RESEARCH ARTICLE

A Simple and Convenient Synthesis of 2, 3-dihydroquinazolin-4(1H)-one Derivatives using $MgFe_2O_4@SiO_2$-SO_3H Catalyst

Anand Shankar Aswar[1,*], *Nilesh Govindrao Salunkhe*[1], *Chandrashekhar Arun Ladole*[1], *Nikita Vinod Thakare*[2], *Jagruti Manish Barabde*[1]

In the present study, use of $MgFe_2O_4@SiO_2$-SO_3H as an efficient, green, magnetically recoverable & recyclable catalyst for micro-wave assisted solvent free synthesis of 2,3-dihydroquinazolin-4(1H)-ones reaction pathway is presented. The superiorities of this method are higher conversion rate, shorter reaction time, easy isolation of product and reusability of catalyst without remarkable loss of activity. The synthesized 2,3-dihydroquinazolin-4(1H)-ones derivatives were assessed for their antimicrobial and antifungal activity; where, the most of these compounds exhibit potent antibacterial and antifungal activities against various bacteria and fungi.

Introduction

The development of green catalyst and its reactions are the necessity for sustainable and green chemistry. Previously we have developed and successfully characterized $MgFe_2O_4@SiO_2$-SO_3H as a magnetically recoverable catalyst as green catalyst and exhibited its effectiveness as catalyst in the microwave-assisted synthesis of benzoxazinones and benzothioxazinones [**1**]. Easy preparation, soft acidic nature, good stability, easy magnetic separation and reusability motivated us to explore its potential to catalyse many other useful reactions such as synthesis of 2,3-dihydroquinazolin-4(1H)-ones derivatives. This reaction is three component acid or base catalysed reactions of aldehyde, isatoic anhydride and ammonium acetate. Quinazolinone and its derivatives are often synthesized because of their widespread biological activities and pharmacological properties such as antibacterial [**2**], antitumor [**3**], anticonvulsant [**4**], antihypertensive agents [**5**] and vasodilating activities [**6**]. Considering wide-range applications of this moiety, several protocols for its synthesis have been developed along with many multicomponent approaches. Various catalysts have been reported for multicomponent synthesis of quinazolinones from isatoic anhydride, aldehydes and amines and ammonium acetate.

In the past, a number of research efforts have been made to synthesis of quinazolinone and its derivatives through one pot three component synthesis involving several catalysts such as; DBSA [**7**], Trifluoroethanol (TFE) [**8**], AcOH [**9**], Thiamine hydrochloride (VB1) [**10**], Aluminium methanesulfonate [**11**], A strong acidic cation-exchange resin [**12**], TiO_2 nanoparticles [**13**], CuO nanoparticles [**14**], Tartaric acid–sodium dodecyl sulfate (SDS) [**15**], MCM-41-SO_3H [**16**], Silica-supported Preyssler nanoparticles [**17**], Nano-In_2O_3 [**18**], Cu-CNTs [**19**], Titanium Silicon Oxide Nanopowder [**20**].Using β-cyclodextrin-SO_3H [**21**], Zirconium (IV) chloride [**22**], $FeCl_3/Al_2O_3$ [**23**] catalysts two components synthesis of 2,3-dihydroquinazolin-4(1H)-ones was carried out using 2-aminobenzamide and substituted aldehyde/ketone. However, most of the synthetic protocols reported so far have several drawbacks such as drastic reaction conditions, longer reaction times, low yields, tedious work-up procedures and the use of hazardous solvents. This not only results in a waste of energy but also causes harm to our environment. Therefore, introducing a clean, greener and eco-friendly protocol has always be a permanent attention of researchers. This reveals that the scope for better methods.

In the present investigation, we successful demonstrated application of our recently developed $MgFe_2O_4@SiO_2$-SO_3H as a magnetically recoverable catalyst a soft solid acid as an alternative, eco-friendly, economical and easy preparation of catalyst for solvent free, microwave assisted synthesis of diverse 2,3-dihydroquinazolin-4(1H)-ones derivatives.

