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# Design and characterization of nano-silica-bonded 3-*n*-propyl-1-sulfonic acid imidazolium chloride {nano-SB-[PSIM]Cl} as a novel, heterogeneous and reusable catalyst for the condensation of arylaldehydes with β-naphthol and alkyl carbamates

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**Abstract** In this research, nano-silica-bonded 3-*n*-propyl-1-sulfonic acid imidazolium chloride {nano-SB-[PSIM]Cl} as a new and efficient Brønsted acidic ionic liquid supported on silica has been synthesized, and characterized using Fourier transform infrared spectroscopy, scanning electron microscopy, transmission electron microscopy, thermogravimetric, differential thermal gravimetric, X-ray diffraction, and energy-dispersive X-ray spectroscopy spectra. The presented silica-supported ionic liquid has been applied as a highly effective, heterogeneous, easy regenerable, and reusable catalytic system for the solvent-free condensation of arylaldehydes with β-naphthol and alkyl carbamates leading to α-carbamatoalkyl-β-naphthols. Once the nanocatalyst was regenerated and reused, no significant loss of its activity was observed.

*Graphical Abstract* Design and characterization of nano-silica-bonded 3-*n*-propyl-1-sulfonic acid imidazolium chloride {nano-SB-[PSIM]Cl} as a novel, heterogeneous and reusable catalyst for the condensation of arylaldehydes with β-naphthol and alkyl carbamates.

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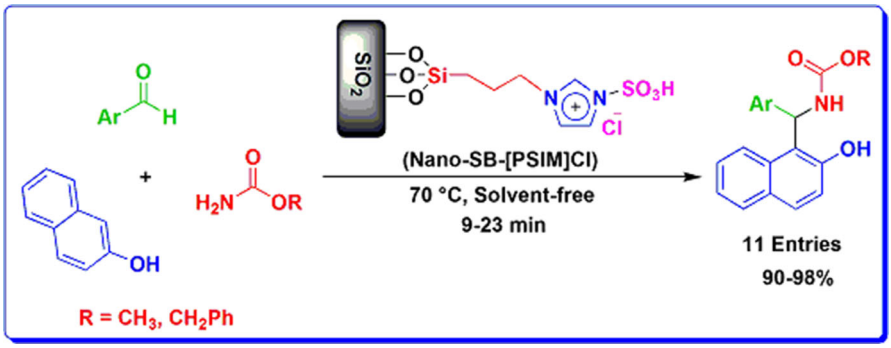



**Keywords** Nano-silica-bonded 3-*n*-propyl-1-sulfonic acid imidazolium chloride {nano-SB-[PSIM]Cl} · Silica-supported Brønsted acidic ionic liquid · Arylaldehyde · β-Naphthol · Alkyl carbamate · α-Carbamatoalkyl-β-naphthol

## Introduction

As catalysts for organic reactions and green reaction media, ionic liquids (ILs) have been applied for many interesting transformations. They have various distinct properties such as non-volatility, a wide liquid-state temperature range, non-flammability, very low vapor pressures, reasonable thermal and chemical stability, excellent ionic conductivities, electrochemical stability, a large electrochemical window, recyclability as well as reusability, eco-friendly, tunable hydrophobicity, and favorable solvation behavior [1–14]. Because of their operational efficacy and selectivity along with their green nature, among different types of ILs, Brønsted acidic ones have shown the potential for ,, progress of environmental friendly acid-catalyzed organic reactions [5–14]. On the other hand, lately, heterogenization of ILs by decorating them on solid supports has grasped an ever increasing attention. The immobilization of IL catalysts can extensively expand their applications in addition to the advantages of their homogeneous counterparts, their application, handling, isolation, and reusing process make them superior to other catalysts. From the economic point of view, it is highly favorable to minimize the amount of IL applied in the potential transformations [15–21], and alternatively solid-supported acidic ILs have been applied as remarkable heterogeneous catalysts for a myriad of acid-catalyzed transformations [15–21].

The chemistry of nano-sized materials is a growing and effective field in the modern technology. Nanocatalysts continue to attract attention for a variety of research disciplines because of their different chemical and physical characteristics once compared with bulk materials. The nano-sized particles maximize the surface area exposed to the reagents facilitating more reactions to take place concurrently, which, therefore, enhances the rate of the chemical reaction [22–27].

Multi-component reactions (MCRs) play a significant role in combinatorial chemistry since they can synthesize desired products with better efficacy and atomic

economy in a single step from three or more starting materials. Furthermore, they decrease energy consuming steps such as separation and purification of intermediates, and improve raw materials consumption. MCRs also present the advantage of synthetic efficiency and simplicity compared with conventional chemical reactions [28–31].

