**ORIGINAL PAPER** 



# New, stable and reusable magnetic Fe<sub>3</sub>O<sub>4</sub>/PEG@ CPTMS-thioaminophenol@Ni nanocatalyst for the synthesis of dispiro-cyclopropanes's Meldrum's acid

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#### Abstract

Magnetic  $Fe_3O_4/PEG@CPTMS$ -thioaminophenol@Ni nanocatalyst as a heterogeneous, stable and reusable nanocatalyst was formed. This nanocatalyst was characterized by FT-IR, XRD, TGA, ICP, SEM, EDS and VSM techniques and was used for six times without decreasing activity. This nanocatalyst was used successfully for the synthesis of 3,3,10,10-tetramethyl-13-aryl(alkyl)-2,4,9,11-tetraoxadispiro[5.0.5<sup>7</sup>.1<sup>6</sup>]tridecane-1,5,8,12-tetraones (dispiro-cyclopropane's based on Meldrum's acid) by the reaction of various aldehydes and Meldrum's acid in the presence of triethylamine and cyanogen bromide in excellent yield.

#### **Graphic abstract**



**Keywords**  $Fe_3O_4 \cdot Dispiro-cyclopropane \cdot Meldrum's acid \cdot 2-Amino thiophenol$ 

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# Introduction

Fe<sub>3</sub>O<sub>4</sub>/polyethylene glycol (PEG-400) as a core–shell magnetic nanoparticles was introduced as a greenly nanocatalyst [1]. Hybrid inorganic–organic magnetized nanoparticles had

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constantly reading case reason to their biomedical utilization, to protein separation [2], therapeutic transfer of DNA and drugs [3], inhibition of their aggregation by building a clog barrier among the prepared biocompatibility, renderer probable for link aim region and avoid the reconnoiter [4]. PEG is a biotic biocompatible polymer and is utilization as cover for nanoparticles which is an egregious between the more proper polymers because of its remarkable attribute for instance, greatly hydrophilic nature (hydrophilicity), flexibility and adaptability, safe and nonimmunogenicity [5]. PEG is sorption to the superficial of the micelle to form a compound micelle. The absorption probably outcomes not just of the hydrophilic sling of polymer moving toward the cations yet additionally from the coat among the cations and the single-electron couple from the  $O_2$  particle in PEG [6]. PEG sorption per absorbing can draw out flow time in a blood circulation system. Indeed, the modification magnetite nanoparticles by PEG is able to sorption contrarily the protein sorption and in this manner protection a strategic distance from their acknowledgment by macrophage cells and yet encourage the nanoparticle take-up to diagnosis malignancy cells in a tumor remedy [7, 8]. The visualization from nanoparticles by PEG expands the particles sparsity, biocompatible and material soundness [9].

Cyclopropane backbone is one of the most important organic structures that can be found in natural product and some organic compounds structures. The cyclopropane is an essential structure in numerous natural compounds which included antifungal, antibacterial, antiviral and some chemical restraint exercises [10–12]. Several routes were reported for the synthesis of some full-substituted cyclopropanes in the absence of catalyst [13–16]. Previously, according to one-pot reaction of Meldrum's acid, aldehydes and cyanogen bromide (BrCN) in the presence of Et<sub>3</sub>N, cyclopropanes were prepared [17]. Instead, in this reaction, the long reaction time, unsatisfactory yields of products were insufficient of the reaction.

Meldrum's acid is a heterocyclic compound and it has organic and pharmaceutical properties. Likewise, it executes as a wellspring of geminal dicarboxylic functional groups which gets a great deal of consideration in the science and pharmacology [18–22]. Owing to the importance of cyclopropanes and dispiro-cyclopropanes, initially, we presented synthesis of MNP–Ni and characterized the nanocatalyst structure. The MNP–Ni is used as an efficient heterogeneous nanocatalyst for the synthesis of dispiro-cyclopropanes from the one-pot reaction of Meldrum's acid with various aldehydes and BrCN in the presence of  $Et_3N$  in excellent yield.

# **Results and discussion**

This article described the synthesis and characterization of new heterogeneous and recyclable nanocatalyst (MNP-Ni) and its using to synthesis of dispiro-cyclopropanes based on Meldrum's acid. The reaction scheme of the magnetically nanocatalyst is shown in Scheme 1.

