

## Fe<sub>3</sub>O<sub>4</sub>@nano-dextrin/Ti(IV) as a bio-based magnetic nano-catalyst for facile synthesis of 2,3-dihydroquinazolin-4(1H)-ones

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### ABSTRACT

A bio-based magnetic nano-catalyst (Fe<sub>3</sub>O<sub>4</sub>@nano-dextrin/Ti(IV)) was synthesized *via* the preparation of Fe<sub>3</sub>O<sub>4</sub>@nano-dextrin as magnetic support and then its treatment with titanium tetrachloride (TiCl<sub>4</sub>). The properties of this catalyst were characterized by different kinds of techniques such as FT-IR, FESEM, TEM, ICP, XRF, VSM and TGA. As a first report, the catalytic activity of Fe<sub>3</sub>O<sub>4</sub>@nano-dextrin/Ti(IV) was investigated in the synthesis of 2,3-dihydroquinazolin-4(1H)-ones *via* condensation between 2-aminobenzamide and aldehyde under mild conditions. Good efficiency, a high percentage of product yields, simplicity of operation and easy recovery and reuse of catalyst are some benefits of this protocol.

**Keywords:** Fe<sub>3</sub>O<sub>4</sub>@nano-dextrin/Ti(IV), Magnetic nano-catalyst, Lewis acid catalyst, Multi-component reactions, 2,3-Dihydroquinazolin-4(1H)-ones.

### 1. Introduction

Unique feature of multi-component reactions (MCRs) is that three or more substrates react to produce products in one pot, producing absolutely no by-products [1-3]. Products can be taken rapidly, in one pot and with fewer steps. Accordingly, in modern organic chemistry attention was given to MCRs, like medicinal chemistry or combinatorial chemistry. Therefore, utilization of MCRs are very common with just the least of effort and provide a variety of products [4,5]. The quinazolinone (QZ) structure is found in various important synthetic or natural compounds [6-9]. Many of these compounds have been known as one of heterocycles which have biological activity such as anti-tumor [10], anti-bacterial [11], antidepressant [12] and anti-cancer [13]. So, synthesis of 2,3-dihydroquinazolinones (DHQZ) are important and a number of new methods have been reported in this way. Some of biologically active compounds that containing QZ structure are shown in Fig. 1 [14].

Already, some procedures for synthesis of various DHQZ *via* cyclisation of 2-aminobenzamide and aldehyde were reported.

These procedures have been carried out in the presence of a catalyst such as  $\beta$ -cyclodextrin [15], sulfanilic acid [16], tetrabutyl ammonium bromide [17] and thionyl chloride [18]. Nonetheless, several methods of them have different problems like more prolonged reaction, usage of harmful and volatile solvents and low yield of product. Hence, the advancement of such green and clean methodologies, for DHQZ derivatives synthesis remains an interesting challenge.

Using heterogeneous catalysts have been interested in organic reactions due to their benefits such as easiness of its storage, purification steps and plainness separation. Investigators have tried to make these catalysts' size smaller to increase their activity and surface areas.

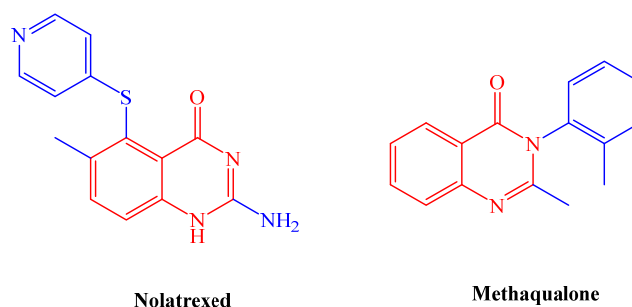


Fig. 1. Some bioactive compounds with QZ structure.

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Nanoparticles (NPs) are mediator of heterogeneous and homogeneous catalysts which can be diffused in the medium of reaction effectively and improve reaction's process [19]. But these kinds of catalysts have a tough procedure to separate due to their tiny measure. Therefore, as a support of catalyst magnetic nanoparticles (MNPs) have been picked [20-23] due to variety of significant advantages like simple synthesis and functionalization, plain procedure to separate by an exterior magnet, high catalytic activity and a high level of chemical stableness [24]. Pure  $\text{Fe}_3\text{O}_4$  NPs are remarkably chemical-active because of their wide surface area to volume proportion. So, it should be noted that they are susceptible to oxidation and aggregate.

