

Synthesis of 2,3-Dihydroquinazolin-4(1H)-ones by Three-Component Coupling of Isatoic Anhydride, Amines, and Aldehydes Catalyzed by Magnetic Fe₃O₄ Nanoparticles in Water

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A simple and efficient protocol for one-pot three-component coupling of isatoic anhydride, amines, and aldehydes in water using magnetically recoverable Fe₃O₄ nanoparticles is reported. This methodology results in the synthesis of a variety of 2,3-dihydroquinazolin-4(1H)-ones in high yields. The catalyst can be recovered and recycled without a significant loss in the catalytic activity.

1. Introduction

Because of increasing environmental concerns, the development of a clean synthetic procedure has become crucial and demanding research. In this sense, heterogeneous organic reactions have many advantages, such as ease of handling, separation, recycling, and environmentally safe disposal.¹ Nanoparticles as heterogeneous catalysts have attracted a great deal of attention in recent years because of their interesting structure and high catalytic activities.² In this context, magnetic particles, in particular, have emerged as one of the most useful heterogeneous catalysts because of their numerous applications in nanocatalysis, biotechnology, and medicine.³ Additionally, the magnetic properties make possible the complete recovery of the catalyst by means of an external magnetic field.⁴ On the other hand, performing organic reactions in aqueous media has several benefits because water would be considerably safe, abundant, nontoxic, environmentally friendly, and economical compared to organic solvents. Moreover, water exhibits unique reactivity and selectivity, which is different from those in conventional organic solvents.⁵ Therefore, the development of a catalyst that is not only stable toward water but also simply recyclable seems highly desirable.

2,3-Dihydroquinazolinones are an important class of bioactive compounds that are prescribed as plant growth regulators, and as anticancer drugs.⁶ In addition, these compounds can easily be oxidized to their quinazolin-4(3H)-one analogues,⁷ which are themselves important biologically active heterocyclic compounds,⁸ and can also be found in some natural products.⁹ Particularly, the quinazolinone core scaffold has been extensively utilized as a drug-like template in medicinal chemistry.¹⁰ In view of their significance, various procedures have been developed for the construction of 2,3-dihydroquinazolin-4(1H)-one frameworks.¹¹ Unfortunately, those methods for the assembly of these heterocycles involve multi-step and low-yielding reaction sequences. A more attractive and atom-efficient strategy for the preparation of 2,3-dihydroquinazolinones is through a one-pot, three-component reaction of isatoic anhydride, amines,

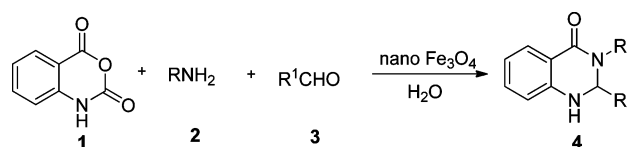
and aldehydes. Multicomponent reactions (MCRs) have some advantages over classic divergent reaction strategies, such as lower costs, shorter reaction time, and less side products, as well as environmentally friendlier aspects.¹² Until now, only a few strong acid catalysts such as montmorillonite K-10,¹³ silica sulfuric acid,¹⁴ zinc(II) perfluorooctanoate [Zn(PFO)₂],¹⁵ KAl(SO₄)₂ · 12H₂O,¹⁶ Al(H₂PO₄)₃,¹⁷ gallium(III) triflate,¹⁸ molecular iodine,¹⁹ MCM-41-SO₃H,²⁰ 1-butyl-3-methylimidazolium tetrafluoroborate ([bmim]BF₄),²¹ and Amberlyst-15²² have been employed to accomplish this three-component reaction. However, most of those methods are associated with different drawbacks such as long reaction time, low yields, strongly acidic conditions, use of expensive reagents, and the requirement of an additional microwave oven. Therefore, there is still a need to develop a simple and convenient approach for the preparation of 2,3-dihydroquinazolinones.

In continuation of our efforts to develop new synthetic methods for important organic compounds,²³ herein we report the synthesis of 2,3-dihydroquinazolin-4(1H)-ones via one-pot three-component condensation of isatoic anhydride, amines, and aldehydes in the presence of a catalytic amount of Fe₃O₄ nanoparticles²⁴ in water (Scheme 1).

