



Ferrocene labelled supported ionic liquid phase (SILP) containing organocatalytic anion for multi-component synthesis

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ABSTRACT

Ferrocene labelled supported ionic liquid phase (SILP) catalyst containing L-prolinate anion has been synthesized by anion metathesis reactions of ionic liquid like unit grafted on Merrifield resin. The multi-component synthesis of 1-amidoalkyl-2-naphthols from 2-naphthol, aryl aldehydes and amides has been effectively achieved under solvent-free conditions using SILP catalyst. Additionally, the catalyst could be reused for several cycles with slight loss of activity.

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1. Introduction

Developments in the field of catalysis are being reported constantly in the form of novel catalysts, new catalytic reactions and alternative methodologies. Much of the pressure for this is driven by the economic demands to develop systems in which easy separation of products and reuse of catalysts are possible along with high reactivity and selectivity. As a part of this effort an ever increasing degree has been focussed on ionic liquids (ILs) as an alternative reaction media in synthetic chemistry [1]. The tempting properties of ILs such as negligible vapour pressure, large liquidus range, high thermal stability, large electrochemical window and ability to solvate compounds of varying polarity have made them an environmentally attractive alternative to organic solvents. The almost limitless combinations of cation–anion pairs allow the synthesis of plethora of diverse ILs, catering to the needs of any particular process [2]. However, despite their well reorganised advantages, a series of drawbacks such as their high cost as compared to traditional solvents, low biodegradability and (eco)toxicological properties still exist [3]. This factor coupled with their recovery that induces energy consumption *via* distillation [4] has been a major obstacle in the preponderance of their use in industrial scale applications. The new concept of supported ionic liquid phase (SILP) catalysis involving immobilization of ILs onto a surface of a porous high area support material [5] offers a highly attractive strategy to

circumvent the drawbacks associated with ILs. This new class of advanced materials dramatically reduces the amount of ILs used, retaining their properties. A number of benefits arise from the use of SILP catalysts, chief of which are the ease of separation of catalyst simply by filtration, as well as facilitating significant advances in selectivity, recycling reproducibility and activity. The concept of SILP catalysis has significantly progressed in the last few years, resulting in new applications for various organic transformations with the ionic liquid either containing a catalyst or being the catalysts phase itself [6]. The bright prospects of the applications of SILP catalysts in organic synthesis spurred us to investigate their ability to catalyze multi-component reactions (MCRs).

The rapid assembly of molecular diversity utilizing multi-component reactions has received a great deal of attention, most notably for the speedy synthesis of novel molecular libraries [7]. These methodologies are of particularly great utility when they lead to the formation of privileged medicinal scaffolds. 1-Amidoalkyl-2-naphthol scaffolds are of significant medical relevance since they can be converted into hypertensive and bradycardiac active 1-aminoalkyl-2-naphthols by amine hydrolysis reactions [8]. 1-Amidoalkyl-2-naphthols are prepared by MCR of 2-naphthol with aryl aldehydes and amides. Although the reaction has been investigated using number of Lewis or Bronsted acid catalysts [9–20], there is still a scope for improvement especially towards developing a green protocol using highly efficient and reusable catalyst.

In continuation of our research devoted to the applications of ferrocene in organic synthesis [21,22], we report herein an improved strategy for the synthesis of a series of 1-amidoalkyl-2-naphthols using ferrocene labelled supported ionic liquid phase

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(SILP) catalyst containing L-prolinate anion under solvent-free conditions.