For controlling the above restrictions, it's necessary to coat the surface of  $\text{Fe}_3\text{O}_4$  nanoparticles by a suitable protective coating [25]. Recently, several coatings for  $\text{Fe}_3\text{O}_4$  nanoparticles like carbon [26], surfactants [27], biopolymers [23, 28] and silica [20, 22] have been utilized. Dextrin, acting as naturally and renewable biopolymer which is a suitable coating substrate for nano- $\text{Fe}_3\text{O}_4$  particles because nanoparticles are stabilized in solution as well as having free OH groups for functionalization aims.

In current research,  $\text{Fe}_3\text{O}_4$ @nano-dextrin/Ti(IV) or (FND-Ti(IV)) was prepared as a magnetic nano-catalyst and consider its effect in reaction of 2-aminobenzamide with different aromatic aldehydes for DHQZ synthesis.

## 2. Experimental

### 2.1. General

Materials were prepared from chemical companies such as Aldrich, Merck and Fluka. The nano-dextrin was prepared by Yazd Pishgam Sarir Chemical Industrial Company (Yazd, Iran). Fourier-transform infrared spectra were done on a Bruker, Equinox 55 spectrometer. In order to record the  $^1\text{H-NMR}$  spectrum, a Bruker (DRX-400 Avance) NMR was used. Buchi melting point B-540 B.V.CHI device has measured melting points. X-ray diffraction (XRD) schema was acquired by a Philips Xpert MPD diffract meter armed with a  $\text{Cu K}\alpha$  anode ( $k = 1.54 \text{ \AA}$ ) in the  $2\theta$  range from  $10$  to  $80^\circ$ . Mira 3-XMU was used to obtain Field Emission Scanning Electron Microscopy (FESEM). Bruker, S4 Explorer instrument has obtained XRF analysis. The vibrating sample magnetometry (VSM) was carried out on Vibrating Sample Magnetometer (Meghnatis Daghigh Kavir Co. Kashan, I.R.IRAN). ICP analyzes were done with Spectro Ciros CCD ICP equipment. Philips CM120 with LaB6 cathode and accelerating voltage of  $120 \text{ kV}$  was used to acquire TEM images.

### 2.2. Preparation of $\text{Fe}_3\text{O}_4$ @nano-dextrin

First of all, nano-dextrin ( $1.5 \text{ g}$ ) is dissolved in acetic acid solution ( $0.05 \text{ M}$ ,  $100 \text{ mL}$ ) and then  $3.51 \text{ g}$  of  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  and  $1.29 \text{ g}$   $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$  are added. Above mixture is stirred for  $6 \text{ h}$  at  $80^\circ \text{C}$ . As a consequence,  $\text{NH}_4\text{OH}$  ( $6 \text{ mL}$ ,  $25\%$ ) is added drop wise to the reaction solution with continual stirring. Then the mixture is stirred for  $30 \text{ min}$  and after that is chilled to the ambient temperature. Afterwards, nano-dextrin which is coated over magnetic nanoparticles are isolated by using exterior magnet and then it is washed 3 times by using distilled water and dried for  $4 \text{ hours}$  at  $80^\circ \text{C}$  [29-33].

### 2.3. Preparation of FND-Ti(IV)

Under a ventilator system,  $5 \text{ mL}$  of  $\text{TiCl}_4$  was injected into nano- $\text{Fe}_3\text{O}_4$ @nano-dextrin ( $5 \text{ g}$ ) in  $20 \text{ mL}$  of chloroform. Acquired solution was stirred for  $1 \text{ h}$  at ambient temperature. Obtained suspended solution was filtrated, then rinsed by using chloroform. Ultimately made dry at ambient temperature.

