

Research Article

M-Type $\text{SrFe}_{12}\text{O}_{19}$ Ferrite: An Efficient Catalyst for the Synthesis of Amino Alcohols under Solvent-Free Conditions

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Magnetically separable strontium hexaferrite $\text{SrFe}_{12}\text{O}_{19}$ was prepared using the chemical coprecipitation method, and the nanostructured material was characterized by X-ray diffraction, scanning electron microscopy (SEM), energy-dispersive spectrometry (EDS), and BET analysis. The SEM images showed the homogeneity of the chemical composition of $\text{SrFe}_{12}\text{O}_{19}$ and uniform distribution of size and morphology. The pore size of the nanomaterial and its specific area were determined by BET measurements. Strontium hexaferrite $\text{SrFe}_{12}\text{O}_{19}$ exhibited a strong magnetic field, which is highly suitable in the heterogeneous catalysis as it can be efficiently separated from the reaction. The magnetic nanocatalyst showed high activity and environmentally benign heterogeneous catalysts for the epoxide ring-opening with amines affording β -amino alcohols under solvent-free conditions. When unsymmetrical epoxides were treated in the presence of aromatics amines, the regioselectivity was influenced by the electronic and steric factors. Total regioselectivity was observed for the reactions performed with aliphatic amines. The magnetically $\text{SrFe}_{12}\text{O}_{19}$ nanocatalyst showed excellent recyclability with continuously good catalytic activities after four cycles.

1. Introduction

β -Amino alcohols are molecules with an interesting role as intermediates in the synthesis of a wide range of biologically active natural and synthetic products [1–4]. The presence of these functional groups with a defined stereochemistry is of great importance in the biological activity of these molecules. They are also widely used as potential chiral auxiliaries and chiral ligands in asymmetric synthesis [5]. The nucleophilic ring-opening of epoxides with amines represents one of the most important and straightforward methods for the preparation of the β -amino alcohols [6]. While the classical methods require a high temperature [7], various methodologies developed the epoxide ring-opening using

homogenous catalysts but suffer from several disadvantages such as low regioselectivity, toxic solvents, toxic metal ions, and nonreusable catalysts [8–14].

On the other hand, only a few reports are focused on the use of heterogeneous catalysts such as silica nanoparticles [15], Fe-MCM-41 [16], zeolites [17], heteropoly acid, and MCM-22 [18, 19].

Recently, green chemistry has gained considerable importance in chemical design processes that utilize environmental-friendly processes to reduce or eliminate the generation of toxic by-products [20]. In addition, heterogeneous catalysts offer interesting advantages compatible with the green chemistry approach in catalytic reactions, such as high activity, atom economy, good regioselectivity,

and especially the reusability of the catalyst. Furthermore, solvent-free conditions have a supplementary advantage in avoiding toxic solvents.

In recent years, magnetic nanoparticles have emerged as efficient heterogeneous catalysts in organic synthesis that offer a major advantage with its easy magnetic separation [21–25]. In particular, magnetic nano- Fe_3O_4 and sulfonic acid-functionalized silica-coated magnetic Fe_3O_4 were used as efficient heterogeneous catalysts for the aminolysis of epoxides by amines [26, 27]. The same reaction was performed under solvent-free conditions using various spinel nanoferrites MFe_2O_4 ($\text{M} = \text{Co}, \text{Ni}, \text{Cu}, \text{Zn}$) [28].

Hexagonal strontium hexaferrite ($\text{SrFe}_{12}\text{O}_{19}$), known as M-type strontium hexaferrite (SrM), was discovered in the 1950s by Philips' laboratories [29]. Strontium hexaferrite is usually used as magnetic recordings, permanent magnets, and microwave devices [30–32]. In organic synthesis, it was applied as a catalyst in the synthesis of 2-amino-4,6-diphenylnicotinonitrile derivatives [33]. The magnetic properties of $\text{SrFe}_{12}\text{O}_{19}$ depend on the shape and size of the particles. Several methods have been used to prepare this nanomaterial, such as sol-gel [34], hydrothermal [35], salt-melt methods [36], ball milling [37], self-propagating high-temperature synthesis [38], and the chemical coprecipitation method [39]. However, the coprecipitation method is the most widely used in the synthesis of magnetic oxides due to its simplicity and good control of the grain size [40].

