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Synthesis and characterization of a novel Fe₃O₄@SiO₂/ bipyridinium dichloride nanocomposite and its application as a magnetic and recyclable phase-transfer catalyst in the preparation of β -azidoalcohols, β -cyanohydrins, and β -acetoxy alcohols

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ABSTRACT

 β -Azidoalcohols, β -cyanohydrins, and β -acetoxy alcohols have been synthesized in the presence of a Fe₃O₄@SiO₂/bipyridinium nanocomposite (Fe₃O₄@SiO₂/BNC) as a novel magnetic and recyclable phase-transfer catalyst (PTC) in water. The catalyst was characterized with FT–IR, SEM, XRD, VSM, and TGA. This methodology offers several advantages, including easy work-up procedure, excellent regioselectivity, high yields, short reaction times, recyclable catalyst, easy separation of the catalyst through an external magnet and eco-friendly procedure.

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1. Introduction

Due to their simplicity of synthesis and their recyclability, magnetic-nanoparticle-supported catalysts, especially iron oxide nanoparticles such as magnetite (Fe₃O₄) and maghemite (γ -Fe₂O₃) have been greatly utilized in organic reactions, including hydrogenation and reduction, oxidation, hydroformylation, C–C coupling, asymmetric, biocatalytic, olefin metathesis, and other catalytic reactions [1–4]. Different catalysts have been immobilized on magnetic nanoparticles (MNPs), including:

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- organic materials such as small molecules and surfactants, polymers, ionic liquids and biological molecules;
- inorganic materials such as silica, metals and nonmetals, metal oxides and sulfides [5,6].

Because of simple experimental operations, mild reaction conditions, inexpensive, environmentally benign reagents and solvents and large-scale reaction, phasetransfer catalysis (PTC) is a very useful approach [7]. One practical limitation of the phase-transfer method, however, is that many of the catalysts used promote the formation of stable emulsions [8]. Immobilization of the phase-transfer catalyst on an insoluble magnetic support can provide a simple solution to this problem.

 β -Azido alcohols are compounds of interest in organic synthesis as either precursors of vicinal amino alcohols or

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in the chemistry of carbohydrates, nucleosides, lactames, and oxazolines [9]. Generally, azidohydrins are prepared through ring opening reactions of epoxides by using different azides in suitable solvents. Even though the classical protocol uses sodium azide and ammonium chloride, the azidolysis reaction requires a long reaction time (12–48 h) and the azidohydrin is often accompanied by isomerization, epimerization, and rearrangement of products [10].

Although a few reagents and catalysts have been reported recently for the conversion of epoxides to β -azido alcohols, β -cyanohydrins, and β -acetoxy alcohols [11–18], disadvantages such as long reaction times, low yields of products, difficulty in the preparation and/or the storage of reagents or catalysts, tedious workup, and, in most cases, low regioselectivity, clearly identify a need for introducing new methods for such functional group transformations.

In continuation of our effort toward the synthesis of novel catalysts, and their application in organic synthesis [19–24], an Fe₃O₄@SiO₂/bipyridinium nanocomposite (Fe₃O₄@SiO₂/BNC) has been introduced as a novel heterogeneous phase-transfer catalyst for the one-pot preparation of β -azido alcohols, β -cyanohydrins, and β -acetoxy alcohols in aqueous medium (Scheme 1).

2. Experimental

2.1. General

All commercially available chemicals were purchased from Fluka and Merck companies and used without further purification. The products were characterized by their physical constant and comparison with authentic samples. Reaction monitoring was accomplished by TLC on silica gel polygram SILG/UV 254 plates. The IR spectra were recorded on a BOMEM MB-Series 1998 FT–IR spectrophotometer using KBr pellets for the samples and the catalyst in the range between 4000 and 400 cm⁻¹. ¹H and ¹³C NMR spectra were recorded in CDCl₃ and DMSO- d_6 on a Bruker Advanced DPX 400 MHz spectrometer using TMS as internal standard. The SEM analyses were carried out using a LEO 1455VP Scanning Electron Microscope, operating from 1 to 30 kV. Thermal stability of the supported catalyst was examined using a BÄHR, SPA 503 Thermo-gravimetric Analyzer (TGA) at a heating rate of 10 °C.min⁻¹ over the temperature range from 40 to 950 °C.

