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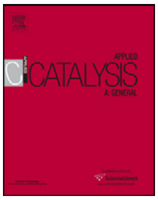

Nano n-propylsulfonated γ-Fe_2O_3 as magnetically recyclable heterogeneous catalyst for the efficient synthesis of β-phosphonomalonates

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ABSTRACT

Nano n-propylsulfonated γ-Fe_2O_3 as a new sulfonated nanomagnetic iron oxide was synthesized directly through ring opening reaction of 1,3-propanesultone with nano magnetic γ-Fe_2O_3 and used as magnetically recyclable heterogeneous catalyst for the efficient one-pot synthesis of β-phosphonomalonates. The catalyst was easily isolated from the reaction mixture by magnetic decantation using an external magnet and reused at least five times without significant degradation in the activity.

1. Introduction

Phosphonates exhibit a wide range of notable biological properties which expand their applications as enzyme inhibitors, metabolic probes [1–3], peptide mimetics [4], antibiotics, and pharmacologic agents [5,6] in addition to their traditional roles as intermediates in organic synthesis [7]. Extensive efforts have been made to introduce convenient and efficient routs to synthesis of phosphonates. Direct phosphorus-carbon bond formation represents the most versatile and powerful technique for the synthesis of phosphonates. Amongst the methods for P–C bond formation, phospha-Michael addition, that is, the addition of a phosphorous nucleophile to an electron-deficient alkene, has evoked remarkable attention of organic chemists [8–22]. Synthesis of β-phosphonomalonates by this method has been commonly promoted by bases [8–13], BrØnsted/Lewis acids [14–16], transition metals [17,18], radical initiators such as AIBN [19,20] and microwave radiation [21]. Even though, phospha-Michael addition could be proceed by these methods, many of these reagents cannot be reused, and in many instances, long reaction time, drastic reaction conditions and sometimes, according to the nature of the catalyst, tedious workup is needed. Therefore, the development of a new method to overcome these shortcomings still remains as an ongoing challenge for the synthesis of these significant scaffolds.

Magnetic nanoparticles (MNPs) have attracted much attention by researchers in different areas including physics, biomedicine, biotechnology, material science and catalysis, because of the large ratio of surface area to volume, paramagnetic behavior and low toxicity [23–25]. In recent years, MNPs acquired organic chemists' attention as a new alternative to porous materials for supporting catalytic transformations [26–34]. The magnetic nature of MNPs allows a convenient method for removing and recycling MNPs-supported catalysts by applying an appropriate magnetic field. In contrast to filtration and centrifugation methods, this kind of separation is not time-consuming and prevents the loss of solid catalyst in the process. It also enhances products purity and optimizes operational costs. In addition, the MNPs-supported catalysts show high dispersion and reactivity with a high degree of chemical stability.

On the other hand, n-propylsulfonated surface materials with propanesulfonate moieties are organic–inorganic hybrid materials that have been applied as effective solid acid catalysts in organic transformations [35–38]. In these types of solid acid catalysts, the reactive centers are highly mobile similar to that of homogeneous catalysts. At the same time these species have the advantage of being recyclable in the same fashion as heterogeneous catalysts. In general, synthesis of n-propylsulfonated surface materials with propanesulfonate moieties was conducted by SH oxidation of supported mercaptopropyl [37–39] or directly through ring opening reaction of 1,3-propanesultone with surface hydroxyl groups [35,36]. In both cases, sulfonic acid groups are introduced on the surface *via* covalently bonds. However, in SH oxidation method, imperfect oxidation of SH groups decreases the efficiency of the catalyst.

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Combining characteristic properties of organic–inorganic hybrid materials and nanomagnetic iron oxides as a supporting material, herein, we report the synthesis of nano *n*-propylsulfonated γ-Fe_2O_3 (NPS-γ-Fe_2O_3) directly through ring opening reaction of 1,3-propanesultone by nano magnetic γ-Fe_2O_3. We have also used these newly synthesized sulfonated nanomagnetic iron oxide as a magnetically recyclable heterogenous catalyst for the efficient one-pot synthesis of β-phosphonomalonates.