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[1] Department of Chemistry, Sant Gadge Baba Amravati University, Amravati, 444602, India
[2] Department of Chemistry, Elphinstone College, Mumbai, M.G. Road, Mumbai, 400032, India

*Corresponding author: aswaranand@gmail.com

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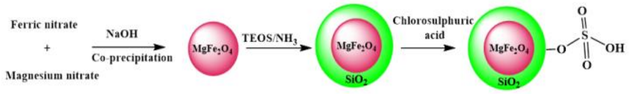
Experimental

Materials/chemicals details

Provide appropriate experimental details in this section in form of subsections:

All solvents and chemicals were of analytical grade and purchased from Sigma Aldrich and used as received. ^{1}H & ^{13}C - NMR spectra were recorded on Bruker Advance spectrometers using $CDCl_3$ and DMSO-d_6 as solvents. A Fourier-transform infrared spectrum was recorded on the Shimadzu FT-IR-8400 spectrometer. The microwave-assisted reaction was exhibit in a scientific Ragatech microwave oven (2450 MHz). This system is fitted with a temperature & power feedback control switch and measure the temperature via highly sensitive IR sensor. The bactericidal activity of the compounds was tested by disc diffusion method as described by Kirby-Bauer [**24,25**]. Catalyst $MgFe_2O_4@SiO_2\text{-}SO_3H$ was synthesized according to our previous report in Ref. [1].

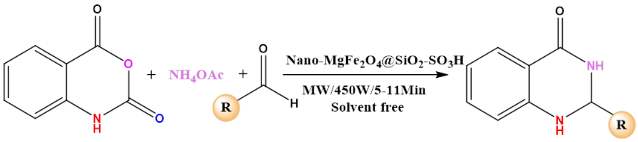


Scheme 1. MNPs-$MgFe_2O_4@SiO_2\text{-}SO_3H$ as catalyst for synthesis of 2,3-dihydroquinazolin-4(1H)-ones.

General procedure for $MgFe_2O_4@SiO_2–SO_3H$ catalyst mediated synthesis of 2,3-dihydroquinazolin-4(1H)-ones

A mixture of Isatoic anhydride (1.0 mol), aldehyde (1.0 mol), ammonium acetate (1.2 mol) and $MgFe_2O_4@SiO_2\text{-}SO_3H$ (10 Wt%) was charged in 50 mL RBF and reaction mixture was irradiated in microwave (450 W at 120°C) for appropriate time. The reaction was monitored by TLC. After the completion of reaction, the reaction mixture was diluted with dichloromethane (DCM) + methanol (1:1 v/v) (25 mL) and isolation of the catalyst was carried out by applying an external magnet. The reaction mixture was concentrated under reduced pressure to get a solid crude product. The obtained crude product was washed with water and recrystallized by ethanol. For the reusability of the recovered catalyst, it was washed with methanol and chloroform. It was dried at 60 °C and reused for the next cycle.

All the synthesized compounds are known and characterized by ^{1}H & ^{13}C-NMR spectroscopy, IR and Mass spectrometric techniques. Melting points of synthesized compounds are compared with reported values. (**Scheme-2**)



Scheme 2. MNPs-$MgFe_2O_4@SiO_2–SO_3H$ as catalyst for synthesis of 2,3-dihydroquinazolin-4(1H)-ones.