Ni-complex supported on magnetic nanoparticle (MNP) is totally characterized by FT-IR, TGA, VSM, ICP, XRD, SEM and EDS analyses. The FT-IR spectrum of MNP was recorded and is shown in Fig. 1 (curve a). According to the information obtained from the IR and according to curve a, the peak observed in the nearly 3500 and 571 cm<sup>-1</sup> regions is related to the vibrational stretching frequencies of OH and Fe–O, respectively. In curve b, due to PEG coupling, the intensity of the peaks of C-H and C-O stretching frequencies are appeared at nearly 2900 and 1100 cm<sup>-1</sup>, respectively. The C-H absorption bands of *n*-propyl linker are also shown at 2922 cm<sup>-1</sup>. The Si–O absorption bands are also observed at 1100, 1200, 1108 cm<sup>-1</sup> and corresponded to the symmetric and asymmetric stretching vibrations of the mesoporous structure of Si–O. In curve c, the vibration band at nearly 1450 and 1603 cm<sup>-1</sup> corresponded to symmetric and unsymmetric C=C stretching frequency and confirmed the linking of 2-aminothiophenol. Apparently, a peak at 2400 cm<sup>-1</sup> attributed to S-H stretching frequency in curves c and d. The chemical analysis of SH group in catalyst indicated that the 2-aminothiophenol is attached to the spacer by the NH<sub>2</sub> group. For these experiments, we used the sodium plumbite solution for the indication of SH group.

Scanning electron microscopy (SEM) images of the synthesized MNP–Ni are shown in Fig. 2. The size and morphology of particles was thought out with SEM. These nanoparticles had a middle size of where about 19–20 nm. As well ready particles include of small particles with regularly sphere morphology. This observation display that the PEG surfactant can sorption adoptive on preferred shape of crystal, which can interdict the loose growth of Fe<sub>3</sub>O<sub>4</sub> nanoparticles.

To indicate the supporting Ni on ligand, energy-dispersive spectrum (EDS) analysis of  $Fe_3O_4/PEG@CPTMS-2$ -aminothiophenol@Ni was carried out (Fig. 3). The results indicate the presence of C, N, O, Si, S, Fe and Ni elements in MNP and furthermore demonstrate the rate level of each individually 14.08%, 4.54%, 26.98%, 6.46%, 19.65%, 27.94% and 0.34%, respectively. The amount of Ni is 1.18% which was determined by inductively coupled plasma-optical emission spectrometry (ICP-OES).

Figure 4 shows the TGA curves for MNP–Ni accommodating with TGA bends, the essential weight dwindles to temperatures less 370 °C is because of the loss of water and natural solvents such as EtOH and the second weight loss is increasing the temperature from 370 to 600 °C is extensively to the decomposition of covalently organic compound (Fig. 4).

The measurement of the magnetization curve of  $Fe_3O_4/PEG@CPTMS-2$ -aminothiophenol@Ni is presented in Fig. 5 and is gained from the vibration sample



Scheme 1 Synthesis of MNPs@2-aminothiophenol@Ni



Wavenumbers (cm-l)







Fig. 2 SEM images of Fe<sub>3</sub>O<sub>4</sub> (a), Fe<sub>3</sub>O<sub>4</sub>/PEG@CPTMS-2-aminothiophenol@Ni (b, c)



Elt	Line	Int	W%	A%
С	Ka	388.7	14.08	25.87
N	Ka	150.8	4.54	7.15
0	Ka	1386.1	26.98	37.21
Si	Ka	927.2	6.46	5.08
s	Ka	3230.6	19.65	13.52
Fe	Ka	4003.6	27.94	11.04
Ni	Ka	45.7	0.34	0.13
v			100.00	100.00

Fig. 3 EDS spectrum of  $Fe_3O_4/PEG@CPTMS-2$ -aminothiophenol@Ni



Fig.4 TGA analysis of  $Fe_3O_4/PEG@CPTMS-2$ -aminothiophenol@Ni

magnetometer (VSM). It is determined that the severity of the saturation magnetization (Ms) of  $Fe_3O_4/PEG@$  CPTMS-2-aminothiophenol@Ni were 7.0 emu g<sup>-1</sup>.







Fig.6 XRD patterns of  $Fe_3O_4$  and  $Fe_3O_4/PEG@CPTMS-2-aminothiophenol@Ni$ 

Moreover, the magnetic particles have super paramagnetism due to the zero forcible energy and since it is free of hysteresis. One can conclude this kind of nanoparticles is paramagnetism. The magnetization saturation (MS) 8000 estimated to be and copious magnetization pointer the high-speed reaction the nanocatalysts in the magnetic part.