2. Experimental

2.1. Synthesis of NPS-γ-Fe_2O_3

The maghemite (γ-Fe_2O_3) nanoparticles were synthesized by a reported chemical co-precipitation technique of ferric and ferrous ions in alkali solution with minor modifications [41,42]. $FeCl_2{\cdot}4H_2O$ (9.25 mmol) and $FeCl_3{\cdot}6H_2O$ (15.8 mmol) were dissolved in deionized water (150 mL) under Ar atmosphere at room temperature. A NH_4OH solution (25%, 50 mL) was then added dropwise (drop rate = 1 mL min^{-1}) to the stirring mixture at room temperature to reach the reaction pH to 11. The resulting black dispersion was continuously stirred for 1 h at room temperature and then heated to reflux for 1 h to yield a brown dispersion. The magnetic nanoparticles were then purified by a repeated centrifugation (1730–3461 g, 20 min), decantation, and redispersion cycle 3 times. The as-synthesized sample was heated at 2 °C min^{-1} up to 200 °C and then kept in the furnace for 3 h to give a reddish-brown powder. The sulfonation of maghemite nanoparticles was conducted using the reaction of γ-Fe_2O_3 with 1,3-propanesultone. γ-Fe_2O_3 (6 g) was suspended in 600 mL of 0.1 M toluene solution of 1,3-propanesultone and the colloidal solution was refluxed for 48 h. The *n*-propylsulfonated γ-Fe_2O_3 was isolated and purified by repeated washing (first in CH_3CN and then in water) and centrifugation.

2.2. Ion-exchange pH analysis of the catalyst

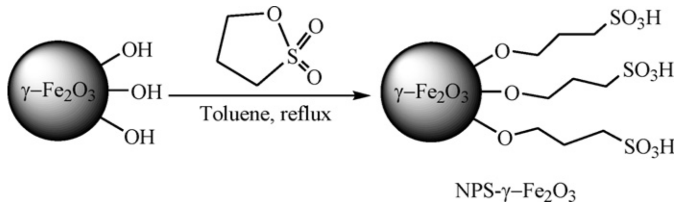
To an aqueous solution of NaCl (1 M, 25 mL) with a primary pH 5.93, the catalyst (500 mg) was added and the resulting mixture was stirred for 2 h after which the pH of solution decreased to 2.25. This is equal to a loading of 0.28 mmol SO_3H g^{-1}.

2.3. Back titration analysis of the catalyst

NaOH solution (10 mL, 0.1 M) was added to the catalyst (100 mg) in an Erlenmeyer flask. Excess amount of the base was neutralized by addition of HCl solution (0.1 M) to the equivalence point of titration. The required volume of HCl to this point was 9.75 mL.

2.4. General procedure for the synthesis of β-phosphonomalonates

NPS-γ-Fe_2O_3 (1 mol%) was added to a mixture of aldehyde (1 mmol), malononitrile (1 mmol) and triethyl phosphite (1 mmol). The mixture was stirred at 60 °C for the appropriate time (Table 2). The reaction was monitored by TLC. After completion, the reaction mixture was diluted with EtOH. The catalyst was separated by an external magnet, washed with EtOH, dried and re-used for a consecutive run under the same reaction conditions. Evaporation of the solvent of the filtrate under reduced pressure gave a crude product. The pure products (1–12) were isolated by chromatography on silica gel eluted with *n*-hexane:EtOAc (1:2).



Scheme 1.

3. Results and discussion

Due to the reasonable needs to clean and green recovery of the heterogenous catalyst, especially acid catalysts, we synthesized NPS-γ-Fe_2O_3 as a new sulfonated nanomagnetic iron oxide simply by a direct method through the ring opening reaction of 1,3-propanesultone with nano magnetic γ-Fe_2O_3 in refluxing toluene (Scheme 1). The synthesized NPS-γ-Fe_2O_3 was then characterized by different methods such as XRD, SEM, TEM and FT-IR. The amount of sulfonic acid loaded on the surface of nano magnetic γ-Fe_2O_3 was determined by TG analysis and confirmed by ion-exchange pH, back titration and elemental analysis.