Previously, we have shown the catalytic efficiency of calcium trifluoroacetate $\text{Ca}(\text{CF}_3\text{CO}_2)_2$ as a highly chemoselective homogenous catalyst in the epoxide ring-opening with amines [9]. As part of our studies directed towards the development of green chemistry protocols by performing organic transformations under solvent-free conditions [41, 42], we have focused our attention on the preparation, characterization, and catalytic application of magnetic strontium hexaferrite $\text{SrFe}_{12}\text{O}_{19}$ nanoparticles. The magnetic behavior of $\text{SrFe}_{12}\text{O}_{19}$ was evaluated by measurement of the hysteresis loops at room temperature. Furthermore, $\text{SrFe}_{12}\text{O}_{19}$ nanoparticles were tested as a heterogeneous catalyst in the ring-opening reactions with various amines.

2. Experimental Details

2.1. Materials. All reagents and solvents were purchased from commercial sources and used as received without further purification (Aldrich, Acros).

The synthesized NPs were characterized by X-ray powder diffraction (XRD) using D8 Discover Bruker (AXS) with $\text{Cu K}\alpha$ radiation ($\lambda_{\text{Cu}} = 1.5407 \text{ \AA}$) model. Microstructural characterization was performed using a Scanning Electron Microscope (SEM) (FEI, Quanta FEG 450) from BRUKER. Aliquots samples from the reaction mixture were monitored by Shimadzu gas chromatography (GC) with a flame ionization detector using nitrogen as a carrier gas. GC parameters for capillary columns BP (25 m \times 0.25 mm, SGE) are injector 250°C; detector 250°C; oven 70°C for 5 min then 3°C/min until 250°C for 30 min; column pressure 20 kPa; column flow 6.3 mL/min; linear velocity 53.1 cm/s; total flow 138 mL/min. The products were confirmed by injecting the

reaction mixture on an ISQ LT single quadrupole mass spectrometer in positive EI mode using a mass scan range of 50 to 400 Da. BET measurements were performed using 3Flex 3500 Micromeritics instruments. The magnetic propriety was determined by the Quantum Design XL-SQUID magnetometer.

2.2. Preparation of the Catalyst. The M-type hexaferrite $\text{SrFe}_{12}\text{O}_{19}$ was obtained by the co-precipitation method of Sr^{2+} and Fe^{3+} in the presence of NaOH as a precipitate agent in an aqueous medium. Briefly, stoichiometric amount of SrCl_2 (0.068 g) and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (2 g), were dissolved in 30 mL of deionized water. To the resulting mixture, 100 mL of NaOH (1.5 M) solution was added dropwise at 60°C. Next, the temperature was increased to 100°C for 2 hours. The precipitated solid was separated magnetically and washed several times to remove the excess of salts and then dried at 80°C overnight. The obtained powder was annealed at 1000°C for 6 hours and subjected to various analyses.

2.3. Catalytic Activity. In a typical reaction, a mixture of epoxide (1.02 mmol), amine (1.1 mmol), and $\text{SrFe}_{12}\text{O}_{19}$ ($9.4 \cdot 10^{-3}$ mmol) was introduced into a 50 mL Rotaflo tube and stirred at 60°C for 17 hours. The evolution of the reaction was monitored by gas chromatography. At the end of the reaction, the catalyst was easily recovered by applying a magnetic field, then washed with acetone and distilled water, and dried in an oven for 6 hours before reuse.

3. Results and Discussion

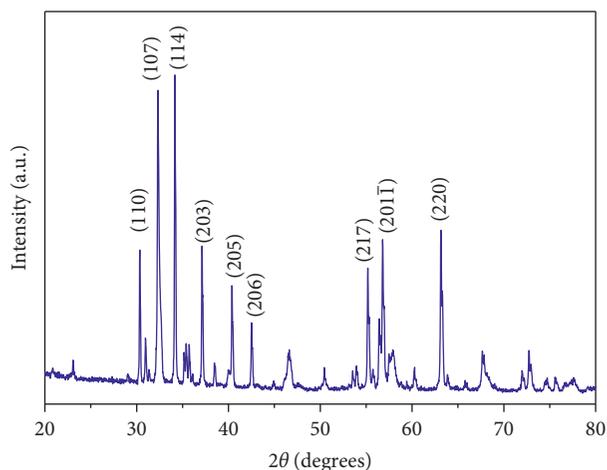
3.1. Characterization. Figure 1 presents the XRD pattern of hexagonal $\text{SrFe}_{12}\text{O}_{19}$ sample with corresponding diffraction lines. The prepared nanomaterial crystallizes in a pure hexagonal structure with a space group $194/P_{63} \text{mmc}$. As shown in Figure 1, no impurity phases were detected from the XRD pattern. Rietveld refinement of the sample was performed by TOPAS software in order to define the lattice parameters of $\text{SrFe}_{12}\text{O}_{19}$ according to JCPDS 01-084-1531. The result is presented in Table 1.

