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Nano-Fe_3O_4@SiO_2 supported ionic liquid as an efficient catalyst for the synthesis of 1,3-thiazolidin-4-ones under solvent-free conditions

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ABSTRACT

A magnetically supported ionic liquid on Fe_3O_4@SiO_2 nanoparticles (MNPs@ SiO_2-IL) was synthesized and evaluated as a recoverable catalyst for the one-pot synthesis of 1,3-thiazolidin-4-ones in high to excellent yield under solvent-free conditions. The MNPs@SiO_2-IL catalyst was characterized via Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), thermal gravimetric analysis (TGA), transmission electron microscopy (TEM) and vibrating sample magnetometer (VSM). Moreover, the catalyst could be easily recovered by magnetic separation and recycled for 10 times without significant loss of its catalytic activity.

1. Introduction

The preparation and the use of nanoparticles (NPs) in organic synthesis has become a subject of intense investigation. In particular, magnetic nanoparticles (MNPs) which offer advantages in clean and sustainable chemistry as they can be non-toxic, readily accessible, and retrievable. Additionally, the activity and selectivity of magnetic nano-catalysts can be manipulated by its surface modification [1]. The use of MNPs as catalysts in chemical synthesis has been extensively studied in recent years as the recovery of expensive catalysts after their use are some of the most noticeable features in the sustainable process development [2–5]. Also, ionic liquids have received considerable interest as eco-friendly solvents, catalysts and reagents in organic synthesis because of their unique properties, such as low volatility, non-flammability, high thermal stability, negligible vapor pressure and ability to dissolve a wide range of materials [6–8]. Though ILs possessed such promising advantages, their widespread practical application was still prevented by several disadvantages such as high viscosity, which resulted in only a minor part of ILs taking part in the catalyzed reaction for chemical production; homogeneous reaction, which was difficult for separation and reuse procedures; consequently high cost for the use of relatively large amounts of ILs as opposed to economic criteria [9,10]. So, in order to reduce these problems, supported IL catalyst as a novel solid catalyst combining the advantageous characteristics of ILs, inorganic acids and solid acids had been designed for catalyzed reactions [11,12].

Thiazolidinones have emerged as an important class of compound because of their biological importance and have shown interesting biological activity profiles such as anti-histaminic [13], anti-inflammatory [14], antioxidant [15], antitubercular [16], anti HIV [17], antibacterial [18] and anticancer [19] agents. One of the commonly used methods reported for the synthesis of thiazolidinones involves one pot three-component condensation of amine, carbonyl compound and thioglycolic acid. This reaction can be carried out in the presence of $ZnCl_2$ [20], DCC [21], HBTU [22], [bmim][PF_6] [23], silica chloride [24], Saccharomyces *cerevisiae* [25], $Bi(SCH_2COOH)_3$ [26], silica gel [27] and supported protic acid [28]. However, some of these methods suffer from at least one of the following disadvantages: longer reaction time, corrosive, hazardous reaction condition, tedious work-up procedures, unsatisfactory yields, and non-recyclable reagents. Therefore, it seems that a major task of current research is to replace less efficient and traditional catalysis procedures with more acceptable methods based on improved, stable, and recoverable catalysts. Herein, nano-Fe_3O_4@SiO_2 supported ionic liquid (MNPs@SiO_2-IL) has been successfully applied to perform the reaction of arylaldehydes,

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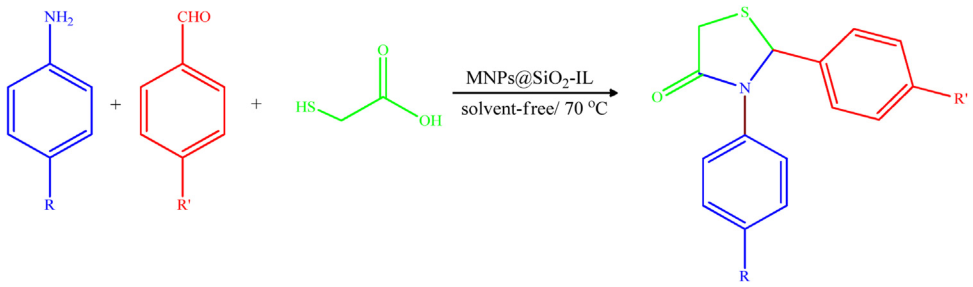


Scheme 1. One-pot synthesis of 1,3-thiazolidin-4-ones catalyzed by MNPs@SiO_2-IL.1.

anilines and thioglycolic acid in solvent-free condition to provide a series of 1,3-thiazolidin-4-one. The method provides rapid and easy access to thiazolidinone compounds in high to excellent yields (Scheme 1).

