

Chromium (VI) Immobilized on Mesoporous Polyaniline (Cr-PANI) as a Catalyst for the Oxidation of Alkanes and Alkenes Under Mild Conditions

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Abstract

A mesoporous polyaniline-supported chromium (VI) composite (Cr-PANI) was synthesized via a straightforward in-situ radical polymerization of aniline, subsequently modified with potassium dichromate. To evaluate its structural, morphological, and thermal properties, the resulting material was characterized using X-ray diffraction (XRD), transmission electron microscopy (TEM), Fourier transform infrared spectroscopy (FT-IR), UV-vis diffuse reflectance spectroscopy, and thermogravimetric analysis (TGA). Structural and morphological analysis via XRD and TEM suggested that the material possesses a mesostructured framework characterized by disordered, worm-like mesopores. When evaluated as a catalyst, Cr-PANI demonstrated excellent efficiency in liquid-phase oxidation of alkanes and alkenes, utilizing aqueous 30% hydrogen peroxide (H_2O_2) as the oxidant. Operating under mild conditions, the catalytic system provided high product selectivity and satisfactory yields. Moreover, the catalyst demonstrated strong reusability, maintaining its performance over five consecutive cycles without significant loss of activity.

Keywords: Alkenes and Alkanes; H_2O_2 ; Mesoporous Polyaniline; Oxidation; Water

Introduction

Catalytic oxidation represents one of the most important transformations in organic synthesis, providing efficient routes to a wide range of value-added oxygenated products such as alcohols, aldehydes, ketones, carboxylic acids and epoxides. In particular, hydrogen peroxide-based oxidation processes are highly attractive owing to the benign nature of water as the only by-product, together with their environmental and economic advantages (Mukherjee *et al.*, 2006; Mandi *et al.*, 2014; Salam *et al.*, 2012). While alkanes and alcohols are readily oxidized, the selective activation of C–H bonds in alkanes remains challenging because of their inherent stability, often leading to overoxidation. The selective oxidation of primary alcohols to aldehydes, in particular, has significant applications in pharmaceuticals, dyes and agrochemicals.

Polymer-bound metal complexes have been widely explored as heterogeneous catalysts for oxidation reactions due to their ability to merge the high catalytic performance and selectivity of homogeneous systems with the practical advantages of heterogeneous catalysts, such as simple separation, catalyst recovery and recyclability (Mondal *et al.*, 2013). Conducting polymer metal composites, especially those based on polyaniline (PANI), have attracted considerable attention due to their synergistic properties, facile synthesis, environmental stability, insolubility in most solvents and wide conductivity range (Banjar *et al.*, 2023; Haral *et al.*, 2024; Nazerian *et al.*, 2025). Polyaniline-supported catalysts show various catalytic activities for organic transformations (Behera, 2024), electrochemical energy conversion (Wu & Li, 2025), enhanced oxygen reduction reactions (Yang *et al.*, 2024), and oxygen evolution activities for rechargeable Zn-air batteries (Zou *et al.*, 2025). PANI-supported metal catalysts, including vanadium, Fe (II), cobalt, Mn (II) porphyrins, FeCl_2 , and CuCl_2 , have demonstrated high efficiency in the selective oxidation of alcohols and alkenes (Reddy *et al.*, 2004; Pielichowski & Kowalski, 2010), oxidation of propanol (Watanabe *et al.*, 2025), oxidation of methanol (Teng *et al.*, 2023), and oxidation of nitrophenols (Khani *et al.*, 2023).

Encouraged by these findings, we prepared mesoporous PANI-supported Cr (VI) catalysts to capitalize on the synergistic benefits of homogeneous and heterogeneous catalytic approaches. The catalysts were characterized by TEM, XRD, FTIR, TGA and UV-visible spectroscopy, confirming their nanostructured nature. The Cr-PANI system showed excellent catalytic activity for the oxidation of alkenes and alkanes using 30% H₂O₂ in an aqueous medium and retained its activity over multiple cycles, demonstrating high stability and recyclability.

This work highlights the potential of PANI-supported Cr(VI) as a sustainable and efficient heterogeneous catalyst for selective oxidation reactions, combining high catalytic performance with environmental and economic benefits.

