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Synthetic Communications

An International Journal for Rapid Communication of Synthetic Organic Chemistry

ISSN: 0039-7911 (Print) 1532-2432 (Online) Journal homepage: http://www.tandfonline.com/loi/lsyc20

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To cite this article: Nilesh N. Korgavkar & Shriniwas D. Samant (2018) 1,3-Dipolar cycloaddition reaction of aryl nitrile oxides with alkenes using imidazole and pyridine containing reusable polymeric base catalysts, Synthetic Communications, 48:4, 387-394, DOI: 10.1080/00397911.2017.1399207

To link to this article: https://doi.org/10.1080/00397911.2017.1399207

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Published online: 18 Jan 2018.

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1,3-Dipolar cycloaddition reaction of aryl nitrile oxides with alkenes using imidazole and pyridine containing reusable polymeric base catalysts

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ABSTRACT

Cross-linked poly-1-(4-vinylbenzyl)imidazole and cross-linked poly-4vinylpyridine have been prepared from respective vinyl monomers using different quantities of divinylbenzene as a cross-linker by radical polymerization. The polymeric bases were characterized by FT-IR, TGA, field emission-scanning electron microscopy (FE-SEM), and Brunauer-Emmett-Teller (BET) surface area analysis. Their swelling behavior was also studied. They were used as base catalysts for 1,3-dipolar cycloaddition reaction of aryl nitrile oxides, generated *in situ* from *N*-hydroxyimidoyl chloride, with *N*-phenylmaleimide and ethyl acrylate, to obtain 3-arylisoxazolines. Both the bases gave excellent results. These polymers were reusable, safe to use, and provided good alternative for organic nonreusable bases like pyridine and triethylamine which are hazardous.



Received 10 August 2017

KEYWORDS

1,3-Dipolar cycloaddition; nitrile oxide; poly-4vinylpyridine; polymeric base catalyst





Introduction

Isoxazoline unit is a part of many natural products. Many of its derivatives show broad spectrum of biological activity.^[1,2] This unit is also important in synthetic chemistry as a precursor for many useful compounds.^[3–5] One of the routes for the synthesis of this heterocycle is the 1,3-dipolar cycloaddition reaction of nitrile oxides (1,3-dipole) with alkenes (dipolarophiles).^[6–8] Alkenes like 3-methylene oxindoles,^[9] chalcones,^[10] α,β -unsaturated carbonyl compounds,^[11,12] acrylates,^[13] N-Boc- Δ^3 -pyrroline,^[14] N-phenylmaleimide,^[15] allyl amines,^[16,17] allyl alcohols,^[18] styrenes,^[19] and fullerenes^[20,21]

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have been used as dipolarophiles in this reaction. Unsaturated compounds like alkynes, imines,^[22] *in situ* generated benzyne,^[23] and 1,3-dicarbonyl compounds in enol form^[24] also react with nitrile oxides.

Nitrile oxides are unstable and dimerize to form furoxans (1,2,5-oxadiazole-2-oxide) and hence are usually prepared *in situ*. Dehydrohalogenation of *N*-hydroxyimidoyl chloride (chloroxime), in the presence of a base, is one of the commonly used methods for the *in situ* generation of nitrile oxides. Nitrile oxides of aromatic nitriles are comparatively more stable than those of aliphatic nitriles.^[25] The steric shielding of the dipole and the electron delocalization provide the stability. To avoid the dimerization, the concentration of *in situ*-generated nitrile oxide is maintained minimum. In one such effort, a syringe pump has been used for slow addition of *N*-hydroxyimidoyl chloride.^[23] Heterogeneous mixtures of inorganic bases with organic solvents have also been used.^[14]

The reaction usually requires stoichiometric amount of a base. The bases used in the reaction are not reusable. Further, the reaction requires 2–4 equiv. of hydroxyimidoyl chloride, and there is a possibility of dimerization of the nitrile oxide. Taking these points into consideration, there is a need to develop a protocol which would require lesser amount of hydroxyimidoyl chloride and a recyclable weak base.

Organic polymeric amines are heterogeneous recyclable weak bases for organic reactions. We have been working on poly-4-vinylpyridine (PVP) and cross-linked PVP and demonstrated successful use of these bases in some reactions.^[26–28] This prompted us to prepare polymeric bases containing pyridine and imidazole and use them for *in situ* generation of nitrile oxides from *N*-hydroxybenzimidoyl chlorides and subsequent reaction of these nitrile oxides with *N*-phenylmaleimide and ethyl acrylate. Cross-linking is required to make the polymer insoluble in organic solvents and thus makes the base reusable. Hence, divinylbenzene (DVB) was used as a cross-linker. DVB cross-linked poly-1-(4-vinylbenzyl)imidazole (CPVBIm) polymers are not reported. There are few reports on imidazole-based polymers used as base catalysts^[29,30] and in electrochemical studies.^[31,32] Cross-linked poly-4-vinylpyridine (CPVP) has been used as base catalyst in some reactions.^[33–36] These cross-linked polymers swell rapidly on interaction with certain solvents, which increase the accessibility of the basic sites. Polymers swell differently in different solvents. Hence we studied the gelling behavior of the polymers.