2. Experimental

High-purity chemical reagents were purchased from the Merck Chemical Company. Melting points were determined using an Electrothermal Mk3 apparatus and are uncorrected. NMR spectra were recorded in DMSO-d_6 on a Bruker Advance DRX-400 MHz instrument spectrometer using TMS as internal standard. FT-IR spectra were obtained with potassium bromide pellets in the range 400–4000 cm^{-1} with a Perkin-Elmer 780 spectrometer. Nanostructures were characterized using a D8-ADVACE (XRD) diffractometer (Cu-K_α, radiation, l = 0.154056 nm), at a scanning speed of 20 min^{-1} from 108 to 1008 (2u). Transmission electron microscopy (TEM) measurements were carried out on a Philips CM10 analyzer operating at 100 kV. Thermal gravimetric analysis (TGA) was recorded on a Shimadzu-50 system at a heating rate of 10 °C/min. The magnetic measurement was carried out in a vibrating sample magnetometer (Model 7300 VSM system, Lake Shore Cryotronic, Inc., Westerville, OH, USA) at room temperature.

2.1. *General procedure for the preparation of Fe_3O_4 nanoparticles*

Typically, 0.01 mol $FeCl_2{\cdot}4H_2O$ and 0.03 mol $FeCl_3{\cdot}6H_2O$ were dissolved into 200 mL distilled water. Thereafter, under rapid mechanical stirring, NaOH was added to the solution and the pH value was controlled in the range $12 \le pH \le 13$. Then, different amounts of hydrazine hydrate ($N_2H_4{\cdot}H_2O$, 80% concentration) was added to the above suspension. The reaction was continued for about 24 h at room temperature. During this period, the pH value was adjusted by NaOH and kept in the range $12 \le pH \le 13$. The black Fe_3O_4 MNPs were then rinsed several times with distilled water and dried at 60 °C under vacuum.

2.2. *General procedure for the preparation of Fe_3O_4@SiO_2 nanoparticles*

0.02 mol of Fe_3O_4 MNPs were dispersed in a mixture of 80 mL of ethanol, 20 mL of deioned water and 2.0 mL of 28 wt% concentrated ammonia aqueous solution, followed by the addition of 0.20 g of tetraethyl orthosilicate (TEOS). After vigorous stirring for 24 h, the final suspension was repeatedly washed, filtered for several times and dried at 60 °C in the air.

Table 1
Synthesis of **4a** by MNPs@SiO_2-IL in different solvents.[a]

Entry	Solvent	Yield (%)[b]
1	EtOH	58
2	H_2O	48
3	CH_3CN	62
4	THF	50
5	CH_2Cl_2	42
6	EtOAc	77
7	DMF	53
8	PhMe	22
9	solvent-free	94[c]

[a] Reaction conditions: benzaldehyde (1 mmol), anilines (1 mmol), thioglycolic acid (1 mmol) and NMPs@SiO_2-IL (0.0007 g) at reflux temperature of solvents after 1 h.
[b] Isolated yields.
[c] Reaction performed at 70 °C.

2.3. *Synthesis of 1-methyl-3-(3-trimethoxysilylpropyl)-1H-imidazol-3-ium chloride (IL)*

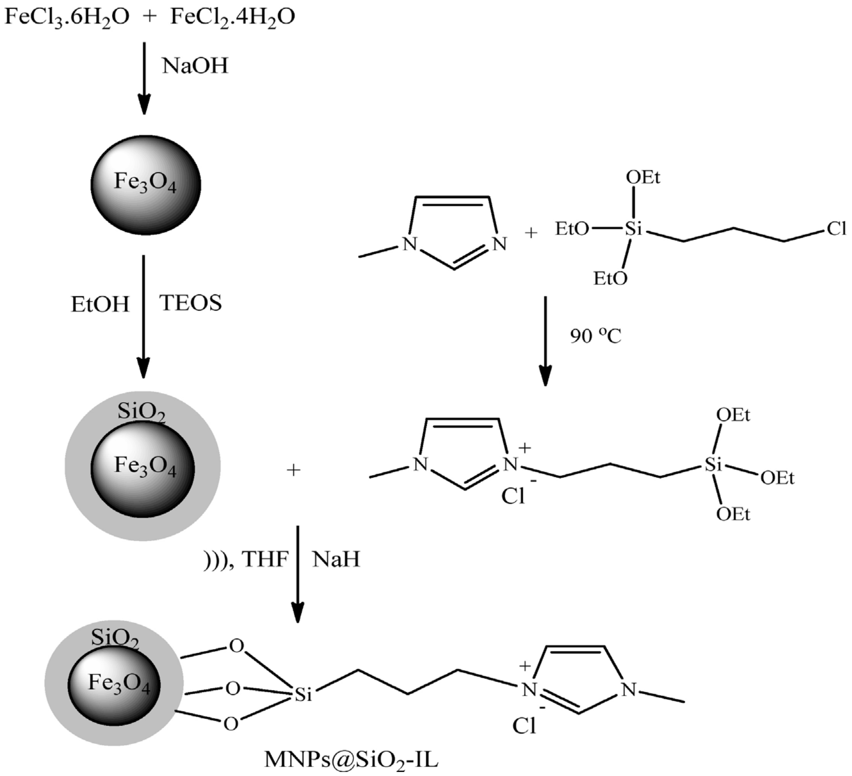
1-Methylimidazole (13.6 mL, 0.17 mol) and (3-chlor-opropyl) trimethoxysilane (31 mL, 0.17 mol) were refluxed at 80 °C for three days in the absence of any catalyst and solvent under Ar atmosphere. The unreacted materials were washed with diethyl ether (3× 8 mL). The diethyl ether was removed under reduced pressure at room temperature, followed by heating under high vacuum, to yield a yellowish viscous liquid. Isolated yield was 98%.