Structures holding α,γ-amino-oxygenated motifs are significant in a variety of biologically distinct natural products and potent pharmaceutical compounds such as nucleoside antibiotics and HIV protease inhibitors like ritonavir and lipinavir [32, 33]. Derivatives of α-carbamatoalkyl-β-naphthol are considered as a class of α,γ-amino-oxygen containing systems. In addition, these structures can be easily transformed into α-aminoalkyl-β-naphthols as a biologically important motif by carbamate hydrolysis. For example, α-aminoalkyl-β-naphthol derivatives have bradycardiac and hypotensive properties [34, 35]. One of the rational synthetic approaches toward α-carbamatoalkyl-β-naphthols is the catalyzed condensation of arylaldehydes with β-naphthol and alkyl carbamates [36–50].

Recently, we introduced sulfonic acid-functionalized imidazolium salts (SAFIS) and quaternary ammonium salts as a new type of Brønsted acidic ILs [9–14]. In these materials, $SO_3H$ functional groups have been bonded to positive nitrogens ($N^+$) for the first time. We applied them as highly efficient and homogeneous catalysts for different organic transformations [9–14]. Although these ILs efficiently catalyzed several organic reactions, recycling and reusing the ILs were difficult. To overcome this drawback, we decided to heterogenize this class of ILs by supporting them on solids. In this regard, we report in the present work, nano-silica-bonded 3-*n*-propyl-1-sulfonic acid imidazolium chloride {nano-SB-[PSIM]Cl} as the first class of heterogenenized SAFIS (Fig. 1), and fully characterize it by Fourier transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM), transmission electron microscopy (TEM), thermogravimetric (TG), differential thermal gravimetric (DTG), X-ray diffraction (XRD), and energy-dispersive X-ray spectroscopy (EDX) analysis. Encouraged by that, we utilize the heterogeneous SAFIS as a highly effective, easy regenerable and reusable nanocatalyst for the solvent-free production of α-carbamatoalkyl-β-naphthols through the condensation between arylaldehydes, β-naphthol, and alkyl carbamates under relatively mild conditions (70 °C). In this research, a helpful organic reaction is reported via the combination of heterogeneous and solid-supported IL nanocatalyst under solvent-free and relatively mild conditions, which represents an interesting approach toward the so-called "ideal synthesis."

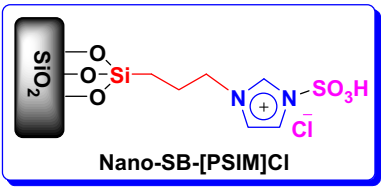



**Fig. 1** The structure of nano-silica-bonded 3-*n*-propyl-1-sulfonic acid imidazolium chloride

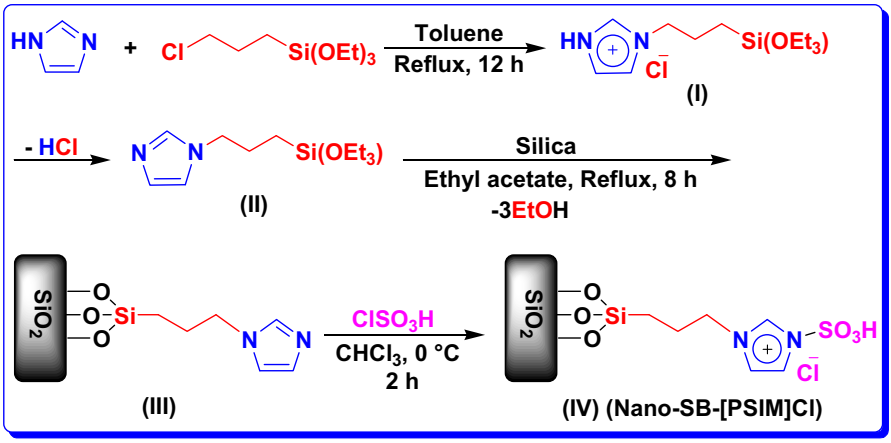
## Experimental

### General

All starting materials were purchased from Merck or Fluka Chemical Companies. The nano-silica gel for the preparation of nano-SB-[PSIM]Cl was synthesized by the reported procedure [51]. For identification of known products, their melting points and spectral data were compared with the reported data in the literature. Progress of the reactions was monitored by TLC using silica gel SIL G/UV 254 plates. The $^{1}H$ NMR (400 MHz) and $^{13}C$ NMR (100 MHz) were run on a Bruker Avance DPX FT-NMR spectrometer ($\delta$ in ppm). Melting points were recorded on a Büchi B-545 apparatus in open capillary tubes. Thermal gravimetry (TG) and differential thermal gravimetric (DTG) were analyzed by a Perkin Elmer (Model: Pyris 1). TG/DTG analysis was done (25–600 °C, temperature increase rate of 10 °C $min^{-1}$, nitrogen atmosphere).