2.2. General procedure for the preparation of nano-Fe₃O₄

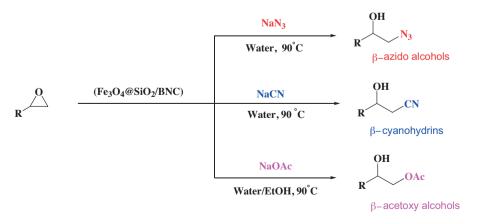
Nano-Fe₃O₄ was prepared via an improved chemical coprecipitation method [23]. A 500-mL round-bottom flask was charged with FeCl₂·4H₂O (3.1736 g, 16 mmol) and FeCl₃·6H₂O (7.57 g, 28 mmol) and dissolved in 320 mL of deionized water. The mixed solution was stirred under nitrogen at 80 °C for 1 h, then 40 mL of NH₃·H₂O (25%) were injected into the reaction mixture rapidly, which was stirred under a nitrogen atmosphere for another 1 h and then cooled to room temperature. The precipitated particles were washed five times with hot water and separated by magnetic decantation. Finally, nano-Fe₃O₄ was dried under vacuum at 70 °C.

2.3. General procedure for the preparation of N-Nbis(triethoxysilylpropyl)-4,4-bipyridinium dichloride precursor, (TEOS)₂ BiPy⁺²2Cl⁻

In a 25-mL three-neck round-bottom flask, bipyridine (8 mmol, 1.25 g) was added to DMF (5 mL), and stirred to make a clear solution. To this solution, (3-chloropropyl)-triethoxy silane (16 mmol, 3.18 g) was added dropwise, and the mixture was stirred at 90 °C for 72 h under an argon atmosphere. Afterwards, the white solid, $(TEOS)_2$ *BiPy*⁺²2*Cl*⁻, was filtered and washed with methanol. This solid was dried for 2 h in an oven at 90 °C [27].

2.4. General procedure for the preparation of Fe $_3\mathrm{O}_4\mathrm{@SiO}_2/\mathrm{BNC}$

In a 250-mL round-bottom flask, 2 g of Fe₃O₄ nanoparticles were dispersed in a solution of 40 mL of distilled water and 120 mL of ethanol via an ultrasonic bath for 15 min. Afterwards, 3 mL of ammonia (25%) were added. In the next step, (TEOS)₂ BiPy⁺²2Cl⁻ (5 g) was added to the suspension. While stirring, 1.5 mL of TEOS in 40 mL of



Scheme 1. (Color online). One-pot preparation of β -azido alcohols, β -cyanohydrins, and β -acetoxy alcohols using Fe₃O₄@SiO₂/BNC as a catalyst.

$$2(\text{MeO})_{3}\text{Si} - (\text{CH}_{2})_{3} - \text{Cl} + N \xrightarrow{N} \sqrt{N} \xrightarrow{\text{DMF}, 90^{\circ}\text{C}} MeO \xrightarrow{\text{MeO}} \text{Si} \xrightarrow{N} \sqrt{N} \xrightarrow{N} \sqrt{N} \xrightarrow{OMe} N \xrightarrow{OMe} N \xrightarrow{N} \sqrt{N} \xrightarrow{OMe} N \xrightarrow{OMe$$

Scheme 2. Preparation of (TEOS)₂ BiPy⁺²2Cl⁻.

ethanol was added dropwise to the suspension. After 48 h of stirring at room temperature, the desired catalyst, $Fe_3O_4@SiO_2/BNC$, was separated via an external magnet and was washed repeatedly with ethanol. Then the catalyst was dried at 60 °C for 2 h.

2.5. General procedure for the preparation of β -azido alcohols in the presence of Fe₃O₄@SiO₂/BNC in water

A 25-mL round-bottom flask was charged with epoxide (1 mmol), sodium azide (3 mmol), Fe₃O₄@SiO₂/bipyridine (0.03 g) and 5 mL of deionized water and was heated at 90 °C in an oil bath for a specific time (10–50 min). After completion of the reaction, which was indicated by TLC [TLC *n*-hexane/ethyl acetate (5:1)] (see Table 2), the catalyst was separated utilizing an external magnet. The product was extracted with diethyl ether (3 × 10 mL), the organic phase was concentrated and dried using CaCl₂. After evaporation of the solvent under reduced pressure with a rotary evaporator, the β -azido alcohols were obtained with 88–95% yield.