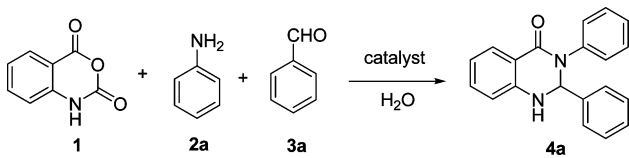
2. Results and Discussion

Initially, the three-component reaction of isatoic anhydride, benzaldehyde, and aniline as a simple model substrate was investigated to establish the feasibility of the strategy and optimize the reaction conditions. The catalytic activity of various nano metal oxides was evaluated for this model reaction, and the results are summarized in Table 1. From Table 1, it was clear that these nanosized particles successfully promoted this A³-coupling reaction with high yields.

Scheme 1. Fe₃O₄ Catalyzed Synthesis of 2,3-Dihydroquinazolin-4(1H)-ones



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Table 1. Effect of Different Catalysts on the Reaction of Isoatoic Anhydride, Benzaldehyde, and Aniline^a


entry	catalyst	yield (%) ^b
1	none	trace
2	ZnO	70
3	TiO ₂	68
4	CuO	75
5	Al(OH) ₃	70
6	ZnFe ₂ O ₄	73
7	γ-Fe ₂ O ₃	72
8	Fe ₃ O ₄	80
9	powdered Fe ₃ O ₄	38

^a Experimental conditions: isoatoic anhydride (1 mmol), benzaldehyde (1 mmol), aniline (1 mmol) and catalyst (0.15 mmol) in 5 mL of H₂O for 2 h under reflux. ^b Isolated yields.

Table 2. Investigation of the Amounts of Catalyst and Solvent Effects on the Reaction of Isoatoic Anhydride, Benzaldehyde, and Aniline^a

entry	catalyst loading (mol %)	solvent	time (h)	yield (%) ^b
1	15	MeCN	2	20
2	15	CH ₂ Cl ₂	2	5
3	15	EtOH	2	78
4	15	THF	2	10
5	15	EtOAc	2	25
6	1	H ₂ O	2	50
7	5	H ₂ O	2	57
8	10	H ₂ O	2	70
9	10	H ₂ O	3	72
10	15	H ₂ O	2	80
11	15	H ₂ O	3	81
12	20	H ₂ O	2	80
13 ^c	15	H ₂ O	2	80 ^d
14 ^c	50	H ₂ O	1.5	84

^a Experimental conditions: isoatoic anhydride (1 mmol), benzaldehyde (1 mmol), aniline (1 mmol), and Fe₃O₄ in 5 mL of solvent under reflux.

^b Isolated yields. ^c Methylamine was used instead of aniline. ^d Yield of 2-amino-*N*-methylbenzamide.

Among the catalysts evaluated, nano Fe₃O₄ was superior to others and produced the best yield of the target 2,3-diphenyl-2,3-dihydro-1*H*-quinazolin-4-one (**4a**). However, powdered Fe₃O₄ displayed low activity (Table 1, entry 9). Only a trace amount of the product was formed in the absence of catalyst (Table 1, entry 1).

We next made a study on the effect of solvents and the catalyst loading in model reaction. In general, nonpolar solvents such as dichloromethane and ethyl acetate led to low yields. The best conversion was observed when the reaction was performed in water. Moreover, we found that the yields were obviously affected by the amount of Fe₃O₄ loaded. It was found that 15 mol % of Fe₃O₄ was sufficient enough to afford **4a** with 80% isolated yield (Table 2, entry 10). The yield remained unaffected when the catalyst loading was increased to 20 mol % (Table 2, entry 12). However, the yield was decreased when the catalyst loading was reduced to 10 mol % (Table 2, entry 8). Prolonging the reaction time did not enhance the yield (Table 2, entry 9). Unexpectedly, only 2-amino-*N*-substituted-benzamide was obtained when aliphatic amine such as methylamine was employed in the presence of 15 mol % of Fe₃O₄ (Table 2,

entry 13). The corresponding 2,3-dihydroquinazolin-4(1*H*)-one could be obtained when higher catalyst loading was used (50 mol %), probably because of the higher basicity of aliphatic amine compared to aniline. Thus, the effective catalyst loading was found to be 15 mol % for aniline and 50 mol % for aliphatic amine, respectively.

To explore the scope and limitation of this A³-coupling reaction, particularly in regard to library construction, we conducted the reactions of diverse amines and aldehydes under the optimized conditions. Selected results are summarized in Table 3. In general, aromatic aldehydes bearing electron-donating or electron-withdrawing groups with isoatoic anhydride proceed smoothly when aniline was employed in this reaction except 4-cyanobenzaldehyde gave lower yield (Table 3, entry 12). Subsequently, the substrate scope of amines was examined for this three-component reaction. Aromatic amines were found to be effective substrates and afforded the respective 2,3-dihydroquinazolin-4(*H*)-one derivatives in high yields. Further, this three-component reaction also proceeded very smoothly when aliphatic amines (Table 3, entries 34–41) were used, and the desired products were obtained in very good yields in the presence of 50 mol % Fe₃O₄.