3.1. X-ray diffraction (XRD) analysis

Fig. 1 shows the XRD pattern of nano *n*-propylsulfonated γ-Fe_2O_3. Diffraction peaks at around $2\theta = 30.4°$, 35.8°, 43.3°, 54.0°, 57.6°, 63.2° corresponding to the (2 2 0), (3 1 1), (4 0 0), (4 2 2), (5 1 1) and (4 4 0) are readily recognized from the XRD pattern. The observed diffraction peaks agree with the cubic structure of maghemite (JCPDS file No 39-1364) with a unit cell dimension of 8.35 Å and the space group of *P*4132 (2 1 3). The average crystallite size was calculated to be 10.7 nm using the Scherrer equation in which $K = 0.9$ and $\lambda = 0.154$.

3.2. SEM and TEM

The size and structure of the NPS-γ-Fe_2O_3 were also evaluated using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). According to the SEM (Fig. 2) and TEM (Fig. 3), it was observed that the synthesized NPS-γ-Fe_2O_3 has nano dimension ranging from ∼35 to 100 nm.

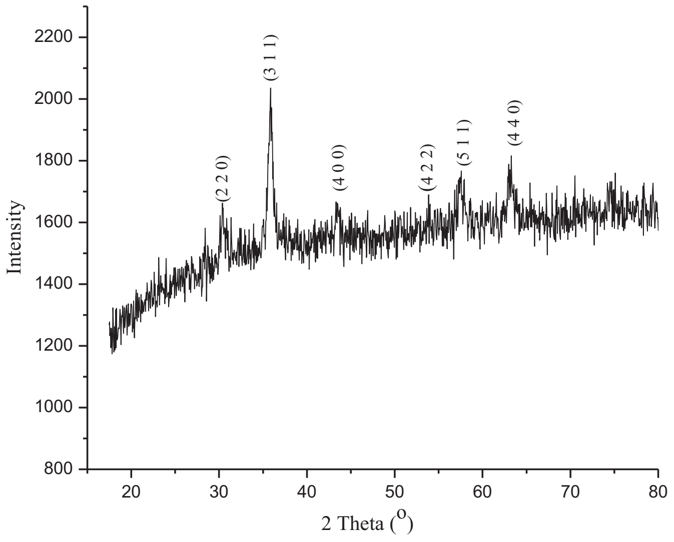


Fig. 1. XRD spectra of NPS-γ-Fe_2O_3.

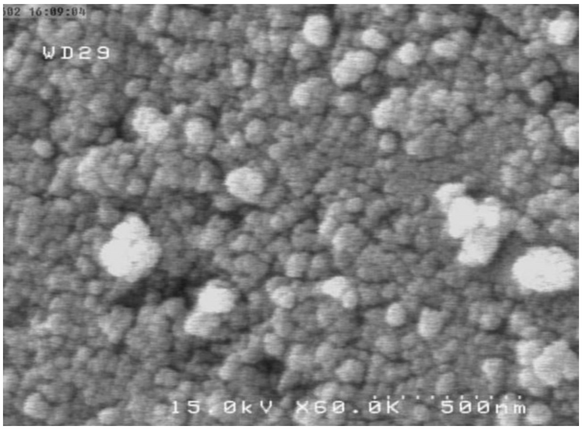


Fig. 2. SEM image of NPS-γ-Fe_2O_3.

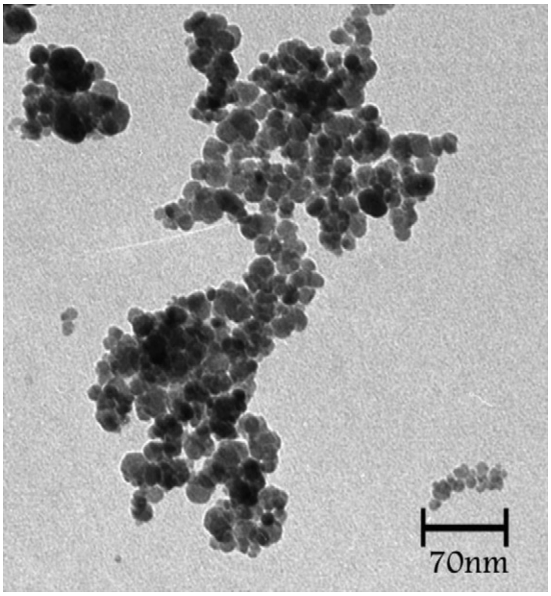


Fig. 3. TEM image of NPS-γ-Fe_2O_3.