2.6. General procedure for the preparation of β -cyanohydrins in the presence of Fe₃O₄@SiO₂/BNC in water

A 25-mL round-bottom flask was charged with epoxide (1 mmol), sodium cyanide (3 mmol), Fe₃O₄@-SiO₂/BNC (0.05 g) and 5 mL of deionized water and was refluxed in an oil bath for a specific time (20–40 min). After completion of the reaction, which was indicated by TLC [TLC *n*-hexane/ethyl acetate (5:1)] (see Table 3), the catalyst was separated utilizing an external magnet. The product was extracted with diethyl ether (3 × 10 mL), the organic phase was concentrated and dried using CaCl₂. After evaporation of the solvent under reduced pressure with a rotary evaporator, the β -cyanohydrins were obtained with 85–95% yield.

2.7. General procedure for the preparation of β -acetoxy alcohols in the presence of Fe₃O₄@SiO₂/BNC in water

A 25-mL round-bottom flask was charged with epoxide (1 mmol), sodium acetate (8 mmol), Fe₃O₄@-SiO₂/BNC (0.07 g) and 5 mL of aqueous ethanol (50:50); the mixture was refluxed in an oil bath for a specific time (25–90 min). After completion of the reaction, which was indicated by TLC [TLC *n*-hexane/ethyl acetate (5:1)] (as indicated in Table 4), the catalyst was separated utilizing an external magnet. The product was extracted with diethyl ether (3 × 10 mL), the organic phase was concentrated and dried using CaCl₂. After evaporation of the solvent under reduced pressure with a rotary evaporator, the β -acetoxy alcohols were obtained with 78–92% yield.

3. Results and discussion

In the first step, Fe_3O_4 magnetic nanoparticles were prepared via the well-known coprecipitation method. Afterwards, *N-N*-bis(triethoxysilylpropyl)-4,4-bipyridinium dichloride, (TEOS)₂ BiPy⁺²2Cl, as a precursor was prepared by the reaction of bipyridine and (3-chloropropyl) triethoxy silane in DMF at 90 °C for 72 h (Scheme 2). In the next step, (TEOS)₂ BiPy⁺²2Cl⁻ and TEOS were added to the suspension of magnetite nanoparticles in the presence of ammonia to prepare the silica shell containing bipyridinium units through a sol–gel method (Scheme 3).

To study the structure of Fe₃O₄@SiO₂/BNC, its characterization was accomplished with FT–IR, SEM, TGA, VSM and XRD techniques.

The FT–IR spectra of Fe₃O₄ nanoparticles. $(TEOS)_2BiPy^{+2}2Cl^-$ and $Fe_3O_4@SiO_2/BNC$ were recorded. In the spectra of Fe_3O_4 nanoparticles and of $Fe_3O_4@SiO_2/$ BNC, the characteristic peak corresponding to the stretching of the Fe–O bond is evident. Peaks in the range from 990 to 1200 cm^{-1} in the spectra of $(\text{TEOS})_2 \text{ BiPy}^{+2}2\text{Cl}^{-}$ and Fe₃O₄@SiO₂/BNC is due to symmetrical and unsymmetrical stretching vibrations of the Si-O bond, which verifies the presence of silica shell around the magnetic nanoparticles. The hydroxyl groups on the surface of the silica shell and also the absorbed water show stretching vibration peaks of the O-H bond in the $3200-3600 \text{ cm}^{-1}$ region. The stretching peaks due to the aromatic moiety are evident in the 1400–1600 cm⁻¹ region. The bending vibration of water is seen at 1640 cm^{-1} (Fig. 1).

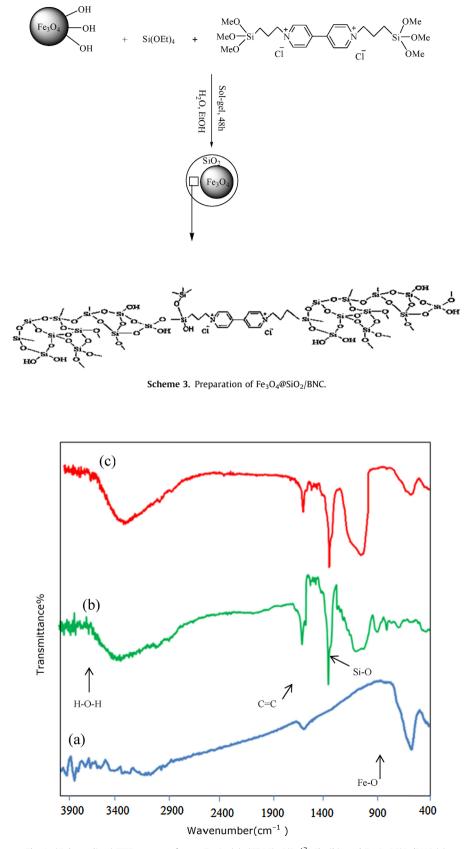
After successful verification of the structure by recording the FT–IR spectrum, it was decided to record the SEM image of the catalyst. According to the SEM image, although the particles have coagulated, the size of the nanocomposite particles is under 100 nm (Fig. 2).