The reusability is one of the important properties of this catalyst. After the reaction was complete, ethyl acetate was added, and the catalyst was absorbed onto the magnetic stirring bar. The catalyst was then washed with ethyl acetate, air-dried, and used directly with fresh substrates under identical conditions without further purification. It was shown that the catalyst could be used for five runs without noticeable drop in the product yield and its catalytic activity (Table 4). A transmission electron microscopy (TEM) image of Fe₃O₄ showed that the average size and distribution of the Fe₃O₄ were not significantly altered, in good agreement with previously reported results^{3m,n} that proved the unusual properties and potential applications of this magnetic material.

To elucidate the reaction mechanism, 2-amino-*N*-phenylbenzamide was prepared from the reaction of isoatoic anhydride with aniline under same condition. Next, 2-amino-*N*-phenylbenzamide reacted with benzaldehyde to give the corresponding product **4a** in the presence of Fe₃O₄. In addition, the reactions of isoatoic anhydride with benzaldehyde and benzaldehyde with aniline did not proceed under same conditions. On the basis of the above results and by referring to the literature,^{15,17} we propose a plausible mechanism for the formation of 2,3-dihydroquinazolin-4(1*H*)-ones (Scheme 2). First, the isoatoic anhydride is activated by Fe₃O₄ followed by the *N*-nucleophilic amine attacks on the carbonyl to form intermediate **I**. Then decarboxylation occurs resulting in generation of 2-amino-*N*-substituted-benzamide (**II**). Iron cations act as Lewis acid and play a significant role in increasing the electrophilic character of the aldehydes. Subsequently, the reaction of activated aldehyde with **II** proceeds to afford intermediate **III** that is converted to product **4** via an intramolecular cyclization.

In conclusion, we have developed an efficient one-pot procedure for the synthesis structurally diverse libraries of 2,3-dihydroquinazolin-4(1*H*)-ones by a three-component condensation of isoatoic anhydride, amines, and aldehydes in water in the presence of Fe₃O₄. The procedure is applicable

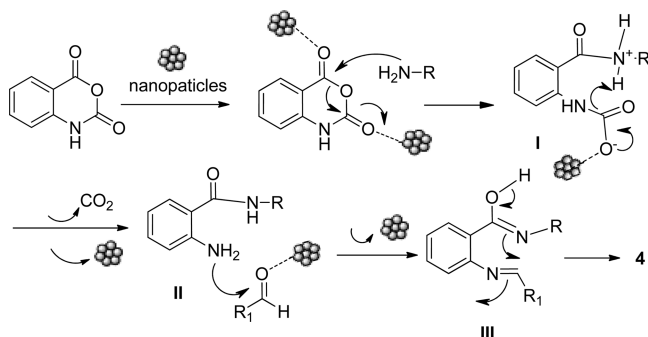
Table 3. Synthesis of 2,3-Dihydroquinazolin-4(1H)-ones in the Presence of Fe₃O₄