2. Experimental

2.1. General remarks

¹H NMR spectra were recorded on a Bruker Avon 300 MHz spectrometer using DMSO-d₆ as solvent using tetramethylsilane (TMS) as an internal standard. Infrared spectra were recorded on a PerkinElmer FTIR spectrometer. The samples were examined as KBr discs ~5% (w/w). Raman spectroscopy was done by means of Bruker FT-Raman (MultiRAM) spectrometer. Mass spectra were recorded on a Shimadzu QP2010 GCMS with an ion source temperature of 280 °C. The materials were analysed by SEM using a JEOL model JSM with 5 kV accelerating voltage. Elemental analyses were performed on EURO EA3000 vectro model. For fluorescence studies, the polymeric samples were suspended in methanolic solutions of pyrene (0.01 M) and stirred for 4 h. After filtration and washing with MeOH, the polymers were vacuum dried (60 °C). The samples were introduced into fluorescence cells and excited at 338 nm (PC based spectrofluorometer FP-750 JASCO, equipped with a 450 W xenon lamp) and emitted light was recorded. The various aryl aldehydes (Aldrich/BDH/Fluka), amides, 2-naphthol (sd Fine, India) and Merrifield resin (2% cross-linked, 200–400 mesh, ca 2–3 mmol/g, Alfa Aesar) were used as received. 1-N-Ferrocenylmethyl-1,2,4-triazole was synthesized following the literature procedure [23].

2.2. Preparation of SILP catalyst

2.2.1. Preparation of [FemSILP]Cl (2)

A mixture of Merrifield resin (1.0 g) and 1-N-ferrocenylmethyl-1,2,4-triazole (3 mmol) in 10 mL of toluene was heated at 80 °C in an oil bath. After 72 h, the polymer was filtered, washed with toluene (3 × 20 mL), MeOH (3 × 20 mL), CH₂Cl₂ (3 × 20 mL) and dried under vacuum at 50 °C for 48 h to afford [FemSILP]Cl (2). IR (neat, thin film): $\nu = 3111, 3014, 2923, 2847, 1601, 1556, 1490, 1450, 1390, 1259, 1187, 821, 699, 540 \text{ cm}^{-1}$; Raman: $\nu = 3186, 3135, 3007, 2891, 1532, 1415, 1383, 992, 792, 459 \text{ cm}^{-1}$; elemental analysis observed: %C 65.77, %H 4.68, and %N 4.44. Loading: 1.05 mmol functional group⁻¹ resin.

2.2.2. Preparation of [FemSILP]OH (3)

[FemSILP]Cl (1.0 g) was suspended in 10 mL of an aqueous solution of NH₃. The system was stirred for 48 h at room temperature. Afterwards the polymer was filtered and washed with MeOH (3 × 20 mL), MeOH:H₂O (1:1) (3 × 20 mL), H₂O (3 × 20 mL) and MeOH (3 × 20 mL) and dried under vacuum at 50 °C for 48 h to afford [FemSILP]OH (3). IR (neat, thin film): $\nu = 3490, 3110, 3021, 2921, 2847, 1601, 1555, 1505, 1490, 1447, 1380, 1273, 1176, 818, 699, 543 \text{ cm}^{-1}$; Raman: $\nu = 3537, 3151, 3031, 2937, 2890, 1534, 1417, 1380, 991, 799, 460 \text{ cm}^{-1}$; Elemental analysis observed: %C 66.06, %H 4.93, and %N 5.01. Loading: 1.18 mmol functional group⁻¹ resin.

2.2.3. Preparation of [FemSILP]L-prolinate (4)

[FemSILP]OH (0.9 g) was suspended in 10 mL of a 1 M solution of L-proline in MeOH:H₂O (1:1). The system was heated at 60 °C for 8 h. The filtration of the reaction mixture followed by washing with MeOH (3 × 20 mL), MeOH:H₂O (1:1) (3 × 20 mL), H₂O (3 × 20 mL) and MeOH (3 × 20 mL) afforded [FemSILP]L-prolinate (4). IR (neat, thin film): $\nu = 3397, 3112, 3072, 2920, 2840, 1633, 1601, 1454, 1419, 1380, 1279, 1172, 818, 739, 551 \text{ cm}^{-1}$; Raman: $\nu = 3380, 3185, 3137, 3008, 2890, 1610, 1531, 1416, 1382, 1049,$

991, 897, 791, 463 cm⁻¹; Elemental analysis observed: %C 66.26, %H 5.17, and %N 5.06. Loading: 0.9 mmol functional group⁻¹ resin.

