

Highly Efficient Epoxidation of Alkenes with Sodium Periodate Catalyzed by Reusable Polystyrene-Bound Ruthenium(III) Salophen

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In this paper, highly efficient epoxidation of alkenes catalyzed by ruthenium(III) salophen chloride, [Ru(salophen)Cl], supported on functionalized chloromethylated polystyrene, PS, is reported. The PS was modified with 1,4-diaminobenzene, 4-aminophenol and 4-aminothiophenol, and [Ru(salophen)Cl] was attached to the supports *via* axial ligation. The prepared catalysts were used for efficient epoxidation of alkenes with NaIO₄ at room temperature. These new heterogenized catalysts were characterized by elemental analysis, FT-IR spectroscopy, scanning electron microscopy and transmission electron microscopy. The heterogeneous catalysts were reusable in the oxidation reactions and were reused several times.

Keywords: Ruthenium(III) salophen, Alkene epoxidation, Sodium periodate, Polystyrene, Heterogeneous catalysts

INTRODUCTION

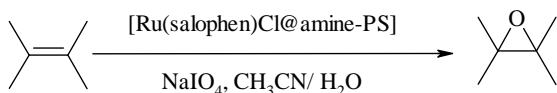
Ruthenium compounds are versatile electron-transfer and energy-transfer compounds [1-22]. Due to these unique properties, ruthenium complexes have a wide range of applications in various research areas, such as artificial photosynthesis, photomolecular devices, probes for biological macromolecules, oxidation catalysts and organic synthesis [8-22].

Salen and salophen ligands and their metal complexes have found extensive applications in the fields of synthesis and catalysis [23-26]. These homogeneous catalysts cannot be recovered and are degraded in the reaction media, which limit their practical applications. On the contrary, their

heterogeneous counterparts offer many advantages such as easy separation and recovery of the catalyst from the reaction media, higher stability of the catalytic species and catalyst protection against destruction. Several methods such as non-covalent immobilization in zeolites, clay or siloxane membranes [27-32], covalent grafting onto inorganic supports such as silica or MCM-41 [33-37], co-polymerization of a functionalized salen monomer into an organic polymer [38], attachment or build-up of a salen structure to a performed polymer [39-44], and axial ligation to surface bound ligands [45-47] have been used for immobilization of Schiff base complexes (salen or salophen) on the supports.

The use of supported Co(salen) as a highly efficient catalyst for hydrolytic kinetic resolution of racemic epoxides and enantioselective parallel synthesis have been reported by Jacobsen group [48,49]. Polystyrene is one of the most popular polymeric supports used in synthetic organic

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Scheme 1. Epoxidation of alkenes with NaIO₄ in the presence of [Ru(salophen)Cl@amine-PS]

chemistry because of its inexpensiveness, ready availability, mechanical robustness, chemical inertness and facile functionalization.

In continuation of our research on the preparation of polystyrene-bound catalysts [46,47,50-53], here, we report the preparation, characterization and investigation of catalytic activity of [Ru(salophen)Cl] supported on chloromethylated polystyrene modified with 1,4-diaminobenzene, 4-aminophenol and 4-aminothiophenol (Scheme 1).

EXPERIMENTAL

Alkenes were obtained from Merck chemical company and passed through a column containing active alumina to remove the peroxidic impurities. Salophen ligand was prepared and metallated according to the literature [54,55]. FT-IR spectra were obtained with potassium bromide pellets in the range 400-4000 cm⁻¹ with a Nicolet Impact 400D spectrometer. Scanning electron micrograph of PS was taken on a Philips XL 30 SEM instrument. Gas chromatography experiments (GC) were performed with a Shimadzu GC-16A instrument using a 2 m column packed with silicon DC-200 or Carbowax 20m. In the GC experiments, *n*-decane was used as internal standard. ¹H NMR spectra were recorded on a Bruker-Avance AQS 300 MHz spectrometer.

Preparation of Polystyrene Supported Ruthenium(III) Salophen, Ru(Salophen)Cl@amine-PS

To a solution of ruthenium(III) salophen (0.45 g, 1 mmol) in acetonitrile (50 ml) was added amine-modified polystyrene (3 g). The mixture was vigorously stirred at 80 °C for 24 h. After cooling, the red-brown resins were collected by filtration, washed thoroughly with acetonitrile, methanol, and ether successively, and dried at room temperature for several

hours. The content of ruthenium(III) salophen on the polymer, which was calculated from its ruthenium content in the heterogenized catalyst, was determined by ICP.