To confirm the nanoparticles formation, Debye–Scherrer formula was used to calculate the crystallite size of the prepared nanoparticles:

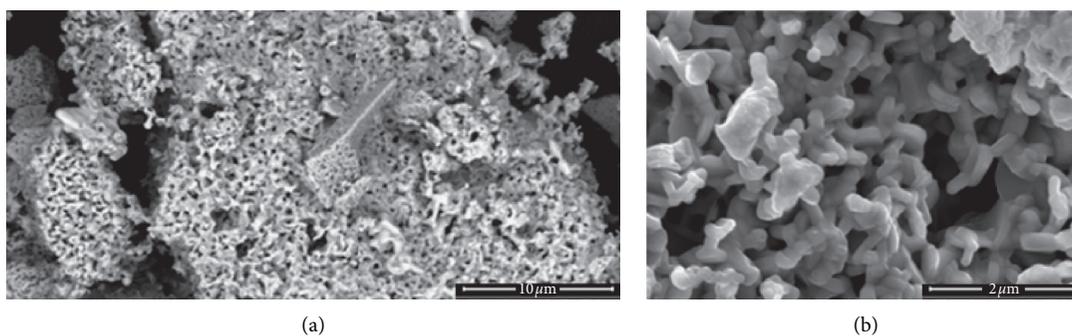
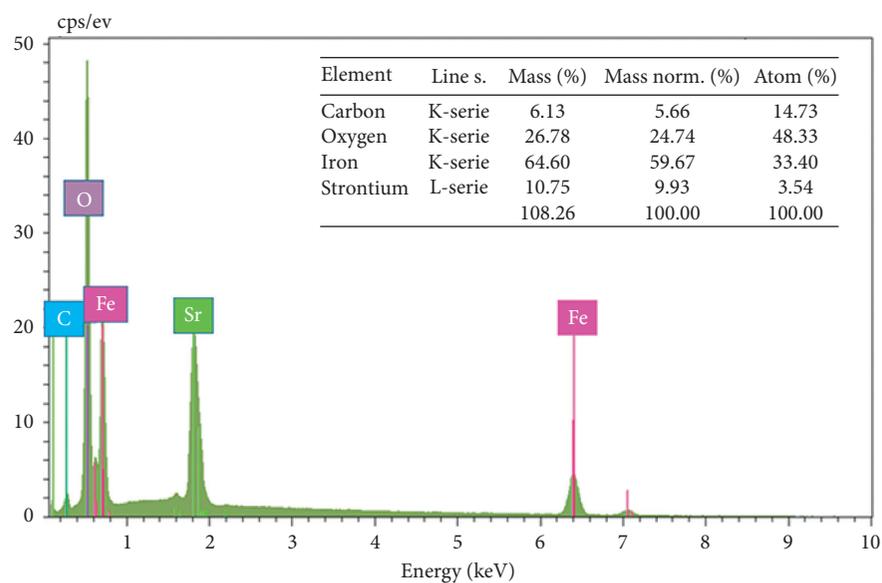
$$d = \frac{0.9\lambda}{\beta \cos(\theta)}, \quad (1)$$

where λ is the wavelength ($\text{Cu K}\alpha$), β is the full width to half-maximum (FWHM) of line broadening, and θ is the Bragg angle of diffraction. The average crystallite size was found to be 85.4 nm.

The scanning electron microscope (SEM) shows the homogeneity of the chemical composition and regular distribution of size and morphology (Figure 2). EDS results confirmed the elements that consist of strontium hexaferrite (Sr, Fe, and O) (Figure 3). The percentage of elements is in good agreement with the chemical composition of $\text{SrFe}_{12}\text{O}_{19}$, which confirms the absence of impurities.