2.3. General method for the synthesis of 1-amidoalkyl-2-naphthols

A mixture of 2-naphthol (1 mmol), aromatic aldehyde (1 mmol), amide (1.5 mmol) and [FemSILP]L-prolinate (50 mg) was stirred at 100 °C in an oil bath. After completion of the reaction as monitored by the TLC, acetone (10 mL) was added and the reaction mixture was filtered. The catalyst was washed with acetone (2 × 10 mL). Evaporation of the solvent followed by column chromatography over silica gel using ethyl acetate/petroleum ether (1:4, v/v) afforded pure 1-amidoalkyl-2-naphthols, which were characterized by spectral methods.

Spectral data for N-[(4-methyl-phenyl)-(2-hydroxy-naphthalen-1-yl)-methyl] benzamide (Table 1, entry 6): ¹H NMR (300 MHz, DMSO-d₆): δ 1.95 (s, 3H), 5.55 (bs, 1H), 6.93 (s, 1H), 7.02 (d, *J* = 7.5 Hz, 2H), 7.05 (s, 1H), 7.13–7.21 (m, 5H), 7.29 (d, *J* = 7.8 Hz, 2H), 7.62 (d, *J* = 9 Hz, 1H), 7.69 (d, *J* = 6 Hz, 1H), 7.89 (d, *J* = 7.9 Hz, 1H), 7.98 (s, 1H), 8.22 (d, *J* = 7.8 Hz, 1H), 9.78 (s, 1H); IR (neat, thin film): $\nu = 3401, 3062, 2922, 1641, 1560, 1514, 1480, 1438, 1380, 1274, 1180, 813, 739 \text{ cm}^{-1}$; MS (EI): *m/z* 366 (M⁺ - 1); anal. calcd. for C₂₅H₂₁NO₂ C 81.72 H 5.76 N 3.81; found: C 81.68 H 5.82 N 3.79.

3. Results and discussion

3.1. Preparation of SILP catalyst

SILP catalysts are prepared either by simple depositing or by covalent attachment of ILs on the surface of high area support like silica or polymeric based materials. We envisioned latter as support for the preparation of SILP catalyst in the present work since they provide reasonable stability to mechanical, chemical and thermal demands under normal operating conditions. As these materials are not able to adsorb ILs as films onto the surface [24], our approach was based on the immobilization by covalent bonding the IL like unit. In the preparation of SILP catalyst (Scheme 1), the 1-N-ferrocenylmethyl-1,2,4-triazole (1) was reacted with Merrifield resin to give the [FemSILP]Cl (2). The resulting compound when treated with aqueous solution of ammonia underwent anion metathesis reaction to form [FemSILP]OH (3). The substitution of hydroxide anion by prolinate by the reaction of [FemSILP]OH with L-proline afforded the ferrocene labelled SILP containing L-prolinate anion, [FemSILP]L-prolinate (4).

The Raman and IR spectroscopy was used to monitor the progress of the reactions involved in the synthesis of SILP catalyst. The reaction of 1 with Merrifield resin was monitored by Raman spectroscopy. The intensities of bands at 639 cm⁻¹ (C–Cl stretching band) and 1266 cm⁻¹ (wagging bands of CH₂–Cl) diminished significantly while the peaks at 459 cm⁻¹ (Fe–Cp stretching band), 1383 cm⁻¹, 1415 cm⁻¹ and 1532 cm⁻¹ (ring stretching modes of triazole ring), 3135 cm⁻¹ and 3186 cm⁻¹ (C–H stretching of Cp rings) increased in intensity after 72 h reflecting the substantial grafting of 1 onto Merrifield resin. Anion metathesis reactions were monitored using IR spectroscopy. Appearance of a band of medium intensity at 3490 cm⁻¹ (O–H stretch) in the IR spectrum of 3 clearly revealed the considerable replacement of Cl by OH. The sharp band at 3397 cm⁻¹ (N–H stretch) and 1633 cm⁻¹ (C=O stretch) in the IR spectrum of 4 confirmed the formation of [FemSILP]L-prolinate.

