

Bis (1(3-trimethoxysilylpropyl)-3-methyl-imidazolium) Nickel Tetrachloride Tethered to Colloidal Silica Nanoparticles as a Superior Catalyst for the Bis-thiazolidinones Preparation

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An easy and rapid method for the synthesis of bis-thiazolidinones is presented by one-pot pseudo-five-component reaction of benzaldehydes, ethylenediamine, 2-mercaptoacetic acid with bis (1(3-trimethoxysilylpropyl)-3-methyl-imidazolium) nickel tetrachloride tethered attached to colloidal silica nanoparticles. Atom economy, low catalyst loading, reusable catalyst, applicability to a wide range of substrates and high yields of products are some of the important features of this protocol.

Keywords: Heterogeneous catalysts, Ionic liquid, Colloidal silica, Bis-thiazolidinones, One-pot

INTRODUCTION

Thiazolidinones exhibit biological properties such as anti-tumor [1], anti-viral [2], antibacterial [3], antituberculous therapy [4], and anti-HIV [5] activities. These activities make them substantial targets in organic synthesis. Therefore, seeking easy and brief techniques for the preparation of thiazolidinones is a significant subject. Among the thiazolidine derivatives, bis-thiazolidinones have been devoted significant attention because of their high biological activities [6-8]. Recently, reports have been developed on synthesis of bis-thiazolidinones using the catalysts such as Zeolite [9], $\text{HClO}_4\text{-SiO}_2$ [10], ChCl (Choline Chloride)/urea based ionic liquid [11], and ZnCl_2 [12]. Despite available techniques, there remains adequate purpose to offer a new way for an efficient, high yielding, and mild approach to achieve such systems. Recently, preparation and immobilization of ionic liquids (ILs) on nanoparticles have been widely considered [13-18]. Although ionic liquids encompass some utilization, their effective applications are limited by some problems in their recovery, leading to economic and environmental difficulties. These limitations can be solved by

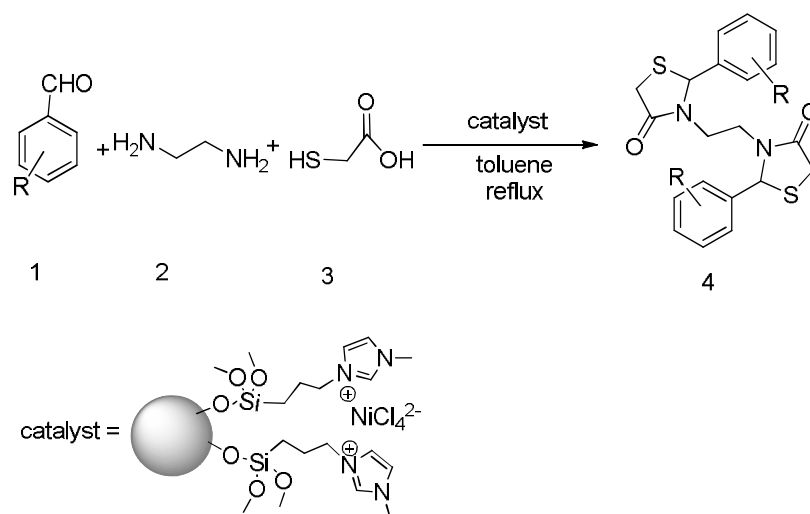
immobilization of ILs on nanoparticles to obtain heterogeneous catalysts [19-25]. The structures of 1-ethyl-3-methylimidazolium (Emim) and 1-butyl-3-methylimidazolium (Bmim) with transition metal chloride anions including NiCl_4^{2-} , CoCl_4^{2-} and PdCl_4^{2-} have been studied [26-30]. Here, we report the preparation of bis (1(3-trimethoxysilylpropyl)-3-methyl-imidazolium) nickel tetrachloride tethered attached to colloidal silica nanoparticles [31-32], and investigate its catalytic application for the preparation of bis-thiazolidinones by one-pot pseudo-five-component reaction of benzaldehydes, ethylenediamine, 2-mercaptoacetic acid under reflux conditions in toluene (Scheme 1).