Results and discussion

Polymeric catalysts, CPVP-n and CPVBIm-n (n = mol% of DVB) were prepared from 4-vinylpyridine and 1-(4-vinylbenzyl)imidazole, respectively, by radical polymerization using AIBN as a radical initiator and DVB as a cross-linker. Both CPVP-n and CPVBIm-n were amorphous white solids. The amount of cross-linker affected the bulk density and swelling property of the polymers.

Characterization of polymers

The polymers were characterized as follows.

FT-IR

FT-IR spectra were recorded in ATR mode. FT-IR of CPVP showed characteristic bands at 1,598, 1,416, and 821 cm^{-1} , while CPVBIm showed characteristic bands at 1,511, 1,100,

900, and 817 cm^{-1} . The bands at 1,230, 1,076, and 1,030 cm⁻¹ were common to both (Fig. S1).

Thermal analysis

Thermogravimetric analysis (TGA) showed that CPVP-3, 5, and 7 and CPVBIm-3, 5, and 7 were stable up to 350 °C and at higher temperatures the polymers decomposed. The amount of cross-linker had no effect on the thermal stability. But, about 2–3% more mass loss was observed around 100 °C with an increase in cross-linker amount. This increase in mass loss suggested the presence of some water and solvent in the polymers having more amount of DVB. This observation was in consonance with the increase in porosity of the material with an increase in the amount of DVB. TGA graphs are given in the supporting information (Figs. S2, S3).

FE-SEM images

FE-SEM images of CPVP-5 (Fig. S4) showed clusters of spherical particles with average diameter $2.5 \,\mu$ m. For CPVBIm-5, irregular shaped particles were observed (Fig. S5). The images showed typical porous polymer morphology for CPVBIm-5.

Swelling behavior of the basic polymers

The materials were highly porous and showed good swelling property. The swelling helps helps to increase the accessibility of the active sites to the substrate molecules. However, a swollen polymer occupies more volume of the reaction vessel and thickens the reaction mass. Thus, it requires more amount of solvent for efficient mixing. The swelling of polymers was tested in various solvents which are commonly used in the synthesis and polymerization. CPVP and CPVBIm polymers showed high swelling in methanol, ethanol, chloroform and DMF. In DMSO, CPVP dispersed throughout the volume of the solvent and it was difficult to separate it from the solvent. Thus, an increase in mass and volume of CPVP-5 in DMSO could not be measured. Swelling of CPVBIm polymers was comparatively higher than that of CPVP (Fig. 1). Both the polymers floated on chloroform, nitrobenzene, 1,2-dichloroethane, and chlorobenzene.

The two polymers were studied with respect to BET surface area analysis. Both the polymers showed type-IV isotherm (Fig. S6). The hysteresis loop at relative pressure $0.5 < P/P_o < 0.9$ indicated the mesoporous nature of CPVBIm.^[37] The BET surface areas of CPVBIm-5 and CPVP-5 were 274.97 and 36.48 m² g⁻¹, respectively, and the pore volumes of both the polymers were 0.323 and 0.046 cm³ g⁻¹, respectively (Table S3). It is reported that the surface area increases with an increase in the amount of DVB.^[36] Thus, the surface area for CPVP-5 was also in agreement with the previous reports.

1,3-Dipolar cycloaddition reaction of N-hydroxybenzimidoyl chloride and N-phenyl-maleimide in the presence of CPVP and CPVBIm

We selected the reaction between *N*-phenylmaleimide chloride (R=H, 1a) and *N*-phenylmaleimide (2) as a model reaction (Scheme 1) and optimized the reaction conditions. In this reaction, 1 undergoes *in situ* base-catalyzed elimination of HCl to form aryl nitrile oxide.

Effect of base

The reaction was performed in ethanol, DMF, and acetonitrile using TEA as a base. Good yield of **3a** was obtained in all solvents. The results in ethanol were comparatively better.



Ratio of volume of swollen polymer to original volume

Figure 1. Swelling study: (a) swelling of CPVP-5 (b) swelling of CPVBIm-5.