General Procedure for Alkene Epoxidation with NaIO₄ Catalyzed by [Ru(salophen)Cl@amine-PS]

To a mixture of alkene (1 mmol), [Ru(salophen)@PS] (600 mg) in CH₃CN (10 ml) was added a solution of NaIO₄ (2 mmol) in H₂O (10 ml). The reaction mixture was stirred magnetically at room temperature. The progress of the reaction was monitored by GC. At the end of the reaction, the reaction mixture was diluted with Et₂O (20 ml) and filtered. The catalyst was thoroughly washed with Et₂O and combined washings and filtrates were purified on a silica-gel plate or a silica-gel column. IR and ¹H NMR spectral data confirmed the identities of the products.

Catalyst Reuse and Stability

The reusability of each catalyst was investigated in the multiple sequential epoxidation of cyclooctene as described above. At the end of each reaction, the catalyst was separated from the reaction mixture by simple filtration, washed with Et₂O and dried before being used in the next run.

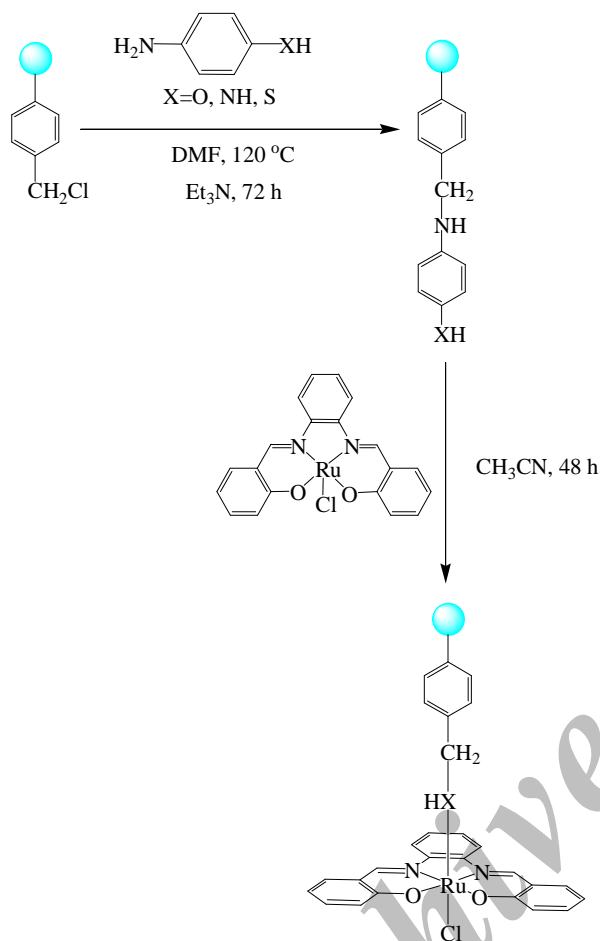
RESULTS AND DISCUSSION

Preparation and Characterization of the Catalysts [Ru(salophen)@DAB-PS], [Ru(salophen)@AP-PS] and [Ru(salophen)@ATP-PS]

Scheme 2 shows the preparation route for [Ru(salophen)@DAB-PS], [Ru(salophen)@AP-PS] and [Ru(salophen)@ATP-PS]. First, amines such as 1,4-diaminobenzene (DAB), 4-aminophenol (AP) or 4-aminothiophenol (ATP) were reacted with chloromethylated polystyrene, (cross-linked with 2% divinylbenzene, 4-5% Cl content, 1.14-1.40 mmol g⁻¹ Cl), to afford the amine-modified PS (DAB-PS, AP-PS and ATP-PS). In the final step, [Ru(salophen)Cl] was attached to these supports *via* axial ligation.

The prepared catalysts were characterized by elemental analysis, FT IR spectroscopy, scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The nitrogen content of the supports, measured by CHNS analysis, showed values of 2.25, 1.15 and 1.20% for DAB-PS, AP-PS

Highly Efficient Epoxidation of Alkenes with Sodium Periodate



Scheme 2. Preparation of [Ru(salophen)Cl@amine-PS]

and ATP-PS, respectively. Based on these values, the amount of nitrogen in DAB-PS, AP-PS and ATP-PS was calculated to be 1.61 (0.80 for coordinated nitrogens), 0.82 and 0.86 mmol per gram of each support, respectively. The Ru content of the catalysts, measured by ICP, was 0.105, 0.111 and 0.122 mmol

g^{-1} for [Ru(salophen)@DAB-PS], [Ru(salophen)@AP-PS] and [Ru(salophen)@ATP-PS], respectively (Table 1).