### 2.4. General method for DHQZ synthesis

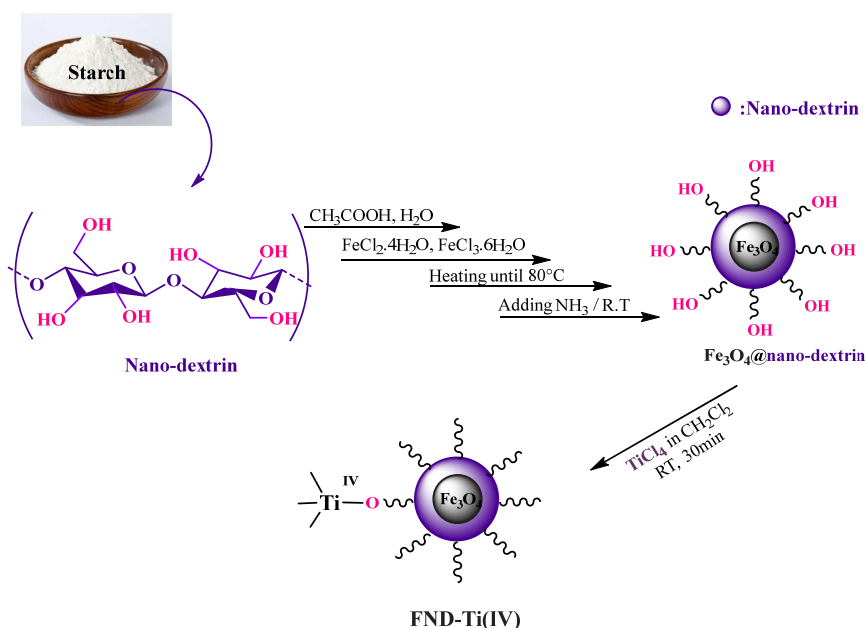
First of all,  $1.0 \text{ mmol}$  of 2-aminobenzamide,  $1.0 \text{ mmol}$  of aromatic aldehyde and  $0.03 \text{ g}$  of FND-Ti(IV) were mixed in ethanol ( $3.0 \text{ mL}$ ). The acquired solution was mixed for  $15 \text{ minutes}$  at room temperature. The reaction progression has been investigated by TLC, and then by using an exterior magnet the catalyst was removed. Finally, water is added to remainder and main product was collected in high yields.

### 2.5. Hot filtration test

To prove recoverability of synthesized nano-catalyst, hot filtration test was carried out. So, a mixture of 2-aminobenzamide ( $1 \text{ mmol}$ ), 4-chlorobenzaldehyde ( $1 \text{ mmol}$ ), ethanol ( $3 \text{ mL}$ ) and FND-Ti(IV) ( $0.03 \text{ g}$ ) was mixed at ambient temperature for  $15 \text{ minutes}$ . Mentioned catalyst was then separated from reaction mixture which was hot, and reaction in filtration was yet checked. No enhancement in conversion was noticed in filtrate.

## 3. Results and discussion

For FND-Ti(IV) synthesis, in a two-step procedure was prepared. First of all,  $\text{Fe}_3\text{O}_4$ @nano-dextrin prepared by co-precipitation of  $\text{Fe}^{3+}$  and  $\text{Fe}^{2+}$  ions in presence of nano-dextrin and was applied as a magnetic support to bond with  $\text{TiCl}_4$  (Scheme 1). The magnetic nano catalyst, FND-Ti(IV), is recognized by various techniques like FE-SEM, FT-IR, TGA, X-Ray Fluorescence, TEM and VSM.