EXPERIMENTAL

Materials

All organic materials were purchased commercially from sigma-Aldrich and Merck and were used without further purification. The FT-IR spectra were recorded with KBr pellets using a Magna-IR, spectrometer 550 Nicolet. The NMR spectra were recorded on a Bruker 400 MHz spectrometer with CDCl_3 as a solvent and TMS as an internal standard. The CHN compositions were measured by Carlo ERBA Model EA 1108 analyzer. The dynamic light

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Scheme 1. Preparation of bis-thiazolidinones using ionic liquid-attached colloidal silica nanoparticles

scattering (DLS) measurement was performed with Malvern. The thermogravimetric analysis (TGA) curves were recorded using a V5.1A DUPONT 2000. To investigate the morphology and particle size of the nanoparticles synthesized, the FE-SEM images and EDS spectrum of the products were visualized by MIRA 3.

Preparation of Nanocatalyst

In a typical procedure, 0.098 ml of colloidal silica nanoparticles (LUDOX SM colloidal silica 30 wt% suspension in H₂O) was diluted in 3 ml of deionized water, and 1.5 mmol of 1-(3-trimethoxysilylpropyl)-3-methylimidazolium chloride IL was slowly added with continuous stirring during 1 h. Then, 0.18 g of NiCl₂·6H₂O was added and refluxed for 24 h. After 24 h, IL functionalized silica nanoparticles were separated by centrifugation and washed with acetone and methanol for four times, then, IL/Ni/silica was dried by lyophilization/ freeze-drying.

The purity of the resultant IL/Ni/silica was confirmed using ¹H NMR spectrum. The Ni loading was measured using XRF to be 3.3 wt%.

General Procedure for the Preparation of Bis-thiazolidinones

A mixture of aldehydes (2 mmol), ethylenediamine (1 mmol), 2-mercaptoacetic acid (2 mmol) and 12 mg of bis (1(3-trimethoxysilylpropyl)-3-methyl-imidazolium) nickel tetrachloride tethered attached to colloidal silica

nanoparticles in toluene (5 ml) was refluxed. The reaction was monitored by TLC (ethyl acetate/*n*-hexan 2:8). After completion of the reaction, EtOAc was added to dilute the reaction mixture after terminating the reaction. The catalyst was insoluble in the solvent and was separated by centrifuging. The EtOAc was evaporated and the crude mixture was separated by silica gel column chromatography (ethyl acetate/*n*-hexan) to get pure product.

3,3'-(Ethane-1,2-diyl)bis(2-(4-chlorophenyl)thiazolidin-4-one) (4a). White solid; IR (KBr) cm⁻¹: 2941, 1661; ¹H NMR (400 MHz, DMSO-*d*₆): δ (ppm): 2.52-2.58 (m, 2H), 3.38-3.45 (m, 2H), 3.52 (d, *J* = 15 Hz, 2H), 3.72 (dd, *J* = 1.7, 15 Hz, 2H), 5.84 (d, *J* = 1.4 Hz, 2H), 7.15 (d, *J* = 7 Hz, 4H), 7.36 (d, *J* = 7 Hz, 4H); ¹³C NMR (100 MHz, DMSO-*d*₆): δ (ppm): 31.7, 40.2, 62.9, 128.2, 130.5, 131.4, 141.2, 170.8; Anal. Calcd. for C₂₀H₁₈Cl₂N₂O₂S₂: C, 52.98; H, 4.00; N, 6.18; S, 14.14; Found: C, 52.75; H, 4.24; N, 6.19; S, 14.03.

3,3'-(Ethane-1,2-diyl)bis(2-(*p*-tolyl)thiazolidin-4-one) (4b). White solid; IR (KBr) cm⁻¹: 2929, 1669; ¹H NMR (400 MHz, CDCl₃): δ (ppm): 2.28 (s, 6H), 2.68-2.75 (m, 2H), 3.53-3.61 (m, 4H), 3.67 (dd, *J* = 1.8, 16 Hz, 2H), 5.46 (d, *J* = 1.5 Hz, 2H), 7.11-7.18 (m, 8H); ¹³C NMR (100 MHz, CDCl₃): δ (ppm): 20.8, 32.1, 39.5, 63.2, 126.7, 129.3, 135.3, 138.9, 171.1; Anal. Calcd. for C₂₂H₂₄N₂O₂S₂: C, 64.05; H, 5.86; N, 6.79; S, 15.54; Found: C, 63.94; H, 5.93; N, 6.83; S, 15.30.