The most informative evidence, which confirmed the anchoring of the Ru(salophen)Cl to the functionalized PS, was obtained by FT IR spectra of [Ru(salophen)@DAB-PS], [Ru(salophen)@AP-PS] and [Ru(salophen)@ATP-PS] (Fig. 1 and Table 1). The azomethene (C=N) stretching band of homogeneous [Ru(salophen)Cl] appeared at 1607 cm^{-1} (Fig. 1A), while in the supported catalysts this band was observed at 1602, 1609 and 1601 cm^{-1} for [Ru(salophen)@DAB-PS], [Ru(salophen)@AP-PS] and [Ru(salophen)@ATP-PS], respectively (Fig. 1B-1D and Table 1). These observations clearly confirmed the attachment of Ru(salophen)Cl to amine-modified PS. The scanning electron micrographs of the catalysts (Fig. 2) show the morphology of the [Ru(salophen)@DAB-PS], [Ru(salophen)@AP-PS] and [Ru(salophen)@ATP-PS] used in this study. The TEM image of the prepared catalysts shows how the Ru(salophen)Cl is distributed on the polystyrene (Fig. 3).

The Effect of Catalysts Amount in the Epoxidation of Cyclooctene

In order to optimize the catalysts amount, different quantities of each catalyst were used in the epoxidation of cyclooctene (0.5 mmol) with NaIO_4 (1 mmol). The best results were obtained with 300 mg of each catalyst (Table 2).

The Effect of Solvent on the Epoxidation of Cyclooctene Catalyzed by [Ru(salophen)@DAB-PS], [Ru(salophen)@AP-PS] and [Ru(salophen)@ATP-PS]

In order to choose the reaction media, different solvents were checked in the epoxidation of cyclooctene with NaIO_4 . From among the different mixtures of acetonitrile, acetone, methanol, ethanol (single-phase systems), dichloromethane,

Table 1. Characteristic of the Prepared Catalysts

Catalyst	N content (mmol g^{-1})	Ru content (mmol g^{-1})	C=N band (cm^{-1})
[Ru(salophen)Cl]	-	-	1607
[Ru(salophen)@DAB-PS]	1.60	0.105	1602
[Ru(salophen)@AP-PS]	0.82	0.111	1609
[Ru(salophen)@ATP-PS]	0.86	0.122	1601