According to the analysis of  $Fe_3O_4/PEG@CPTMS-2$ -aminothiophenol@Ni and its comparison with the XRD patterns of  $Fe_3O_4$ , the peaks at 30.2°, 35.5°, 40.1°, 57.3°, and 62.9° were respectively equivalent to the (220), (311), (400), (511), and (440) crystal planes of a pure  $Fe_3O_4$  with

a cubic spinal structure. According to XRD pattern after having properties of crystalline catalyst maintained and treated with Fe<sub>3</sub>O<sub>4</sub> courier region are in place [23, 24]. According the XRD pattern of Fe<sub>3</sub>O<sub>4</sub>/PEG@CPTMS-2-aminothiophenol@Ni, all the peaks of Fe<sub>3</sub>O<sub>4</sub> have been observed. It should be noted that the peaks at 34°, 39.6°, 56.1°, 62.9° and 63.5° correspond to orthorhombic crystal system of Fe<sub>3</sub>O<sub>4</sub>/PEG@CPTMS-2-aminothiophenol@Ni nanocatalyst (Fig. 6).

Synthesis of 3,3,10,10-tetramethyl-13-aryl(alkyl)-2,4,9,11-tetraoxadispiro[ $5.0.5^7.1^6$ ]tridecane-1,5,8,12tetraone compounds by catalytic amounts of Fe<sub>3</sub>O<sub>4</sub>/PEG@ CPTMS-2-aminothiophenol@Ni

We performed the cyclopropanation reaction in the presence of new heterogeneous catalyst of Fe<sub>3</sub>O<sub>4</sub>/PEG@ CPTMS-2-aminothiophenol@Ni. We checked catalytic activity as a new heterogeneous catalyst in the one-pot synthesis of dispiro-cyclopropanes's Meldrum's acid. Optimization of the catalyst for the synthesis of the desired cyclopropane scaffold in the attendance of various catalytic amount of Fe<sub>3</sub>O<sub>4</sub>/PEG@CPTMS-2-aminothiophenol@Ni is shown in Table 1. The best results were gained in the presence of Fe<sub>3</sub>O<sub>4</sub>/PEG@CPTMS-2-aminothiophenol@Ni (0.03 mg), EtOH (3 min) and/or MeOH (10 min) at 0 °C (Table 1, entries 3 and 5, respectively). It should be noted that the use of higher amounts of catalyst (0.07 mg) gained 80% product (Table 1, entries 2 and 4, respectively). The maximum reaction yield in the absence of catalyst was 60% (Table 1, entry 1). We concluded that the 0.03 mg of catalyst was the best optimum condition. After successful synthesis of cyclopropane scaffold in the presence of catalyst with good performance of various aldehydes in the reactions with Meldrum's acid, the results are reported in Table 2. Results indicated Table 1 The effect of different parameters<sup>a</sup> on the synthesis of compounds 3



Entry	Cat. (mg)	Solvent	Base	Temp. (°C)	Time (min)	Yield (%)
1	0	EtOH	Et <sub>3</sub> N	0	400	60
2	0.07	EtOH	Et <sub>3</sub> N	0	30	80
3	0.03	EtOH	Et <sub>3</sub> N	0	3	95
4	0.07	MeOH	Et <sub>3</sub> N	0	30	80
5	0.03	MeOH	Et <sub>3</sub> N	0	10	95
6	0.03	EtOH	NaOAc	0	7	92
7	0.03	EtOH	DABCO	0	20	72
8	0.03	EtOH	N-methylmorpholine	0	15	83

Bolded cases indicated the best condition

<sup>a</sup>The molar ratio of aldehyde 1, Meldrum's acid 2, BrCN, and Et<sub>3</sub>N were 1: 2: 1: 1, respectively

that the yields of aldehydes including electron-withdrawing substituents were higher than that of electron-donating substituents (Table 2). Comparison of several methods for synthesis of cyclopropane is shown in Table 3. For example, the yield of 3a in the presence of catalyst was better than to the absence of catalyst under the same reagents and condition.

The reaction mechanism of the cyanogen bromide (BrCN) is shown in Scheme 2. In this reaction, the BrCN plays a major role. The reaction of BrCN with Meldrum's acid 2 formed the salt of 4. Previously, the structure of 4 was characterized (Scheme 2) [25, 27–29]. Similar to the synthesis of dispirocyclopropanes 3 under catalyst-free condition [17], a proposed reaction mechanism for the formation of 3 in the presence of nanocatalyst is shown in Scheme 3. There are two possible pathways for continuation of reaction via an intermediate **A**. The product of C-attack was observed (path *a*). Owing to the inductive effect of oxygen atom into ring, the O-attack cannot occur and compounds 5 were not observed (Scheme 3) [17].

