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# The use of SrFe<sub>12</sub>O<sub>19</sub> magnetic nanoparticles as an efficient catalyst in the modified Niementowski reaction

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Ghodsi Mohammadi Ziarani, Department of Chemistry, Alzahra University, Vanak Square, P.O. Box 1993893973, Tehran, Iran. Email: gmziarani@hotmail.com Nanomagnetic  $SrFe_{12}O_{19}$  was synthesized through a simple sol–gel auto-combustion and then characterized by FT-IR, XRD, EDXA, VSM, BET and SEM image. Moreover, it was used as catalyst for the first time in the modified Niementowski reaction to investigate its catalytic activity. According to the high yield of quinazolinone products obtained within short reaction times, it was found that  $SrFe_{12}O_{19}$  can be used as an effective and green nanocatalyst in organic reactions. The nanomagnetic catalyst can be easily separated from the reaction mixture using an external magnet.

#### KEYWORDS

magnetic nanoparticles, nanocatalyst, Niementowski reaction, Quinazolinone, SrFe<sub>12</sub>O<sub>19</sub>

# **1** | **INTRODUCTION**

Ferromagnetic ceramics or ferrites are a composition of iron (III) oxide  $(\alpha$ -Fe<sub>2</sub>O<sub>3</sub>) and another element(s) including Mn, Ni, Si, Zn, Cu, Co, Ba, Mg and Sr. According to some advantages of ferrites such as inexpensive cost, simple preparation procedure, high corrosion and heat resistance, they are used more than ferromagnetic alloys, whereas the saturation magnetization of ferrite compounds is very lower than that of ferromagnetic alloys.<sup>[1]</sup> Strontium hexaferrite or SrFe<sub>12</sub>O<sub>19</sub> is a type of magnetic ceramics which was discovered by Went and coworkers in 1952.<sup>[2]</sup> SrFe<sub>12</sub>O<sub>19</sub> was the first ferrite prepared for commercial production around 1980s since its anisotropic constant, remanence, and Curie temperature is relatively high.<sup>[1]</sup> Thus, up to now, various preparation approaches have been reported for the synthesis of SrFe<sub>12</sub>O<sub>19</sub> magnetic nanoparticles (MNPs) including hydrothermal,<sup>[3]</sup> chemical co-precipitation,<sup>[4]</sup> sol-gel,<sup>[5]</sup> salt melt methods,<sup>[6]</sup> and ball milling.<sup>[7]</sup> The sol-gel auto-combustion procedure is the best choice for this aim due to its relatively simple procedure, low reaction temperature which leads to reducing energy consumption, and short reaction time. This approach involves gelling and subsequent

combustion of an aqueous solution containing the desired metal salts and organic complexant, which produces a voluminous and fluffy product with high specific surface area.

In 1894, Niementowski published a synthetic procedure for the preparation of quinazoline derivatives through the reaction of anthranilic acid and ketones.<sup>[8]</sup> According to the importance of quinazoline and quinazolinone compounds in medicinal chemistry,<sup>[9–11]</sup> Niementowski reaction conditions were developed and modified to gain a diverse library of such compounds.<sup>[12–16]</sup> According to our previous research in the field of catalysis,<sup>[17–24]</sup> and also due to the importance of nanomagnetic catalysts in organic syntheses,<sup>[25–35]</sup> herein, we want to introduce the first application of prepared SrFe<sub>12</sub>O<sub>19</sub> MNPs as catalyst in the modified Niementowski reaction.