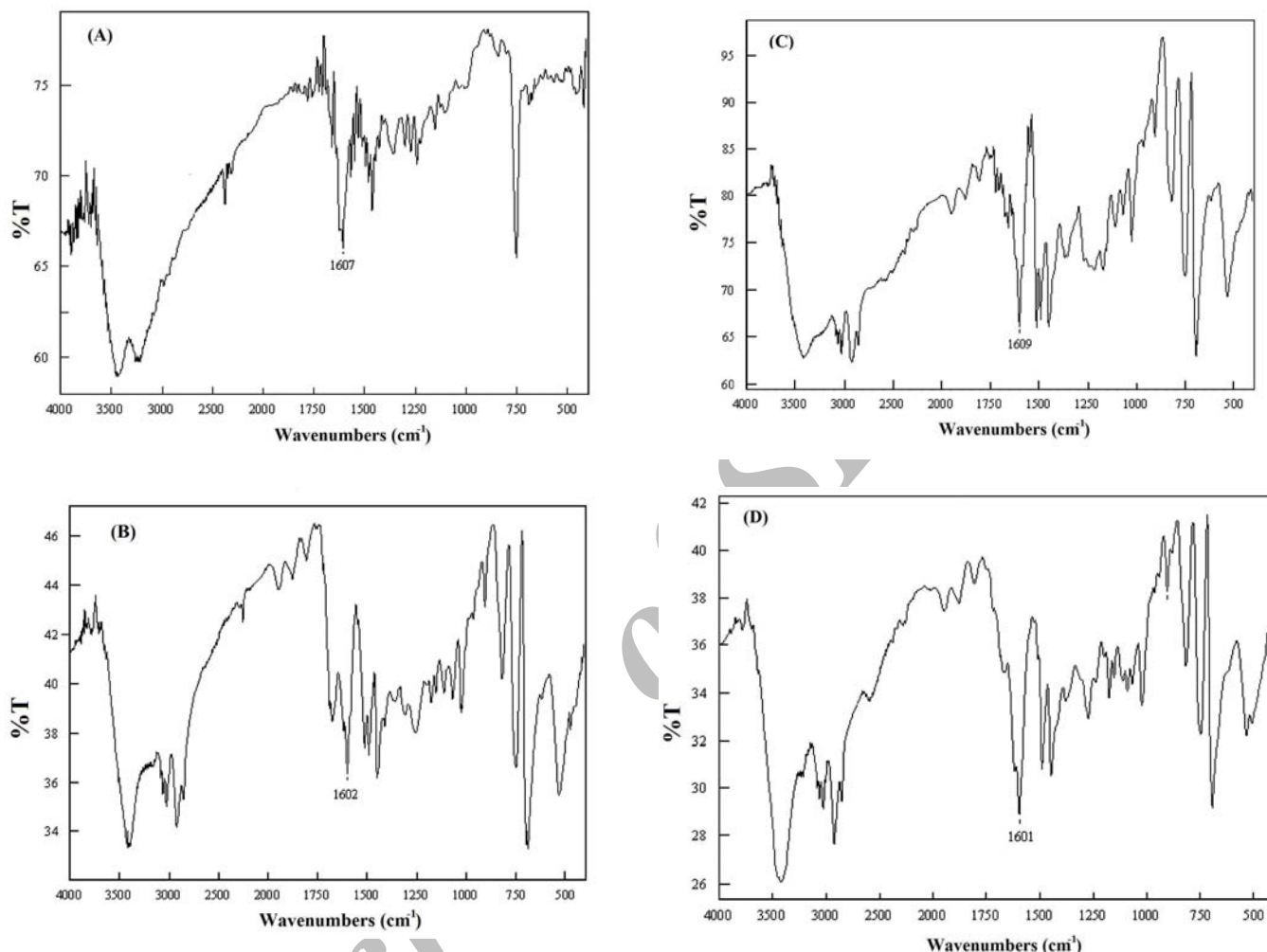


Fig. 1. FT IR spectra of: (A) [Ru(salophen)Cl], (B) [Ru(salophen)@DAB-PS], (C) [Ru(salophen)@AP-PS] and (D) [Ru(salophen)@ATP-PS].

chloroform and carbon tetrachloride (two-phase systems with Bu_4NBr as phase transfer catalyst), the 1:1 mixture of acetonitrile, water was chosen as the reaction medium because the higher catalytic activity was observed in this experimental condition (Table 3). The higher catalytic activity in acetonitrile/water mixture is attributed to the polarity of the solvent and solubility of NaIO_4 in this medium.

The Effect of Oxidant on the Epoxidation of Cyclooctene Catalyzed by [Ru(salophen)@DAB-PS], [Ru(salophen)@AP-PS] and [Ru(salophen)@ATP-PS]

Single oxygen donors such as NaIO_4 , Oxone, H_2O_2 , urea-

H_2O_2 (UHP), *tert*-BuOOH and *n*-Bu $_4$ NIO $_4$ were used for the epoxidation of cyclooctene. The results, which are summarized in Table 4, showed that NaIO_4 was the best oxygen source because this oxidant, which is inert in the absence of the catalyst, affords better oxidation conversion in $\text{CH}_3\text{CN}/\text{H}_2\text{O}$.

Epoxidation of Alkenes with NaIO_4 Catalyzed by [Ru(salophen)@DAB-PS], [Ru(salophen)@AP-PS] and [Ru(salophen)@ATP-PS]

The supported catalysts were used for the epoxidation of olefins with NaIO_4 under optimized conditions. The [Ru(salophen)@DAB-PS], [Ru(salophen)@AP-PS] and