Selected spectra for the synthesized compounds

2-(4-methoxyphenyl)-2,3-dihydroquinazolin-4(1H)-one(4f):

IR (cm^{-1}): 3297, 3176, 1650, 1608, 1504, 1484;^{1}H-NMR (400 MHz, DMSO-d_6): 3.75 (s, 3H), 5.70 (s, 1H), 6.66(s, 1H), 6.73 (d, 1H), 6.90 (m, 3H), 7.19 (t, 1H), 7.41 (d, 2H), 7.63 (d, 1H), 8.12 (s, 1H) ^{13}C NMR (100 MHz, DMSO-d_6): δ 55.04, 66.45, 78.85, 113.49, 113.82, 114.35,114.93, 116.99, 127.28, 128.17, 133.06, 133.29, 147.98, 163.76; MS: m/z 254(M).

2-(5-bromo-2-hydroxyphenyl)-2,3-dihydroquinazolin-4(1H)-one(4i)

IR (cm^{-1}): 3412, 3193, 2968, 2880, 1653, 1611, 1490;^{1}H-NMR (400 MHz, DMSO-d_6): 5.9 (s, 1H), 6.6 (t, 1H), 6.78 (d, 1H), 6.84 (d, 1H),7.23 (t, 1H), 7.32(dd, 1H), 7.4(s, 1H), 7.63(dd, 1H), 8.05(s, 1H), 10.3(s, 1H);^{13}C NMR (100 MHz, DMSO-d_6): δ 61.3, 110.3, 114.9, 115.0, 117.9, 118.2, 127.9,130.0, 130.2, 132.3, 133.9, 148.1, 154.4, 164.3; MS: m/z 320(M+2)

2-(2-hydroxynaphthalen-1-yl)-2,3-dihydroquinazolin-4(1H)-one(4l):

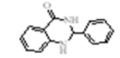
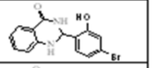
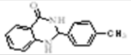
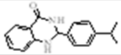
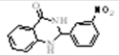
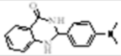
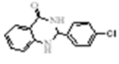
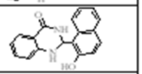
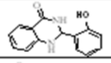
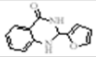
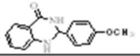
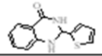
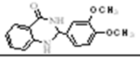
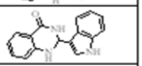
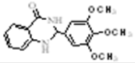
IR (cm^{-1}): 3383, 3166, 2360, 1663, 1608, 1589, 1450;^{1}H-NMR (400 MHz, DMSO-d_6): 6.91 (d, 1H), 7.33-7.38 (m, 2H), 7.52-7.66 (m, 4H),7.76 (d, 1H), 7.89 (d, 2H), 8.02 (s, 1H), 8.46 (d, 1H), 9.46 (s, 1H), 15.07 (s, 1H);^{13}C NMR (100 MHz, DMSO-d_6): δ 60.22, 108.77, 119.02, 120.21, 123.09, 123.43, 125.51, 126.53, 128.13, 128.31, 128.98, 131.18, 133.54, 137.39, 141.90, 153.65, 169.12; MS: m/z 290(M).

Antimicrobial activity

All the synthesized 2,3-dihydroquinazolin-4(1H)-ones derivatives were screened for antimicrobial activities in vitro by using disc diffusion method. The anti-microbial activity of synthesized compounds were explored against *Escherichia coli* (Gram negative) (MTCC443) and *Staphylococcus aureus* (Gram positive) (MTCC96) bacterial strain using streptomycin as standard. The antifungal activity was studied against *Candida albicans* (MTCC227) and *Aspergillus niger* (MTCC282) fungal strain using ketoconazole as standard. Initial screening of prepared products and standard drugs were carried out using 2000 μg mL^{-1} as a fixed concentration. The zone of inhibition was measured the end of 24 h for bacteria and 20-24 h for fungi at temperature 35^{0}C. Screening results are summarized in **Fig. 1** and **Fig. 2** and compound code mentioned in **Table 1**.

Among all tested compounds, D4 and D12 was found to be exhibits good activity against both bacteria in which D12 was most potent against *Escherichia coli*. The compound D7 was found to be more potent against *A.nigaer* and *C.albicans* fungal strains. The detailed results of antibacterial and antifungal preliminary investigation are mentioned in **Fig. 1** and **Fig. 2**.