2.4. *General procedure for the preparation of Fe_3O_4@SiO_2-3-chloropropyltriethoxysilane nanoparticles (MNPs@SiO_2-IL)*

2 mmol of Fe_3O4@SiO_2 MNPs and 20 mL THF were mixed together in a beaker, and then 20 mmol of NaH was dispersed in to the mixture by ultrasonication. 22 mmol 1-methyl-3-(3-trimethoxysilylpropyl)-1H-imidazol-3-ium chloride was added drop-wise at room temperature and stirred for another 16 h at 60 °C. The resultant products were collected and washed with ethanol and deionized water in sequence, and then dried under vacuum at 60 °C for 2 h for further use.

2.5. *General synthesis of 1,3-thiazolidin-4-ones*

A mixture of aromatic aldehyde (1 mmol), anilines (1 mmol), thioglycolic acid (1 mmol) and MNPs@SiO_2-IL (0.0007 g) was stirred at 70 °C for the appropriate time, as shown in Table 1. Completion of the reaction was indicated by TLC monitoring. The reaction mixture was cooled to ambient temperature, and the crude solid residue was recrystallized from *n*-hexan/ethyl acetate to afford pure crystals of the proper 1,3-thiazolidin-4-ones in 86–95% yields. The products were characterized by FT-IR, NMR and physical constants.



Scheme 2. Preparation steps for synthesis of 1-methyl-3-(3-trimethoxysilylpropyl)-1H-imidazol-3-ium chloride supported nano-Fe_3O_4@SiO_2.

Spectral data for the representative compounds:

2-Phenyl-3-*p*-tolyl-1,3-thiazolidin-4-one (4b): White solid; mp 115–118 °C; FT-IR cm^{-1} (KBr); 2914, 1675, 1565, 1425, 1232, 1089, 815, 670; ^{1}H NMR (400 MHz, DMSO-d$_6$): δ_H 7.30 (1H, t, *J* = 7.4), 7.29 (2H, d, *J* = 7.4), 7.27 (2H, t, *J* = 7.4), 7.14 (2H, d, *J* = 8.4), 7.12 (2H, d, *J* = 8.4), 6.06 (1H, d, *J* = 1.5), 4.03 (1H, dd, *J* = 15.8, 1.5), 3.90 (1H, d, 15.8), 2.21 (3H, s); ^{13}C NMR (100 MHz, DMSO-d$_6$): δ_C 21.30, 33.50, 72.60, 120.20, 126.90, 128.60, 129.20, 133.40, 138.20, 139.20, 142.80, 174.20.

2,3-Di-*p*-tolyl-1,3-thiazolidin-4-one (4e): White solid; mp 119–121 °C; FT-IR cm^{-1} (KBr); 2924, 1670, 1567, 1425, 1212, 1086, 813, 672; ^{1}H NMR (400 MHz, DMSO-d$_6$): δ_H 7.18 (2H, d, *J* = 7.6), 7.08 (2H, d, *J* = 7.5), 7.06 (2H, d, *J* = 7.6), 7.02 (2H, d, *J* = 7.5), 6.01 (1H, d, *J* = 1.7), 3.98 (1H, dd, *J* = 15.7, 1.7), 3.87 (1H, d, *J* = 15.7), 2.26 (3H, s),