3.3. *FT-IR spectroscopy*

In FT-IR of NPS-γ-Fe_2O_3 (Fig. 4), characteristic adsorption bands due to the stretching vibrations of Fe–O bond in γ-Fe_2O_3 are observed at 638.94 cm^{-1}. The peaks placed in 1047 and 1220 cm^{-1} are related to the stretching of the S=O bonds. A peak appeared at 3470 cm^{-1} is due to the stretching of OH groups in the SO_3H.

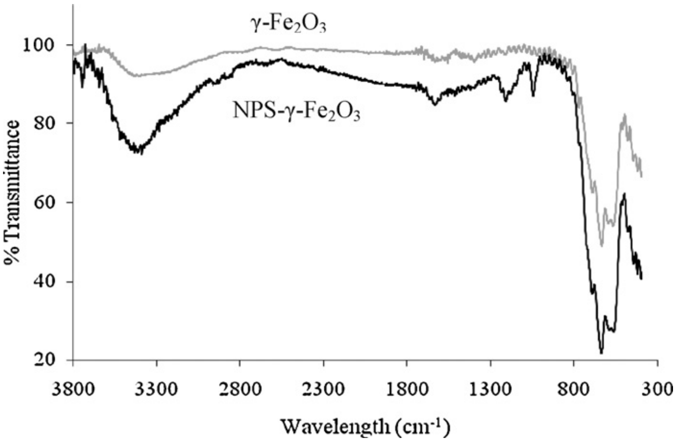


Fig. 4. FT-IR spectra of γ-Fe_2O_3 and NPS-γ-Fe_2O_3.

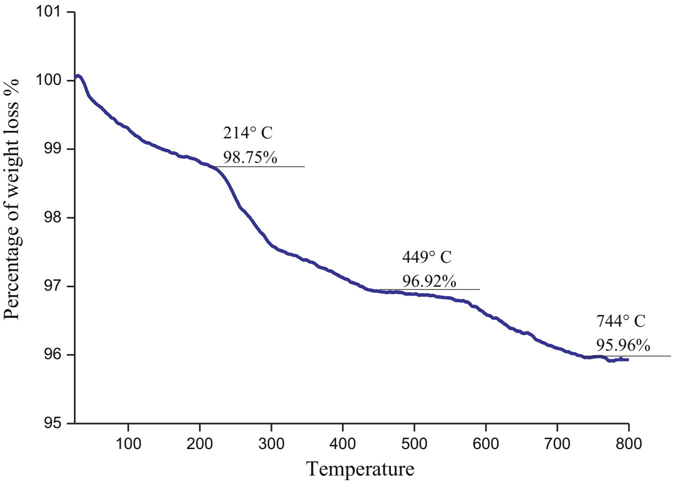


Fig. 5. TGA diagram of NPS-γ-Fe_2O_3.

3.4. *Thermo-gravimetric analysis (TGA)*

The thermal behavior of NPS-γ-Fe_2O_3 is shown in Fig. 5. A significant decrease in the weight percentage of the *n*-propylsulfonated γ-Fe_2O_3 at about 150 °C is related to desorption of water molecules from the catalyst surface. This was evaluated to be ∼1.3% according to the TG analysis. In addition, the analysis showed two other decreasing peaks. First peak appears at temperature around 300 °C due to the decomposition of sulfuric acid and formation of sulfur dioxide. This is followed by a second peak at 660 °C, corresponding to the loss of the organic spacer group. According to the TGA, the amount of sulfuric acid functionalized on γ-Fe_2O_3 is evaluated to be 0.22 mmol g^{-1}. These results are in agreement with those of ion-exchange pH, back titration and elemental analysis (S=0.721% and C=0.928%).