But, the poor solubility of **3a** restricted the use of ethanol under heterogeneous condition. Considering the ease in workup and recovery of solvent, we performed the reaction using polymeric bases in acetonitrile (Table 1). The reaction took place smoothly at room temperature and gave good yield of **3a** within 1 h. For comparison, the reaction was also performed using classical organic bases, TEA, pyridine, imidazole; when comparable results were obtained.

There was no difference in the yield of 3a using CPVBIm-3, 5, and 7 (Table 1, entries 1–3) and similar observation was made using CPVP-3, 5, and 7 (Table 1, entries 4–7). The elemental analysis showed the nitrogen content in the range of 11–12% for



Scheme 1. 1,3-Dipolar cycloaddition reaction of 1a-h with 2 in the presence of polymeric bases.

| Sr. No. | Catalyst (wt%) | Yield of 3a ^a (%) |
|---------|-----------------------------|-------------------------------------|
| 1 | CPVBIm-3 ^b | 80 |
| 2 | CPVBIm-5 ^b | 80 |
| 3 | CPVBIm-7 ^b | 78 |
| 4 | CPVP-3 ^b | 74 |
| 5 | CPVP-5 ^b | 75 |
| 6 | CPVP-7 ^b | 75 |
| 7 | TEA ^c | 85 |
| 8 | Pyridine ^c | 78 |
| 9 | Imidazole ^c | 85 |
| 10 | N–Me imidazole ^c | 80 |

Table 1. Effect of catalyst on the reaction of 1a and 2.

CPVBIm, cross-linked poly-1-(4-vinylbenzyl)imidazole; CPVP, cross-linked poly-4-vinylpyridine. *Reaction conditions*: **1a** (R=H, 3.4 mmol), **2** (1.7 mmol), solvent—acetonitrile (10 mL), rt (27–30 °C), reaction time—1 h.

^alsolated yield calculated w.r.t. 2.

^bPolymeric base 100 wt% w. r. t. 2.

^c1.7 mmol of homogeneous base.

CPVBIm-3, 5, and 7 and in the range of 10–11% for CPVP-3, 5, and 7. As there was no difference, the 5% cross-linked polymers were selected for further studies.

Effect of solvent and molar ratio of 1a and 2

The effect of solvent was studied using CPVBIm-5 as a base catalyst in ethanol, methanol, acetonitrile, ethyl acetate, THF, DMF, and chloroform. Acetonitrile (80%) and DMF (78%) were the best solvents. The 2:1 molar ratio of **1a** to **2** (yield = 80%) was selected; increasing the amount of **1a** did not increase the yield (Table S4).

Effect of catalyst loading

The catalyst was increased from 50 to 150 wt% w.r.t *N*-phenylmaleimide (2). 120 wt% of catalyst was found to be satisfactory (Table S5). The results are also comparable to homogeneous bases under identical conditions (Table 1, entries 7–10). However, polymeric bases are safe to handle, free of odor, and recyclable.

Reaction of 1a-h with N-phenylmaleimide (2)

To study the scope of the new protocol, the reaction of substituted N-hydroxyimidoyl chlorides (1a-h) with N-phenylmaleimide was performed (Table 2).

| | The reaction of it in which 2. | | |
|--------|--------------------------------|--------------------------------|-------------|
| | | Yield of 3a-h ^a (%) | |
| Sr. No | Reactant 1 'R' | With CPVBIm-5 | With CPVP-5 |
| 1 | Н | 85 | 72 |
| 2 | 2-Cl | 80 | 72 |
| 3 | 4-Me | 82 | 77 |
| 4 | 4-Cl | 78 | 70 |
| 5 | 4-OMe | 74 | 65 |
| 6 | 4-NO ₂ | 85 | 78 |
| 7 | 3-Br | 68 | 58 |
| 8 | 3-NO ₂ | 68 | 60 |
| | | | |

Table 2.The reaction of 1a-h with 2.

Reaction conditions: 1a-h (3.4 mmol), 2 (1.7 mmol), solvent—acetonitrile (10 mL), CPVBIm-5 and CPVP-5 (120 wt% w.r.t 2), rt (27–30 °C), reaction time 1 h.

^{*a*}lsolated yield, calculated w.r.t. 2.



Scheme 2. Reaction of 1a-h with ethyl acrylate (4).

| Table 3. Reaction of Ta-h with | 4 | 4. |
|--------------------------------|---|----|
|--------------------------------|---|----|

| Sr. No | Reactant 1 "R" | Yield of 5a–h ^a (%) |
|--------|-------------------|--------------------------------|
| 1 | Н | 74 |
| 2 | 2-Cl | 80 |
| 3 | 4-Me | 82 |
| 4 | 4-Cl | 78 |
| 5 | 4-OMe | 74 |
| 6 | 4-NO ₂ | 85 |
| 7 | 3-Br | 68 |
| 8 | 3-NO ₂ | 68 |

Reaction conditions: 1a-h (3.4 mmol), 4 (1.7 mmol), solvent—acetonitrile (10 mL), CPVBIm-5 (120 wt% w.r.t. 4), rt (27–30 °C), reaction time 6 h.