# 2 | EXPERIMENTAL

#### 2.1 | Materials and methods

The chemical substances employed in this work were obtained from Merck Company and used with no purification. Melting points were determined by capillary tube method with an Electrothermal 9200 apparatus. Infrared (IR) spectra were recorded on KBr disks using a Fouriertransform (FT)-IR Bruker Tensor 27 instrument. <sup>1</sup>H NMR (250 MHz) and <sup>13</sup>C NMR (62.5 MHz) spectra were run on a Bruker DPX using tetramethylsilane (TMS) as an internal standard in DMSO- $d_6$ . Elemental analyses were performed on a ThermoFinigan Flash EA 1112 series elemental analyzer. The X-ray powder diffraction (XRD) pattern of the prepared SrFe<sub>12</sub>O<sub>19</sub> nanoparticles was collected by a Philips X'pert MPD diffractometer using Cu Ka radiation  $(\lambda = 0.15478 \text{ nm})$ . The scanning electron microscopy (SEM) image was taken with SEM (MV2300). Surface area and pore size distribution of the magnetic nanospheres were determined by N<sub>2</sub> adsorption-desorption at 77 K with Brunauer-Emmett-Teller (BET) and Barret-Joyner Halenda (BJH) methods using a BELSORP analysis software. The magnetic measurement of sample was carried out in a vibrating sample magnetometer (VSM) (Meghnatis Daghigh Kavir Co.; Kashan Kavir; Iran) at room temperature.

# 2.2 | Preparation of SrFe<sub>12</sub>O<sub>19</sub> nanoparticles

The synthetic procedure was directed using the method published before.<sup>[36]</sup> Briefly,  $Fe(NO_3)_3.9H_2O$  and  $Sr(NO_3)_3$  powders (Fe/Sr = 10) were dissolved in deionized water (100 ml for 0.1 mole of Fe<sup>3+</sup>) and stirred at 60 °C. After mixing the solutions together (at 60 °C), citric acid was added to it in a proper molar ratio with metal nitrates and stirred vigorously. Then, the solution was cooled down to room temperature. Subsequently, ammonium hydroxide (NH<sub>4</sub>OH) was added dropwise till the pH was adjusted at 7. Then, a cationic surfactant (surfactant/strontium =0.4) was added to the solution. After evaporation of the solvent, upon ignition, a fluffy powder was obtained which then, ground and calcined at 900 °C for 1 h.

# 2.3 | General procedure of modified Niementowski reaction

SrFe<sub>12</sub>O<sub>19</sub> nanoparticles (0.02 g) was activated under reduced pressure at 100 °C for removal of the adsorbed water and then cooled to room temperature. Then, isatoic anhydride (1 mmol, 0.16 g) and phenyl hydrazine (1 mmol, 0.1 ml) was added to the catalyst and heated at 120 °C under stirring for about 5 min. Afterward, benzaldehyde derivative (1 mmol) was added to the mixture and the reaction was continued at 120 °C for appropriate time as shown in Table 2. The reaction completion was monitored by TLC. The resulting crude product was dissolved in hot EtOH and then, SrFe<sub>12</sub>O<sub>19</sub> nanoparticles were easily separated from the solution using an external magnet. The solution was cooled to gain the pure crystalline product. The products were characterized using melting point and FT-IR spectra and also new compound was analyzed through the mentioned techniques in addition to the  ${}^{1}$ H and  ${}^{3}$ C NMR spectra.

# **2.3.1** | **2-(4-chlorophenyl)-3-(phenylamino)-2,3-dihydroquinazolin-4(1***H***)-one 4 g**

FT-IR (KBr,  $\nu$ , cm<sup>-1</sup>) = 3299 (NH stretching), 3048 (aromatic =C-H stretching), 2929 and 2835 (asymmetric and symmetric stretching of aliphatic C-H, respectively), 1653 (amide C = O stretching), 1607, 1493 and 1435 (aromatic C = C Stretching), 1304 (NH bending vibration), 750 (C-Cl stretching). <sup>1</sup>H NMR (250 MHz, DMSO- $d_6$ ,  $\delta_{\rm H}$ , ppm) = 4.75 (s, 1H, NH), 5.93 (s, 1H, CH), 6.45–7.92 (m, 13H, ArH and NH), and 7.95 (d, 1H, ArH). <sup>13</sup>C NMR (62.5 MHz, DMSO- $d_6$ ,  $\delta_{\rm C}$ , ppm) = 74.29, 113.97, 114.38, 119.54, 121.73, 127.95, 128.83, 129.00, 134.40, 135.18, 137.81, 145.40, 146.34, and 163.54. Anal. Calcd. for C<sub>20</sub>H<sub>16</sub>N<sub>3</sub>OCl (349.10): C, 68.67; H, 4.61; N, 12.01. Found: C, 67.88; H, 4.89, N, 11.84.