FIGURE 1: X-ray patterns of $\text{SrFe}_{12}\text{O}_{19}$.TABLE 1: Lattice parameters of $\text{SrFe}_{12}\text{O}_{19}$.

Catalyst	Space group	Structure	a (Å)	c (Å)	D_{drx} (nm)	V (Å ³)
$\text{SrFe}_{12}\text{O}_{19}$	194/ $P_{63}mmc$	Hexagonal	5.8856 (± 0.0047)	23.0790 (± 0.0082)	95.4 (± 0.4)	692.1410 (± 0.0056)

FIGURE 2: SEM images of $\text{SrFe}_{12}\text{O}_{19}$.FIGURE 3: EDS of $\text{SrFe}_{12}\text{O}_{19}$.

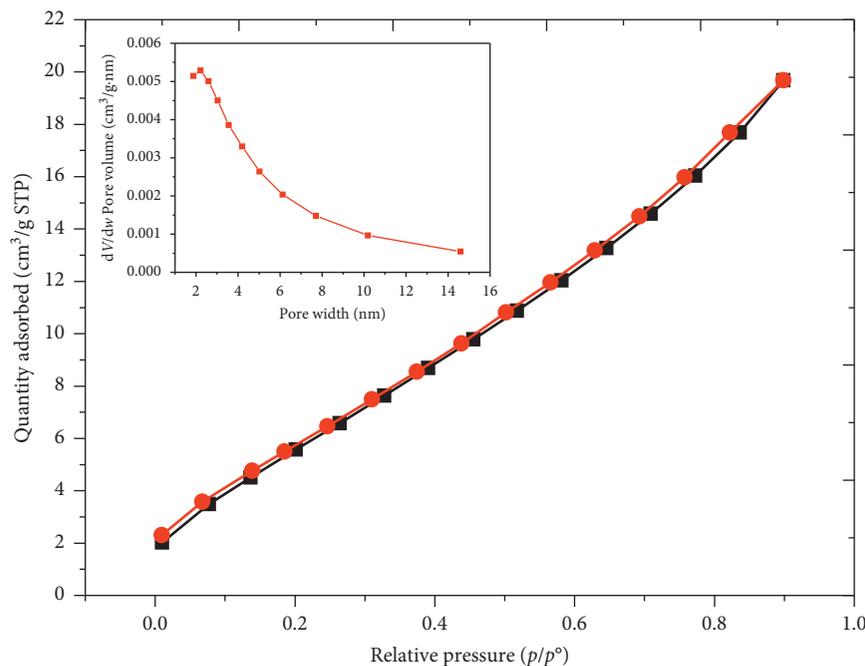


FIGURE 4: N₂ adsorption-desorption isotherm and BJH pore diameter distribution of SrFe₁₂O₁₉.

Figure 4 shows the isotherm of M-type hexaferrite SrFe₁₂O₁₉ material. According to the IUPAC classification, the isotherm of the catalyst is assigned to type II, which is characteristic of nonporous materials, due to the high calcination temperature. This was confirmed by the pore size distribution curve determined by the Barrett–Joyner–Halenda (BJH) method, which showed a centered pore size at 2.21 nm. The BET surface area of the SrFe₁₂O₁₉ material was found to be 25 m²/g.

3.2. Magnetic Measurement. The magnetic propriety of SrFe₁₂O₁₉ at room temperature was investigated (Figure 5). The hysteresis loops of the samples showed that strontium hexaferrite exhibits a ferrimagnetic behavior characterized by high coercivity (5.37 KOe), high magnetization saturation (81.79 emu/g), and remanence magnetization of about 39.74 emu/g. The values are higher than those found when the material was prepared by other methods and confirm the hard magnetic behavior of SrFe₁₂O₁₉ [43–45].

3.3. Catalytic Application. The catalytic activity of the SrFe₁₂O₁₉ nanocatalyst was evaluated for the nucleophilic ring-opening of cyclohexene oxide with aniline (Scheme 1). The scope and limitation of the catalytic system were examined by testing different parameters (Table 2).