### Procedure for the production of nano-silica-bonded 3-*n*-propyl-1-sulfonic acid imidazolium chloride {nano-SB-[PSIM]Cl}

A mixture of imidazole (0.34 g, 5 mmol), (3-chloropropyl)triethoxysilane (1.125 g, 5 mmol) and toluene (15 mL) in a 50 mL round-bottomed flask connected to a reflux condenser, was stirred for 12 h under reflux conditions. The obtained white precipitate was filtered, washed with toluene (2 × 5 mL), and dried to give intermediate **II** (Scheme 1). In the next step, the obtained intermediate **II** reacted with silica gel (0.30 g, 5 mmol) in refluxed ethyl acetate (15 mL) for 8 h. The formed precipitate was separated by centrifuging and decanting, washed by ethyl acetate (2 × 5 mL), and dried to afford intermediate **III**. Subsequently, a solution of chlorosulfonic acid (5 mmol) in chloroform (10 mL) was added dropwise to




**Scheme 1** The production of nano-SB-[PSIM]Cl

intermediate **III** at 0 °C (ice-water bath), and stirred for 2 h at this temperature. The solvent was removed by centrifuging and decanting, and the residue was triturated with chloroform (3 × 10 mL), and dried under powerful vacuum at 90 °C to give nano-SB-[PSIM]Cl as a white precipitate in a 95 % yield.

### General procedure for the production of α-carbamatoalkyl-β-naphthols

To a mixture of β-naphthol (0.144 g, 1 mmol), arylaldehyde (1 mmol) and alkyl carbamate (1.3 mmol) in a test tube, was added nano-SB-[PSIM]Cl (0.01 g), and the resulting mixture was stirred magnetically at 70 °C, and after solidification of the reaction mixture with a small rod at that temperature. The mixture was cooled to room temperature, then warm EtOAc (5 mL) was added and stirred for 1 min followed by centrifugation and decanting to separate nano-SB-[PSIM]Cl (the silica-bonded IL is not soluble in warm EtOAc, but the unreacted starting materials and the product are soluble in it). The separated EtOAc was evaporated, and the solid residue was recrystallized from hot EtOH (95 %) to give the pure α-carbamatoalkyl-β-naphthol.

**Note** The spectral data of α-carbamatoalkyl-β-naphthols have been given in the Supplementary Material.

## Results and discussion

### Production of nano-SB-[PSIM]Cl (Scheme 1)

At first, imidazole reacted with (3-chloropropyl)triethoxysilane under to give **I**, which transformed to **II** as a white precipitate by releasing HCl gas. In the next step, **II** reacted with silica in ethyl acetate to afford **III**. Subsequently, by the reaction of **III** with chlorosulfonic acid, nano-SB-[PSIM]Cl(**IV**) was produced as a white precipitate.

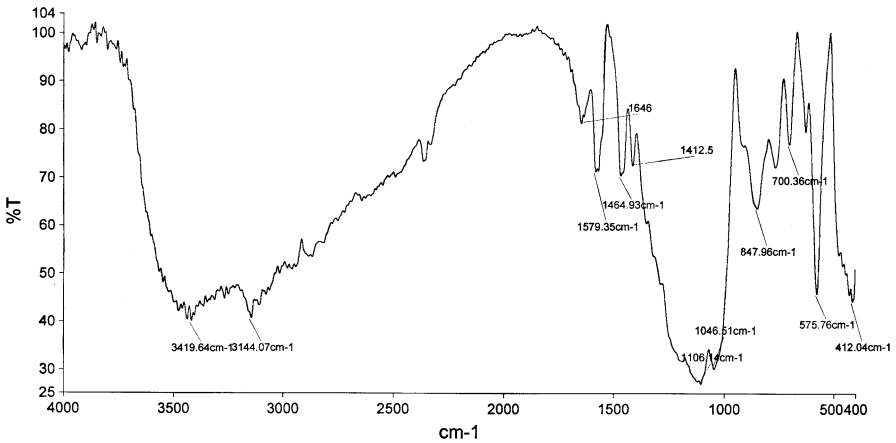



**Fig. 2** The FT-IR spectrum of nano-SB-[PSIM]Cl

## Characterization of the nanocatalyst

The nanocatalyst structure was identified by FT-IR, SEM, TEM, TG, DTG, XRD, and EDX analysis.

*IR spectrum of nano-SB-[PSIM]Cl*

The IR spectrum of the catalyst revealed a broad peak at 2500–3600 $cm^{-1,}$ which could be related to O–H stretching of the $SO_3H$ group. Moreover, the two peaks observed at 1046 and 1106 $cm^{-1}$ corresponded to vibrational modes of N-$SO_2$ and O-$SO_2$ bonds (Fig. 2).