3.2. Morphology of SILP catalyst

An attempt was made to investigate the morphology of SILP catalyst using scanning electron microscopy. The grafting of IL like

Table 1
[FemSILP]L-prolinate catalyzed multi-component synthesis of 1-amidoalkyl-2-naphthols.^{a,b}

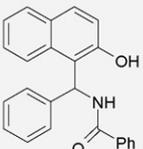
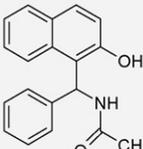
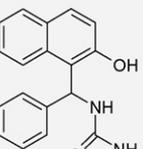
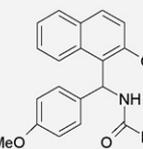
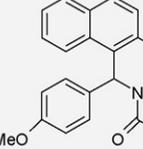
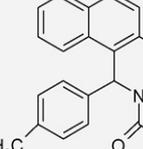
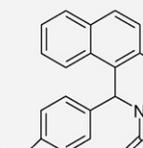
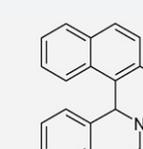
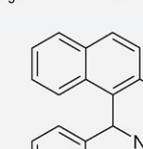
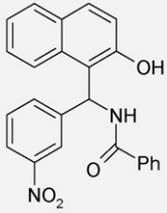
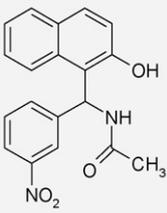
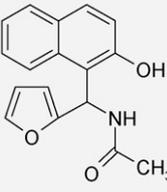
Entry	Aryl aldehyde	Amide	Product	Yield ^c (%)	TON	TOF h ⁻¹
1	Benzaldehyde	C ₆ H ₅ CONH ₂		82	1640	3.64
2	Benzaldehyde	CH ₃ CONH ₂		80	1600	3.95
3	Benzaldehyde	H ₂ NCONH ₂		83	1660	3.35
4	<i>p</i> -Methoxy benzaldehyde	C ₆ H ₅ CONH ₂		69	1380	2.04
5	<i>p</i> -Methoxy benzaldehyde	CH ₃ CONH ₂		71	1420	2.25
6	<i>p</i> -Methyl benzaldehyde	PhCONH ₂		70	1400	2.59
7	<i>p</i> -Methyl benzaldehyde	H ₂ NCONH ₂		74	1480	2.98
8	<i>p</i> -Methyl benzaldehyde	CH ₃ CONH ₂		73	1460	2.70
9	<i>p</i> -Chloro benzaldehyde	PhCONH ₂		84	1680	4.14

Table 1 (Continued)

Entry	Aryl aldehyde	Amide	Product	Yield ^c (%)	TON	TOFh ⁻¹
10	<i>m</i> -Nitro benzaldehyde	PhCONH ₂		86	1720	3.82
11	<i>m</i> -Nitro benzaldehyde	CH ₃ CONH ₂		87	1740	4.29
12	2-Furfuraldehyde	CH ₃ CONH ₂		62	1240	1.62

^a All products were characterized by ¹H NMR and IR spectroscopy as well as by mass spectrometry.

^b No chirality was observed.

^c Isolated yields after chromatography.