# 2.3.2 | 2-(2,3-dichlorophenyl)-3-(phenylamino)-2,3-dihydroquinazolin-4(1*H*)-one 4 h

FT-IR (KBr,  $\nu$ , cm<sup>-1</sup>) = 3350 (NH stretching), 3059 (aromatic =C-H stretching), 2986 and 2955 (asymmetric and symmetric stretching of aliphatic C-H, respectively), 1649 (amide C = O stretching), 1609, 1511 and 1419 (aromatic C = C Stretching), 1321 (NH bending vibration), 755 (C-Cl stretching). <sup>1</sup>H NMR (250 MHz, DMSO- $d_6$ ,  $\delta_{\rm H}$ , ppm) = 6.32 (s, 1H, NH), 6.58 (s, 1H, CH), 6.61–7.92 (m, 12H, ArH), and 7.95 (s, 1H, NH). <sup>13</sup>C NMR (62.5 MHz, DMSO- $d_6$ ,  $\delta_{\rm C}$ , ppm) = 71.96, 113.77, 114.33, 114.97, 119.38, 121.63, 125.07, 127.70, 128.51, 129.33, 130.71, 134.41, 138.57, 144.93, 146.33, and 164.20. Anal. Calcd. for C<sub>20</sub>H<sub>15</sub>N<sub>3</sub>OCl<sub>2</sub> (349.10): C, 62.51; H, 3.93; N, 10.94. Found: C, 62.69; H, 4.01, N, 10.86.

#### **3 | RESULTS AND DISCUSSION**

#### **3.1** | Application of SrFe<sub>12</sub>O<sub>19</sub> magnetic nanoparticles in the modified Niementowski reaction

In order to study the catalytic activity of the  $SrFe_{12}O_{19}$  nanoparticles in the modified Niementowski reaction, initially, the reaction of isatoic anhydride **1**, phenyl hydrazine **2** and 2-nitrobenzaldehyde **3a** was selected and optimized as the model reaction. As shown by the results in Table 1, the use of solvents for this reaction was not effective as well as a solvent free system. Therefore, among the tested conditions, solvent-free system at 120 °C provided the best results in the presence of  $SrFe_{12}O_{19}$ , with the shortest reaction time (15 min) in high yield of the

TABLE 1 The optimization of reaction condition for the synthesis of dihydroquinazolinone 4a



Entry	Solvent	Catalyst*	Conditions	Time (min)	Yield (%)
1	H <sub>2</sub> O	SrFe <sub>12</sub> O <sub>19</sub> MNPs	Reflux	120	N.R.
2	EtOH	SrFe <sub>12</sub> O <sub>19</sub> MNPs	Reflux	60	80
3	H <sub>2</sub> O/EtOH	SrFe <sub>12</sub> O <sub>19</sub> MNPs	Reflux	80	Trace
4	-	SrFe <sub>12</sub> O <sub>19</sub> MNPs	120 °C	15	91
5	-	-	120 °C	60	N.R.

\*0.02 g



TABLE 2 Synthesis of dihydroquinazolinone products 4a-h in the presence of SrFe<sub>12</sub>O<sub>19</sub> MNPs

Entry	No.	R	Time (min)	Yield (%)	m.p.	m.p. [ref.]
1	<b>4</b> a	2-NO <sub>2</sub>	15	91	217-219	180–182 <sup>[37]</sup>
2	4b	4-OMe	30	88	220-223	216-218 <sup>[38]</sup>
3	4c	3-NO <sub>2</sub>	105	70	180–184	177–179 <sup>[37]</sup>
4	4d	Н	15	87	195–197	179–180 <sup>[38]</sup>
5	<b>4</b> e	4-NO <sub>2</sub>	30	85	224–227	220-222 <sup>[37]</sup>
6	<b>4f</b>	4-F	15	88	210-214	205-207 <sup>[37]</sup>
7	4 g	4-cl	30	85	211–214	New
8	4 h	2,3-Cl <sub>2</sub>	15	90	185–186	New