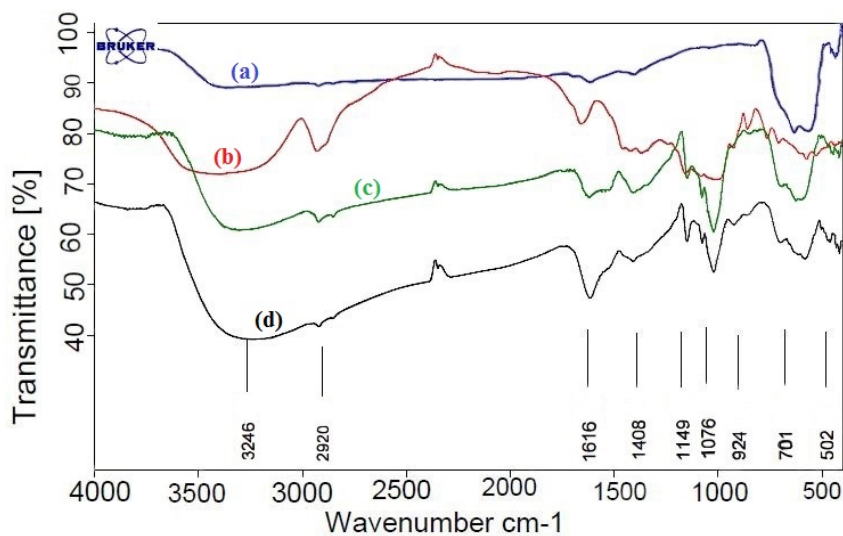


**Scheme 1.** Preparation of FND-Ti(IV).

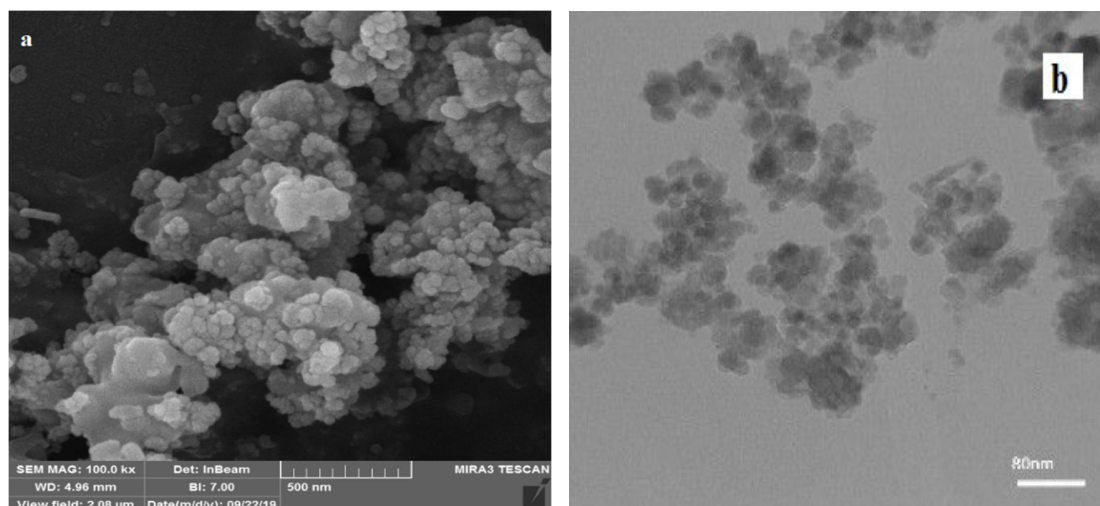
To identify FND-Ti(IV) structure, we have studied FT-IR (ATR) nano-Fe<sub>3</sub>O<sub>4</sub> spectra, nano-dextrin, Fe<sub>3</sub>O<sub>4</sub>@nano-dextrin and FND-Ti(IV) (Fig. 2). A band at 3200-3400 cm<sup>-1</sup> was seen in nano-dextrin FT-IR spectrum which is related to stretching vibrations of OH groups. The absorption bands about 1370-1450 cm<sup>-1</sup> are attributed to CH<sub>2</sub> group's bending stretching bands. C-H aliphatic bonds are appeared 2900 cm<sup>-1</sup>. Absorption band around 1076 cm<sup>-1</sup> indicate stretching vibrations of C-O bonds. For Fe<sub>3</sub>O<sub>4</sub>@nano-dextrin, dextrin absorptions are revealed in addition to Fe/O groups stretching vibrations at 560 cm<sup>-1</sup> showing that nano-dextrin covered magnetic Fe<sub>3</sub>O<sub>4</sub>@nano-dextrin.

The 1616 cm<sup>-1</sup> band probably is result of H-O-H bending vibration of adsorbed water [34]. In accordance with formerly announced FT-IR of Ti(OBut)<sub>4</sub>, the band in 794 cm<sup>-1</sup> is ascribed to C-O-Ti group (stretching vibration) [35,36]. Meanwhile, the bending vibration of 405, O-Ti-O was appeared to 405 cm<sup>-1</sup> [37]. In order to study size of the particle and morphology of nano-dextrin and FND-Ti(IV), TEM and FESEM images of nano-catalyst were shown in Fig. 3. As a consequence, it indicates that the size of catalyst was attained below 100 nm.

The magnetic features of Fe<sub>3</sub>O<sub>4</sub> and FND-Ti(IV) was determined by usage of vibrating sample magnetometer (VSM) (Fig. 4).



**Fig. 2.** FT-IR spectra of (a) nano-Fe<sub>3</sub>O<sub>4</sub> (b) nano-dextrin (c) Fe<sub>3</sub>O<sub>4</sub>@nano-dextrin, (d) FND-Ti(IV).



**Fig. 3.** (a) FESEM and (b) TEM images of FND-Ti(IV).

Both measured samples indicate a super paramagnetic manner, as recognized in comparison with zero coercivity and durability on the magnetization loop. Saturation magnetization amounts of  $\text{Fe}_3\text{O}_4$  and FND-Ti(IV) are  $\sim 50 \text{ emu g}^{-1}$  and  $\sim 10 \text{ emu g}^{-1}$ , severally. It can be seen from results that bonding  $\text{Fe}_3\text{O}_4$  to nano-dextrin/Ti(IV) has negligible efficacy on  $\text{Fe}_3\text{O}_4$  magnetic properties.