3.5. *Catalytic activity of NPS-γ-Fe_2O_3 for the one-pot synthesis of β-phosphonomalonates*

As a part of our ongoing program directed towards the development of new methods for the synthesis of phosphonate derivatives, we have recently introduced micellar solution of sodium stearate for the one-pot synthesis of β-phosphonomalonates [43] and heterogeneous catalysts [$H_3PMo_{12}O_{40}$, $HClO_4/SiO_2$, aminopropylated silica (AP-SiO_2)] for the two-pot synthesis of β-phosphonomalonates [44–46]. In this relation, in order to show the merit of synthesized NPS-γ-Fe_2O_3 as magnetically recyclable heterogeneous catalyst in organic reactions, we used this new sulfonated nanomagnetic iron oxide for the one-pot synthesis of β-phosphonomalonates directly from aldehydes, malononitrile and trialkyl phosphites.

For this purpose, one-pot reaction of benzaldehyde, malononitrile and triethyl phosphite was chosen to optimize the reaction conditions such as temperature and solvent (Table 1). We found that in the presence of NPS-γ-Fe_2O_3, the best yield of the corresponding β-phosphonomalonate was obtained at 60 °C under solvent-free conditions (entry 1). In order to show the role of the catalyst, similar reactions in the absence of the catalyst or in the presence of nanomagnetic γ-Fe_2O_3 were also examined. Under these conditions, the reactions led to the formation of the desired product in low yields after a long reaction time (entries 8 and 9).

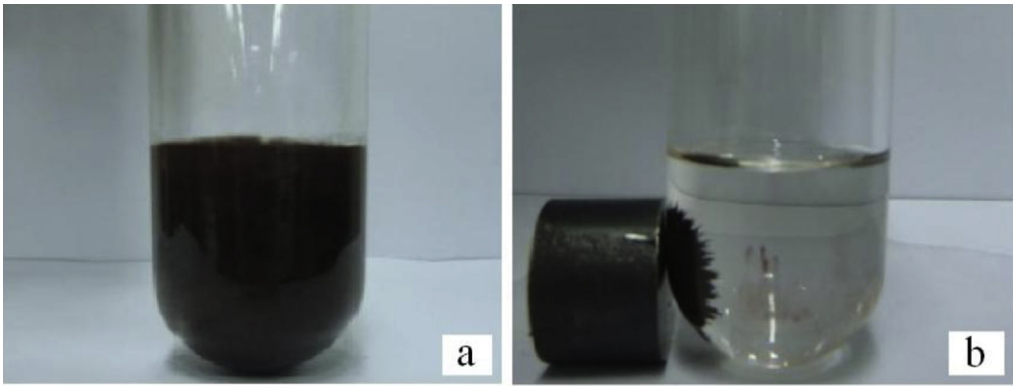


Fig. 6. (a) Reaction mixture, (b) Separation of NPS-γ-Fe_2O_3 from the reaction mixture by an external magnetic field.

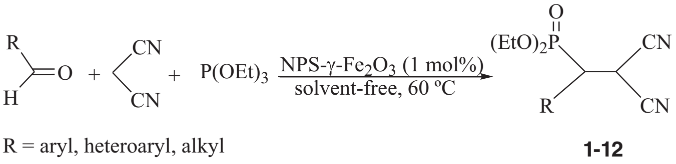
Table 1
One-pot reaction of benzaldehyde, malononitrile and triethyl posphite under different conditions.

Entry	Catalyst	Solvent	Time (h)	Yield[a] (%)
1	NPS-γ-Fe_2O_3	–	1	90
2	NPS-γ-Fe_2O_3[b]	–	1	65
3	NPS-γ-Fe_2O_3	H_2O	3	51
4	NPS-γ-Fe_2O_3	Toluene	24	84
5	NPS-γ-Fe_2O_3	*n*-Hexane	24	52
6	NPS-γ-Fe_2O_3	CH_3CN	24	71
7	NPS-γ-Fe_2O_3	EtOH	24	65
8	γ-Fe_2O_3	–	24	62
9	–[c]	–	24	64

[a] Isolated yield, conditions: benzaldehyde (1 mmol), malononitrile (1 mmol), triethyl phosphite (1 mmol), catalyst (1 mol%, except for entry 9), 60 °C (except for entry 3).
[b] Conditions: room temperature.
[c] No catalyst.