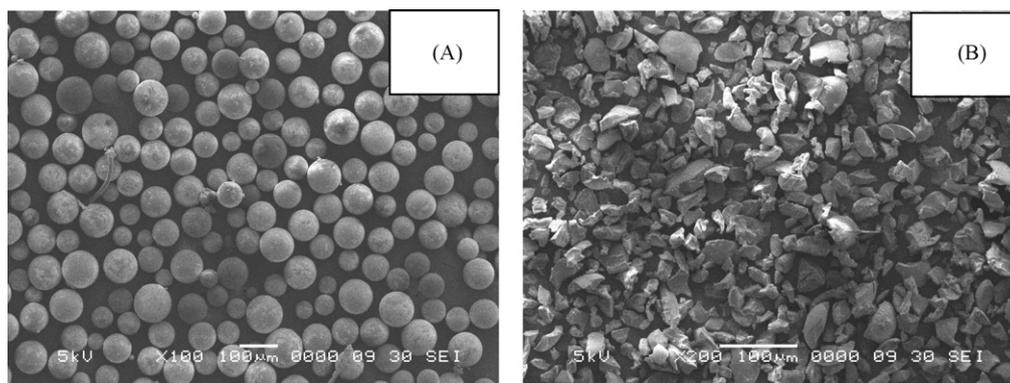


Fig. 1. Scanning electron micrographs (SEM images) of (A) Merrifield resin and (B) Fem[SILP]L-prolinate.

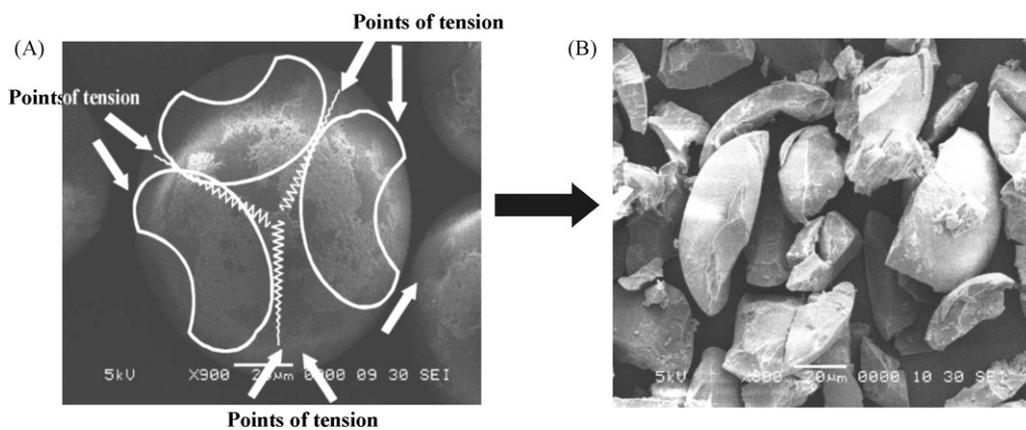
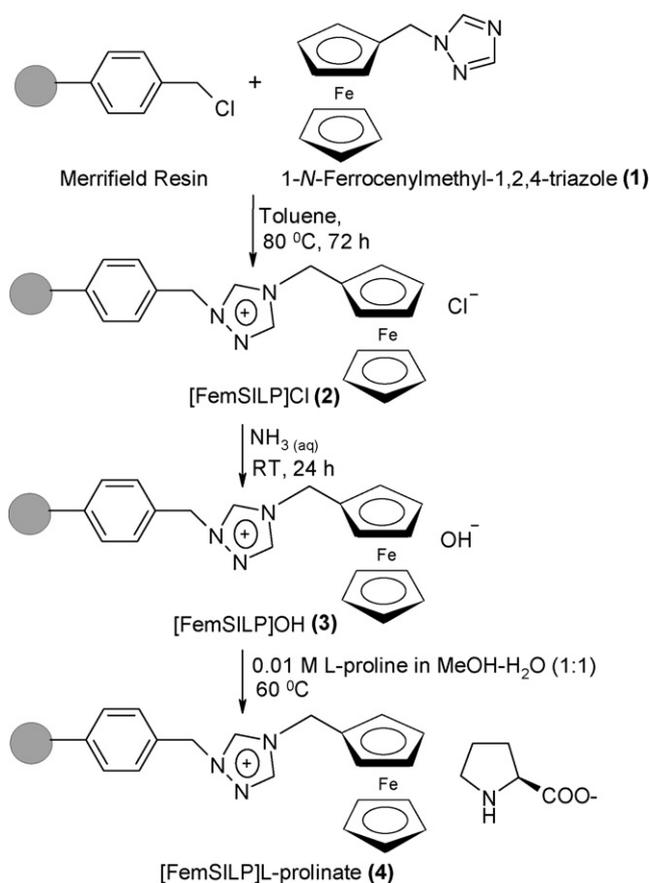


Fig. 2. Sketch of the tension suffered by the Merrifield resin beads (A) and high resolution SEM of totally destroyed beads in [FemSILP]L-prolinate (B).