Preliminary experiments were conducted at 80°C with 1.02 mmol of cyclohexene oxide (1) and 1.1 mmol of aniline (2a) catalyzed by SrFe₁₂O₁₉ NPs (0.01 g, 0.92 mol%) in various solvents or under solvent-free conditions (Table 2). The reaction was high stereoselective and afforded the trans amino alcohol 3a. In protic polar solvents, amino alcohol derivative 3a was obtained in good yield (entries 2 and 3). However, in an aprotic solvent such as THF or acetonitrile,

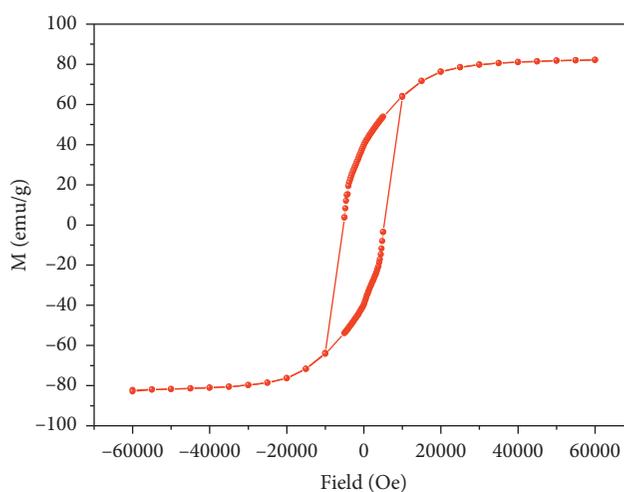
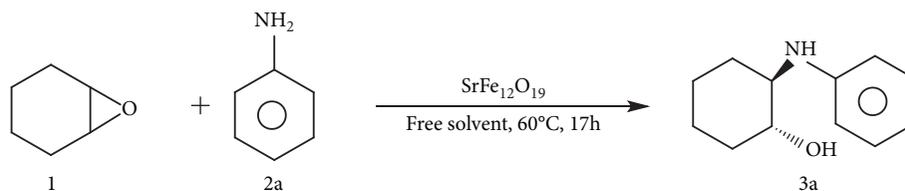


FIGURE 5: Hysteresis loop of SrFe₁₂O₁₉.

no reaction took place (entries 4 and 5). In solvent-free conditions, total conversion and high selectivity were achieved (entry 1). In the absence of catalysts, only 19% of selectivity was obtained (entry 6). The optimal catalytic amount was found to be 0.92 mol% and the optimum temperature was 60°C (entry 10).

A kinetic study was performed with 0.01 g of the nanocatalyst SrFe₁₂O₁₉ (Figure 6). Based on Figure 6, the evolution of the reaction versus time shows that a maximum of the desired amino alcohol 3a was obtained after 17 h of reaction time. Under the optimized conditions, various aromatic and aliphatic amines were examined. The results are summarized in Table 3.

With all amines used, high stereoselectivity was observed. Exclusive trans amino alcohol derivatives were



SCHEME 1: Catalytic ring-opening of cyclohexene oxide with aniline.

TABLE 2: Catalytic epoxide ring-opening of cyclohexene oxide as symmetric epoxide.

Entry	Solvent	C/S mol (%)	T (°C)	Conversion ^a (%)	Selectivity ^b (%)
1	Solvent-free	0.92	80	100	100
2	Water	0.92	80	80	80
3	Methanol	0.92	80	80	80
4	THF	0.92	80	None	None
5	Acetonitrile	0.92	80	None	None
6	Solvent-free	None	80	19	19
7	Solvent-free	0.64	80	28	28
8	Solvent-free	1.38	80	96	96
9	Solvent-free	1.84	80	79	79
10	Solvent-free	0.92	60	100	100
11	Solvent-free	0.92	50	96	96
12	Solvent-free	0.92	40	96	95
13	Solvent-free	0.92	25	20	20

Reaction conditions: solvent (1 mL), reaction time 17 h, cyclohexene oxide (1.02 mmol), and aniline (1.1 mmol). ^aConversion and ^bselectivity were determined by GC.