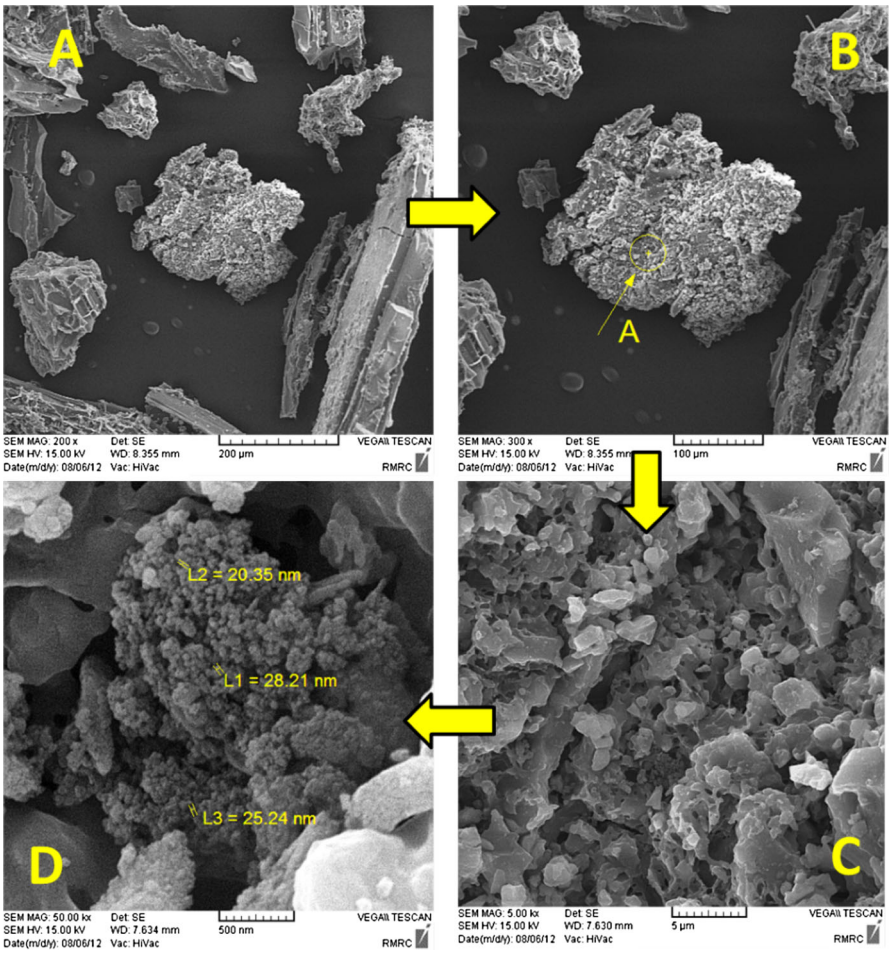



**Fig. 3** The SEM micrographs of nano-SB-[PSIM]Cl

*SEM of the nanocatalyst*

The SEM micrographs of the catalyst are shown in Fig. 3. As is clear from this Fig., the particles of nano-SB-[PSIM]Cl have not completely agglomerated, and the particles were observed in nano-scale.

*TEM of nano-SB-[PSIM]Cl*

To further confirm the nanostructure of nano-SB-[PSIM]Cl, TEM measurements were performed as shown in Fig. 4. The TEM micrograph proved the presence of more or less spherical nanoparticles with a mean diameter of 12.0 nm.

*TG and DTG of the nanocatalyst*

The TG and DTG diagrams of the catalyst showed weight loss in one step at 325 °C (Fig. 5).

*XRD study of nano-SB-[PSIM]Cl*

XRD pattern of nano-SB-[PSIM]Cl was investigated in a domain of 0–90 degree (Fig. 6). As shown in this Fig., XRD patterns exhibited diffraction lines of a high crystalline nature at about $2\theta \approx 9.6°$, 12.1°, 19.5°, 20.2°, 22.8°, and 81.7°.

*EDX of nano-SB-[PSIM]Cl*

EDX of the obtained nanomaterials (Fig. 7) provided the presence of the expected elements in the catalyst structure, namely carbon, oxygen, nitrogen, sulfur, chlorine, and silicon.