Scheme 1. Synthesis of ferrocene labelled SILP catalyst containing L-prolinate anion, [FemSILP]L-prolinate (4).

unit on Merrifield resin had a pronounced effect on its morphology. The Merrifield resin beads in SILP catalyst did not appear spherical like the original support beads (Fig. 1).

In fact the bead degradation was observed which may be plausibly due to stress on the bead [25]. High ratios of grafted IL like units mass to bead volume lead to a situation where most of the beads experience enormous tension (Fig. 2A: graphic work has been superimposed on high resolution SEM of Merrifield resin to show points of tension due to grafting of IL like unit). These points of tension evolve and give rise to stress in the bead which eventually bursts (Fig. 2B). The bead degradation did not have any influence on the catalytic activity of SILP catalyst.

3.3. Micropolarity of SILP catalyst

The polarity of ILs is one of the fundamental parameters that ascertain their final properties and this can also be a determinant for SILPs. It has been suggested that the advanced properties of SILP catalysts are, in part, directly related to the change of polarity of SILPs as compared to their standard supports. The polarity of

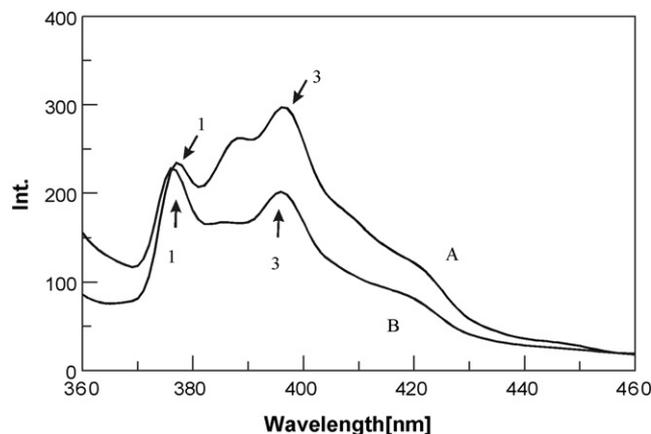


Fig. 3. Fluorescence spectra of pyrene in Merrifield resin (A) and in [FemSILP]L-prolinate (B).

Table 2

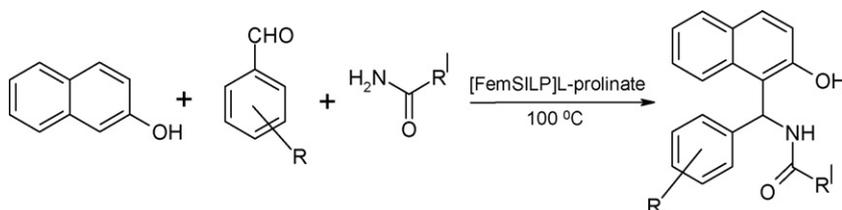
Fluorescence data of pyrene in Merrifield resin and [FemSILP]L-prolinate. *Py* values (I_1/I_3) and positions of peaks I and III.

Entry	Polymer	I_1/I_3	λ_1 (nm)	λ_3 (nm)
1	Merrifield resin	0.789	376	396
2	Fem[SILP] L-prolinate	1.13	377	396