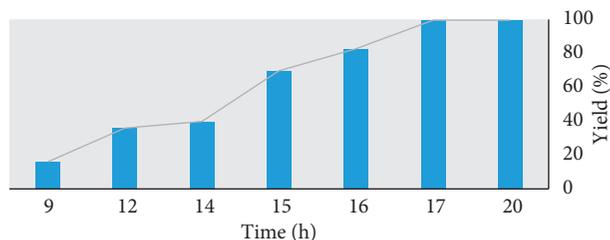
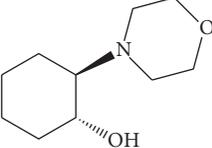
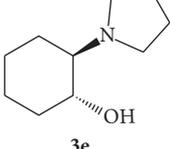
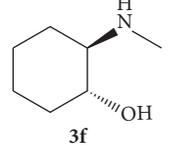
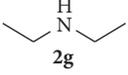
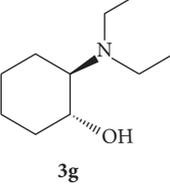
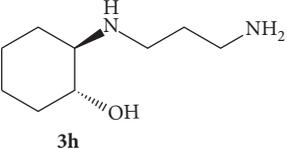
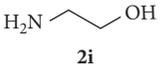
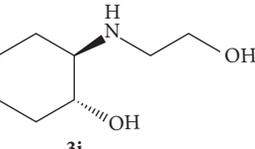


FIGURE 6: Effect of the reaction time.

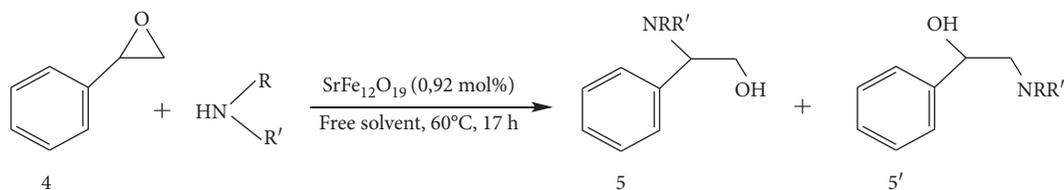
TABLE 3: Catalytic ring-opening of cyclohexene oxide with various amines.

Entry	Amine	Product	Conversion (%) ^a	Yield (%) ^b
1	<chem>Nc1ccccc1</chem> 2a	<chem>N[C@@H]1CCCC[C@H]1Oc2ccccc2</chem> 3a	100	97
2	<chem>Nc1ccc(Cl)cc1</chem> 2b	<chem>N[C@@H]1CCCC[C@H]1Oc2cc(Cl)ccc2</chem> 3b	49	49
3	<chem>Nc1ccc(F)cc1</chem> 2c	<chem>N[C@@H]1CCCC[C@H]1Oc2ccc(F)cc2</chem> 3c	42	42

TABLE 3: Continued.

Entry	Amine	Product	Conversion (%) ^a	Yield (%) ^b
4	 2d		82	82
5	 2e	 3e	84	84
6	$\text{H}_3\text{C}-\text{NH}_2$ 2f	 3f	100	97
7	 2g	 3g	10	10
8	 2h	 3h	90	87
9	 2i	 3i	97	95

Reaction conditions: cyclohexene oxide (1.02 mmol), amine (1.1 mmol), C/S = 0.92 mol%, solvent-free, and 17 h 60°C. ^aConversion was determined by GC; ^bisolated yield.

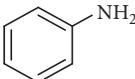
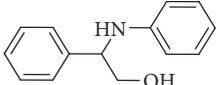
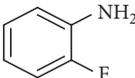
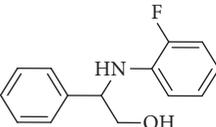
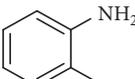
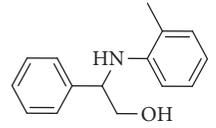
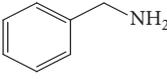
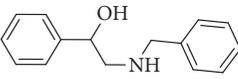
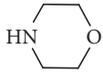
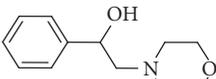
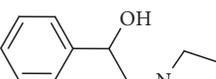


SCHEME 2: Catalytic ring-opening of styrene oxide with different amines.

obtained in the cleavage of the epoxide ring of cyclohexene oxide which is in good agreement with the literature [46, 47]. Table 3 shows that electron-withdrawing effects resulted in the decrease of the nucleophilicity of the aromatic amines (entries 2 and 3). In the presence of cyclic secondary amines,

a slight decrease in yield was detected (entries 4 and 5). All the primary amines afford the corresponding amino alcohols in high yields under solvent-free conditions (entries 6, 8, and 9). Among aliphatic amines, diethylamine exhibited a low activity due to its low boiling temperature (55°C) (entry 7).