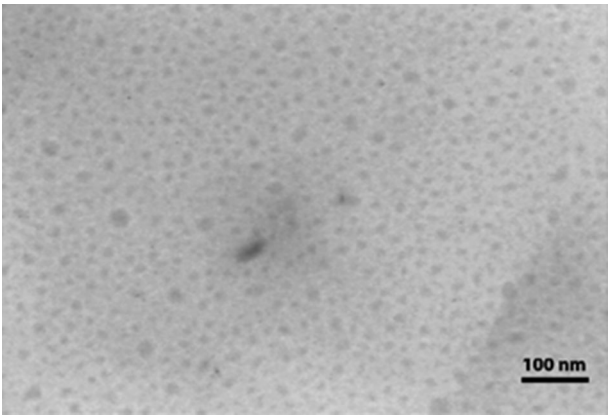



**Fig. 4** The TEM image of nano-SB-[PSIM]Cl

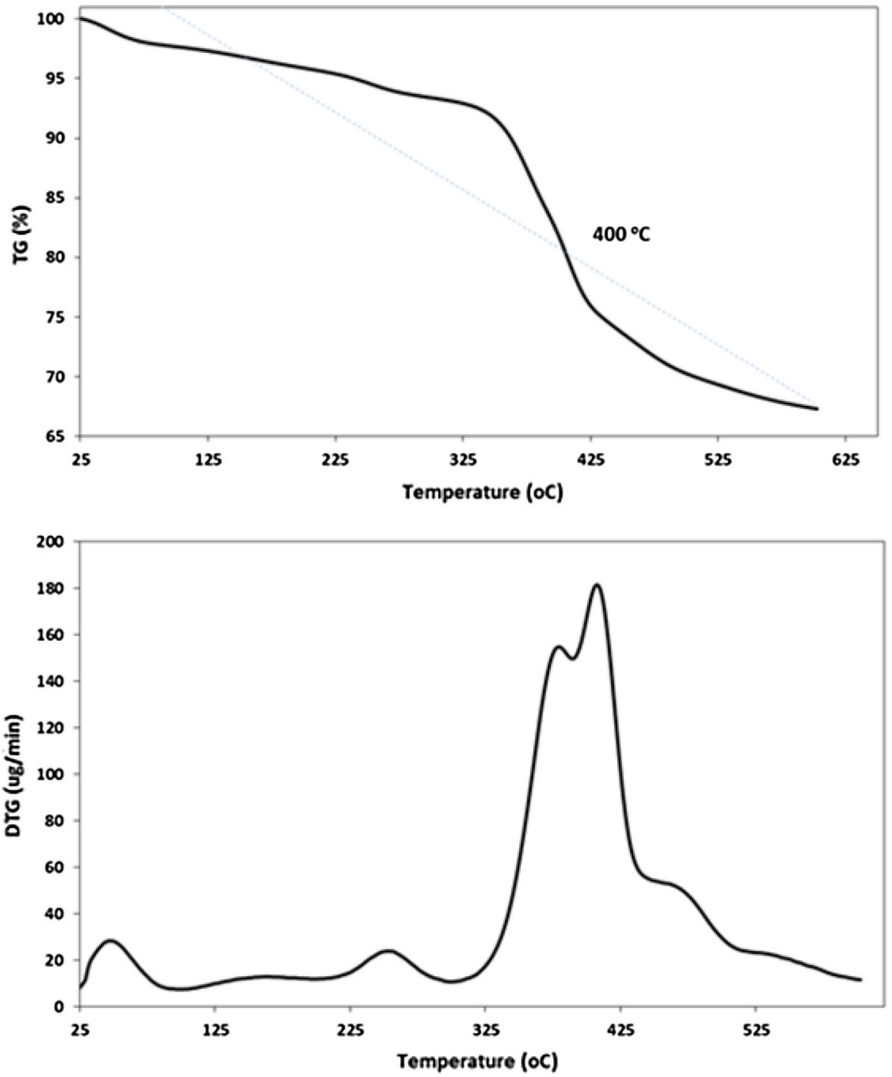



**Fig. 5** The TG and DTG diagrams of nano-SB-[PSIM]Cl at range of 25–600 °C, with a temperature increase rate of 10 °C $min^{-1}$

## Examining catalytic activity of nano-SB-[PSIM]Cl to promote the reaction between arylaldehydes, β-naphthol and alkyl carbamates (Scheme 2)

After the nano-SB-[PSIM]Cl structure was identified, its catalytic activity was examined for the production of an important class of organic compounds, i.e., derivatives of α-carbamatoalkyl-β-naphthols. Hence, *p*-nitrobenzaldehyde (1 mmol) reacted with β-naphthol (1 mmol) and methyl carbamate (1.3 mmol) in the presence of 0.0025–0.0125 g of the nanocatalyst at 50–75 °C in solventless conditions; the