#### Hot filtration

To test the saturation of Ni in nanocatalyst for cyclopropanation, we performed the product in this test, and in the middle of this time, we isolated 45% of the catalyst from the reaction mixture, resulting from the absence of any advance in the reaction. The yield of reaction in this step was 45%, emphasizing the leaching of Ni was insignificant and is outlined in Fig. 7.

#### **Recyclability of the catalyst**

Renewable catalysts in commercial use are important after completion, and the catalyst reaction is discretized by a magnet and washed with  $Et_2O$  and for six runs without the decrease in activity used, as shown in Fig. 8. Ni percentage is reused six runs specified by (ICP-OES) Ni percentage before using in reaction (1.18%) and after 6 runs (1%). According to the SEM analysis, the particle size before the reaction was 19 nm, and after six times 32 nm was determined.

#### **Experimental**

#### **Materials and instruments**

All starting materials and solvents used in this work purchased from Merck, Aldrich and Fluka Companies. FT-IR spectra were done with KBr pellets utilizing a Nexus 670 FT-IR spectrometer (Urmia University, Urmia, IRAN). Thin-layer chromatography (TLC) was used for the control of reaction progression was Al sheet-coated  $G/UV_{254}$  plates. The nanocatalyst morphology was investigated by an examining SEM (Day Petronik Company, Tehran, IRAN) using FESEM-TESCAN MIRA3. The <sup>1</sup>H (300) and <sup>13</sup>C NMR (75 MHz) spectra were recorded on BRUKER NMR-Spectrometer (Urmia University, Urmia, IRAN). The

 Table 2
 Synthesis of 3 with the reaction of various aldehydes (1) with Meldrum's acid (2) in the presence of nanocatalyst



Entry	Product (3)	Time (min)	M.P. (°C) <sup>a</sup> , Lit.	Yield (%) <sup>b</sup>
1	$\rightarrow$	4	216–218 217–218 [17]	90
2		5	217–219 216–218 [17]	88
3		5	212–213 212–214 [17]	90
4	$\rightarrow$	5	205–207 204–205 [17]	92
5		2	191–192 192–194 [17]	95

#### Table 2 (continued)

Entry	Product (3)	Time (min)	M.P. (°C) <sup>a</sup> , Lit.	Yield (%) <sup>b</sup>
6		2	202–204 202–203 [17]	96
7		3	197–199 198–199 [17]	94
8	g	3	205–206 204–205 [17]	91
9	CN h	4	192–194 192–193 [17]	80

#### Table 2 (continued)

Entry	Product (3)	Time (min)	M.P. (°C) <sup>a</sup> , Lit.	Yield (%) <sup>b</sup>
10		3	177–179 178–180 [17]	95
11		3	179–180 178–179 [17]	93
12		5	177–178 178–180 [17]	89
13	o o o o o o o o o o o o o o	5	127–129 126–128 [17]	91

<sup>a</sup>All products are identified by comparing their physical and spectral data with valid compounds

<sup>b</sup>Yield refers to obtained product

thermogravimetric examination (TGA) bends are recorded utilizing a Shimadzu DTG60 instrument (Day Petronik Company, Tehran, IRAN). The EDX (energy-dispersive X-ray) of the MNPs were recorded using a FESEM-TESCAN MIRA3 (Day Petronik Company, Tehran, IRAN). The percentage of Ni was measured by inductively coupled plasma-optical emission spectrometry (ICP-OES) (Tarbiat Modares University, Tehran, IRAN). PEG with molecular weight 400 was applied in whole reaction. Vibrating sample magnetometer **Table 3** Comparison of severalmethods for the synthesis ofcyclopropane

Entry	Cat. (mg)	Reagent and condition	Yield (%)	References
1	_	Dimedone, BrCN, Et <sub>3</sub> N	75	[25]
2	Fe <sub>3</sub> O <sub>4</sub> /PEG@CPTMS- 2-aminothiophenol@Ni	BrCN, Et <sub>3</sub> N, 0 °C	95	This work
3	-	Meldrum's acid, BrCN, Et <sub>3</sub> N	60	[17]
4	Rh <sub>2</sub> (Opiv) <sub>4</sub>	70 °C, 3 h, PhF	94	[26]
5	-	BrCN, EtONa	80	[27]



Scheme 2 Mechanism of the formation of salt 4 in the presence of BrCN [17, 25, 27–29, 32]

(VSM)Lbkfb modeh-Meghnatis Daghigh Kavir company (Azad University of Mahabad, Mahabad, IRAN, X-ray Powder Diffraction (XRD) was measured and accomplished by a X'PertPro (University of Tabriz, Tabriz, IRAN). The nomenclature of compounds 3 was done by means of Chem-Draw Professional version 15.0.