Table 1. Structure and code of synthesized compounds.

Comp. code	Compound structure	Comp. code	Compound structure
D1		**D9**	
D2		**D10**	
D3		**D11**	
D4		**D12**	
D5		**D13**	
D6		**D14**	
D7		**D15**	
D8		**STR**	**Streptomycin**
		KET	**Ketoconazole**

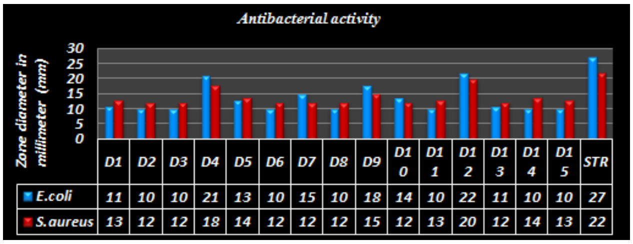


Fig. 1. Graphical representation of antibacterial activity of quinazolines (D1 to D15) compounds series.

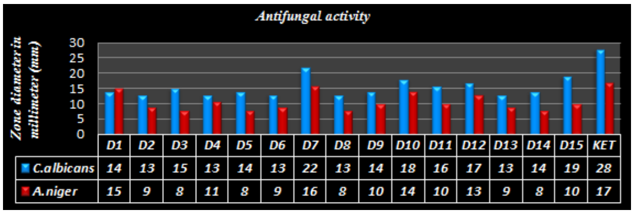


Fig. 2. Graphical representation of antifungal activity of quinazolines (D1 to D15) compounds series.

Catalytic application of $MgFe_2O_4@SiO_2$-SO_3H

During preliminary stage of investigation, it was decided to study catalytic activity of $MgFe_2O_4@SiO_2$-SO_3H for synthesis of 2,3-dihydroquinazolin-4(1H)-ones derivatives (Scheme 2). The three-component reaction of isatoic anhydride (1mol), benzaldehyde (1mol), and ammonium acetate (1.2 mol), was selected as model reaction. The efficacy of $MgFe_2O_4@SiO_2$-SO_3H catalyst was investigated for the preparation of the 2,3-dihydroquinazolin-4(1H)-ones against the other acid catalysts. The related comparative data are presented in **Table 2**. Among the other acid catalysts $MgFe_2O_4@SiO_2$-SO_3H was found to be superior in terms of catalyst amount as well as yield and time of reaction. Results of [bmim] BF_3 and β-cyclodextrin-SO_3H catalyst were comparable but however required higher amount of the catalysts as well as longer reaction time (Entries 4 and 10, **Table 2**).

Entry	Catalyst	Amount Wt% or Mol%	Molar Ratio[a]	Condition/ Solvent	Time (min)/ Yield[b] (%)	Ref.
1	Montmorillonite K-10	283 wt%	1:1:1.2	Reflux/EtOH	240/72	29
2	β-cyclodextrin	214 wt%	1:1:1.2	Reflux/H_2O	120/86	30
3	β-cyclodextrin	107 wt%	1:1:1	$65^0C/H_2O$	-/83	31
4	[bmim]BF_4	188 wt%	1:1:1	70^0C/solvent free	90/92	32
5	$KA1(SO_4)_2$-$12H_2O$	141 wt%	1:1:1.2	Reflux/EtOH	240/71	28
6	$Zn(PFO)_2$	25 wt%	1:1:1.2	Reflux/EtOH:H_2O	360/78	33
7	Cyanuric chloride	17 wt%	1:1:1.1	$60^0C/CH_3CN$	60/90	34
8	nano-In_2O_3	13 wt%	1:1:1.2	80^0C/EtOH:H_2O	240/89	18
9	$Fe_3O_4@SiO_2$-imid-PMA	38 wt% 28 wt%	1:1:1.2 1:1:1.2	Reflux/EtOH:H_2O Ultrasound/EtOH	120/82 12/90	35
10	β-cyclodextrin-SO_3H	15 mol%	1:1:1.2	Water	30/90	21
11	$MgFe_2O_4@SiO_2$-SO_3H	10 wt%	1:1:1.2	Microwave (120°C)/solvent free	5/96	This work