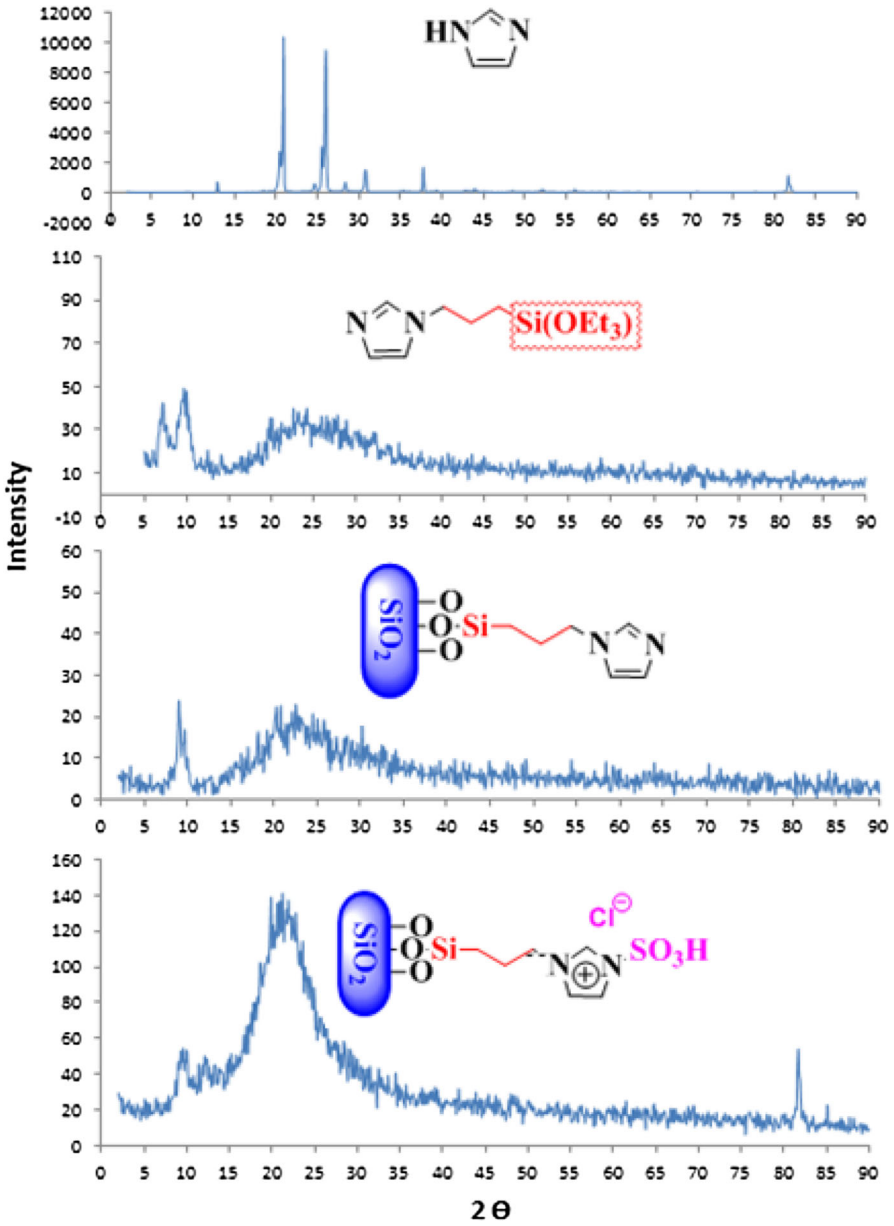



**Fig. 6** The XRD diagram of the nanocatalyst compared with the starting materials

related results are indicated in Fig. 8. As this Fig. shows, the production of the desired α-carbamatoalkyl-β-naphthol was efficiently achieved at 70 °C using 0.01 g of nano-SB-[PSIM]Cl (entry 4).

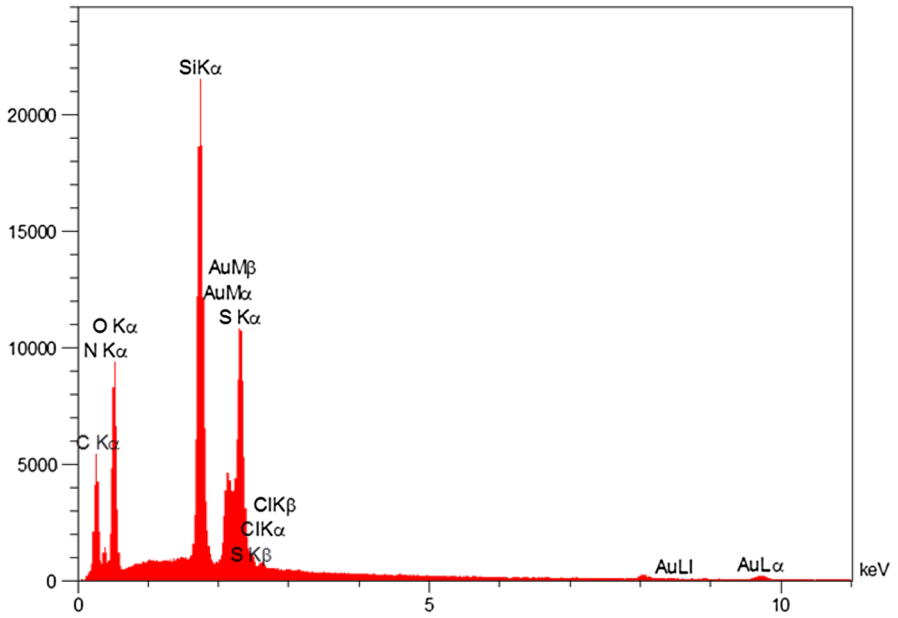
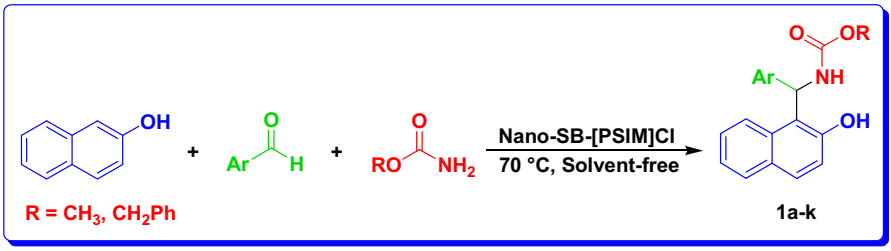



