_6H_4	Dimedone	1	92

Note: Reaction condition: aldehyde (1 mmol), 2-aminotetrazole (1 mmol), dimedone (1 mmol), and 0.1 g of Fe_3O_4@C/Ph—SO_3H in 3 ml H_2O at 80°C.

TABLE 3 Fe_3O_4@C/Ph—SO_3H catalyzed the MC-synthesis of tricyclic tetrazoloquinazolines

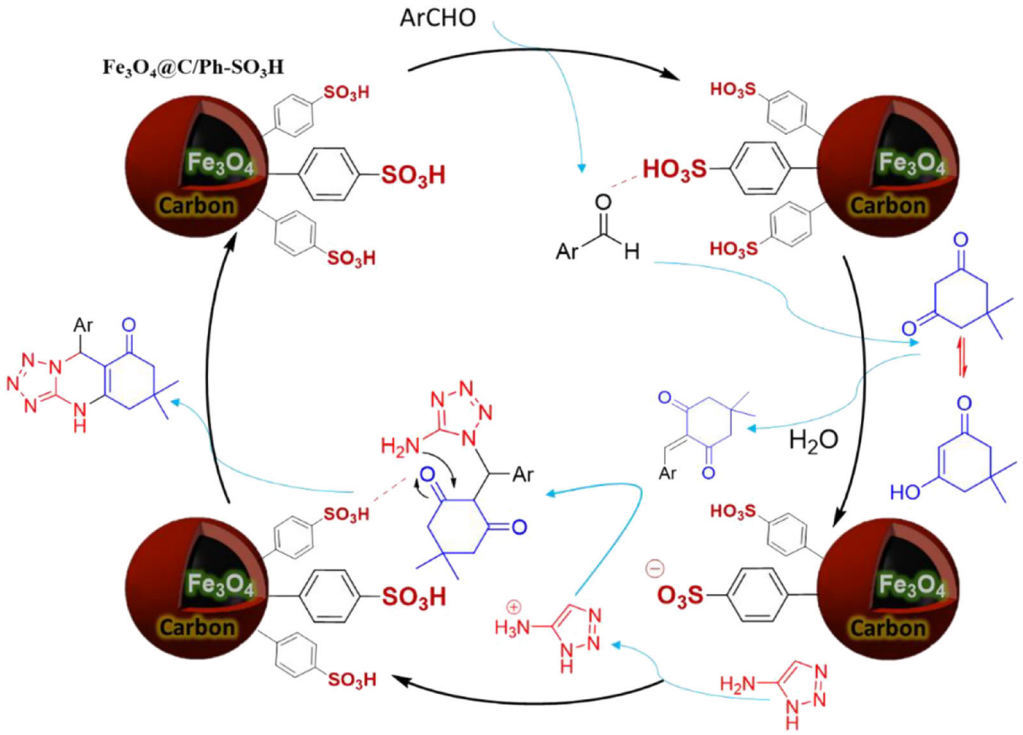


Entry	Ar	Ketone	Time (h)	Yield (%)
1	Ph	1,3-Cyclohexanedione	1	96
2	4-Br—C_6H_4	1,3-Cyclohexanedione	1	95
3	2-Cl—C_6H_4	1,3-Cyclohexanedione	1	88
4	4-Cl—C_6H_4	1,3-Cyclohexanedione	1	92
5	2,4-Cl_2—C_6H_3	1,3-Cyclohexanedione	1	92
6	3-O_2N—C_6H_4	1,3-Cyclohexanedione	1	95
7	4-O_2N—C_6H_4	1,3-Cyclohexanedione	1	95
8	2-O_2N—C_6H_4	1,3-Cyclohexanedione	1	90
9	4-OCH_3—C_6H_4	1,3-Cyclohexanedione	1	88
10	4-CH_3—C_6H_4	1,3-Cyclohexanedione	1	90

Note: Reaction condition: aldehyde (1 mmol), 2-aminotetrazole (1 mmol), 1,3-cyclohexanedione (1 mmol), and Fe_3O_4@C/Ph—SO_3H in 3 mL H_2O at 80°C.

condensation of aldehyde with dimedone resulted in the formation of a benzididine compound. After the reaction of Michael and the addition of 2-aminetetrazole to the reaction cycle, subsequent cyclization leads to the formation of the desired product.[16,17,28–30]

The ability to recover and reuse the catalyst according to the optimized conditions in Table 1 was investigated for subsequent experiments up to 8 cycles (Figure 4a). As shown in Figure 4b, the catalytic power and structure of Fe_3O_4@C/Ph—SO_3H were maintained during successive



