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Efficient Synthesis of 2, 3-Dihydroquinazolin-4(1*H*)-ones in the Presence of Ferrite/Chitosan as a Green and Reusable Nanocatalyst

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Abstract

In this work, an efficient and facile method has been developed for the synthesis of 2,3-dihydroquinazolin-4(1H)-ones from a condensation reaction of 2-aminobenzamide with various alkyl, aryl and alicyclic aldehydes or ketones using Fe_3O_4*/chitosan as an environmentally benign, magnetically recoverable nanocomposite catalyst in ethanol at room temperature in high to excellent yields under mild reaction conditions. The* Fe_3O_4*/chitosan composite nanocatalyst was first prepared by a sol-gel method and characterized by using a variety of conventional techniques including Fourier transforms infrared spectroscopy (FT-IR), scanning electron microscopy (SEM), energy-dispersive X-ray (EDX) and X-ray diffraction (XRD) analyses. The present work include several advantages such as avoiding the use of toxic solvents or expensive catalysts, generality, high yields, short reaction times, clean reaction profile, ease of product isolation, simplicity, recyclability of the catalyst and finally agreement with the green chemistry protocols. All make it a useful and attractive protocol for the synthesis of heterocyclic compounds like quinazolinone derivatives.*

Keywords: *Magnetic nanoparticles,* Fe_3O_4*, Chitosan, Dihydroquinazolinone, Heterogeneous catalysts.*

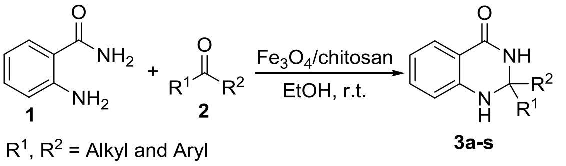
1. INRODUCTION

Chitosan is a very plentiful polysaccharide biopolymer that can be achieved via the alkaline deacetylation of chitin [1]. It is applied widely in several industrial and manufacturing processes including wastewater treatment, pharmaceutical, medical, food processes, agriculture and textile industries [2]. It has not only good catalytic activity for some reactions, but also, could be used as a support for the preparation of heterogeneous catalysts. The insertion of metals such as iron into the chitosan polymer matrix may improve its capacity of interaction by complexation. Chitosan-based composites are increasingly important as biochemical, antimicrobial and catalytic reagents [3].

Quinazolinone derivatives are a class of fused heterocyclic compounds with diverse pharmacological and biological activities. 2,3-Dihydro-4(1*H*)-quinazolinones are very interesting because of their various pharmaceutical activities such as antibacterial, diuretic, antifungal, antitumor, mono amine oxidase inhibitor and potent poly(ADP-ribose)polymerase inhibitors [4-7]. 3, 4-Dihydroquinazolin-4-ones have exhibited a broad range of biological properties such as anti-cancer, anti-inflammatory, anti-convulsant and anti-hypertensive activities [8]. They have been reported as potent antimycobacterial and chemotherapeutic agents in the treatment of tuberculosis [9, 10].

Due to importance of quinazolinone derivatives, a few synthetic methods have been reported for the their synthesis from the condensation of 2-aminobenzamide with aromatic aldehydes catalyzed by Lewis and Brönsted acids, or one-pot condensation of isatoic anhydride, amines and aldehydes under various conditions [11-16]. But, some of these reactions have drawbacks such as using expensive reagents, toxic solvents, excess amounts of catalysts, long reaction times and high temperatures along with the use of non-recyclable catalysts and low yields. Furthermore, the preparation of required catalysts is an important trouble in some cases. Therefore, design and development of a new, simple, environmentally benign, high-yielding and clean synthetic route toward this direction is of prime importance.

In this regard, superparamagnetic nanocomposites such as Fe_3O_4/chitosan possess unique features such as environmental compatibility, magnetically recoverable, non-toxicity, reusability, non-corrosivity and chemical and physical stability [17-19]. As a result, due to remarkable properties of Fe_3O_4/chitosan nanocatalyst and importance of quinazolinone scaffold, and in continuation of our recent researches on development of green, simple and environmentally benign protocols for the synthesis of heterocyclic compounds [20-25], herein, we wish to report the synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones **3a-s** by a condensation reaction of 2-aminobenzamide **1** with various aldehydes or ketones **2** in the presence of Fe_3O_4/chitosan as a recyclable heterogeneous superparamagnetic composite nanocatalyst in EtOH at room temperature under mild reaction conditions (Scheme 1).



Scheme 1.** Synthesis of 2, 3-dihydroquinazolin-4(1H)-ones **3a-s.