**Fig. 7** The EDX spectrum of nano-SB-[PSIM]Cl




**Scheme 2** The synthesis of α-carbamatoalkyl-β-naphthols catalyzed by nano-SB-[PSIM]Cl

After the reaction conditions were optimized, the efficiency and generality of the first member of heterogenized SAFIS on the production of α-carbamatoalkyl-β-naphthols was assessed by the reaction of different aromatic aldehydes with β-naphthol and alkyl carbamates, and the results are depicted in Table 1. As is indicated in this Table, nano-SB-[PSIM]Cl was highly capable and general for the production of α-carbamatoalkyl-β-naphthols. All substituents on the arylaldehydes including nitro, methyl, methoxy, chloro, bromo, and fluoro afforded the relevant products in short reaction times and in high yields.

In a plausible mechanism, which is illustrated in Scheme 3, we suggest that at first, arylaldehyde is activated by the acidic group of nano-SB-[PSIM]Cl. Afterward, β-naphthol is condensed with the activated arylaldehyde to afford *ortho*-quinone methide (*o*-QM). Subsequently, alkyl carbamate reacts with the

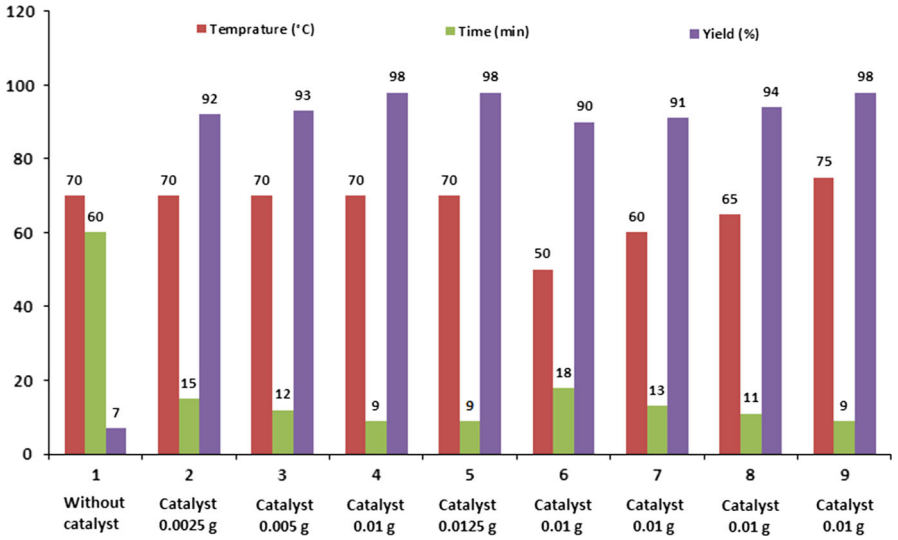



**Fig. 8** Optimization of the catalyst amount and temperature on the reaction of β-naphthol with *p*-nitrobenzaldehyde (1 mmol) and methyl carbamate (1.3 mmol) under solvent-free conditions

activated *o*-QM to give α-carbamatoalkyl-β-naphthol. This mechanism is supported by the literature [38, 40, 43, 44].

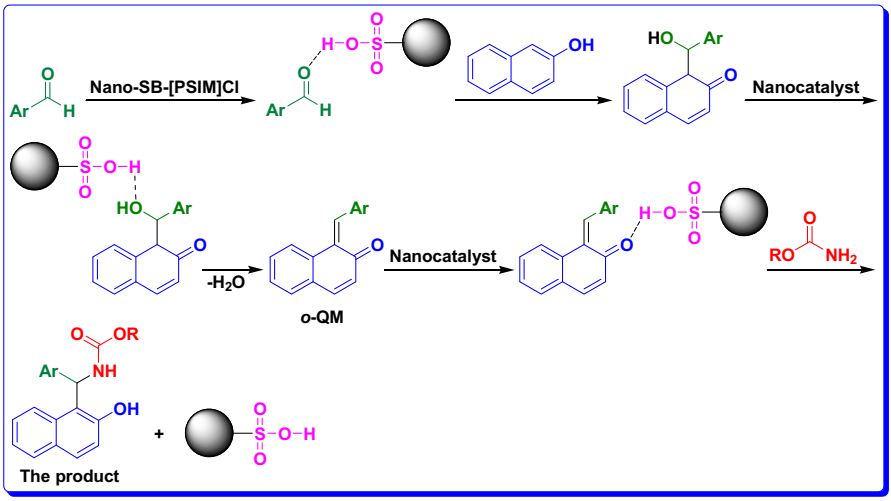
## Regenerability and reusability of the nanocatalyst

The regenerability and reusability of nano-SB-[PSIM]Cl were checked by running the reaction between *p*-nitrobenzaldehyde (1 mmol), β-naphthol (1 mmol) and