SCHEME 2 The possible mechanism for the synthesis of tetrazoloquinazolines over catalysis of Fe_3O_4@C/Ph—SO_3H

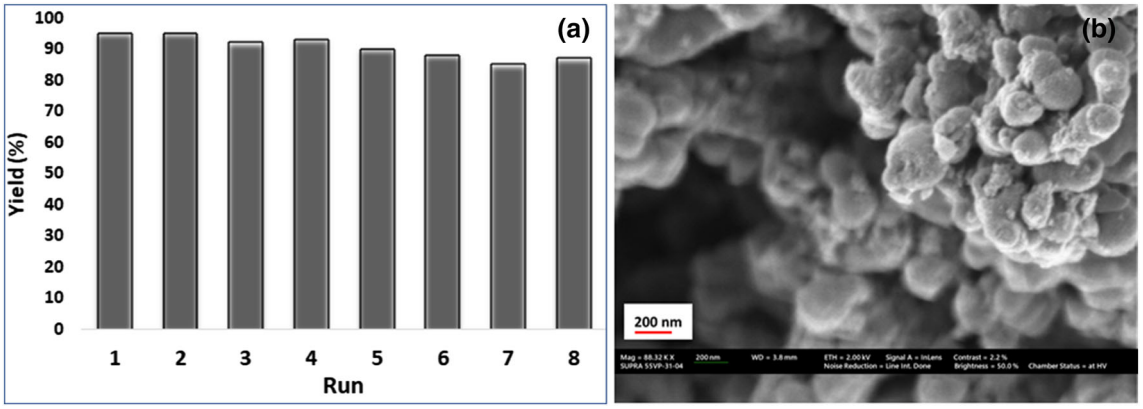


FIGURE 4 (a) Reusability of Fe_3O_4@C/Ph—SO_3H in the synthesis of tetrazoloquinazolines and (b)FE-SEM image of Fe_3O_4@C/Ph—SO_3H after 8 cycles

TABLE 4 Comparison of different methods in the synthesis of tetrazoloquinazolines

Entry	Catalyst	Conditions	Time (h)	Yield (%)/Ref
1	TsOH (10 mol%)	Solvent-free, 30 °C	8	84[28]
2	$AlCl_3$ (20 mol%)	CH_3CN, Reflux	4	92[29]
3	MNPs@SiO_2—Pr—ANDSA (0.2 g)	H_2O/EtOH (1:1)/100 °C	5 min	94[30]
4	I_2 (10 mol%)	^{i}PrOH, Reflux	20 min	83[16]
5	Fe_3O_4@C/Ph—SO_3H (0.1 g)	H_2O, 80 °C	1	96 (this work)

cycles, which SEM pattern obtained after eight cycles also confirms this.

Table 4 summarizes the results of our work compared to the previously reported catalysts in the literature. As

can be seen, the Fe_3O_4@C/Ph—SO_3H catalysts with acidic sites exhibited high catalytic activity with excellent yield (96%) in shorter time than most of the reported works.

4 | CONCLUSION

In summary, we developed the synthesis of novel Fe_3O_4@C/Ph—SO_3H heterogeneous catalyst using an environmentally friendly and atom-economical procedure with aromatic sulfonic acid-active sites as an effective and efficient catalyst for direct synthesis of tetrazoloquinazolines. Various derivatives of tetrazoloquinazolines were synthesized through our method using a one-pot three component couplings of aromatic aldehydes and dimedone and 1,3-cyclohexanedione ketones in the presence of 2-aminotetrazole. The molecular structure of synthesized tetrazoloquinazolines was identified by ^1H and ^{13}C NMR spectroscopy and other physical properies. The method was green, with high yields of products, which the catalyst system avoids the use of dangerous liquid acids.

ACKNOWLEDGMENTS
The authors are thankful for financial supports from Iran National foundation of Science (INSF).

CONFLICT OF INTEREST
The authors declare no competing interests.

AUTHOR CONTRIBUTIONS
Asadollah Hassankhani: Conceptualization; supervision. **Behnam Gholipour:** Investigation; methodology. **Sadegh Rostamnia:** Supervision.

DATA AVAILABILITY STATEMENT
The data that support the findings of this study are available in the supporting information of this article.

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How to cite this article: A. Hassankhani, B. Gholipour, S. Rostamnia, E. Zarenezhad, N. Nouruzi, T. Kavetskyy, R. Khalilov, M. Shokouhimehr, *Appl Organomet Chem* **2021**, e6346. https://doi.org/10.1002/aoc.6346