3,3'-(Ethane-1,2-diyl)bis(2-phenylthiazolidin-4-one)

(4c). White solid; IR (KBr) cm^{-1} : 2923, 1660; ^1H NMR (400 MHz, DMSO- d_6): δ (ppm): 2.52-2.58 (m, 2H), 3.41-3.47 (m, 2H), 3.52 (d, $J = 16$ Hz, 2H), 3.72 (dd, $J = 1.8$, 16 Hz, 2H), 5.59 (d, $J = 1.6$ Hz, 2H), 7.19-7.33 (m, 10H); ^{13}C NMR (100 MHz, DMSO- d_6): δ (ppm): 32.3, 41.2, 62.8, 127.1, 127.3, 128.6, 140.4, 171.4; Anal. Calcd. for $\text{C}_{20}\text{H}_{20}\text{N}_2\text{O}_2\text{S}_2$: C, 62.47; H, 5.24; N, 7.29; S, 16.68; Found: C, 62.33; H, 5.15; N, 7.17; S, 16.55.

3,3'-(Ethane-1,2-diyl)bis(2-(4-nitrophenyl)thiazolidin-4-one) (4d). Yellow Solid; IR (KBr) cm^{-1} : 2935, 1670, 1521; ^1H NMR (400 MHz, DMSO- d_6): δ (ppm): 2.64-2.73 (m, 2H), 3.53-3.63 (m, 2H), 3.65-3.74 (m, 2H), 3.92 (dd, $J = 1.9$, 16 Hz, 2H), 6.03 (d, $J = 1.9$ Hz, 2H), 7.46 (d, $J = 8$ Hz, 4H), 8.07 (d, $J = 8$ Hz, 4H); ^{13}C NMR (100 MHz, DMSO- d_6): δ (ppm): 32.7, 41.6, 63.4, 123.9, 130.1, 147.2, 150.2, 171.6; Anal. Calcd. for $\text{C}_{20}\text{H}_{18}\text{N}_4\text{O}_6\text{S}_2$: C, 50.62; H, 3.82; N, 11.81; S, 13.51; Found: C, 50.54; H, 3.78; N, 11.64; S, 13.43.

3,3'-(Ethane-1,2-diyl)bis(2-(3-nitrophenyl)thiazolidin-4-one) (4e). Cream solid; IR (KBr) cm^{-1} : 2933, 1664, 1516; ^1H NMR (400 MHz, DMSO- d_6): δ (ppm): 2.63-2.69 (m, 2H), 3.53-3.59 (m, 2H), 3.62-3.68 (m, 2H), 3.88 (dd, $J = 1.7$, 15 Hz, 2H), 5.95 (d, $J = 1.9$ Hz, 2H), 7.53-7.72 (m, 4H), 8.01-8.09 (m, 4H); ^{13}C NMR (100 MHz, DMSO- d_6): δ (ppm): 32.4, 41.4, 63.1, 126.2, 129.4, 130.8, 134.1, 143.9, 148.4, 171.5; Anal. Calcd. for $\text{C}_{20}\text{H}_{18}\text{N}_4\text{O}_6\text{S}_2$: C, 50.62; H, 3.82; N, 11.81; S, 13.51; Found: C, 50.54; H, 3.68; N, 11.74; S, 13.42.

3,3'-(Ethane-1,2-diyl)bis(2-(pyridin-2-yl)thiazolidin-4-one) (4f). Cream solid; IR (KBr) cm^{-1} : 2934, 1675; ^1H NMR (400 MHz, CDCl_3): δ (ppm): 2.66 (dd, $J = 8$, 18 Hz, 2H), 3.53-3.59 (m, 2H), 3.73-3.80 (m, 2H), 3.91 (dd, $J = 8$, 18 Hz, 2H), 5.81 (d, $J = 1.2$ Hz, 2H), 7.17 (dd, $J = 5$, 8 Hz, 2H), 7.20 (d, $J = 10$ Hz, 2H), 7.62 (ddd, $J = 2$, 8, 10 Hz, 2H), 8.49 (d, $J = 5$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ (ppm): 32.9, 40.1, 63.3, 121.1, 123.5, 138.1, 150.5, 158.4, 171.5; Anal. Calcd. for $\text{C}_{18}\text{H}_{18}\text{N}_4\text{O}_2\text{S}_2$: C, 55.94; H, 4.69; N, 14.50; S, 16.59; Found: C, 55.85; H, 4.75; N, 14.40; S, 16.71.