Highly Efficient Epoxidation of Alkenes with Sodium Periodate

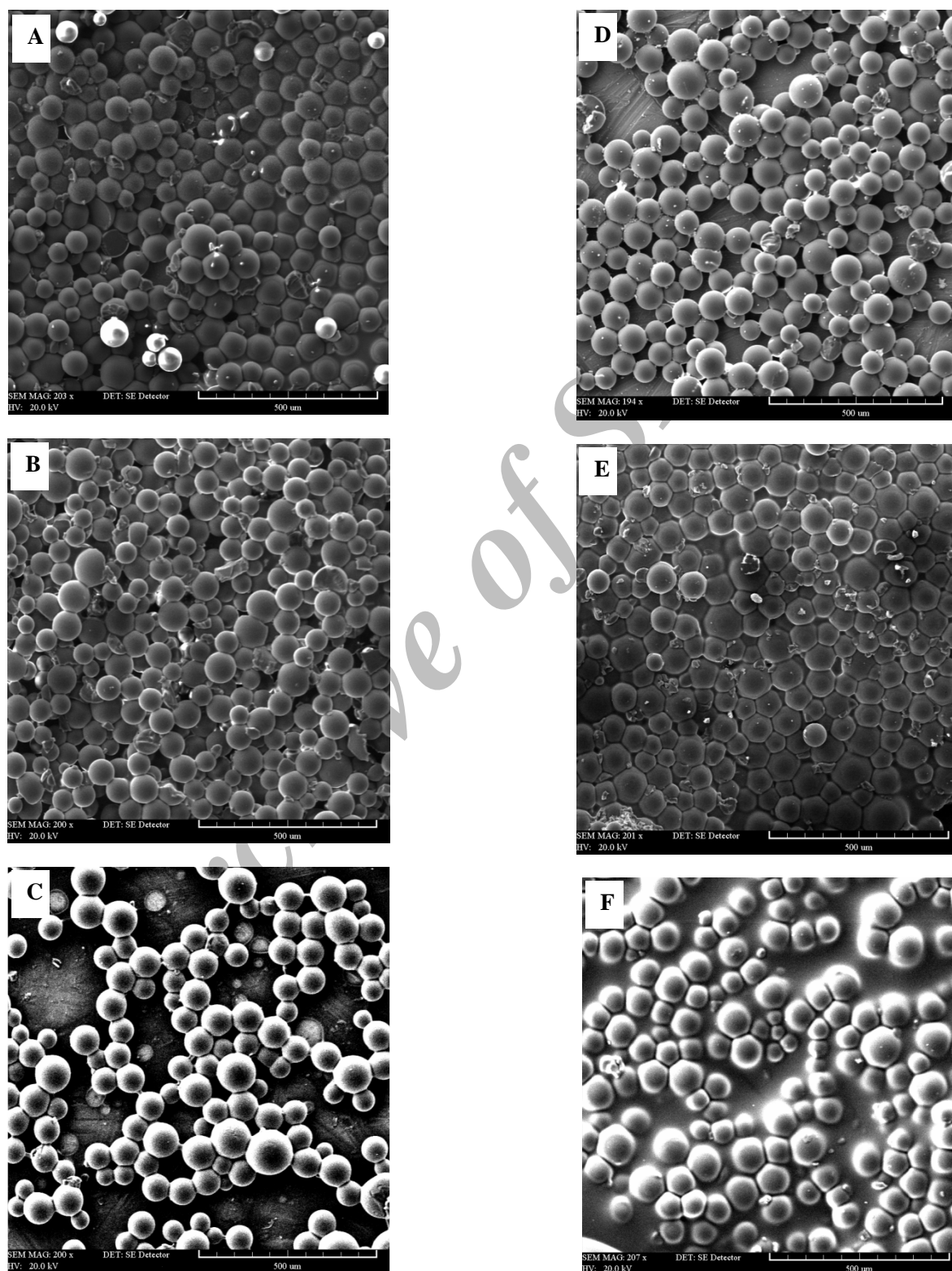


Fig. 2. SEM image of: (A) DAB-PS, (B) [Ru(salophene)@DAB-PS], (C) AP-PS, (D) [Ru(salophene)@AP-PS], (E) ATP-PS and (F) [Ru(salophen)@ATP-PS].