Subsequently, the effect of solvent was examined. The reaction was also studied in different solvents such as Ethanol, THF, Acetonitrile, DMF, Water and Toluene. As regards use of solvent, solvent free condition was found to be most suitable with respect to both yields and reaction time. It was found that reaction accelerate in solvent free condition at 110°C in microwave in the presence of 7.5W% $MgFe_2O_4@SiO_2$-SO_3H catalyst gives 70% yield.

Initial results was encouraged us to optimize reaction, at different catalytic concentration of $MgFe_2O_4@SiO_2$-SO_3H catalyst. An experiment was performed in the absence of catalyst on selected model reaction between benzaldehyde (1mol), ammonium acetate (1.2 mol) and isatoic anhydride (1mol) 110°C /MW in solvent free condition. It was observed that the product obtained in trace quantity. By keeping other reaction parameters same, the catalyst amount was varied stepwise by 2.5 Wt% and the observed results were for 2.5Wt% (25 min and Yield 35%), 5Wt% (18 min and Yield 62%),7.5Wt% (13 min and Yield 70%), 10Wt% (8 min and Yield 88%), 12.5Wt% (8 min and Yield 87%) and 15Wt% (8 min and Yield 88%). Above observation indicates that there were no significant improvement in reaction yield and time after 10 Wt% catalyst. So, we used 10 Wt% catalyst for further investigations.

In the next stage of investigation, to reach the optimal conditions, the reaction was carried out at different temperatures ranging from 0 to 130°C in microwave synthesizer oven. It was observed that at temperature 30°C in microwave oven the reaction did not take place, even trace amount of the product was not observed. Further increasing the temperature of microwave reactor up to 90°C it gives 62 % yield of the desired product. Reaction was accelerated in presence of $MgFe_2O_4@SiO_2$-SO_3H (10 Wt%) at 120°C. within 5 min reaction undergone completion with 95 % yield. Furthermore, no increase in yield was observed even reaction temperature rise up to 130°C in microwave.

In the end, the effect of microwave irradiation was studied; the maximum conversion has recorded at 450W within 5 min. A 95% product yield was observed for the model reaction in the optimized conditions.

After the investigation of the several reaction parameters, the catalytic system was used for the synthesis of 2,3-dihydroquinazolin-4(1H)-one derivatives using different aldehydes with ortho-, meta- and para-substituted groups. The related compounds were prepared with a shorter reaction time and excellent to good yield of 78%-95%. (**Fig. 3**). The use of different aromatic aldehydes does not significantly affect the yield of products. Even with several sensitive functional groups bearing aldehydes such as Cl, Br, OH, CH_3, OCH_3, and OC_2H_5 the reaction works smoothly. It gives excellent yields of the corresponding products (**Fig. 3** entry 1-11). In the presence of sensitive heterocyclic aldehydes such as 2-thiophene, furfural, 2OH- napthyl, and 3-indole, the $MgFe_2O_4$@SiO_2-SO_3H catalyst worked well even without the formation of any side products (Fig. 3 entry 12-15). Longer reaction time (11 min) was needed for the reaction of 3,4,5- trimethoxy benzaldehyde (Fig. 3 entry 8). It is well known that 2,3- dihydroquinazolin-4(1H)-ones's synthesis works very well on aryl aldehydes.