3,3'-(Ethane-1,2-diyl)bis(2-(pyridin-3-yl)thiazolidin-4-one) (4g). White solid; IR (KBr) cm^{-1} : 2934, 1667; ^1H NMR (400 MHz, DMSO- d_6): δ (ppm): 2.68 (dd, $J = 6$ Hz, 15, 2H), 3.49 (dd, $J = 6$, 15 Hz, 2H), 3.62 (d, $J = 16$ Hz, 2H), 3.9 (dd, $J = 1.9$, 16 Hz, 2H), 5.78 (d, $J = 1.7$ Hz, 2H), 7.35-7.41 (m, 2H), 7.75-7.81 (m, 2H), 8.51-8.58 (m, 4H);

^{13}C NMR (100 MHz, DMSO- d_6): δ (ppm): 32.1, 39.6, 61.4, 130.2, 134.8, 135.1, 148.5, 150.2, 171.1; Anal. Calcd. for $\text{C}_{18}\text{H}_{18}\text{N}_4\text{O}_2\text{S}_2$: C, 55.94; H, 4.69; N, 14.50; S, 16.59; Found: C, 55.83; H, 4.75; N, 14.44; S, 16.65.

3,3'-(Ethane-1,2-diyl)bis(2-(2-chlorophenyl)thiazolidin-4-one) (4h). White solid; IR (KBr) cm^{-1} : 2935, 1664; ^1H NMR (400 MHz, CDCl_3): δ (ppm): 2.48-2.53 (m, 2H), 3.34-3.4 (m, 2H), 3.43-3.49 (m, 2H), 3.68 (dd, $J = 1.6$, 16 Hz, 2H), 5.77 (d, $J = 1.4$ Hz, 2H), 7.13-7.21 (m, 6H), 7.64 (dd, $J = 2$, 5 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3): δ (ppm): 31.7, 39.5, 62.2, 124.4, 125.3, 126.4, 128.1, 128.9, 135.1, 170.8; Anal. Calcd. for $\text{C}_{20}\text{H}_{18}\text{Cl}_2\text{N}_2\text{O}_2\text{S}_2$: C, 52.98; H, 4.00; N, 6.18; S, 14.14; Found: C, 53.65; H, 4.12; N, 6.08; S, 14.05.

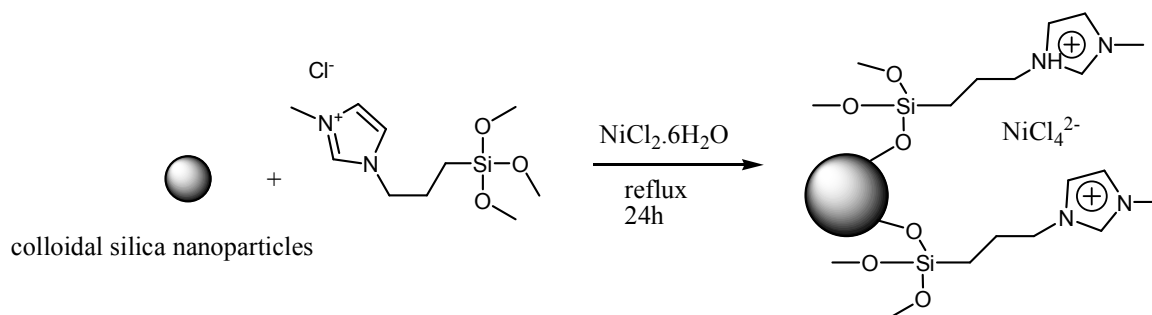
3,3'-(Ethane-1,2-diyl)bis(2-(4-isopropylphenyl)thiazolidin-4-one) (4i). White solid; IR (KBr) cm^{-1} : 2956, 1662; ^1H NMR (400 MHz, CDCl_3): δ (ppm): 1.17 (d, $J = 7$ Hz, 12H), 2.69-2.79 (m, 2H), 2.80-2.89 (m, 2H), 3.55-3.64 (m, 4H), 3.68 (dd, $J = 1.8$, 16 Hz, 2H), 5.49 (d, $J = 1.4$ Hz, 2H), 7.14 (d, $J = 8$ Hz, 4H), 7.18 (d, $J = 8$ Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ (ppm): 23.4, 32.2, 33.4, 39.6, 63.1, 126.6, 126.7, 135.6, 149.8, 171.1; Anal. Calcd. for $\text{C}_{26}\text{H}_{32}\text{N}_2\text{O}_2\text{S}_2$: C, 66.63; H, 6.88; N, 5.98; S, 13.68; Found: C, 66.44; H, 6.77; N, 6.05; S, 13.54.