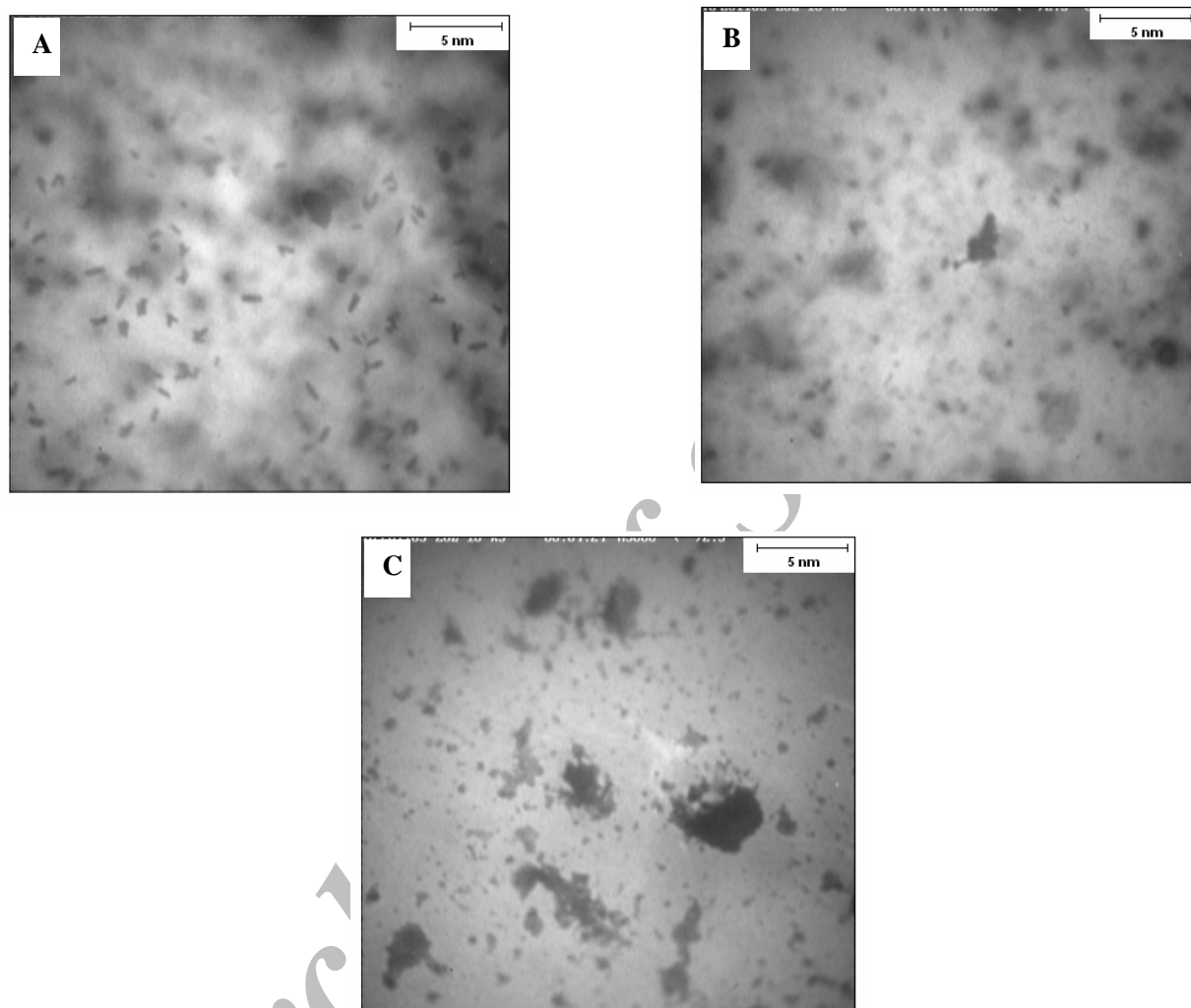


Fig. 3. TEM image of: (A) [Ru(salophen)@DAB-PS], (B) [Ru(salophen)@AP-PS] and (C) [Ru(salophen)@ATP-PS].

Table 2. Optimization of the Catalysts Amount in the Epoxidation of Cyclooctene with NaIO_4^a

Catalyst amount (mg)	Epoxide yield (%) ^b after 5 h		
	[Ru(salophen)@DAB-PS]	[Ru(salophen)@AP-PS]	[Ru(salophen)@ATP-PS]
0	5	5	5
200	62	61	60
250	74	71	70
300	93	87	89
350	93	87	89

^aReaction conditions: cyclooctene (0.5 mmol), oxidant (1 mmol), catalyst, $\text{CH}_3\text{CN}/\text{H}_2\text{O}$ (5 ml/5 ml). ^bGC yield based on the starting alkene.

Highly Efficient Epoxidation of Alkenes with Sodium Periodate

Table 3. The Effect of Solvent on the Epoxidation of Cyclooctene with NaIO₄ Catalyzed by [Ru(salophen)Cl] Supported on Modified PS at Room Temperature^a

Row	Solvent	Epoxide yield (%) ^b after 5 h		
		[Ru(salophen)@DAB-PS]	[Ru(salophen)@AP-PS]	[Ru(salophen)@ATP-PS]
1	CH ₃ CN/H ₂ O (1:1)	93	87	89
2	CH ₃ CN/H ₂ O (2:1)	73	64	66
3	CH ₃ OCH ₃ /H ₂ O	52	39	43
4	CH ₃ OH/H ₂ O	67	47	54
5	CH ₃ CH ₂ OH/H ₂ O	59	55	62
6	CH ₂ Cl ₂ /H ₂ O	34	25	27
7	CHCl ₃ /H ₂ O	23	14	19
8	CCl ₄ /H ₂ O	15	10	11

^aReaction conditions: cyclooctene (1 mmol), NaIO₄ (2 mmol), catalyst (600 mg), solvent/H₂O (10 ml/5 ml). ^bGC yield based on the starting alkene.