To evaluate the thermal stability of $Fe_3O_4@SiO_2/BNC$, thermogravimetric analysis (TGA) was conducted. According to the TGA diagram, the grafted copolymer is stable up to 250 °C (Fig. 3).

A mass loss of a ca. 6% is seen at 200 °C, which can be attributed to the elimination of water. After 250 °C, it seems that the organic part starts to decompose and a mass loss of about 20% is seen in the range from 250 to 600 °C. Further weight loss in the range between 650 and 800 °C can be attributed to the phase transition from Fe_3O_4 to FeO [28].

The X-ray diffraction pattern of Fe₃O₄@SiO₂/BNC was recorded (Fig. 4). It can be seen that the positions and the intensity of the diffraction peaks are consistent with the standard pattern for JCPDS Card No. (19–629) for magnetite, with six peaks at 2θ = 30.5, 35.8, 43.4, 53.8, 57.5, and 63.1°.

In order to investigate the magnetic properties of Fe₃O₄@SiO₂/BNC, a vibrating sample magnetometer (VSM)



 $\label{eq:Fig. 1. (Color online.) FTIR spectra of nano-Fe_{3}O_{4} (a); (TEOS)_{2} \ BiPy^{+2}2Cl^{-} (b); \ and \ Fe_{3}O_{4}@SiO_{2}/BNC (c).$

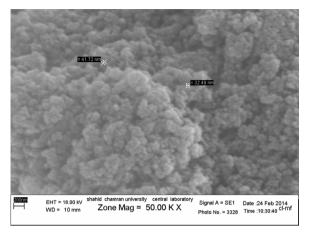


Fig. 2. (Color online.) SEM image of Fe₃O₄@SiO₂/BNC.

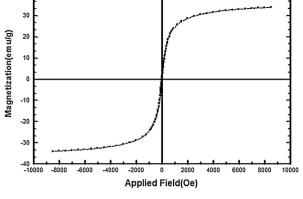


Fig. 5. VSM curve of Fe₃O₄@SiO₂/BNC.

was used. The saturation magnetization of $Fe_3O_4@SiO_2/BNC$ was 33.8 emu/g (Fig. 5).

After successful characterization of $Fe_3O_4@SiO_2/BNC$ using FT–IR, SEM, TGA, VSM and XRD techniques, it was

decided to evaluate its catalytic activity as a phase-transfer catalyst in the ring opening of epoxides.

In the beginning, a model reaction of phenyl glycidyl ether (1 mmol) and sodium azide (2–3 mmol) in the presence of $Fe_3O_4@SiO_2/BNC$ in 5 mL of deionized water

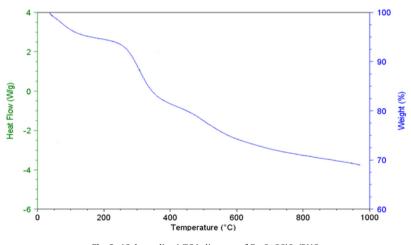


Fig. 3. (Color online.) TGA diagram of Fe₃O₄@SiO₂/BNC.

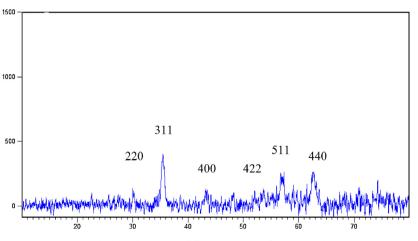


Fig. 4. (Color online.) X-ray diffraction pattern of Fe₃O₄@SiO₂/BNC.

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Table 1

Optimization of reaction conditions for the preparation of β -azido alcohol from phenyl glycidyl ether, in the presence of Fe_3O_4@SiO_2/BNC in aqueous medium.

Entry	NaN ₃ (mmol)	Nanocomposite (g)	Temperature (°C)	Time (min)
1	2	0.07	90	60
2	2	0.1	90	50
3	3	0.03	90	50
4	3	0.05	90	50
5	3	0.07	90	50
6	3	-	90	360
7	3	0.07	60	240
8	3	0.07	r.t	300

In all cases the time indicates the total conversion except for entries 6 and 8, which the reaction did not complete.

was conducted under different conditions. According to TLC, the optimized amount of reactants and of the catalyst was found: 3 mmol of sodium azide and 0.03 g of the catalyst complete the reaction at 90 °C in a minimum time (Table 1).