RESULTS AND DISCUSSION

Characterization of the Nanocatalyst

A scheme for the synthesis process of catalyst is presented in Scheme 2. Figures S1a and S1b (see supporting information) show the ^1H NMR spectra for the 1(3-trimethoxysilylpropyl)-3-methyl-imidazolium chloride and bis (1(3-trimethoxysilylpropyl)-3-methyl-imidazolium) nickel tetrachloride tethered attached to colloidal silica nanoparticles in dimethyl sulfoxide (DMSO), respectively. The NMR spectra of both materials are constant with the expected results for untethered and silica-tethered ionic liquids.

Figure S2 (see supporting information) shows the FE-SEM image of bis (1(3-trimethoxysilylpropyl)-3-methyl-imidazolium) nickel tetrachloride tethered attached to colloidal silica nanoparticles (nanocatalyst). The SEM images show particles with diameters in the nanometer range.



Scheme 2. A scheme for process synthesis of catalyst

Table 1. Optimization of Reaction Conditions Using Different Solvents and Catalysts^a

Entry	Catalyst (mg)	Solvent (reflux)	Time (min)	Yield (%) ^b
1	No catalyst	Toluene	300	7
2	Nano-CaO (5 mol%)	DMF	270	36
3	Nano-CuO (4 mol%)	Toluene	150	50
4	Nano-CeO ₂ (2 mol%)	Toluene	200	32
5	Nano-CuO (4 mol%)	EtOH	200	21
6	Nano-Fe ₃ O ₄	Toluene	240	30
7	Nano-NiO	Toluene	150	54
8	Ni(OAC) ₂	Toluene	200	57
9	Ionic liquid/nanoSiO ₂ (12 mg)	CH ₂ Cl ₂	80	61
10	Ionic liquid/nanoSiO ₂ (12 mg)	THF	80	69
11	Ionic liquid/nanoSiO ₂ (10 mg)	Toluene	60	88
12	Ionic liquid/nanoSiO ₂ (12 mg)	Toluene	60	92
13	Ionic liquid/nanoSiO ₂ (14 mg)	Toluene	60	92

^a4-Chlorobenzaldehyde (2 mmol), ethylenediamine (1 mmol), 2-mercaptoacetic acid (2 mmol). ^bIsolated yields.

In order to study the size distribution of nanocatalysts, DLS (dynamic light scattering) measurements of the nanoparticles are shown in Fig. S3 (see supporting information). This size distribution is centered at a value of 15.8 nm.

The elemental compositions of the nanocatalyst were analyzed by energy dispersive spectroscopy (EDS). The EDS confirmed the presence of Si, O, N, Cl and Ni in the compound (Fig. S4 (see supporting information)).

Thermogravimetric analysis (TGA) considers the thermal stability of the ionic liquid of untethered to SiO₂ (pure ionic liquid) and silica-tethered ionic liquids (Fig. S5 in supporting information). The curve shows a weight loss about 57.12% and 31.73% for ionic liquid/nanosilica with molar ratio 7.5 and 2.5, respectively, from 240 to 620 °C, resulting from the destruction of organic spacer grafting to the nanoparticles. Hence, the nanocatalyst was stable up to 240 °C, confirming that it could be stably utilized in organic reactions at temperature ranges of 80-140 °C.

Preparation of Bis-thiazolidinones Using Nanocatalyst

After the characterizations of nanocatalyst, we studied and optimized different reaction parameters for the preparation of bis-thiazolidinones by reaction of 4-chlorobenzaldehyde (2 mmol), ethylenediamine (1 mmol), 2-mercaptoacetic acid (2 mmol). The model reaction was examined using various catalysts including nano-CaO, nano-CuO, nano-CeO₂, nano-NiO, nano-Fe₃O₄, ionic liquid/nano-SiO₂. We optimized different conditions and found that the reaction gives satisfying result in the presence of ionic liquid/nano-SiO₂ (12 mg) under reflux conditions in toluene for 60 min (Table 1).

With these promising results in hand, we then explored the possibility of the reaction using various aromatic aldehydes as substrates. The results show that the present catalytic method is extensible to a wide diversity of substrates to create a variety-oriented library of bis-thiazolidinones (Table 2).