**SCHEME 2** The proposed mechanism for the modified Niementowski reaction in the presence of the  $SrFe_{12}O_{19}$  MNPs



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TABLE 3 Comparison of different conditions in the Niementowski reaction to obtain derivative 4b

Entry	Catalyst	Solvent	Condition	Time (min)	Yield (%)	Ref.
1	Bentonite	H <sub>2</sub> O	)))*/60 °C	30	75	[38]
2	KAl(SO <sub>4</sub> ) <sub>2</sub> .12H <sub>2</sub> O	EtOH	Reflux	420	91	[37]
3	CeO <sub>2</sub>	EtOH	Reflux	300	73	[44]
4	Citric acid/Al <sub>2</sub> O <sub>3</sub>	-	Grinding	10	90	[45]
5	SrFe <sub>12</sub> O <sub>19</sub> MNPs	-	120 °C	15	91	This work

\*))) = ultrasonic radiation



FIGURE 1 Separated  $SrFe_{12}O_{19}$  from the reaction mixture using an external magnet

product. Moreover, since the reaction was not progressed in the absence of the catalyst (Entry 5, Table 1), the importance of  $SrFe_{12}O_{19}$  nanoparticles as the catalyst in this reaction was proved.

Likewise, to assess the generality and versatility of this reaction, different dihydroquinazolinone products **4a–h** were prepared successfully under solvent-free condition in the presence of  $SrFe_{12}O_{19}$  MNPs (Scheme 1). The obtained results (Table 2) were good in terms of the reaction times and yields. The new products were characterized by FT-IR, CHN and NMR spectroscopy data.

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According to the previous organic reactions catalyzed by different types of Strontium,<sup>[3,39–42]</sup> it was found that Strontium as an alkaline earth metal can active carbonyl groups through the formation of Sr-O bond.<sup>[39]</sup> Accordingly, the probable mechanism for this reaction is proposed in Scheme 2. Initially, Sr atoms of  $SrFe_{12}O_{19}$  MNPs<sup>[43]</sup> activate both carbonyl group of the isatoic anhydride 1. Then, NH<sub>2</sub> group of phenyl hydrazine 2 attacks to the activated carbonyl group followed by decarboxylation to give intermediate 6. Afterwards, the NH<sub>2</sub> groups of the latter is added to the activated carbonyl group of aldehyde 3 to produce imine 7. Furthermore, an intramolecular Michael addition is occurred to afford the desired product 4.



FIGURE 2 XRD pattern of SrFe<sub>12</sub>O<sub>19</sub> nanoparticles

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FIGURE 3 SEM image of SrFe<sub>12</sub>O<sub>19</sub> nanoparticles

Moreover, the modified Niementowski reaction (according to the Scheme 1) has been considered in several conditions in literature as shown in Table 3. In comparison with the mentioned methods, the present methodology has several advantages such as the use of inexpensive magnetic catalyst, simple procedure, shorter reaction time and excellent yields with high purity of the products. For the main advantage of the present technique, we can mentioned that the nanomagnetic catalyst is separable from the reaction mixture by the use of an external magnet (Figure 1).

# **3.2** | Characterization of the SrFe<sub>12</sub>O<sub>19</sub> nanoparticles

The XRD pattern of the prepared  $SrFe_{12}O_{19}$  MNPs is displayed in Figure 2. The XRD pattern of  $SrFe_{12}O_{19}$  crystal with a magnetoplumbite structure showed twelve characteristic peaks including  $2\theta = 30.55^{\circ}$ ,  $32.53^{\circ}$ ,  $34.39^{\circ}$ ,  $35.47^{\circ}$ ,  $37.33^{\circ}$ ,  $40.63^{\circ}$ ,  $42.67^{\circ}$ ,  $55.51^{\circ}$ ,  $57.01^{\circ}$ ,  $63.37^{\circ}$ ,  $67.87^{\circ}$  and  $72.85^{\circ}$  which can be indexed to the (110), (107), (114), (201), (203), (205), (206), (217), (304), (220), (204) and (317) lattice planes, respectively. The results firmly prove the successful formation of  $SrFe_{12}O_{19}$  MNPs.