TABLE 4: Catalytic ring-opening of styrene oxide with various amines.

Entry	Amine	Conversion (%) ^a	Product	Selectivity (%) ^b		Yield (%) ^c	
				5	5'	5	5'
1		95		74	26	70	12
2		74		83	17	61	10
3		76		74	26	56	14
4		99		0	100	0	98
5		85		0	100	0	84
6		100		0	100	0	99

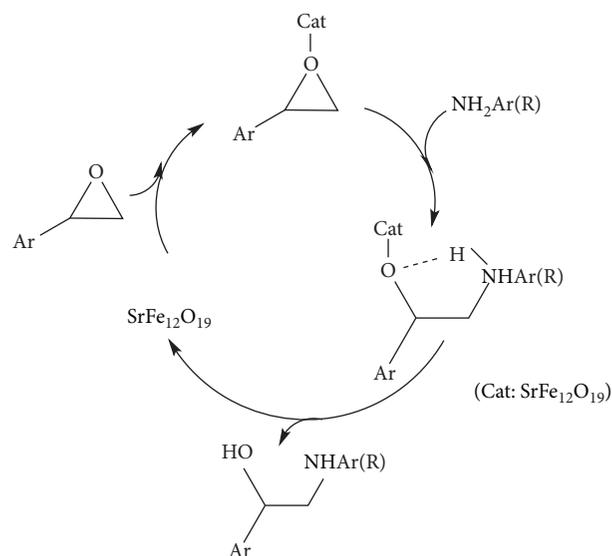
Reaction conditions: styrene oxide (0.8 mmol), amine (1 mmol), C/S = 0.92 mol%, solvent-free, 17 h, and 60°C. ^aConversion and ^bselectivity were determined by GC; ^c isolated yield.

A study on the regioselectivity of the nucleophilic ring-opening reaction was evaluated in the presence of styrene oxide 4 as unsymmetrical epoxide with various aromatic and aliphatic amines (Scheme 2). The result is summarized in Table 4.

As was described in the literature with styrene oxide, the regioselectivity of the reaction is influenced by the electronic and steric factors associated with the epoxides and the amines [47]. Table 4 shows that, with aromatic amines, the reaction afforded the amino alcohols from nucleophilic attack at the benzylic carbon atom of the epoxide ring as the major products (entries 1–3). In the case of aliphatic amines, the major/exclusive product was the regioisomeric amino alcohol produced by nucleophilic attack at the less hindered carbon atom of the epoxide ring (entries 4–6).

It is commonly recognized that the epoxide ring-opening reaction under basic or neutral conditions proceeds via the S_N2 mechanism, but under acidic conditions, a borderline S_N2 mechanism has been invoked to justify the electronic pull on the oxygen by an acid [48, 49].

Sundararajan et al. have discovered that anilines act as superior ring-opening reagents even when the reactions were performed with CoCl₂·6H₂O or under aerobic conditions, thereby ruling out the possibility of the mediation by free radicals [50]. Moreover, they suggest that CoCl₂ coordinates with the oxirane oxygen promote the nucleophilic



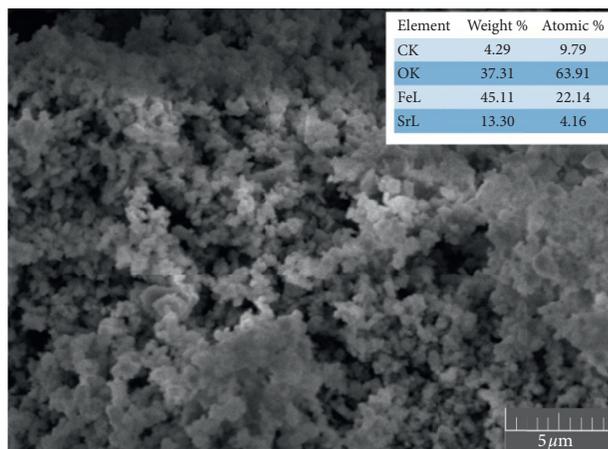
SCHEME 3: Proposed mechanism of the catalytic ring-opening of styrene oxide.

attack of the anilines leading to two regioisomers. In the case of metal coordinated styrene oxide, the positive charge on oxygen appears to be localized on the more highly substituted benzylic carbon and the nucleophile attacks the benzylic carbon of the styrene oxide.