A proposed mechanism for the synthesis of bis-thiazolidine derivatives using ionic liquid/nano SiO₂, is shown in Scheme 3. The mechanism is outlined *via* primary imine intermediate formation followed by the attack of the sulfur atom of 2-mercaptoacetic acid on the activated

imine groups followed by intramolecular cyclization with the elimination of H₂O giving rise to the cyclized product bis-thiazolidines. In this mechanism, the surface atoms of bis (1(3-trimethoxysilylpropyl)-3-methyl-imidazolium) nickel tetrachloride tethered attached to colloidal silica nanoparticles activate the C=O, C=N groups for better reaction with nucleophiles (Scheme 3). The proposed mechanism has also been supported by the literature [33-35].

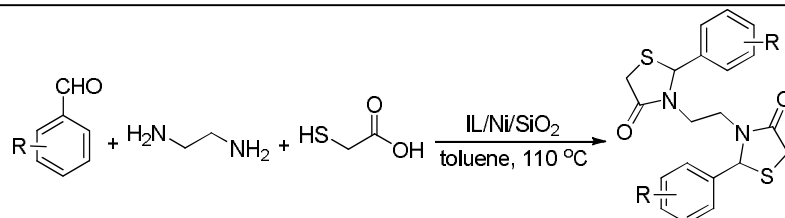
Reusability of the Catalyst

We also studied the reusability of ionic liquid/nano SiO₂ as a nanocatalyst using the reaction of 4-chlorobenzaldehyde (2 mmol), ethylenediamine (1 mmol), 2-mercaptoacetic acid (2 mmol) as the model substrates under optimized conditions. After completion of the reaction, the catalyst was washed with acetone, dried in an oven at 50 °C for 90 min and used directly with new substrates under the same conditions. The results showed that the ionic liquid/nano SiO₂ can be reused several times (Yields 92 to 88%) (Fig. 1). The SEM of ionic liquid/nano SiO₂ before and after the reaction showed identical shape (Fig. S9). The morphology of the nanoparticles remained unchanged before and after reaction. We believe that, this is also the possible reason for the extreme stability of the nanocatalyst presented herein.

To determine the exact Ni species responsible for the reactions observed and to measure the extent of Ni leaching after the reactions, we used the hot filtration test [37]. To do so, we studied the model reaction between 4-chlorobenzaldehyde (2 mmol), ethylenediamine (1 mmol) and 2-mercaptoacetic acid (2 mmol) under optimized condition. The reaction mixture was filtered after 50% conversion to remove the catalyst. Continuation of the reaction under the same conditions showed 54% conversion after 90 min. This result shows that the amount of catalyst leaching into the reaction mixture should be low, confirming that the catalyst acts heterogeneously in the reaction.