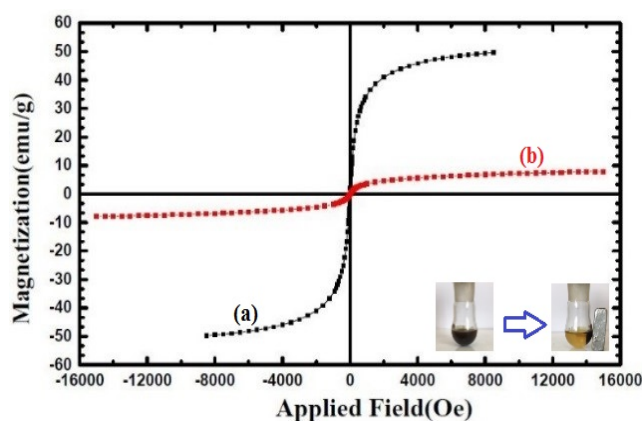
Thermal gravimetric analysis (TG-DTA) schema of FND-Ti(IV) was also performed in range of heating from  $50 \text{ }^\circ\text{C}$  to  $800 \text{ }^\circ\text{C}$  which indicated weight loss percentage (Fig. 5). In accordance with the curve, the 5% losing weight in the temperature ranges  $75\text{--}150 \text{ }^\circ\text{C}$  that can be attributed to the moisture and about 6% weight loss in range of  $140\text{--}195 \text{ }^\circ\text{C}$  attributed to bonding water on catalyst. Two further catalyst weight loss are corresponded to 20% and 15% in temperature in order of  $200\text{--}400$  and  $600\text{--}800 \text{ }^\circ\text{C}$ . Hence, stability of catalyst is ultimately  $100 \text{ }^\circ\text{C}$ .

For obtaining the Fe: Cl: Ti ratio in FND-Ti(IV) by XRF analysis, Kilo Counts Per Seconds values of elements in catalyst were evaluated with KCPS values of same elements in pure samples NaCl,  $\text{TiO}_2$  and Fe. By comparing these results, quantity of Fe, Cl and Ti were acquired in order of 8.71 g (0.15 mol), 5.08 g (0.14 mol) and 4.52 g (0.1 mol). So, molar ratio of Fe: Cl: Ti in catalyst is approximately 1.5: 1.5: 1. Measurement of Fe and Ti amounts in catalyst by usage of Inductively Coupled Plasma shows amount of 195 mg/g for Fe and 105 mg/g for Ti. So, in accordance of ICP, molar ratio of Fe: Ti has gained 1.6: 1.

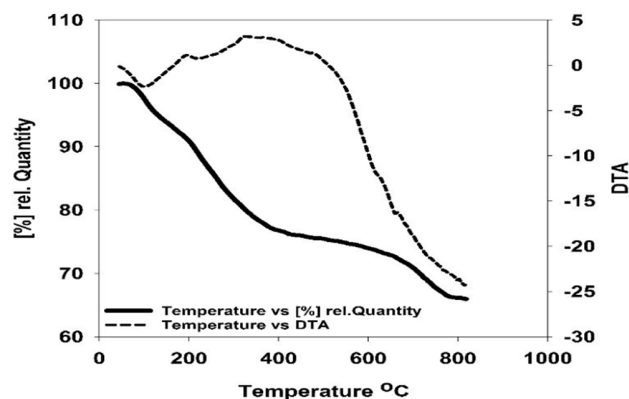
By using X-ray fluorescence (XRF) analysis, catalyst chemical combination has been evaluated (Table 1).

The FND-Ti(IV) efficiency was inquired for DHQZ derivatives synthesis via reaction of 2-aminobenzamide

and aromatic aldehydes. At first, to evaluate FND-Ti(IV) activity, in DHQZ preparation, the condensation of 2-aminobenzamide and 4-chlorobenzaldehyde was done as a model reaction in existence of it under different conditions (Table 2).



**Fig. 4.** The vibrating sample magnetometry (VSM) of (a)  $\text{Fe}_3\text{O}_4$  and (b) FND-Ti(IV).



**Fig. 5.** Thermal gravimetric analysis (TGA) schema of FND-Ti(IV).

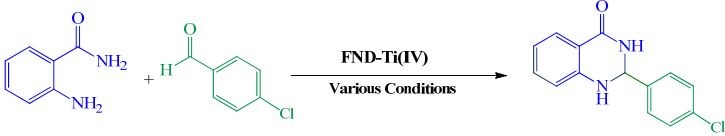
**Table 1.** XRF analysis of catalyst and pure samples NaCl, Fe and TiO<sub>2</sub>.