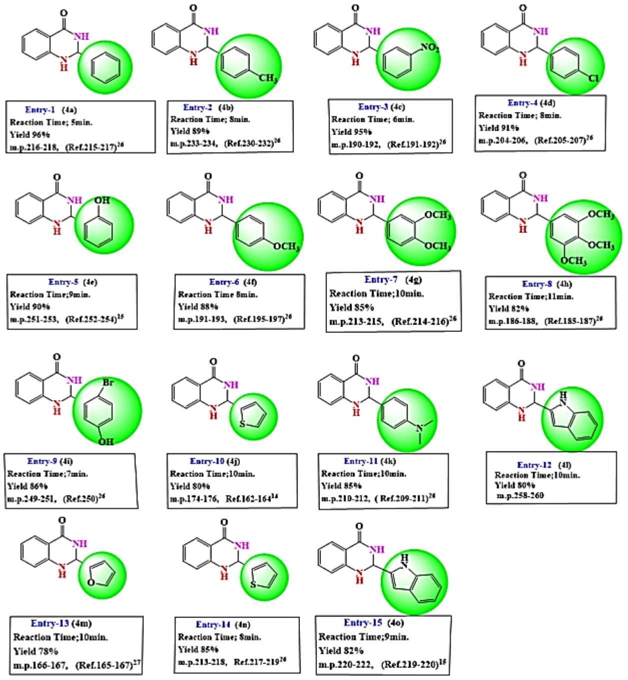


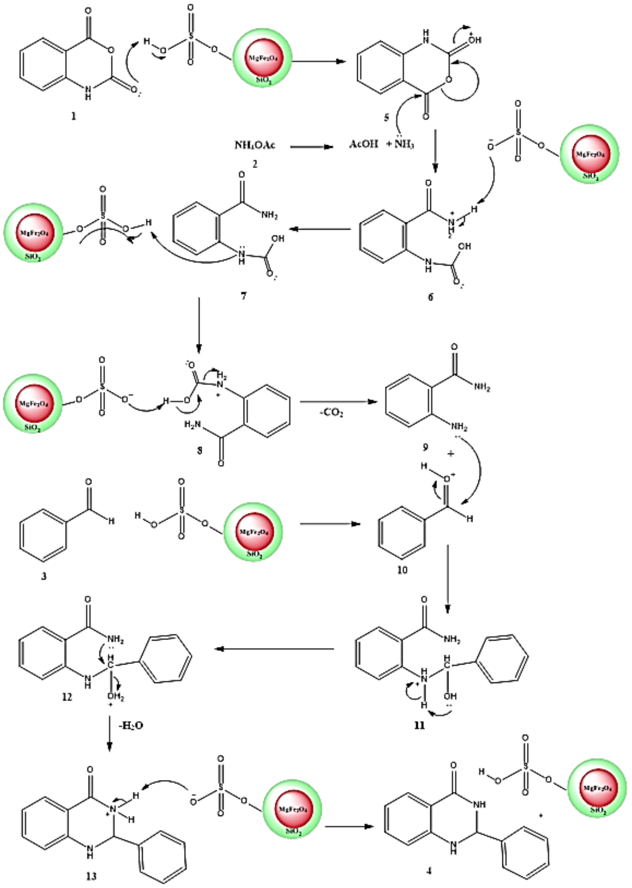
Fig. 3. Scope for substrate.

[a]Reaction conditions: isatoic anhydride (1mol); aldehyde (1 mol); ammonium acetate (1.2 mol) in the presence of 10 wt% $MgFe_2O_4$@SiO_2-SO_3Hcatalyst in microwave irradiation (450W).

[b]All synthesized compounds are known and were identified by their m.p., ^1H &^{13}C-NMR and IR spectra according to literature.

[c]Isolated yield.