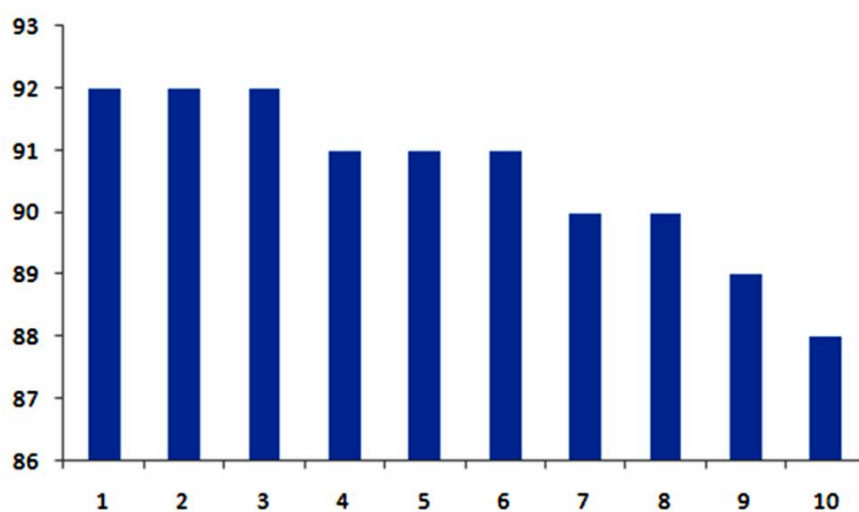
CONCLUSIONS

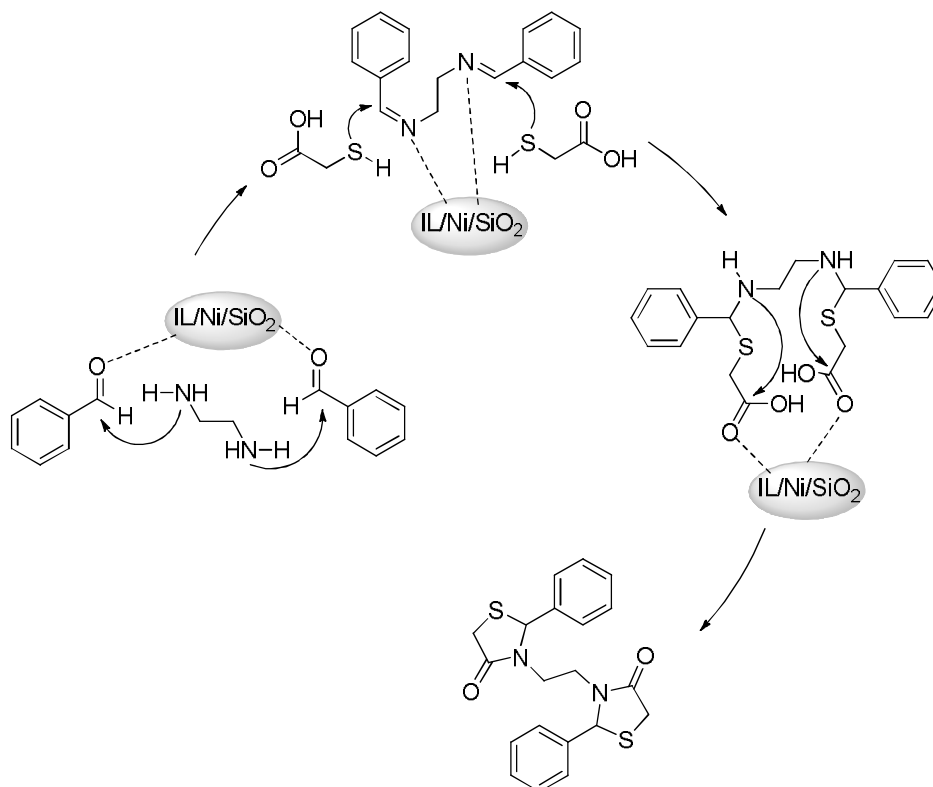
We demonstrated an efficient method for the synthesis of bis-thiazolidinones using ionic liquid tethered attached to colloidal silica nanoparticles under reflux conditions in

Table 2. Synthesis of Bis-thiazolidinones (Racemate as Major Product)^a

Entry	Aldehyde	Product	Time (min)	Yield (%) ^b	M. p. (°C) [Ref]	M.p. (°C) Found
1	4-Cl-C ₆ H ₅	4a	60	92	285-288 [33]	150-152
2	4-Me-C ₆ H ₄	4b	70	82	158-160 [36]	158-160
3	C ₆ H ₅	4c	70	84	152-155 [10]	155-157
4	4-NO ₂ -C ₆ H ₄	4d	60	96	164-166 [36]	164-166
5	3-NO ₂ -C ₆ H ₄	4e	60	91	222-224 [36]	222-224
6	Pyridin-2-yl	4f	70	87	167-169 [34]	170-172
7	Pyridin-3-yl	4g	70	85	198-200 [34]	191-193
8	2-Cl-C ₆ H ₅	4h	60	90	210-211 [33]	143-145
9	4-Isopropyl-C ₆ H ₄	4i	70	80	163-165 [36]	163-165

^aBenzaldehyde (2 mmol), ethylenediamine (1 mmol), 2-mercaptoacetic acid (2 mmol) and ionic liquid/Ni/SiO₂ (12 mg) under reflux conditions in toluene for 60 min. ^bIsolated yield.

**Fig. 1.** Reusability of nanocatalyst for the preparation of 4a.



Scheme 3. Probable mechanism for the formation of bis-thiazolidinones

toluene. The immobilized metal ion-containing ionic liquid catalyst was prepared by the reaction between silyl-functionalized imidazolium ionic molecules and silanol groups of silica surfaces, followed by addition of nickel chloride. The present catalyst provides active sites for the synthesis of bis-thiazolidinones. The advantages of this method are the use of an efficient catalyst, retrievability of the catalyst, little catalyst loading, low reaction times, simple procedure, high atom economy and excellent yields.

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SUPPLEMENTARY DATA

Supplementary data associated with this article can be found, in the online version, at this web site.

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