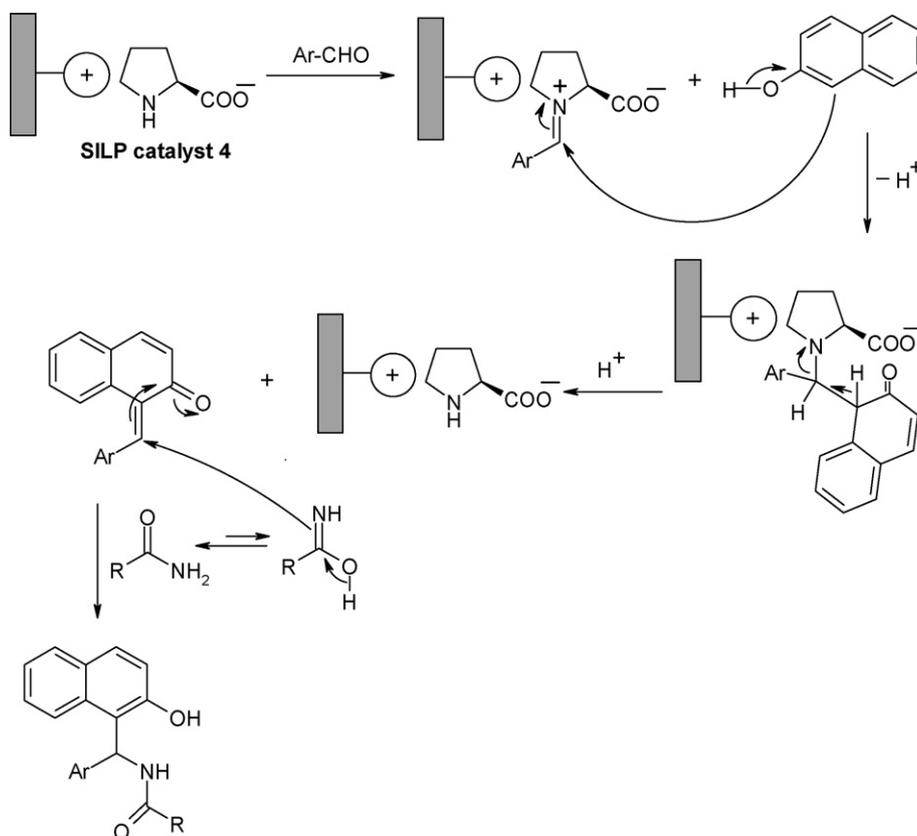
4 was evaluated by steady state fluorescence spectroscopy, using pyrene as probe. In the fluorescence spectra of pyrene, the first band corresponds to the transition $S_1^{v=0} \rightarrow S_0^{v=0}$, and its intensity (I_1) is dependent on the polarity of the medium, provided the existence of vibronic coupling between S_1 and S_2 states while the third band corresponds to the $S_1^{v=0} \rightarrow S_0^{v=2}$ transition and its intensity (I_3) is independent of the polarity. Hence, the ratio of I_1/I_3 (referred as *py* value) has been used to estimate the polarity of several media including SILPs [26]. The fluorescence spectra of pyrene adsorbed on the Merrifield resin and 4 were measured (Fig. 3). The spectrum of Merrifield resin showed the *py* value of 0.789 while that of 4 showed the *py* of 1.13 (Table 2). The increase in the *py* value clearly reflects the increased polarity of 4 as compared to the Merrifield resin. The substantially higher *py* value of SILP catalyst (ca 43%) as compared to Merrifield resin can be attributed to the grafting of ferrocene labelled triazolium unit in the matrix of Merrifield resin. The presence of ferrocene leads to unsymmetrical charge distribution on the triazole ring resulting in the significant enhancement of overall polarity of SILP catalyst.

3.4. Synthesis of 1-amidoalkyl-2-naphthols using SILP catalyst

The synthesis of 1-amidoalkyl-2-naphthols was achieved by multi-component condensation of 2-naphthol with aromatic aldehydes and amides in the presence of catalytic amount of 4 (Scheme 2). To find the optimum conditions, the reaction between 2-naphthol, benzaldehyde, and benzamide in the presence of SILP catalyst (0.05 g) as a model was performed under solvent-free con-



Scheme 2. [FemSILP]L-prolinate promoted synthesis of 1-amidoalkyl-2-naphthols.



Scheme 3. Mechanism of 1-amidoalkyl-2-naphthol synthesis using SILP catalyst.