It is important to note that the magnetic property of NPS-γ-Fe_2O_3 facilitates its efficient recovery from the reaction mixture during work-up procedure. In the presence of an external magnet, NPS-γ-Fe_2O_3 moved onto the magnet steadily and the reaction mixture turned clear within 10 s (Fig. 6). The catalyst was isolated by simple decantation. After washing with EtOH and then drying for 30 min at 110 °C, NPS-γ-Fe_2O_3 was reused without any significant deactivation even after five runs. The average isolated yield for five successive runs was 85.6%, which clearly demonstrates the practical recyclability of this catalyst (Fig. 7).

The reaction of a variety of aldehydes with malononitrile and triethyl phosphite was then investigated to confirm the generality of the present method (Scheme 2). The results of this study are depicted in Table 2.



1-12

Scheme 2.

As shown in Table 2, differently substituted benzaldehydes with electron-donating and electron-withdrawing groups underwent successful Knoevenagel-phospha-Michael reaction with malononitrile and triethyl phosphite (entries 1–8). The catalyst was compatible with functional groups such as Cl, Br and O–Me. No competitive nucleophilic methyl ether cleavage was observed in the substrate possessing an aryl–O–Me group (entry 7), despite the strong nucleophilicity of phosphites. Acid-sensitive aldehydes, such as furan-2-carbaldehyde and pyridine-3-carbaldehyde underwent smooth reactions without any decomposition or polymerization under the present reaction conditions (entries 9 and 10). This method is also applicable for the synthesis of β-phosphonomalonates from the reaction of triethyl phosphite with aliphatic aldehydes and malononitrile (entries 11 and 12). The reaction of ketones with malononitrile and triethyl phosphite was also studied under the same conditions. However, the results were not as positive as those presented above.

Furthermore, we have evaluated the generality of the presented method for the one-pot synthesis of β-phosphonomalonates from

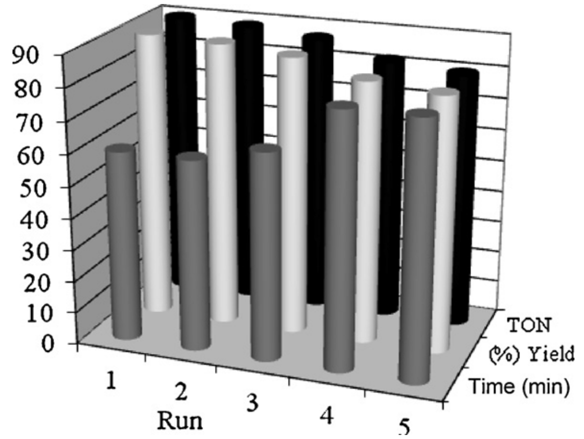
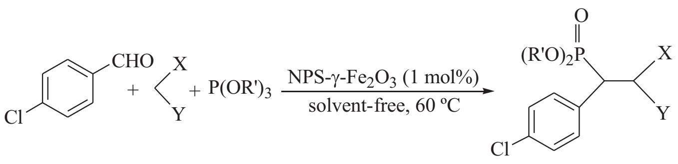


Fig. 7. Reusability of NPS-γ-Fe_2O_3 as a magnetically recyclable heterogeneous catalyst for the synthesis of β-phosphonomalonate 1.

Table 2
One-pot synthesis of different β-phosphonomalonates catalyzed by NPS-γ-Fe_2O_3.