Element	Catalyst		NaCl		TiO <sub>2</sub>		Fe	
	KCPS	wt%	KCPS	wt%	KCPS	wt%	KCPS	wt%
Cl	43.8	5.08	516.4	62 (Cl)				
Ti	175.0	4.52			2318.3	59.8 (Ti)		
Fe	163.7	8.71					1820.5	97(Fe)

By using different kinds of solvents, ethanol had effective role to form products in excellent yield in ambient temperature (Table 2, Entry 10). Different solvents like H<sub>2</sub>O, CH<sub>3</sub>CN, CHCl<sub>3</sub> and MeOH had low effect on producing products even after extended reaction times in an identical temperature and the yields were low (Table 2, Entries 3-6). As the reaction was completed with no solvent, in temperature of ambient and 60 °C, low yields were obtained (Table 2, Entries 1 and 2). To optimize reaction condition, model reaction was done by usage of various catalyst amount in different times (Table 2). By using no catalyst amount, low product yield was gained (Table 2, Entry 12). But then, more products were gained by adding more amount of catalyst (Table 2, Entries 7-11). Best yield was attained within 15 min by using 0.03 g catalyst (Table 2, Entry 10). On the other hand, when the catalyst amount was increased up to 0.10 g, the products yields didn't change noticeably (Table 2,

Entries 7-9). According to ICP data, 0.03 g of catalyst is containing of  $3.15 \times 10^{-3}$  g of Ti(IV) and equal to  $6.5 \times 10^{-5}$  mol or 0.065 mmol of Ti(IV). Thus, TON and TOF are catalyst are equal to  $14.3 \times 10^5$  and  $95000 \text{ min}^{-1}$ , respectively.

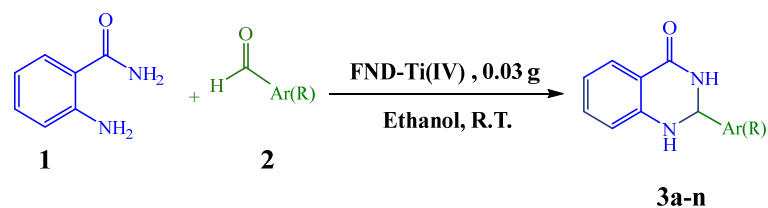
After optimizing reaction conditions, to indicate generality of present work, a variety of aliphatic and aromatic aldehydes have been selected for DHQZ derivatives synthesis by FND-Ti(IV) as catalyst and results are presented in Table 3. This magnetic catalyst could be reused easily. This catalyst property was checked on model reaction. Catalyst magnetism allowed its simple separation by magnetic decantation, washed by CHCl<sub>3</sub> then dried at ambient temperature to arrange a situation for recycling experiments. This process was performed over five runs with no notable catalytic activity loss (Fig. 5). Partial titanium leaching from catalyst and/or catalyst active sites blockage probably are resulted in decreasing catalytic function.

**Table 2.** The reaction of 2-aminobenzamide and 4-chlorobenzaldehyde in the presence of FND-Ti(IV) under different conditions.<sup>a</sup>


Entry	Catalyst (g)	Solvent	Conditions	Time (min)	Yield (%) <sup>b</sup>
1	FND-Ti(IV) (0.04)	-	r.t.	120	30
2	FND-Ti(IV) (0.04)	-	60 °C	70	36
3	FND-Ti(IV) (0.04)	H <sub>2</sub> O	reflux	180	50
4	FND-Ti(IV) (0.04)	CH <sub>3</sub> CN	r.t.	180	40
5	FND-Ti(IV) (0.04)	CHCl <sub>3</sub>	r.t.	120	35
6	FND-Ti(IV) (0.04)	MeOH	r.t.	120	40
7	FND-Ti(IV) (0.10)	EtOH	r.t.	10	65
8	FND-Ti(IV) (0.06)	EtOH	r.t.	10	76
9	FND-Ti(IV) (0.04)	EtOH	r.t.	10	89
10	FND-Ti(IV) (0.03)	EtOH	r.t.	15	93
11	FND-Ti(IV) (0.02)	EtOH	r.t.	15	90
12	None	EtOH	r.t.	240	20

<sup>a</sup>The molar ratios are 2-aminobenzamide (1mmol) and benzaldehyde (1mmol).

<sup>b</sup>Isolated yield.