In the FT–IR spectrum, the stretching vibration of azide group is obvious at 2106 cm^{-1} (Fig. 6).

In the next step, with optimum conditions in hand (1 mmol epoxide: 3 mmol azide: 0.03 g Fe₃O₄@SiO₂/BNC: 5 mL of deionized water at 90 °C), the generality and synthetic scope of this coupling protocol were demonstrated by synthesizing a series of β -azido alcohols (Table 2) in 10 to 50 min with 88–95% yield.

A glimpse to Table 2 makes clearly evident the generality of this methodology. Different functional groups, including ether, ester and double bond, are easily tolerated in the reaction conditions. The results clearly indicate the high regioselectivity of epoxide ring-opening by the azide anion. Steric and electronic effects direct regioselectivity; for example, in styrene oxide, the phenyl group acts as an electron-donating group via resonance, which in turn stabilizes the carbocation. As a result, the

azide attack is directed by electronic effects and the reaction is thought to be pseudo $S_N 1$. In the other cases, due to steric hindrance of bulky groups, the unsubstituted carbon is more accessible to the azide attack and the reaction proceeds with a $S_N 2$ mechanism.

After the success of Fe₃O₄@SiO₂/BNC in the preparation of β -azido alcohols, we decided to explore the catalytic activity of this catalyst in the preparation of β -cyanohydrins. Subsequently, the ring opening of epoxides (1 mmol) was conducted with sodium cyanide (3 mmol) in the presence of Fe₃O₄@SiO₂/BNC (0.05 g) as the magnetic PTC at 90 °C in aqueous medium. In the FT–IR spectrum, the stretching vibration of the cyanide group is obvious at 2253 cm⁻¹ (Fig. 7).

All epoxides reacted almost equally well to afford the corresponding β -cyano hydrins in high to excellent yields (Table 3).

With the same method, we decided to explore the catalytic activity of this catalyst in the preparation of β -acetoxy alcohols. Thus, the ring opening of epoxides (1 mmol) was conducted with sodium acetate (8 mmol) in the presence of Fe₃O₄@SiO₂/BNC (0.07 g) as the magnetic PTC in a 50:50 mixture of ethanol/water under reflux conditions. In the FT–IR spectrum, the stretching vibration of carbonyl group is obvious at 1719 cm⁻¹ (Fig. 8).

All epoxides reacted almost equally well to afford the corresponding β -acetoxy alcohols in high to excellent yields (Table 4).

Due to the importance of green chemistry and catalyst recycling, the recyclability of Fe₃O₄@SiO₂/BNC was assessed in the reaction of phenyl glycidyl ether with the azide anion. The result is shown in Fig. 9. The activity of the nanocomposite is slightly decreased after 4 times of reuse.

To demonstrate the superiority of $Fe_3O_4@SiO_2/BNC$ over the reported catalysts, the azidolysis reaction of styrene oxide was considered as a representative example (Table 5). In all of these cases (except for entry 1, 2), a low yield of the desired product was obtained. It is also noteworthy to mention that all of the reported procedures required long

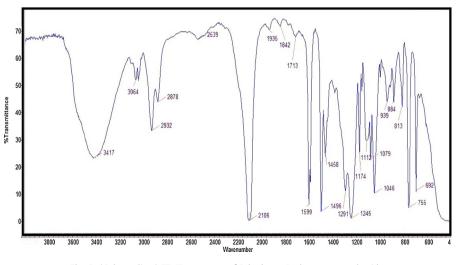


Fig. 6. (Color online.) FT-IR spectrum of 2-hydroxy-3-phenoxy propyl azide.



Preparation of β -azido alcohols from corresponding epoxides and sodium azide in the presence of Fe₃O₄@SiO₂/BNC in aqueous medium at 90 °C.

Entry	Epoxide	Product	Time (min)	Yield (%)
1		N ₃ OH	15	95
2		OH	50	95
3		ОН	10	91
4		~ 0 N_3	10	92
5	\sim	\sim	10	89
6		OH OH N ₃	20	90
7		ООН	20	88
8 ^a		N ₃	30	90
d Due to low	boiling point of 1.2-enovy butane, the re-			

^a Due to low boiling point of 1,2-epoxy butane, the reaction was conducted at 60 °C.