Entry	Carbonyl compound	β-phosphonomalonate	Time (min)	Yield[a] (%)
1	benzaldehyde	1	60	90
2	2-chlorobenzaldehyde	2	60	80
3	3-chlorobenzaldehyde	3	30	82
4	4-chlorobenzaldehyde	4	30	88
5	3-bromobenzaldehyde	5	45	87
6	4-bromobenzaldehyde	6	15	85
7	4-methoxybenzaldehyde	7	240	85
8	4-methylbenzaldehyde	8	120	91
9	furfural	9	60	83
10	3-pyridincarbaldehyde	10	5	96
11	butyraldehyde	11	60	85
12	heptanaldehyde	12	60	84

[a] Isolated yield, conditions: aldehyde (1 mmol), malononitrile (1 mmol), triethyl phosphite (1 mmol), catalyst (0.01 mmol), 60 °C, solvent-free conditions. All the products were characterized by spectroscopic methods and compared with the authentic spectra [13,47–49].



Scheme 3.

Table 3
One-pot synthesis of β-phosphonomalonates from different *in situ* generated Michael acceptors and trialkyl phosphites catalyzed by NPS-γ-Fe_2O_3.

Entry	R	X	Y	Product	Time (h)	Yield[a] (%)
1	Et	CN	CN	4	0.5	88
2	Me	CN	CN	13	2	85
3	*iso*-Pr	CN	CN	14	3	80
4	Et	CN	CO_2Et	15	8	75[b]
5	Et	CO_2Et	CO_2Et	–	24	–[c]
6	Et	H	NO_2	–	24	0

[a] Isolated yield, 4-chlorobenzaldehyde (1 mmol), active methylene group (1 mmol), trialkyl phosphite (1 mmol), catalyst (0.01 mmol), 50 °C, solvent-free conditions. All the products were characterized by spectroscopic methods and compared with the authentic spectra [13,40,47].

[b] d.r. = 50:50, according to NMR.

[c] Trace.

Table 4
Comparison of the catalytic efficiency of NPS-γ-Fe_2O_3 with some catalysts.

Entry	Catalyst	Time (h)	Yield[a] (%)
1	NPS-γ-Fe_2O_3	1	90
2	$HClO_4$-SiO_2	1	30
3	AP-SiO_2	1	42
4	$H_3PMo_{12}O_{40}$	1	35
5	Sodium stearate	1	51

[a] Isolated yield, conditions: catalyst (0.01 mmol), benzaldehyde (1 mmol), malononitrile (1 mmol), triethyl phosphite (1 mmol), 60 °C, solvent-free.

different *in situ* generated Michael acceptors and trialkyl phosphites (Scheme 3, Table 3).

As it is shown in Table 3, the catalytic one-pot reaction of 4-chlorobenzaldehyde and malononitrile proceeded well with trialkyl phosphites such as triethyl/trimethyl/tri-*iso*-propyl phosphite (entries 1–3). These results demonstrate that both the yields and the reaction time are relatively independent of the phosphorus compound. In addition to malononitrile, some other *in situ* generated Michael acceptors were also examined to carry out the reaction with triethyl phosphite (entries 4–6). The results showed that the reaction involving ethyl cyano acetate worked well and the desired product was obtained in 75% yield. However, no product was produced when diethyl malonate or nitromethane were used in this one-pot reaction under the same conditions.

In order to show the unique catalytic behavior of NPS-γ-Fe_2O_3 in these reactions, we have performed one-pot reaction of benzaldehyde, malononitrile and triethyl phosphite in the presence of a catalytic amount of $HClO_4$-SiO_2, AP-SiO_2, $H_3PMo_{12}O_{40}$ and sodium stearate (Table 4). As it is evident from Table 4, NPS-γ-Fe_2O_3 is the most effective catalyst for this purpose, leading to the formation of β-phosphonomalonate (1) in a good yield.

4. Conclusion

In summary, NPS-γ-Fe_2O_3 as a new sulfonated nanomagnetic iron oxide was synthesized directly through ring opening reaction of 1,3-propanesultone with nano magnetic γ-Fe_2O_3. The synthesized NPS-γ-Fe_2O_3 was used as a magnetically recyclable heterogeneous catalyst for the efficient one-pot synthesis of β-phosphonomalonates from the reaction of phosphite esters with aldehydes and malononitrile. The magnetic catalyst was easily isolated from the reaction mixture by magnetic decantation using an external magnet and reused at least five times without significant degradation in the activity.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.apcata.2011.09.039.

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