**Table 3.** Synthesis of DHQZ derivatives using FND-Ti(IV) in ethanol.<sup>a</sup>

Entry	Ar (R)	Product	Time (min)	Yield (%) <sup>b</sup>	TON (TOF) (min <sup>-1</sup> )	m.p.(°C)	Ref.
1	Ph-	3a	15	89	14.3 × 10 <sup>5</sup> (90000)	219-220	[38]
2	4-NO <sub>2</sub> -C <sub>6</sub> H <sub>4</sub> -	3b	12	92	14.1 × 10 <sup>5</sup> (117948)	309-312	[17]
3	4-Cl-C <sub>6</sub> H <sub>4</sub> -	3c	11	90	13.8 × 10 <sup>5</sup> (125874)	193-194	[39]
4	4-Br-C <sub>6</sub> H <sub>4</sub> -	3d	15	93	14.3 × 10 <sup>5</sup> (95384)	199-200	[40]
5	4-Isopropyl-C <sub>6</sub> H <sub>4</sub> -	3e	13	86	13.2 × 10 <sup>5</sup> (101775)	158-160	[38]
6	2-NO <sub>2</sub> -C <sub>6</sub> H <sub>4</sub> -	3f	10	92	14.1 × 10 <sup>5</sup> (141538)	191-192	[41]
7	2-Cl-C <sub>6</sub> H <sub>4</sub> -	3g	12	90	13.8 × 10 <sup>5</sup> (115384)	206-208	[39]
8	3-NO <sub>2</sub> -C <sub>6</sub> H <sub>4</sub> -	3h	13	89	14.3 × 10 <sup>5</sup> (105325)	193-195	[41]
9	3-Br-C <sub>6</sub> H <sub>4</sub> -	3i	15	91	14.0 × 10 <sup>5</sup> (93333)	223-224	[42]
10	2,4-(Cl) <sub>2</sub> -C <sub>6</sub> H <sub>3</sub> -	3j	11	88	13.5 × 10 <sup>5</sup> (123076)	166-169	[43]
11	2,4-(OMe) <sub>2</sub> -C <sub>6</sub> H <sub>3</sub> -	3k	15	84	12.9 × 10 <sup>5</sup> (86153)	185-187	[44]
12	( <i>n</i> -Pentyl-)	3l	10	93	14.3 × 10 <sup>5</sup> (143076)	154	[45]
13	(Cyclohexyl-)	3m	15	90	13.8 × 10 <sup>5</sup> (92307)	174-175	[46]
14	4-HCO-C <sub>6</sub> H <sub>4</sub> -	3n	15	95	14.6 × 10 <sup>5</sup> (97435)	245-246	[40]
15	(H)	3o	8	89	14.3 × 10 <sup>5</sup> (171153)	>160	[47]
16	(Ph-CH <sub>2</sub> CH <sub>2</sub> -)	3p	10	90	13.8 × 10 <sup>5</sup> (138461)	200-202	[47]
17	(Ph-CH=CH-)	3q	10	91	14.0 × 10 <sup>5</sup> (140000)	158-159	[47]

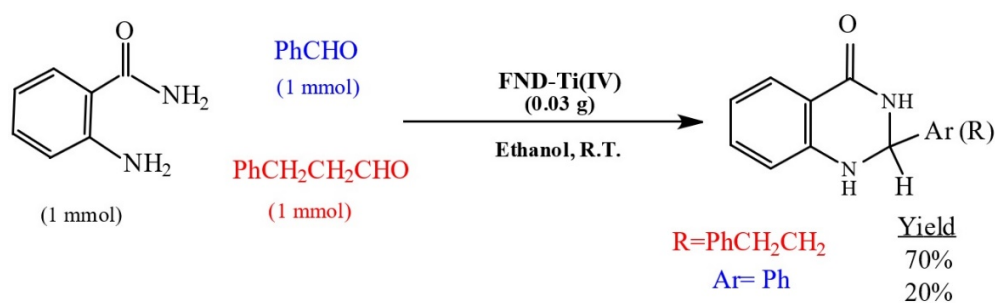
<sup>a</sup>Reaction conditions: 2-aminobenzamide (1.0 mmol), aldehyde (1.25 mmol) and FND-Ti(IV) (0.03 g).

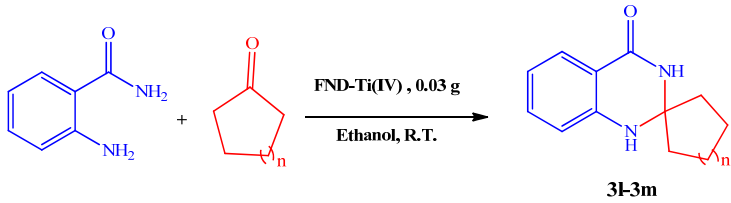
As competition reaction between aromatic and aliphatic aldehydes, we have done the reaction between 3-phenylpropionaldehyde and benzaldehyde with 2-aminobenzamide by using

FND-Ti(IV) as catalyst in EtOH at room temperature for 10 minutes (Scheme 2). In this competitive experiment we have found that after 10 minutes, the conversion

yield for 3-phenylpropionaldehyde is higher than benzaldehyde.