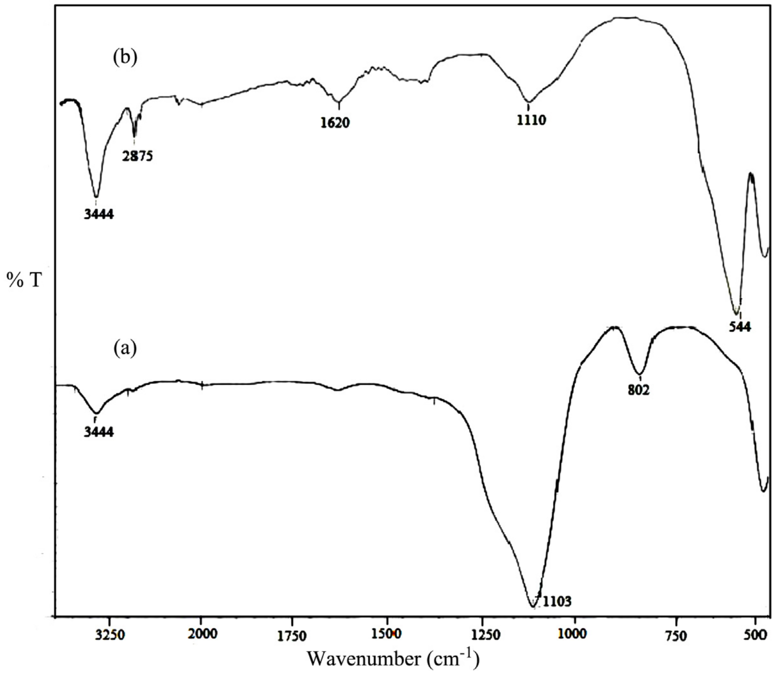


Fig. 1. The FT-IR spectra of MNPs@SiO_2 (a) and MNPs@SiO_2-IL (b).

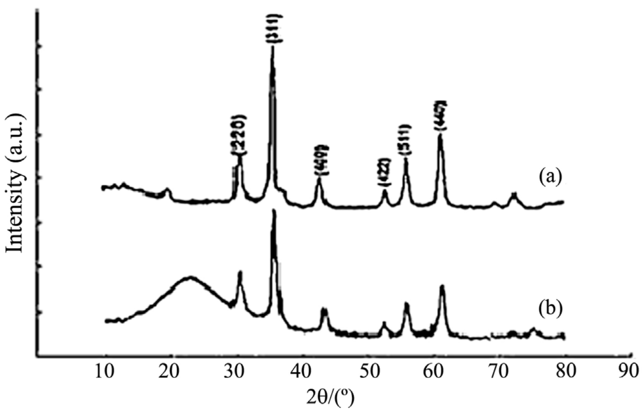


Fig. 2. X-ray powder diffraction patterns of (a) Fe_3O_4 MNPs, (b) MNPs@SiO_2-IL.

2.22 (3H, s); ^{13}C NMR (100 MHz, DMSO-d_6): δ_C 20.23, 22.48, 39.22, 70.80, 119.75, 127.90, 129.38, 131.65, 144.13, 146.34, 167.48.

2-(4-Chlorophenyl)-3-phenyl-1,3-thiazolidin-4-one (**4g**): White solid; mp 124–126 °C; FT-IR cm^{-1} (KBr); 2884, 1676, 1570, 1455, 1212, 1086, 816, 675; ^{1}H NMR (400 MHz, DMSO-d_6): δ_H 3.87 (d, *J* = 15.7 Hz, 1H), 3.98 (d, *J* = 15.7 Hz, 1H), 6.07 (s, 1H, CH), 7.12–7.34 (m, 9H, Ar); ^{13}C NMR (100 MHz, DMSO-d_6): δ_C 34.80, 65.29, 126.10, 126.60, 127.61, 128.81, 128.92, 129.50, 129.56, 129.59, 129.60, 135.12, 137.73, 138.47, 171.20.

3. Results and discussion

3.1. Characterization of the prepared MNPs@SiO$_2$-IL

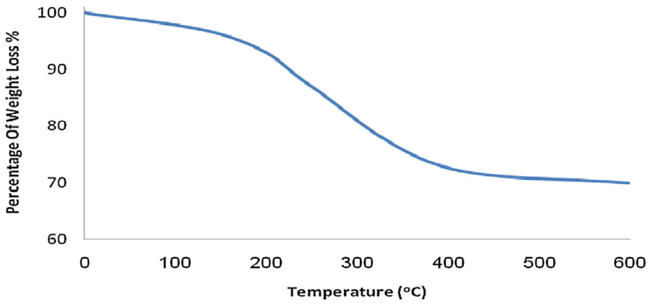


Fig. 4. TGA diagram of MNPs@SiO_2-IL.

The magnetic nanoparticle supported ionic liquid catalyst (MNPs@SiO_2-IL) was prepared following the procedure shown in Scheme 2. Magnetite (Fe_3O_4) nanoparticles were easily prepared via the chemical co-precipitation of Fe^{2+} and Fe^{3+} ions in basic solution. These were subsequently coated with silica (Fe_3O_4@SiO_2) through the well-known Stober method [29].