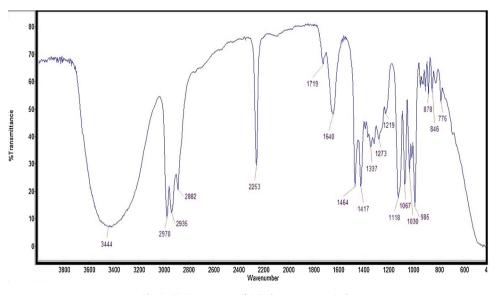


Fig. 7. FT-IR spectrum of 2-hydroxy-pentanenitrile.

8^a

Entry	Epoxide	Epoxide Product		Yield (%)
1		OH OH C	40 'N	94
2		CN OH	20	95
3		ОН	25	92
ŀ			N 25	87
			CN 25	95
i			30 N	91
7		ОН	20	85

'′′CN

CN

90

40

^a Due to low boiling point of 1,2-epoxy butane, the reaction was conducted at 60 $^{\circ}$ C.

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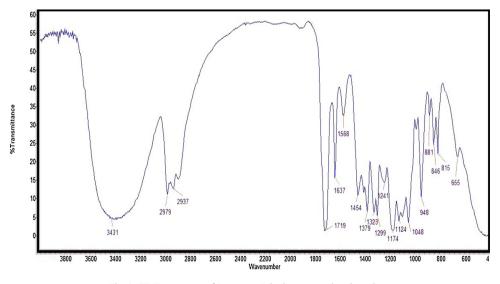


Fig. 8. FT-IR spectrum of 3-acetoxy-2-hydroxy-propylmethacrylate.

Table 4

Preparation of β -acetoxy alcohols from corresponding epoxides and sodium acetate in the presence of Fe₃O₄@SiO₂/BNC in ethanol/water (50:50) under reflux conditions.

Entry	Epoxide	Product	Time (min)	Yield (%)
1		OH OAc	90	92
2		OAc OH	35	90
3		OH OH	30	87
4		OH OH OAc	30	85
5		OAc OAc	30	82
6		OH OH OH OAc	50	78
7		O O O H	25	88
8 ^a		OAc OAc	45	79

^a Due to low boiling point of 1,2-epoxy butane, the reaction was conducted at 60 °C.

 Table 5

 Comparison of azidolysis of styrene oxide with other reported methods in the literature.

Entry	Catalyst	Solvent	Temperature (°C)	Time (h)	Yield (%)	Ref
1	LiClO ₄	CH₃CN	80	5	92	[25]
2	NH ₄ Cl	MeOH-H ₂ O	70	5	90	[25]
3	(TBA) ₄ PFe ₁₁ MO ₃₉ ·3H ₂ O	CH ₃ CN-H ₂ O	80	4.5	85	[26]
4	Dowex-PEG	H ₂ O	100	0.5	85	[19]
5	PEG300	PEG300	60	1.5	85	[20]
6	SiO ₂ -PEG	H_2O	100	1	80	[23]
7	MPTC ^a	H ₂ O	90	0.5	85	[24]
8	Network polymer	H ₂ O	80	1.5	85	[22]
9	Fe ₃ O ₄ @SiO ₂ /BNC	H ₂ O	90	0.25	95	This wor

^a Multi-site phase-transfer catalyst.

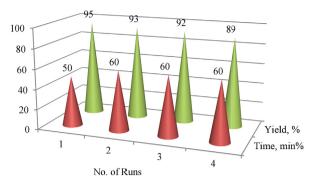


Fig. 9. (Color online.) Reusability of the nanocomposite in the reaction of phenyl glycidyl ether with the azide anion.

reaction times (except for entries 4, 7). These results clearly demonstrate that $Fe_3O_4@SiO_2/BNC$ is an equally or more efficient catalyst for this reaction.

4. Conclusion

In conclusion, a novel $Fe_3O_4@SiO_2/BNC$, has been synthesized and its application as a magnetic and recyclable phase-transfer catalyst in the preparation of β -azidoalcohols, β -cyanohydrins, and β -acetoxy alcohols was investigated. The catalyst was characterized with FT– IR, SEM, XRD, VSM and TGA. According to the experimental results, the regioselective ring opening reaction of epoxides, in the presence of azide, cyano and acetate anions in aqueous medium, leads to the corresponding products in a short reaction time and with high yields. This methodology offers several advantages, including an easy work-up procedure, excellent regioselectivity, high yields, short reaction times, recyclability of the catalyst, easy separation of the catalyst through an external magnet; it is also an eco-friendly procedure.

Acknowledgments

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