As well as aldehydes, ketones have been used in DHQZ production. If cyclic ketones like cyclopentanone or cyclohexanone have been applied, DHQZ with spiro skeleton have been produced (Table 4).

**Scheme 2.** Competitive reaction between aromatic and aliphatic aldehydes.

**Table 4.** Spiro-2,3-dihydroquinazolin-4(1*H*)-ones synthesis catalyzed by FND-Ti(IV) in ethanol.<sup>a</sup>


Entry	n	Time (min)	Yield (%)	TON (TOF) (min <sup>-1</sup> )	m.p.(°C)		Ref.
					Found	Reported	
1	1	20	93	14.3 × 10 <sup>5</sup> (71538)	270-275	258-259	[11]
2	2	20	90	13.8 × 10 <sup>5</sup> (69230)	227-230	220-221	[11]

<sup>a</sup>Reaction conditions: 2-Aminobenzamide (1.0mmol), Ketone (1.25mmol) and FND-Ti(IV) (0.03g).

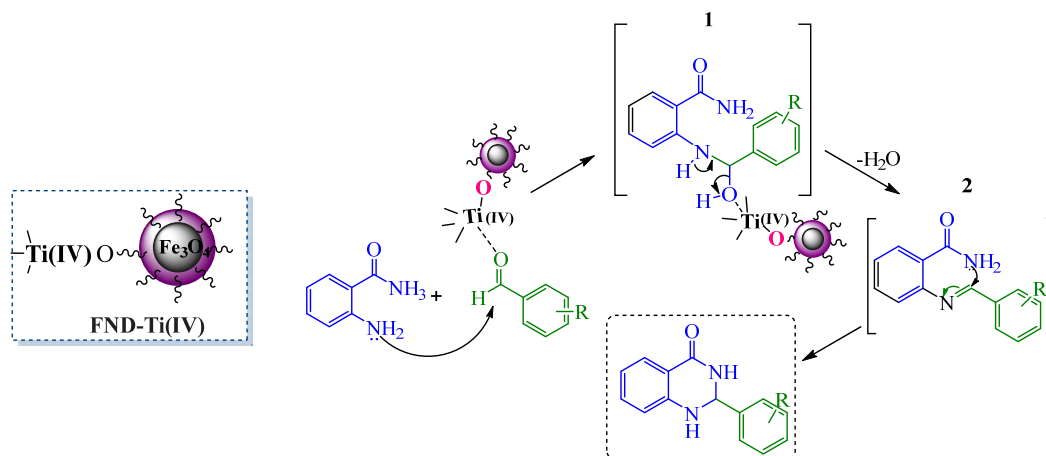
A presumable mechanism for DHQZ preparation in presence of FND-Ti(IV) was depicted in scheme 3. TiCl<sub>4</sub> in FND-Ti(IV) that can act in the role of Lewis acid activates C=O group in benzaldehyde and after that intermediate 1 and then intermediate 2 are obtained from Mannich-type condensation of 2-aminobenzamide and benzaldehyde. Ultimately, by an intramolecular cyclization DHQZ derivatives were prepared.

As shown in Table 5, FND-Ti(IV) usability compared with other catalyst that have been reported. Yields,

times of reaction and terms of conditions have been analyzed.

#### 4. Conclusions

In conclusion, we have reported an effortless method for DHQZ derivatives synthesis in existence of a new heterogeneous catalyst. FND-Ti(IV) has been used as an efficacious magnetic catalyst to promote the reactions such as multicomponent reactions. Some privileges of the presented protocol are simplicity of operation, mild reaction condition and high yields of products.

**Scheme 3.** Proposed mechanism for DHQZ derivatives synthesis 3a-k.**Table 5.** Comparison of FND-Ti(IV) with formerly reported procedures.

Entry	Catalyst	Solvent	Conditions	Time (h)	Yield (%) <sup>b</sup>	Ref.
1	NaHSO <sub>4</sub>	EtOH	r.t.	0.5-5.5	97	[48]
2	H <sub>3</sub> BO <sub>3</sub>	-	120 °C	0.08	90	[49]
3	KI/I <sub>2</sub>	H <sub>2</sub> O	r.t.	2-12	95	[50]
4	HgCl <sub>2</sub>	EtOH	60 °C	1-2	94	[51]
5	ZrO <sub>2</sub> -Al <sub>2</sub> O <sub>3</sub>	EtOH	80 °C	2.5-3.5	94	[52]
6	FND-Ti(IV)	EtOH	r.t.	0.25	93	This work

## Acknowledgements

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