Furthermore, as shown in the SEM image (Figure 3) the  $SrFe_{12}O_{19}$  MNPs are spherical particles with average size of 70 nm. The FT-IR spectrum of the  $SrFe_{12}O_{19}$  MNPs is shown in Figure 4 in which the sharp bands at 441, 551, 601 cm<sup>-1</sup> are related to the  $SrFe_{12}O_{19}$ .<sup>[46]</sup>

To study the hysteresis loops and magnetic properties, SrFe<sub>12</sub>O<sub>19</sub> nanoparticles was characterized by VSM at room temperature. As shown in Figure 5 the magnetic saturation of the compound mentioned above is about 79 emu/g. According to the hysteresis loops in the magnetization curves of SrFe<sub>12</sub>O<sub>19</sub> nanoparticles, it is a typical feature of ferromagnetic compound.

Figure 6 shows the  $N_2$  adsorption–desorption isotherm of the SrFe<sub>12</sub>O<sub>19</sub> nanoparticles. The observed isotherm is classified as III-type by IUPAC; accordingly, SrFe<sub>12</sub>O<sub>19</sub> nanoparticles are non-porous materials which only have external



FIGURE 4 FT-IR spectrum of the SrFe<sub>12</sub>O<sub>19</sub> nanoparticles



**FIGURE 5** The room temperature hysteresis loops of  $SrFe_{12}O_{19}$  nanoparticles



**FIGURE 6**  $N_2$  adsorption/desorption isotherms and BJH pore size distributions of the SrFe<sub>12</sub>O<sub>19</sub> nanoparticles

surface. The BET surface area of  $SrFe_{12}O_{19}$  nanoparticles was calculated as 10.4 m<sup>2</sup>/g.

The catalyst recyclability was also examined under the optimized conditions for the model reaction. In this regard, synthesis of compound **4a** was performed in the first run for four times to recover about 0.07 gr SrFe<sub>12</sub>O<sub>19</sub>. Subsequently, the catalyst was washed with hot EtOH and then reused. As shown in Table 4, the catalytic activity drops slightly from the first use to the second use, which means this catalyst is recyclable. In addition, the element leaching was studied using Energy-dispersive X-ray spectroscopy (EDX analysis) after first run (Table 5). The ratio of Fe to Sr in the fresh catalyst is about 12.4:1.4 while in the recycled catalyst it is 11.75:1.75. It means that the catalyst elements was not significantly leached to the reaction mixture during synthesis of compound **4a**. The presence of carbon and nitrogen atoms in the EDXA of the recycled

TABLE 4 Reuse of SrFe<sub>12</sub>O<sub>19</sub> MNPs for the synthesis of 1a

Entry	Time (min)	Yield (%)
1	15	91
2	15	84

TABLE 5 The results of EDXA for catalyst

	Elements (weight%)				
Sample	Fe	Sr	0	С	Ν
Fresh SrFe <sub>12</sub> O <sub>19</sub>	62	7	31	-	-
Recycled SrFe <sub>12</sub> O <sub>19</sub>	0.47	0.07	9.42	18.32	71.72

catalyst is for the remained organic compounds which were not removed during simple washing process.

#### 4 | CONCLUSION

As a conclusion, nanomagnetic  $SrFe_{12}O_{19}$  was prepared through a simple sol–gel auto-combustion method. Characterization of  $SrFe_{12}O_{19}$  MNPs was performed using XRD, BET, VSM, EDXA, SEM and FT-IR analyses. SEM image showed their average particle size of 70 nm. Afterward, the catalytic activity of such MNPs was studied in the modified Niementowski reaction; the quinazolinone products were obtained in high yields within shorter reaction time compared to the results reported in literature. Therefore,  $SrFe_{12}O_{19}$ MNPs can be used as the green and eco-friendly efficient catalyst which are easily separated from the reaction mixture by the use of an external magnet.

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