Then, 1-methyl-3-(3-trimethoxysilylpropyl) imidazolium chloride (IL), was prepared from the reaction of *N*-methyl imidazole with (3-chloropropyl)trimethoxysilane at 80 °C [30]. Finally, the external surface of MNPs was coated with IL to obtain MNPs@SiO_2-IL.

Fig. 1 shows the Fourier transform infrared (FT-IR) spectra of the MNPs@SiO_2 (a) and MNPs@SiO_2-IL (b). The Fe—O stretching vibration near 540 cm^{-1}, the Si—O—Si symmetric and asymmetric stretching vibrations at 802 cm^{-1} and 1103 cm^{-1} and the O—H stretching vibration at 3444 cm^{-1} were observed for the Fe_3O_4@SiO_2 Fig. 1 (a). The Fe—O stretching vibration near 544 cm^{-1}, the O—H stretching vibration at 3444 cm^{-1}, the Si—O stretching at 1110 cm^{-1}, the C=N stretching at 1620 cm^{-1} and —CH_2 stretching at 2875 cm^{-1} were observed for the Fe_3O_4@SiO_2-IL in Fig. 1(b). These results provided the evidences that IL were successfully attached to the surface of Fe_3O_4@SiO_2 nanoparticles.

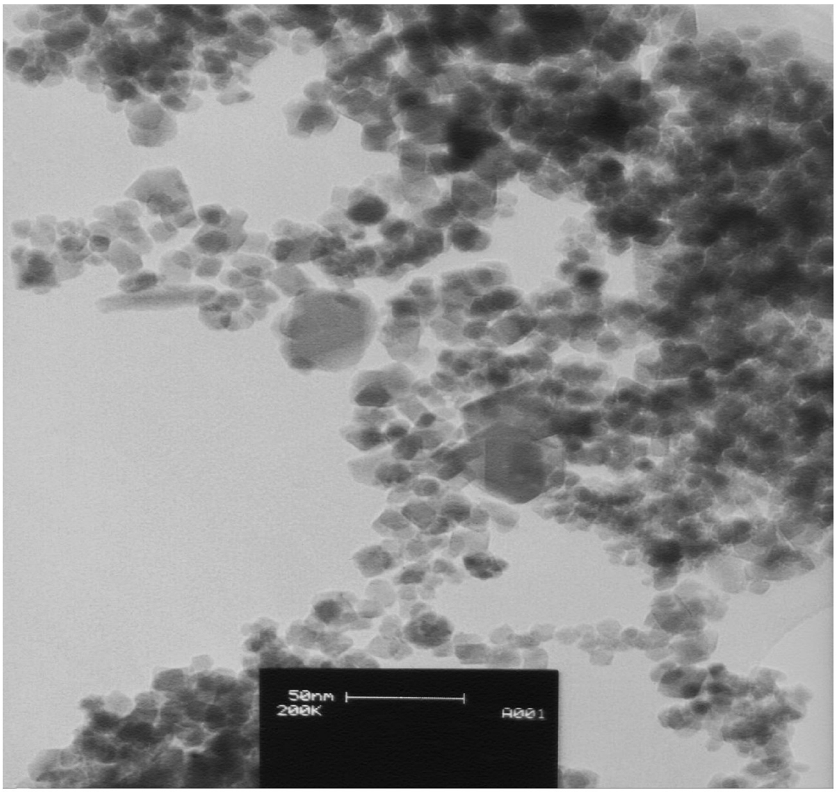


Fig. 3. TEM image of MNPs@SiO_2-IL nanoparticles.

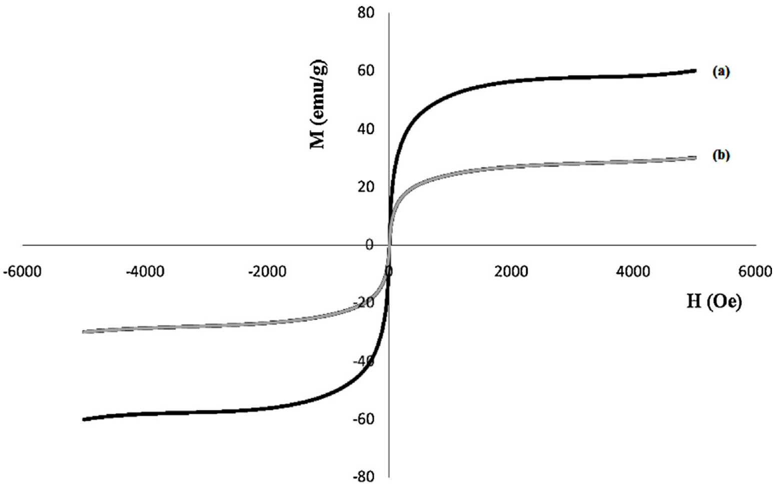


Fig. 5. Magnetic curve of MNPs (a) and MNPs@SiO_2-IL (b).