2. EXPERIMENTAL

2.1. General

All solvents, chemicals and reagents were purchased from Merck, Fluka and Aldrich international chemical companies. Melting points were measured on an Electrothermal 9100 apparatus, UK and are uncorrected. Fourier transforms infrared spectroscopy (FT-IR) spectra were recorded on a Shimadzu IR-470 spectrometer, Japan. Scanning electron microscopy (SEM) images were obtained on a Seron AIS 2100 equipped with energy-dispersive X-ray spectroscopy (EDX) spectra were recorded on Numerix DXP–X10P, Korea. X-ray diffraction (XRD) measurements were carried out using a JEOL JDX–8030 (30 kV, 20 mA), Japan.

2.2. General Procedure for the Preparation of Fe_3O_4/Chitosan Nanocomposite

First, 1 g of chitosan powder was added into a 100 mL of 0.1 M acetic acid and the mixture was stirred to form a 1% (w/v) clear chitosan solution. Then, the homogeneous solution was filtered and degassed by keeping the solution into vacuum oven for 3 h. After that, 2 mL of glycolic acid and calculated amount of Fe_3O_4 nanoparticles (black powder, average particle size 30 nm (TEM), purity 99% and specific surface area 55 m^2/g) were dispersed in the chitosan solution by stirring at room temperature. Next, it was sonicated for 1 h. The resulting solution was stirred overnight at room temperature. Then, the resulted gel separated from the reaction mixture by a permanent magnet, washed several times with EtOH, and dried on a ceramic plate at room temperature for 48 h to obtain Fe_3O_4/chitosan.

2.3. General Procedure for the Synthesis of 2, 3-Dihydroquinazolin-4(1H)-ones 3a-s

A mixture of 2-aminobenzamide (1 mmol) and an aldehyde or ketone (1 mmol) in the presence of Fe_3O_4/chitosan nanocatalyst (10 mg) in 5 mL of EtOH was stirred in a round bottomed flask at room temperature for the appropriate time. After completion of the reaction, as indicated by TLC (ethyl acetate/*n*-hexane, 3/1), the catalyst was recycled easily by an external magnetic stirring bar when the stirring was stopped. Then, the reaction solution was filtered off and the residue was purified by washing further with water, and then crystallized from EtOH to give pure crystalline 2, 3-dihydroquinazolin-4(1*H*)-ones **3a-s**. The recycled nanocatalyst was washed with EtOH (96%), air-dried (6 h) and reused for several times in other fresh reactions without considerable loss of efficiency.

3. RESULTS AND DISCUSSION

The Fe_3O_4/chitosan composite nanocatalyst was first prepared by a sol-gel method and characterized by using a variety of conventional techniques including SEM, FT-IR, EDX and XRD [24, 25].

As shown in Figure 1, the particle size was studied by SEM and the identification of Fe_3O_4/chitosan morphology was based on the analysis of SEM images. The obtained SEM images of nanoparticles showed that average size of the supported nanoparticles was about 50-100 nm.

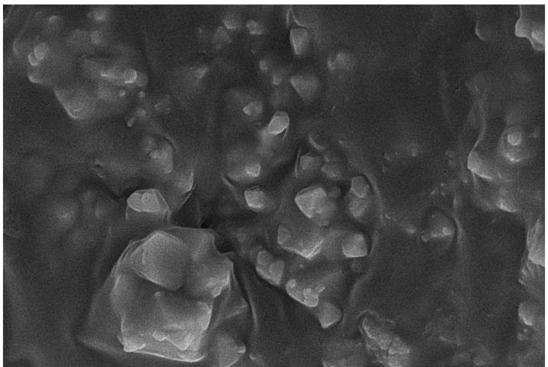


Figure 1. *SEM image of Fe_3O_4/chitosan composite nanocatalyst.*

The FT-IR spectrum of Fe_3O_4/chitosan nanocatalyst is shown in Figure 2. It confirms the presence of functional groups and accordance of predicted bonds.