#### Preparation of nanoparticle Fe<sub>3</sub>O<sub>4</sub>@PEG (MNPs)

In this method, a  $Fe_3O_4$  nanoparticle fusion was synthesized using  $FeCl_3 \cdot 6H_2O$  and  $FeCl_2 \cdot 4H_2O$  [30]. The magnetic nanoparticles of  $Fe_3O_4$  synthesized in combination with solvent and PEG 400 (1.0 g) were mixed into ultrasonic apparatus for 30 min, and then the mixture was added to the ultrasonic apparatus for 1 h and then the magnetic product was separated by a magnet and dried for 9 h at 87 °C [23, 31].

#### Preparation of MNPs@(CH<sub>2</sub>)<sub>3</sub>Cl

After formation of magnetic deposition (5.3 g), 3-chloropropyl trimethoxysilane (CPTMS) (5.0 g) to 90 ml n-hexane was added and the reaction mixture was combined with nitrogen gas for 24 h under reflux, then the sediment was separated by magnet. It was dried at 67 °C for 6 h.

#### Preparation of Fe<sub>3</sub>O<sub>4</sub>@PEG-2-aminothiophenol

In a round-bottom flask, a mixture of MNPs@(CH<sub>2</sub>)<sub>3</sub>Cl (1 g) with 2-aminothiophol (1 g) with Et<sub>3</sub>N (2 ml) in EtOH (50 ml) was refluxed and the resulting cooking was separated from the EtOH by magnet. For 12 h, it was dried at 70 °C.

### Preparation of Fe<sub>3</sub>O<sub>4</sub>@PEG-2-aminothiophol-Ni

The precipitate from the previous step was mixed with the solution of 20 ml of Ni  $(NO_3)_2 \cdot 6H_2O$  in ethanol and was refluxed for 20 h and dried at a temperature of 60 °C. The final MNPs–Ni catalyst was synthesized.

# General procedure for the preparation of dispiro-cyclopropane compounds (3)

In a 25-ml round-bottom flask equipped with stirrer, 0.05 g (0.48 mmol) cyanogen bromide (BrCN) was dissolved in 5 ml methanol at 0 °C. Then, 0.288 g (0.96 mmol) Meldrum's acid, 0.106 g (0.48 mmol) aldehyde were dissolved in 10 ml methanol, added 0.04 g (0.63 mmol) triethylamine and then, the amount of 0.0003 g of the catalyst  $Fe_3O_4@$  PEG-2-aminothiophenol was added into a funnel. This solution was then added dropwise into the BrCN solution in round-bottom flask at 0 °C to room temperature (Attention!! The cyanogen bromide is toxic. Reactions should be carried out in a well-ventilated hood). The reaction control was monitored by TLC. After completion of the reaction, the catalyst was separated by an outer magnet and washed with few ml of methanol. After 5 min, the white precipitate formed, filtered off, washed by fresh methanol and dried.



Scheme 3 Proposed reaction mechanism for the formation of 3 in the presence of nanocatalyst



Fig. 7 Hot filtration of  $Fe_3O_4/PEG@CPTMS-2$ -aminothiophenol@Ni



Fig. 8 Recycling of  $Fe_3O_4$ /PEG@CPTMS-2-aminothiophenol@Ni

In summary, we prepared magnetically nanoparticles  $Fe_3O_4$  by core–shell method in excess PEG as the protective significant advantages of the nanocatalyst consist of the short reaction time and high yields of products, recyclable catalyst at minimum six times without decrease in catalyst activity. This nanocatalyst is an efficient heterogeneous catalyst for the synthesis of dispiro-cyclopropanes from the one-pot response of Meldrum's acid with various aliphatic and aromatic aldehydes and BrCN in the presence of triethylamine.

#### **Compliance with ethical standards**

**Conflict of interest** The authors declare that they have no conflict of interest.

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