The degree of crystallinity of magnetic Fe_3O_4 and the MNPs@SiO_2-IL catalyst was obtained from XRD measurements (Fig. 2). The XRD data of the synthesized magnetic nanoparticles show diffraction peaks at $2\theta = 30.4°$, $35.7°$, $43.3°$, $53.9°$, $57.3°$ and $63.0°$ which can be assigned to the (2 2 0), (3 1 1), (4 0 0), (4 2 2), (5 1 1) and (4 4 0) planes of Fe_3O_4, respectively, indicating that the Fe_3O_4 particles in the nanoparticles were pure Fe_3O_4 with a cubic spinel structure; these match well with the standard Fe_3O_4 sample (JCPDS card no. 85-1436). The broad peak from $2\theta = 20°$ to $27°$ (Fig. 2b) is consistent with an amorphous silica phase in the shell of the silica-coated Fe_3O_4 nanoparticles (Fe_3O_4@SiO_2) [31]. The same peaks were observed in both of the MNPs and MNPs@SiO_2-IL XRD patterns, indicating that the resultant nanoparticles are pure Fe_3O_4 with a spinel structure and that the grafting process did not induce any phase change of Fe_3O_4.

The size and structure of the MNPs@SiO_2-IL were also evaluated using transmission electron microscopy (TEM) (Fig. 3). TEM analysis shows a dark nano-Fe_3O_4 core surrounded by a gray silica shell thick and the average size of the obtained particles is 30–40 nm.

The thermal behavior of Fe_3O_4@SiO_2/1-methyl-3-(3-trimethoxysilylpropyl)-1H-imidazol-3-ium chloride MNPs is shown in Fig. 4. The analysis showed two decreasing peaks. First peak appears at temperature around 120–140 °C due to desorption of water molecules from the catalyst surface. This is followed by a second peak at 390–450 °C, corresponding to the loss of the organic spacer group.

The room temperature magnetization curves (Fig. 5) proved that the MNPs@SiO_2-IL is superparamagnetic. Saturation magnetization of MNPs was 58.9 emu/g and saturation magnetization of MNPs@SiO_2-IL was 23.7 emu/g. Compared with the uncoated Fe_3O_4 particles, the saturation magnetization of the MNPs@SiO_2-IL obviously decreased because the diamagnetic contribution of the thick SiO_2 and organic matter resulted in a low mass fraction of the Fe_3O_4 magnetic substance. Even with this reduction in the saturation magnetization, the solid could still be efficiently separated from solution with a permanent magnet.

3.2. *Evaluation of the catalytic activity of IL-MNPs through the synthesis of 1,3-thiazolidin-4-ones*

In order to optimize the reaction conditions and obtain the best catalytic activity, the reaction of benzaldehyde (1 mmol), aniline (1 mmol), and thioglycolic acid (1 mmol) was used as a model, and was conducted under different reaction parameters such as solvent and amount of catalyst. Initially, the model reaction was carried out in several solvents such as EtOH, H_2O, CH_3CN, THF, CH_2Cl_2, EtOAc, DMF, Toluene, and under solvent-free conditions, to investigate the efficiency of the catalyst. In this study, it was found that conventional heating at 70 °C under solvent-free conditions is more efficient than using organic solvents, with respect to reaction time and yield of the desired 1,3-thiazolidin-4-ones (Table 1).

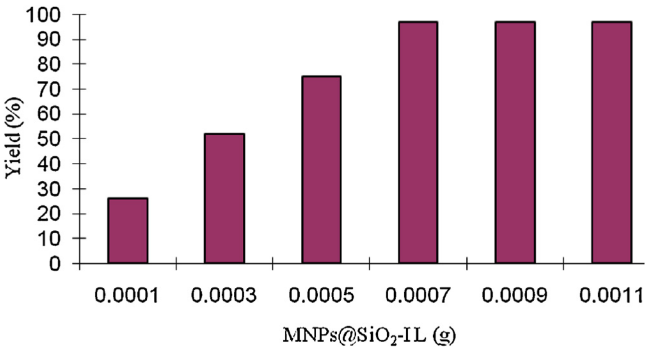


Fig. 6. Optimization of the conditions for synthesis of 2,3-diphenyl-thiazolidin-4-one. Reaction conditions: benzaldehdye (1 mmol), aniline (1 mmol), thioglycolic acid (1 mmol), in different amounts of MNPs@SiO_2-IL at 70 °C.