**Table 1** The solvent-free production of α-carbamatoalkyl-β-naphthols from arylaldehydes, β-naphthol, and alkyl carbamates using nano-SB-[PSIM]Cl at 70 °C (Scheme 2)

| Ar | R | Product | Time (min) | Yield[a] (%) | M.p. (°C) | |
|---|---|---|---|---|---|---|
| | | | | | Found | Reported |
| $C_6H_5$ | $CH_3$ | **1a** | 13 | 98 | 221–223 | (220–222) [38] |
| *p*-$NO_2C_6H_4$ | $CH_3$ | **1b** | 9 | 98 | 202–204 | (200–202) [44] |
| *m*-$NO_2C_6H_4$ | $CH_3$ | **1c** | 8 | 98 | 248–250 | (249–251) [38] |
| *o*-$NO_2C_6H_4$ | $CH_3$ | **1d** | 14 | 90 | 241–243 | (241–242) [44] |
| *p*-$CH_3C_6H_4$ | $CH_3$ | **1e** | 23 | 95 | 189–191 | (187–189) [45] |
| *p*-$ClC_6H_4$ | $CH_3$ | **1f** | 13 | 97 | 201–203 | (198–200) [41] |
| *m*-$ClC_6H_4$ | $CH_3$ | **1g** | 12 | 97 | 195–197 | (196–198) [41] |
| *p*-$BrC_6H_4$ | $CH_3$ | **1h** | 11 | 95 | 173–175 | (172–174) [38] |
| $C_6H_5$ | $C_6H_5CH_2$ | **1i** | 18 | 93 | 178–180 | (180–182) [44] |
| *m*-$CH_3OC_6H_4$ | $C_6H_5CH_2$ | **1j** | 20 | 94[b] | 181–183 | (182–184) [41] |
| *p*-$FC_6H_4$ | $C_6H_5CH_2$ | **1k** | 18 | 94 | 187–189 | (185–186) [41] |

[a] Isolated yield. [b] In this reaction, 0.015 g of the catalyst was used




**Scheme 3** The proposed mechanism for the synthesis of α-carbamatoalkyl-β-naphthols

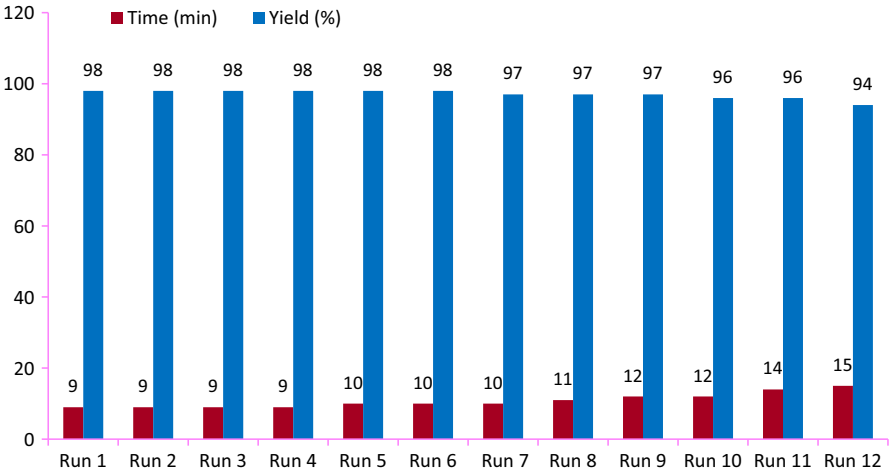



**Fig. 9** The results on regenerability and reusability of nano-SB-[PSIM]Cl

methyl carbamate (1.3 mmol) for several times in the presence of 0.01 g of the nanocatalyst at 70 °C under solventless conditions. After completion of the reaction, the reaction mixtures were cooled to room temperature, and combined. Warm EtOAc was added to the combined reaction mixtures, and stirred for 1 min followed by centrifugation and decanting to separate nano-SB-[PSIM]Cl. Afterward, the separated catalyst was washed with EtOAc, and separated from the solvent by centrifuging and decanting. The recovered catalyst was charged by chlorosulfonic acid according to procedure 2.2 to provide regenerated nano-SB-[PSIM]Cl. The catalytic activity of the regenerated catalyst after 12 runs indicated no significant loss compared with the fresh one. The results are displayed in Fig. 9.

## Conclusions

In summary, we have introduced a novel, heterogeneous and attractive nanocatalyst, namely nano-silica-bonded 3-*n*-propyl-1-sulfonic acid imidazolium chloride {nano-SB-[PSIM]Cl}, for organic synthesis. In this work, we have utilized the nanocatalyst to promote the solvent-free condensation of arylaldehydes with β-naphthol and alkyl carbamates providing α-carbamatoalkyl-β-naphthols. The advantages of nano-SB-[PSIM]Cl include performing the reaction in high yields and short times in the absence of solvent under relatively mild conditions, generality, efficiency, non-toxicity, and with a green nature.

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