TABLE 5: Catalyst recycling effect.

Entry	Cycle	Conversion (%) ^a	Selectivity (%) ^b
1	Fresh	100	100
2	1	83	83
3	2	80	80
4	3	75	75
5	4	71	71

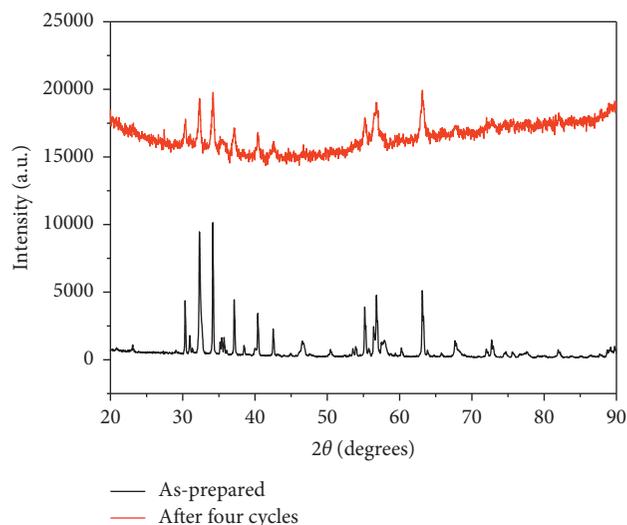
Reaction conditions: cyclohexene oxide (1.02 mmol), aniline (1.1 mmol), C/S = 0.92 mol%, solvent-free, 17 h, and 60°C. ^aConversion and ^bselectivity were determined by GC.

FIGURE 7: SEM image of magnetic SrFe₁₂O₁₉ after five cycles.

Chakraborti et al. have reported that selective formation of the regioisomeric product arising from nucleophilic attack at the benzylic carbon was observed during the reactions with aromatic amines which are less nucleophilic [46, 47]. This preference may be accounted by the fact that the phenyl group in styrene oxide assists in the stabilization/accumulation of carbocationic character at the benzylic carbon. Meanwhile, in the case of aliphatic amines, a preference for nucleophilic attack at the terminal carbon may be explained by the increased nucleophilicity of aliphatic amines favoring a more SN₂ process. According to literature, a similar result was obtained during our present work and a proposed mechanism is given in Scheme 3.

3.4. Recyclability. To investigate the reusability of SrFe₁₂O₁₉ nanoparticles, the catalytic performance in the epoxide ring-opening reaction of cyclohexene oxide with aniline was evaluated in five consecutive cycles (Table 5). After each cycle, the catalyst was separated from the reaction mixture using an external magnet, washed with water and acetone, and dried at 100°C for 6 hours while being reused.

As shown in Table 5, the catalyst still exhibits a good catalytic activity after the third consecutive cycle. However, a noticeable drop in cyclohexene oxide conversion was observed after the fourth consecutive run due to agglomerated particles. SEM image of SrFe₁₂O₁₉ after five cycles showed particle agglomeration and surface change with an increase

FIGURE 8: DRX patterns of magnetic SrFe₁₂O₁₉ fresh and recycled.

of the oxygen content on the surface of the materials (Figure 7).

Moreover, XRD analysis of SrFe₁₂O₁₉ nanocatalyst after five cycles exhibited less intensities of the diffraction planes compared to the fresh one (Figure 8).

4. Conclusion

In summary, SrFe₁₂O₁₉ nanoparticles were prepared by the coprecipitation method and characterized using various techniques. The prepared nanomaterial exhibited a strong magnetic field and marked activity, good reusability, and stability as a heterogeneous catalyst for the regioselective ring-opening of epoxides with various amines under free solvent conditions. The easy magnetic separation of the catalyst and its good reusability make it a useful and attractive process for the synthesis of β-amino alcohols.

Data Availability

SEM, EDS, XRD, BET, and the device types used for recording spectra and other analytical data used to support the findings of this study are included within the manuscript.

Disclosure

The present research is a part of a Ph.D. thesis work of the author Mouhsine Laayati.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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