Table 4. The Effect of Oxidant on the Epoxidation of Cyclooctene Catalyzed by [Ru(salophen)Cl] Supported on Modified PS at Room Temperature^a

Row	Oxidant	Solvent	Epoxide yield (%) ^b after 5 h		
			[Ru(salophen)@DA B-PS]	[Ru(salophen)@AP-PS]	[Ru(salophen)@AT P-PS]
1	NaIO ₄ (2 mmol)	CH ₃ CN/H ₂ O	93	87	89
2	NaIO ₄ (1 mmol)	CH ₃ CN/H ₂ O	61	56	58
3	Oxone	CH ₃ CN/H ₂ O	83	77	79
4	H ₂ O ₂	CH ₃ CN	49	32	34
5	UHP	CH ₃ CN	30	13	21
6	<i>tert</i> -BuOOH	CH ₃ CN	23	19	25
7	(<i>n</i> -Bu) ₄ NIO ₄	CH ₃ CN	57	41	43

^aReaction conditions: cyclooctene (1 mmol), oxidant (2 mmol), catalyst (600 mg), CH₃CN/H₂O (10 ml/10 ml). ^bGC yield based on the starting alkene.







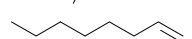
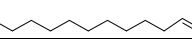
[Ru(salophen)@ATP-PS] were found to be efficient catalysts for the epoxidation of alkenes with NaIO₄ (Tables 5-7). In three catalytic systems, the cyclohexene was oxidized in high yield to cyclohexene oxide and 2-cyclohexen-1-one was obtained in 5-9% yields. In the epoxidation of styrene, the major product was styrene oxide and only small amounts of benzaldehyde was produced (6-8%). In the oxidation of α -methylstyrene, the corresponding epoxide was produced in high yield and acetophenone was obtained as the minor product (3-8%). Linear alkenes such as 1-octene and 1-dodecene were epoxidized in high yields with 100% selectivity. In the case of α -pinene, the α -pinene oxide was produced in 76-78% and allylic oxidation products, Verbenol and Verbenone, were produced in 10-11%.

Catalyst Reuse and Stability







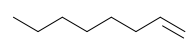
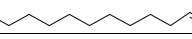
The reusability of a heterogeneous catalyst is of great importance from synthetic and economical points of view. The homogeneous [Ru(salophen)Cl] cannot be recovered even once; in contrast, the PS-supported catalysts can be filtered and reused several times.

The reusability of [Ru(salophen)@DAB-PS], [Ru(salophen)@AP-PS] and [Ru(salophen)@ATP-PS] was investigated in the multiple sequential epoxidation of cyclooctene with NaIO₄. At the end of each reaction, the catalyst was separated by simple filtration, washed with Et₂O and dried carefully before being used in the next run. After using each catalyst for five consecutive times, the epoxide yields were 55, 47 and 43% for [Ru(salophen)@DAB-PS],

Table 5. Epoxidation of Alkenes with NaIO₄ Catalyzed by [Ru(salophen)@DAB-PS]^a

Row	Alkene	Conversion (%) ^a	Epoxide yield (%) ^b	Time (h)
1		93	93	5
2		95 ^c	86	5
3		90 ^d	84	5
4		89 ^e	81	5
5		86	86	5
6		88 ^f	77	5
7		66	66	10
8		55	55	10



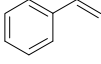
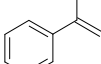
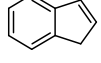


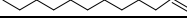
^aReaction conditions: alkene (1 mmol), NaIO₄ (2 mmol), catalyst (0.063 mmol), CH₃CN/H₂O (10 ml/10 ml).^bGLC yield based on the starting alkene. ^cThe by-product is allylic ketone. ^dThe by product is benzaldehyde.^eThe by-product is acetophenone. ^fIn this case, 7% ketone and 4% alcohol were produced.**Table 6.** Epoxidation of Alkenes with NaIO₄ Catalyzed by [Ru(salophen)@AP-PS]^a

Row	Alkene	Conversion (%) ^a	Epoxide yield (%) ^b	Time (h)
1		87	87	5
2		87 ^c	82	5
3		88 ^d	80	5
4		82 ^e	79	5
5		82	82	5
6		86 ^f	76	5
7		58	58	10
8		44	44	10

^aReaction conditions: alkene (1 mmol), NaIO₄ (2 mmol), catalyst (0.067 mmol), CH₃CN/H₂O (10 ml/10 ml).^bGLC yield based on the starting alkene. ^cThe by-product is allylic ketone. ^dThe by product is benzaldehyde.^eThe by-product is acetophenone. ^fIn this case, 7% ketone and 3% alcohol were produced.