^alsolated yield, calculated w.r.t. 4.

The structures of 3a-h were confirmed by FT-IR and ¹H NMR spectroscopy, and the data were compared with the reported characterization data of same compounds for verification.^[38-42] The **3a-h** were obtained as the racemic mixtures.

Reaction of 1a-h with ethyl acrylate (4)

The reaction of nitrile oxides with ethyl acrylate (Scheme 2) is known to give 5-substituted isoxazolines (5).^[13] We performed the reactions of 1a-h with ethyl acrylate (4) using CPVBIm-5 in acetonitrile (Table 3). This reaction was slower as compared to the reaction of 1a with *N*-phenylmaleimide.

The structures for **5a**-**h** were confirmed by FT-IR and ¹H NMR spectroscopy, and data were also compared with the previous literature.^[40,42–45] The **5a**-**h** were obtained as the racemic mixtures. Spectral data for all the compounds (**3a**-**h** and **5a**-**h**) have been given in the Supporting information.

Recyclability study

The recyclability of CPVBIm-5 was studied for both the reactions. The spent catalyst was washed with aqueous saturated sodium bicarbonate solution and then with acetonitrile before being used in the next cycle. The recyclability was studied up to five cycles and there was no significant decrease in the yield of the product (Figs. S7, S8). Only decrease of about 4-5% in the isolated yield after the fifth recycle was observed. Some basic sites on surface may be blocked with CO₂ or water, or some basic sites reacted with HCl in reaction medium (acetonitrile) and are difficult to regenerate.

Experimental

Starting materials were purchased from Sigma-Aldrich, Loba Chemie, and Alfa Acer and used as such. TGA was performed on PerkinElmer STA 6000 with heating rate

 20 °C min^{-1} . FE-SEM analysis was performed using Tescan MIRA 3 model. Nitrogen adsorption-desorption isotherms were recorded on BET-PMI sorptometer (porous material integrated Pvt. Ltd., USA). Samples were degassed at 150 °C for 5 h before analysis. FT-IR spectra were recorded on a PerkinElmer Spectrum Two spectrophotometer. ¹H NMR spectra were recorded on Agilent 400 MHz NMR spectrometer. Elemental analysis (C, H, N) was performed on vario MICRO select elemental analyzer. Experimental procedures for the preparation of CPVP-*n* and CPVBIm-*n* have given the Supporting information.

1,3-Dipolar cycloaddition reaction of N-hydroxybenzimidoyl chloride (1a) and N-phenylmaleimide (2)

N-hydroxybenzimidoyl chloride (3.4 mmol) and *N*-phenylmaleimide (1.7 mmol) were added to acetonitrile (10.0 mL) in a round-bottom flask. The polymeric base (0.3 g) was added to it. The mixture was stirred at 27–30 °C for 1 h. The base was separated from the reaction mixture by filtration. Acetonitrile was removed under reduced pressure at 50 °C. Ethanol (3.0 mL) was added to the reddish brown oily residue and was allowed to stand at ambient temperature (27–30 °C) for 20–30 min. A white solid product was precipitated, which was isolated by filtration, and dried at 80 °C.

The same procedure was followed with ethyl acrylate. The reaction time in this case was 6 h. The products were purified by column chromatography with silica as a stationary phase and ethyl acetate (0-5% v/v): PET ether as a mobile phase.

Conclusion

Cross-linked basic polymers were prepared by the polymerization of 4-vinylpyridine (4-VP) and 1-(4-vinylbenzyl)imidazole using 3, 5, and 7 mol% of DVB as a cross-linker. The resulting polymers CPVP-*n* and CPVBIm-*n* were mesoporous and excellent heterogeneous catalysts for *in situ* generation of aryl nitrile oxides from *N*-hydroxyimidoyl chlorides and their 1,3-dipolar cycloaddition reaction with *N*-phenylmaleimide and with ethyl acrylate. CPVBIm-5 showed excellent results compared to CPVP-5. The much high activity of CPVBIm-5 can be explained on the basis of high basicity of imidazole than pyridine, and much high surface area and porosity of CPVBIm-5. These parameters should be kept in mind for further developments of related catalysts. The main advantages of this protocol were replacement of homogeneous, pungent smelling, hazardous organic bases with recyclable polymeric bases.

Acknowledgment

Author Nilesh N. Korgavkar is thankful to the University Grant Commission (UGC), New Delhi, India for UGC/SAP/DRS fellowship.

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