To investigate the effect of catalyst loading, the model reaction was carried out in the presence of different amounts of catalyst. It was observed that the variation for MNPs@SiO_2-IL had an effective influence. The best amount of MNPs@SiO_2-IL is 0.0007 g which afforded the desired product in 94% yields (Fig. 6).

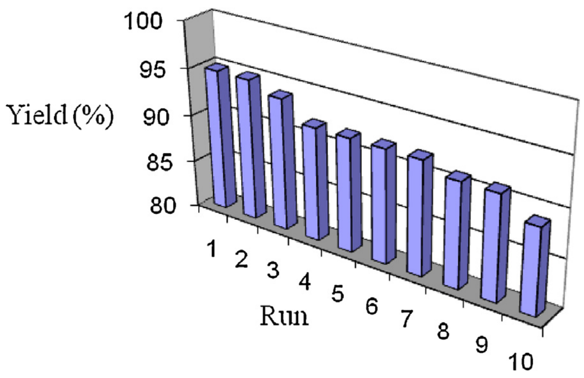
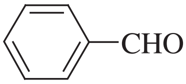
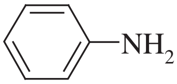
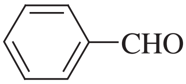
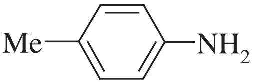
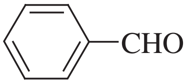
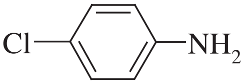
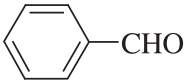
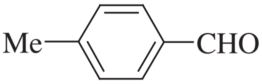
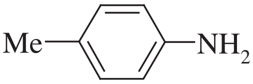
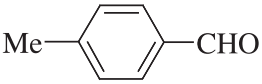
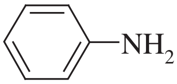
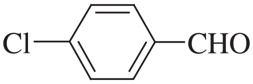
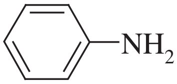
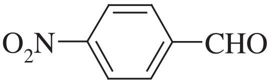
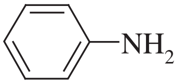
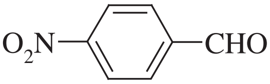
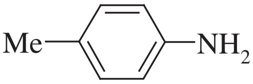
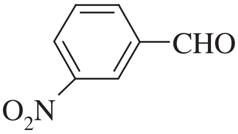
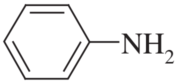


Fig. 7. Recyclability of catalyst in the reaction of benzaldehdye (1 mmol), aniline (1 mmol), thioglycolic acid (1 mmol) and MNPs@SiO_2-IL (0.0007 g) at 70 °C.

Table 2
Synthesis of 1,3-thiazolidin-4-one derivatives by MNPs@SiO_2-IL.[a]

Entry	Aldehyde	Amine	Product	Time (min)	Yield (%)[b]
1			**4a**	60	94
2			**4b**	60	90
3			**4c**	60	90
4			**4d**	70	86
5			**4e**	60	93
6			**4f**	60	88
7			**4g**	60	95
8			**4h**	55	92
9			**4i**	55	90
10			**4j**	55	89

[a] Reaction conditions: Aldehyde (1 mmol), amine (1 mmol), thioglycolic acid (1 mmol) and NMPs@SiO_2-IL (0.0007 g) at 70 °C.
[b] Isolated yields.

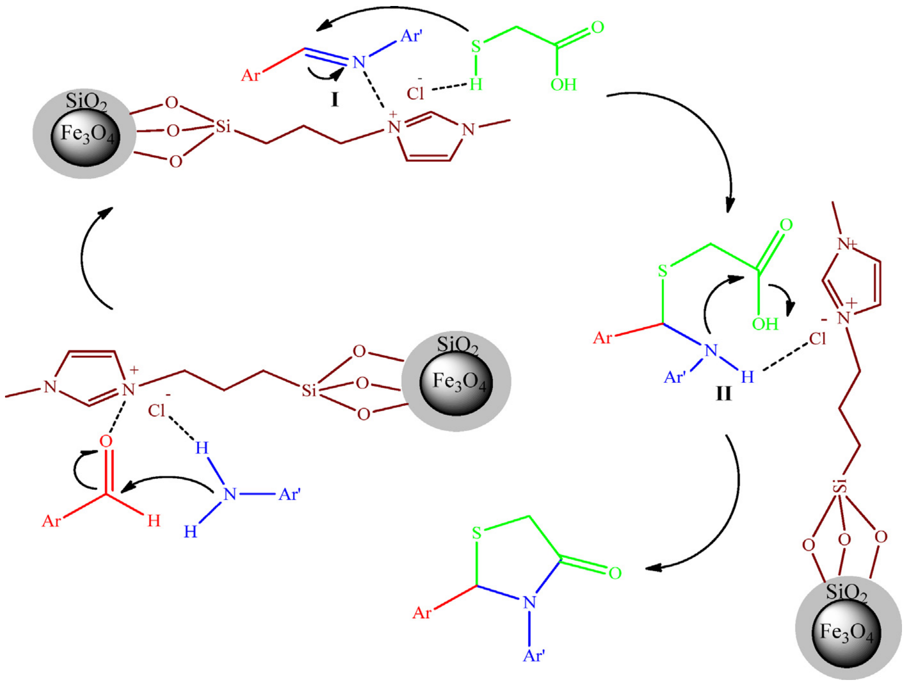
In order to show the efficiency of this catalytic system, a variety of 1,3-thiazolidin-4-ones, was prepared from aldehydes, anilines, thioglycolic acid in the presence of MNP@SiO_2-IL under solvent-free conditions at 70 °C in excellent yields (Table 2, entries 1–10). It is worth mentioning that the corresponding 1,3-thiazolidin-4-ones was isolated by crystallization from the crude filtrate. Also, the reactions worked well with electron donating and electron withdrawing aldehydes.