Highly Efficient Epoxidation of Alkenes with Sodium Periodate

Table 7. Epoxidation of Alkenes with NaIO₄ Catalyzed by [Ru(salophen)@ATP-PS]^a

Row	Alkene	Conversion (%) ^a	Epoxide yield (%) ^b	Time (h)
1		89	89	5
2		92 ^c	85	5
3		89 ^d	82	5
4		85 ^e	79	5
5		84	84	5
6		89 ^f	78	5
7		59	59	10
8		43	43	10

^aReaction conditions: alkene (1 mmol), NaIO₄ (2 mmol), catalyst (0.073 mmol), CH₃CN/H₂O (10 ml/10 ml). ^bGLC yield based on the starting alkene. ^cThe by-product is allylic ketone. ^dThe by product is benzaldehyde. ^eThe by-product is acetophenone. ^fIn this case, 7% ketone and 4% alcohol were produced.

Table 8. The results of Catalysts Recovery and the Amounts of Ruthenium Leached in the Epoxidation of Cyclooctene with Sodium Periodate^a

Run	[Ru(salophen)@DAB-PS]		[Ru(salophen)@AP-PS]		[Ru(salophen)@ATP-PS]	
	Yield (%) ^b	Ru leached (%) ^c	Yield (%) ^b	Ru leached (%) ^c	Yield (%) ^b	Ru leached (%) ^c
1	93	1.5	87	2.1	89	2.9
2	89	1.0	82	1.7	85	2.0
3	84	0.6	78	1.0	78	1.1
4	79	0.4	65	0.8	62	0.9
5	55	0	47	0.0	43	0

^aReaction conditions: alkene (1 mmol), NaIO₄ (2 mmol), catalyst (600 mg), CH₃CN/H₂O (10 ml/10 ml). ^bGLC yield based on the starting alkene. ^cDetermined by ICP.

[Ru(salophen)@AP-PS] and [Ru(salophen)@ATP-PS], respectively (Table 8 and Fig. 4). The filtrates were collected for the determination of Ru leaching (measured by ICP). The results showed that in the first three runs, the ruthenium catalyst was leached from supports and, therefore, the epoxide yield decreased (Table 8).

CONCLUSIONS

In conclusion, Ru(salophen)Cl supported on amine-modified polystyrene was prepared by axial ligation of metallocomplex to the support. Ruthenium(III) salophen, supported on modified PS, [Ru(salophen)@DAB-PS],

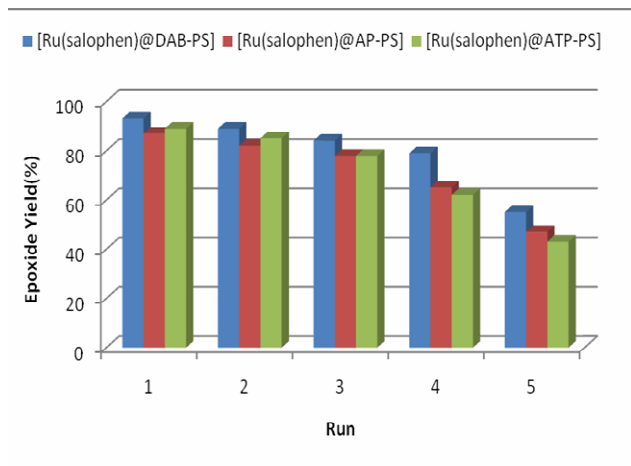


Fig. 4. The results obtained for reusing of [Ru(salophene)@DAB-PS], [Ru(salophene)@AP-PS], and [Ru(salophene)@ATP-PS] in the oxidation of cyclooctene with NaIO₄.

[Ru(salophen)@AP-PS] and [Ru(salophen)@ATP-PS], are highly efficient catalysts for the epoxidation of alkenes with sodium periodate under agitation with magnetic stirring. These new catalysts have the advantages such as easy preparation and handling of the catalysts, commercial availability of the support, and facile and effective recovery and recycling of the supported catalysts.

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Highly Efficient Epoxidation of Alkenes with Sodium Periodate

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