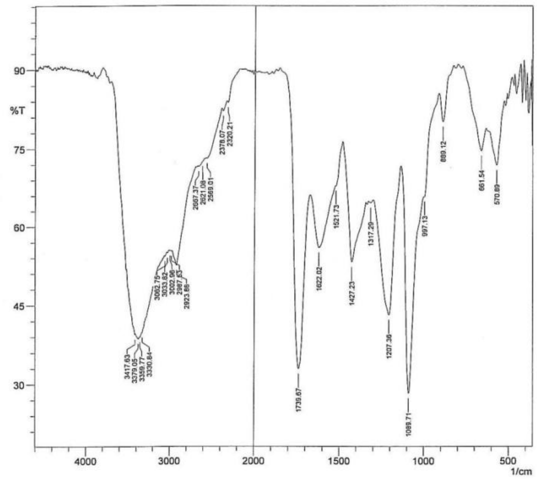


Figure 2. *FT-IR spectrum of Fe_3O_4/chitosan composite nanocatalyst.*

To investigate the catalytic activity of the prepared Fe_3O_4/chitosan nanocatalyst in the synthesis of heterocyclic compounds, a model experiment was carried out using 1 equivalent of each of 2-aminobenzamide and 4-chlorobenzaldehyde stirring at room temperature in EtOH in the presence or absence of the nanocatalyst. The progress of the reaction was monitored by TLC. As indicated in Table 1, by decreasing the catalyst loading, the reaction time was increased (entries 1 and 2). Increasing of the catalyst loading did not significantly improve the reaction time and yield (entry 5). Also, high catalyst loading leads to the formation of side products (observed on TLC), which eventually reduces the yield of the desired product. The best results were obtained with 10 mg of the nanocatalyst (entry 3).

Table 1. *Effect of the amount of the nanocatalyst on the model reaction.*[a]

Entry	Catalyst (mg)	Time (min)	Yield[b] (%)
1	2	40	91
2	5	30	89
3	10	12	97
4	20	20	85
5	30	20	78

[a] Reaction conditions: 2-aminobenzamide (1 mmol), 4-chlorobenzaldehyde (1 mmol), EtOH (5 mL), r.t.
[b] Isolated yields.

To show merit of the present work, the reaction of 2-aminobenzamide and 4-chlorobenzaldehyde was compared with literature data. As shown in Table 2, the reported procedures suffer from one or more disadvantages such as elevated reaction temperatures or longer reaction times (entries 1-5). Therefore, we believe the present method is an improvement with respect to other procedures.

To compare the effect of various solvents in the pilot reaction, 4-chlorobenzaldehyde was reacted with 2-aminobenzamide in the presence of 10 mg of Fe_3O_4/chitosan in CH_2Cl_2 at room temperature for 30 min. The expected 2, 3-dihydroquinazolin-4(1*H*)-one product was obtained in 87% yield (Table 2, entry 6). The use of other solvents such as EtOAc, MeCN and MeOH was examined but did not improve the product yields (Table 2, entries 7-9). The results showed that the efficiency and the yield of the model reaction in EtOH were higher than those obtained in other solvents (Table 2, entry 5). In addition, to study the importance of Fe_3O_4@chitosan nanocomposite structure in this reaction, two blank experiments were conducted in the presence of Fe_3O_4 and chitosan under optimized reaction conditions. The yields in these cases were only 38 and 55% after 30 min, respectively (Table 2, entries 10 and 11).

Table 2. *Comparison of the efficiency of Fe_3O_4/chitosan and the reaction conditions with some other reports on the model reaction.*

Entry	Catalyst	Solvent	Temp. (°C)	Time (min)	Yield[a] (%)	Lit.
1	NH_2SO_3H	H_2O	70	35	89	[26]
2	-	H_2O	reflux	60	94	[27]
3	CAN	H_2O	60	120	92	[28]
4	Amberlyst-15	MeCN	r.t.	30	98	[29]
5	Fe_3O_4/chitosan	EtOH	r.t.	12	97	Present work
6	Fe_3O_4/chitosan	CH_2Cl_2	r.t.	30	87	Present work
7	Fe_3O_4/chitosan	EtOAc	r.t.	45	79	Present work
8	Fe_3O_4/chitosan	MeCN	r.t.	35	83	Present work
9	Fe_3O_4/chitosan	MeOH	r.t.	30	84	Present work
10	Fe_3O_4	EtOH	r.t.	30	38	Present work
11	Chitosan	EtOH	r.t.	30	55	Present work

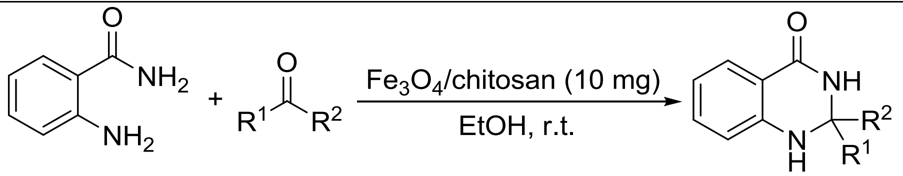
[a] Isolated yields.

To study the scope and limitations of this promising reaction, 2-aminobenzamide **1** was reacted with commercially available aromatic aldehydes or ketones **2** bearing both electron-donating and electron-withdrawing substituents. A series of dihydroquinazolinones **3a–o** were successfully obtained (Table 3, entries 1–15). Neither electronic nor steric effects have significant negative impact on the reaction rate.