A proposed mechanism for the synthesis of 2,3-dihydroquinazolin-4(1H)-one has been outlined in **Scheme 3** according to literature. Firstly, the isatoic anhydride (1) was activated by $MgFe_2O_4$@SiO_2-SO_3H to give intermediate (5), and then dissociation of ammonium acetate occurs in presence of H^+ ion. Nucleophilic nitrogen of ammonia, which is obtained from ammonium acetate (2) attack on activated isotoic anhydride to produce an intermediate (6), which in turn gives an intermediate (7). Then intermediate (8) was obtained in the presence of $MgFe_2O_4$@SiO_2-SO_3H. Further decarboxylation of intermediate (8) gives 2-aminobenzamide (9). Simultaneously, aldehyde was activated by $MgFe_2O_4$@SiO_2- SO_3H to give intermediate (10). Afterward, the reaction was carried out with 2-aminobenzamide (9) and intermediate (10) to get intermediate (11). Intermediate (12) was obtained by transferring proton from intermediate (11). At the end, ring closing results into intermediate (13) which on dehydration turns into the target product (4).



Scheme 3. Plausible mechanism for synthesis of 2,3-dihydroquinazolin-4(1H)-ones derivatives.

The reusability of $MgFe_2O_4$@SiO_2-SO_3H was studied by choosing the model reaction of isatoic anhydride, ammonium acetate and benzaldehyde in the presence of $MgFe_2O_4$@SiO_2-SO_3H under solvent-free condition. After completion of the reaction, crude product was made soluble in dichloromethane (DCM) + methanol (CH_3OH) (1:1 v/v). The separation and recovery of catalyst was easily carried out from the reaction mixture, simply by an external magnet and followed by decantation of the reaction solution. The remaining catalyst was washed with mixture of dichloromethane (DCM) + methanol (1:1 v/v) solvent to remove residual product. Finally, recovered catalyst was dried under vacuum and reused in subsequent reactions. The recovered catalyst was reused for fifth cycles under the same reaction conditions for preparation of product. The relationship between the number of cycles of reactions and the catalytic activity in terms of yields of products is presented in **Fig. 4**.

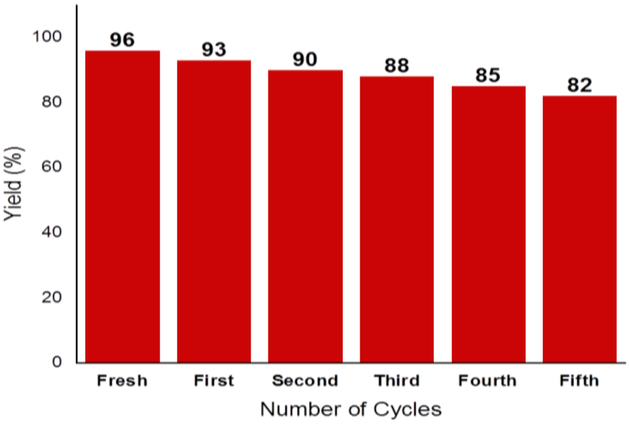


Fig. 4. Recyclability of Catalyst.

Conclusion

In summary, we have explored the catalytic activity of magnetically recoverable $MgFe_2O_4@SiO_2\text{-}SO_3H$ catalyst for synthesis of 2,3-dihydroquinazolin-4(1H)-ones and its derivatives in a green media. The promising features for reported methodologies are high activity, mild reaction conditions, variety of substrate scope, excellent yield, effortless work up and recyclability of a catalyst, making the present protocol beneficial for environmental as well as industrial point of view. Along with this some of the synthesized compounds manifested promising antibacterial activities against E. coli and S. aureus. Some of them exhibited very good antifungal activity against C. albicans and A. niger. We believe that this protocol will be a valuable addition to the existing methods in the field of synthesis of 2,3-dihydroquinazolin-4(1H)-ones and also in biological evaluation. Concerning observed satisfactory catalytic properties, we look further for investigation of use of it in other Multicomponent reactions.

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Keywords

Magnetically recoverable catalyst, Microwave assisted, 2,3-dihydroquinazolin-4(1H)-ones, antibacterial and antifungal activity.

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