Table 3

Optimization of the temperature in the preparation of 1-amidoalkyl-2-naphthols.^{a,b}

Entry	Amount of catalyst (g)	Temp. (°C)	Time (h)	Yield ^c (%)	TON	TOF h ⁻¹
1	0.05	RT	5	32	640	1.42
2	0.05	40	5	40	800	1.77
3	0.05	60	5	42	840	1.86
4	0.05	80	5	68	1360	3.02
5	0.05	100	5	82	1640	3.64

^a The molar ratios of benzaldehyde/2-naphthol/benzamide is 1/1/1.5.

^b No chirality was observed.

^c Yields refer to pure isolated product.

ditions at different temperatures. As can be seen from Table 3, the best results were achieved at 100 °C. Having established the reaction conditions, a series of 1-amidoalkyl-2-naphthols were synthesized by the reaction of 2-naphthol with various aromatic aldehydes and amides in excellent yields with the TON in the range of 1240–1740 and TOF in the range of 1.62–4.29 h⁻¹. The results are summarized in Table 1. In all cases amidoalkyl naphthols were the sole products and no by-product was observed. For aryl aldehydes, the effect of the electronic factor on the yield appeared to be greater than that of steric effect. The aryl aldehydes with electron withdrawing groups led to the higher yields as compared with those having electron donating groups. This observation can be rationalized on the basis the mechanistic details of the reaction [12]. The reaction involves *in situ* generation of *ortho*-quinone methides (o-QMs) by the condensation of aromatic aldehyde with 2-naphthol. The 1,4-nucleophilic addition of amides on o-QMs affords the synthesis of 1-amidoalkyl-2-naphthols. Electron withdrawing groups on aryl aldehyde in the o-QMs facilitates the rate of 1,4-nucleophilic addition as the alkene LUMO is at lower energy in their presence compared with electron donating groups. The identity of all the compounds was ascertained on the basis of IR, ¹H NMR and mass

spectroscopy data as well as on the basis of elemental analysis. The physical and spectroscopic data were in full agreement with the literature data [9–20]. The plausible mechanism for the formation of amidoalkyl naphthols using SILP catalyst is shown in Scheme 3. The

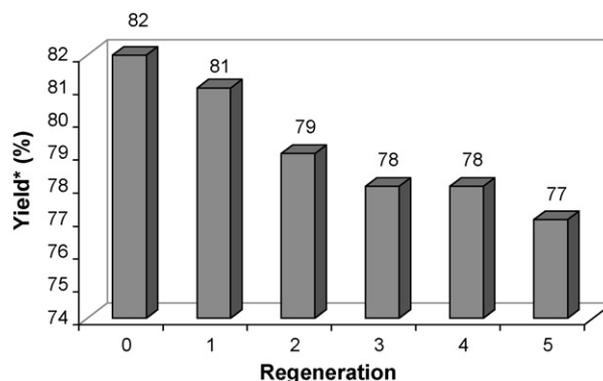


Fig. 4. Recyclable use of [FemSILP]_L-prolinate in 1-amidoalkyl-2-naphthol synthesis.

L-prolinate anion plays a vital role in the catalytic activity of SILP catalyst. It initially facilitates the formation of *o*-QM from aryl aldehyde and 2-naphthol. The subsequent 1,4-nucleophilic addition of amide on *o*-QM furnishes the corresponding 1-amidoalkyl-2-naphthol.

3.5. Reusability of SILP catalyst

We observed that the catalyst can be easily recovered after the reaction simply by filtration and can be reused without any significant loss in chemical yield of the products. The reusability of the catalyst was investigated by the reaction of benzaldehyde, 2-naphthol and benzamide. As shown in Fig. 4, the yield of the product decreased slightly from the first run to fifth run.

4. Conclusion

In summary, we have developed an efficient method for multi-component synthesis of 1-amidoalkyl-2-naphthols using ferrocene labelled SILP catalyst containing L-prolinate anion under solvent-free conditions. Short reaction times, high yields, clean reactions, easy recovery and efficient reusability of catalyst not only make this protocol an alternative to the existing methods but also become significant under the roof of environmentally greener and safer processes.

Acknowledgements

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