entry	R	R ¹	product	time (h)	yield (%) ^a	Mp (°C)	
						found	reported
1	Ph	Ph	4a	2	80	205–206	203–205 ¹⁵
2	Ph	3-Me-C ₆ H ₄	4b	5	84	205–207	194–196 ¹⁵
3	Ph	4-Me-C ₆ H ₄	4c	5	85	215–216	214 ²⁵
4	Ph	4-MeO-C ₆ H ₄	4d	5	80	203–204	204–205 ¹⁵
5	Ph	2,3-(MeO) ₂ -C ₆ H ₃	4e	5	75	170–173	
6	Ph	3,4-(MeO) ₂ -C ₆ H ₃	4f	5	87	244–246	
7	Ph	2,3,4-(MeO) ₃ -C ₆ H ₂	4g	5	86	177–179	
8	Ph	3,4,5-(MeO) ₃ -C ₆ H ₂	4h	5	73	194–196	
9	Ph	3-Br-C ₆ H ₄	4i	5	74	188–190	
10	Ph	4-Br-C ₆ H ₄	4j	5	80	223–225	222–225 ¹⁵
11	Ph	4-NO ₂ -C ₆ H ₄	4k	5	78	194–196	195–196 ¹⁵
12	Ph	4-CN-C ₆ H ₄	4l	5	51	203–205	
13	2-MeC ₆ H ₄	Ph	4m	6	76	175–176	173–175 ²⁶
14	4-MeC ₆ H ₄	Ph	4o	5	80	197–198	196–198 ¹⁵
15	4-Me ₃ CC ₆ H ₄	Ph	4p	5	83	247–248	
16	2-OMeC ₆ H ₄	Ph	4q	5	78	191–192	190 ²⁷
17	4-OMeC ₆ H ₄	Ph	4r	5	82	213–214	215–216 ¹⁷
18	4-OEtC ₆ H ₄	Ph	4s	5	85	196–198	
19	2-ClC ₆ H ₄	Ph	4t	5	76	215–216	215–217 ²⁸
20	4-ClC ₆ H ₄	Ph	4u	5	85	217–219	216–218 ¹⁵
21	4-BrC ₆ H ₄	Ph	4v	5	72	228–229	224–226 ¹⁵
22	4-NO ₂ C ₆ H ₄	Ph	4w	5	70	186–188	185–187 ¹⁵
23	4-MeC ₆ H ₄	4-MeO-C ₆ H ₄	4x	5	73	247–249	
24	4-MeC ₆ H ₄	4-F-C ₆ H ₄	4y	5	69	241–243	
25	4-Me ₃ CC ₆ H ₄	4-MeO-C ₆ H ₄	4z	5	70	196–198	
26	4-Me ₃ CC ₆ H ₄	4-F-C ₆ H ₄	4aa	5	73	244–246	
27	4-Me ₃ CC ₆ H ₄	4-Br-C ₆ H ₄	4ab	5	68	229–230	
28	4-OMeC ₆ H ₄	3-Me-C ₆ H ₄	4ac	5	85	212–213	
29	4-OMeC ₆ H ₄	3,4-(MeO) ₂ -C ₆ H ₃	4ad	5	87	252–254	
30	4-OMeC ₆ H ₄	2,3,4-(MeO) ₃ -C ₆ H ₂	4ae	5	86	190–192	
31	4-OEtC ₆ H ₄	2,3,4-(MeO) ₃ -C ₆ H ₂	4af	5	87	193–195	
32	4-OEtC ₆ H ₄	4-Br-C ₆ H ₄	4ag	5	88	244–246	
33	4-ClC ₆ H ₄	3,4-Me ₂ -C ₆ H ₃	4ah	5	80	229–231	
34 ^b	H	Ph	4ai	2	82	220–221	219–222 ^{14b}
35 ^b	H	4-Me-C ₆ H ₄	4aj	2	85	234–135	233–234 ^{14b}
36 ^b	H	4-MeO-C ₆ H ₄	4ak	2	86	180–182	180–181 ^{14b}
37 ^b	Me	Ph	4al	1.5	84	161–162	160–163 ^{14b}
38 ^b	Me	4-MeO-C ₆ H ₄	4am	1.5	85	146–147	145–146 ^{14b}
39 ^b	Et	Ph	4an	2	85	135–137	134–137 ^{14b}
40 ^b	Et	4-MeO-C ₆ H ₄	4ao	2	88	125–126	124–126 ^{14b}
41 ^b	Et	4-Cl-C ₆ H ₄	4ap	3	83	133–135	132–135 ^{14b}

^a Isolated yield. ^b 50 mol % Fe₃O₄ was used.

Table 4. Recovery and Reuse of Fe₃O₄ Nanoparticles for the Synthesis of **4a**

	cycle				
	1	2	3	4	5
yield (%)	80	78	76	76	75

Scheme 2. Proposed Mechanism for the Synthesis of 2,3-Dihydroquinazolin-4(1H)-ones

to a wide range of aromatic aldehydes and amines, and the products are obtained in high yields. The catalyst can be readily recovered by magnetic separation and reused.

General Procedure for Synthesis of 2,3-Dihydroquinazolin-4(1H)-ones (4). Isatoic anhydride (1 mmol), amine (1 mmol), aldehyde (1 mmol), and Fe₃O₄ (0.15 or 0.50 mmol)

were mixed in H₂O (5 mL). The mixture was stirred under reflux in an air atmosphere for an appropriate time indicated in Table 3. Then ethyl acetate (5 mL) was added. The Fe₃O₄ nanoparticles were absorbed on to the magnetic stirring bar. The organic and aqueous layers were separated. The organic phase was dried over MgSO₄, and the solvent was evaporated under vacuum. The residue was purified by short column chromatography on silica gel.

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Note Added after ASAP Publication. There were errors in Table 3 in the version of this paper published ASAP July 23, 2010. The correct version published on August 4, 2010.

Supporting Information Available. Experimental procedure, characterization data for compound **4** and catalyst. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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