We also investigated the synthesis of 2, 3-dihydroquinazolin-4(1*H*)-ones (**3p–s**) starting from various aliphatic and alicyclic ketones. They afforded the corresponding products in high yields and relatively short reaction times (Table 3, entries 16–19). This reaction proceeds very cleanly and no undesired side reactions were observed.

As shown in Figure 3, the magnetic recoverability of the nanocatalyst was investigated in the pilot experiment for the synthesis of **3a**. The recycled catalyst was reused in the subsequent fresh reactions without any treatment and no considerable loss of its catalytic activity was observed. As a result, it can be classified as an excellent catalyst in industry for large-scale synthesis [40-45].

Table 3. *Synthesis of 2,3-dihydroquinazolin-4(1H)-ones* ***3a-s.***



Entry	Aldehyde/ketone	Product	Time (min)	Yield[a] (%)	Mp (°C)	
					Found	Lit.
1	4-Cl-C_6H_4-CHO	**3a**	12	97	208-209	206-208 [29]
2	2,4-Cl_2-C_6H_3-CHO	**3b**	12	96	189-191	181-185 [29]
3	2-Cl-C_6H_4-CHO	**3c**	15	96	213-214	213-214 [30]
4	4-F-C_6H_4-CHO	**3d**	90	90	200-202	202-204 [31]
5	4-NO_2-C_6H_4-CHO	**3e**	20	90	188-190	190-192 [32]
6	3-NO_2-C_6H_4-CHO	**3f**	15	95	205-207	208-210 [32]
7	4-CN-C_6H_4-CHO	**3g**	35	92	250-252	250-253 [32]
8	2-OH-C_6H_4-CHO	**3h**	19	96	240-242	235-238 [33]
9	4-OH–C_6H_4-CHO	**3i**	36	79	328-330	332 [34]
10	4-MeOC_6H_4-CHO	**3j**	30	89	177-179	179-180 [35]
11	4-$(Me)_2N$-C_6H_4-CHO	**3k**	120	89	223-225	228 [36]
12	furfural	**3l**	66	89	169-171	167-168 [37]
13	4-OH-3-MeO-C_6H_3-CHO	**3m**	120	96	227-229	226-227 [38]
14	4-Me-C_6H_4-CHO	**3n**	105	97	233-236	232-234 [39]
15	C_6H_5-CHO	**3o**	90	81	214-216	216-218 [39]
16	acetone	**3p**	17	82	182-185	183-184 [39]
17	cyclopentanone	**3q**	15	91	260	259-260 [39]
18	cyclohexanone	**3r**	30	80	227-229	225-226 [39]
19	cycloheptanone	**3s**	60	95	201-203	204-205 [39]

[a] Isolated yields.

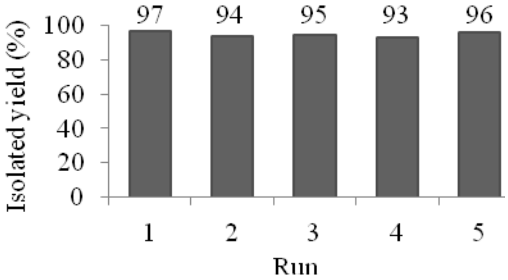
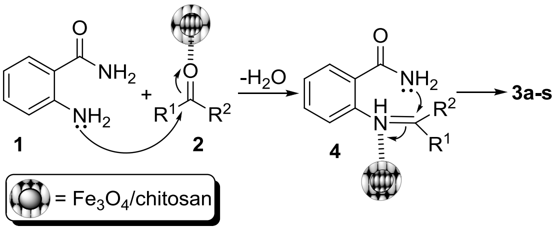


Figure 3. *Recovery results of the nanocatalyst in the model reaction.*

A possible mechanism of the reaction is shown in Scheme 2. The first step involves the condensation of amine moiety of 2-aminobenzamide **1** with aldehyde or ketone **2** promoted in the presence of the nanocatalyst to produce intermediate **4**. Then, the product **3** is formed from an intramolecular nucleophilic attack of amide to imine of **4**.



Scheme 2. *A proposed mechanism for the synthesis of products* ***3a–s****.*

4. CONCLUSION

In summary, a highly efficient and green procedure was developed for the synthesis of structurally diverse quinazolinone derivatives by using 2-aminobenzamide and various aldehydes or ketones in the presence of Fe_3O_4/chitosan as an efficient and environmentally benign composite nanocatalyst. The present work include several advantages such as avoiding the use of toxic solvents or expensive catalysts, high yields, short reaction times, ease of product isolation, simplicity, recyclability of the catalyst and good agreement with the green chemistry protocols. It can be classified as a useful, practical and attractive protocol for the synthesis of heterocyclic compounds like quinazolinone derivatives.

ACKNOWLEDGEMENTS

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