It is important to note that the magnetic property of MNPs@SiO_2-IL facilitates its efficient recovery from the reaction

Table 3
Comparison of our results with previously reported data for the synthesis of **4a**.

Entry	Catalyst	Reaction conditions	Time/h	Yield (%)	Ref.
1	[a] DDC (1.3 eq)	THF/r.t.	0.83	91	[21]
2	[b] Baker's yeast (2 g)	THF/r.t.	40	60	[25]
3	[c] SiO_2 (0.5 g)	DCM/r.t.	7	78	[27]
4	[d] $HClO_4—SiO_2$ (0.125 g)	PhMe/100 °C	5	85	[28]
5	[e] $H_2SO_4—SiO_2$ (10 mol%)	PhMe/100 °C	5	55	[28]
6	[e] TfOH—SiO_2 (10 mol%)	PhMe/100 °C	5	72	[28]
7	[a] $Bi(SCH_2COOH)_3$(10 mol%)	70 °C	2	75	[26]
8	[e] NMPs@SiO_2-IL (0.0007 g)	70 °C	1	94	This work

Reaction conditions: [a] benzldehyde (1 mmol), aniline (1 mmol), thioglycolic acid (1.2 mmol); [b] benzaldehyde (10 mmol), aniline (10 mmol), thioglycolic acid (10 mmol); [c] benzaldehyde (1.05 mmol), aniline (1 mmol), thioglycolic acid (2 mmol); [d] benzaldehyde (2.5 mmol), aniline (2.5 mmol), thioglycolic acid (2.5 mmol); [e] benzaldehyde (1 mmol), aniline (1 mmol), thioglycolic acid (1 mmol).



Scheme 3. Possible mechanism for the synthesis of 1,3-thiazolidin-4-ones by MNPs@SiO$_2$-IL.

mixture during work-up procedure. The activity of the recycled catalyst was also examined under the optimized conditions. After the completion of reaction, the catalyst was separated by an external magnet, washed with ethanol and dried. The recovered catalyst was reused for 10 consecutive cycles without significant loss in catalytic activity (Fig. 7).

A plausible mechanism for the formation of 1,3-thiazolidin-4-ones catalyzed by MNPs@SiO$_2$-IL, is shown in Scheme 3. It can be speculated that the methylimidazolium cation [MIM]$^+$ in the MNPs@SiO$_2$-IL favors the interact on oxygen atom of the carbonyl group of the aldehyde and facilitates the formation of imine intermediate **I** by increasing the electrophilicity of the carbonyl group of the aldehyde. Then, free Cl$^-$ ions interact with thioglycolic acid proton and sulfur atom of thioglycolic acid attacks to the activated imine group of intermediate **I** and affords intermediate **II**. Next, The MNPs@SiO$_2$-IL again activates intermediate **II** and by removing water from intermediate **II**, 1,3-thiazolidin-4-ones is prepared.

In order to examine the efficiency of the present method for the synthesis of 1,3-thiazolidin-4-ones, compound **4a** was compared with some of those reported in the literature (Table 3). As one can see, our results have better performance with previously reported data when all terms including yields, reaction times, and reaction conditions are taken into account.

4. Conclusions

We have developed a simple, clean, efficient and environmentally friendly approach for the one-pot synthesis of 1,3-thiazolidin-4-ones by the three-component condensation of arylaldehydes, anilines and thioglycolic acid using MNPs@SiO$_2$-IL as a recyclable catalyst under solvent-free conditions. This procedure offers several advantages, including high yield, short reaction time, simple work-up procedure, and ease of separation of catalyst by an external magnet.

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