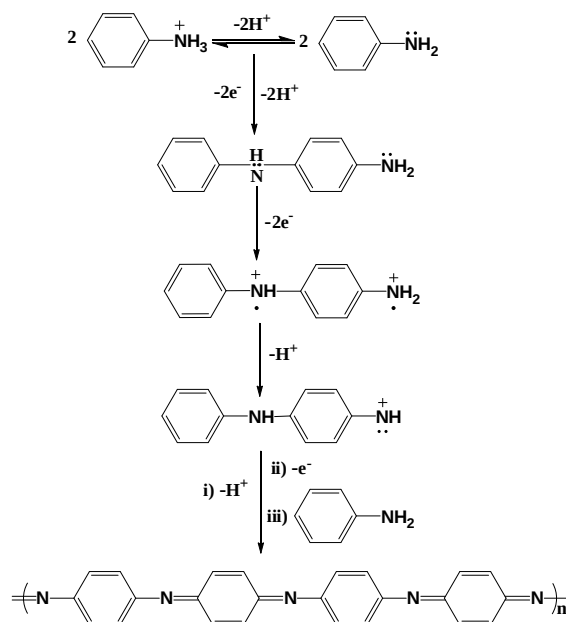
Synthetic Procedure

Materials

Aniline, potassium dichromate (K₂Cr₂O₇), ammonium persulphate ((NH₄)₂S₂O₈), and concentrated hydrochloric acid (HCl) were sourced from Merck. Ammonium persulphate served as the oxidizing initiator during the aqueous-phase polymerization of aniline. Sodium dodecyl sulphate (SDS), obtained from Loba Chemie, was utilized as a structure-directing agent (SDA) to facilitate the formation of the desired polymer morphology. Alkenes, alkanes, and alcohols were supplied by Merck or Fluka.

Synthesis of Mesoporous Polyaniline (PANI)

The meso-PANI (Scheme 1) was prepared through template-assisted polymerization of aniline. For the synthesis, 0.02 mol of aniline was first dissolved in 5 mL of aqueous HCl containing 0.02 mol acid. This solution was then added to 20 mL of an aqueous SDS solution (0.01 mol) with continuous stirring. The reaction mixture was cooled in an ice-salt bath and maintained at 4-5°C. Subsequently, 20 mL of an ice-cold ammonium persulphate solution (0.04 mol) was introduced gradually into the mixture. Once a homogeneous solution was obtained, stirring was halted, and the mixture was left undisturbed at 4-5°C for 48 hours to allow aging. The solid product formed was separated by filtration, rinsed several times with deionized water, and finally dried under vacuum at room temperature. The mesoporous PANI was finally extracted using an aqueous ammonium acetate solution.



Scheme 1: Schematic Diagram Showing the Polymerization of Aniline

Synthesis of Cr-PANI

In a typical procedure, 500 mg of mesoporous polyaniline and 100 mg of potassium dichromate were placed in a 50 mL round-bottom flask. Subsequently, 10 mL of double-distilled water was added to obtain a homogeneous suspension. The mixture was stirred vigorously at room temperature for 24 hours. After completion of the reaction, the resulting precipitate was collected by filtration, thoroughly washed with double-distilled water, and dried under vacuum.

Results and Discussion

Characterization of Synthesized Cr-PANI

X-ray Diffraction (XRD) Studies

Figure 1 presents the small-angle XRD pattern of the dichromate-loaded polyaniline material (Cr-PANI). A distinct diffraction peak appears at $2\theta = 2.41^\circ$, indicating the presence of a mesophase structure in the material. This observation suggests that the incorporation of Cr (VI) species does not significantly alter the structural integrity of the mesophase.

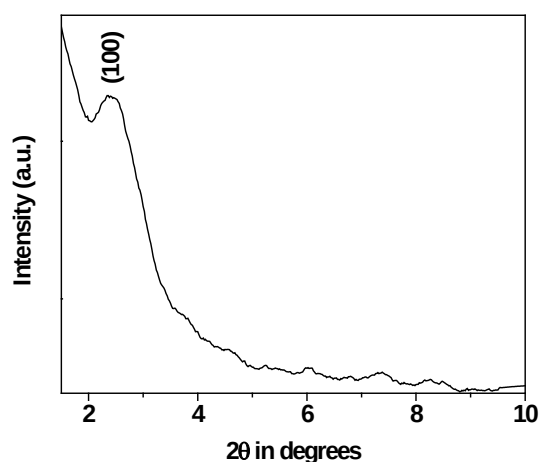


Figure 1: Small Angle X-ray Diffraction (XRD) Pattern of Cr-PANI

Transmission electron microscopy (TEM) Analysis

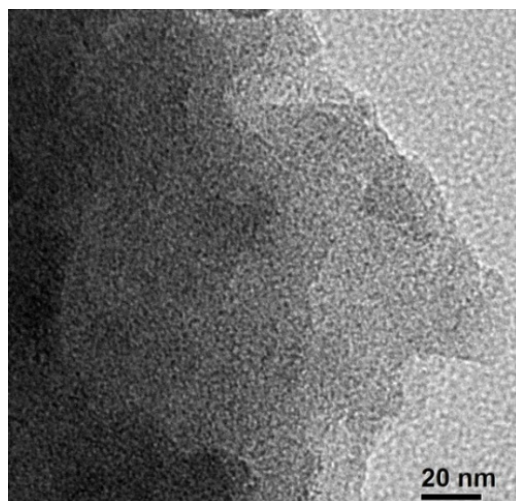


Figure 2: Transmission Electron Microscopy (TEM) Image of Cr-PANI

The representative transmission electron microscopy (TEM) image of the material is shown in Figure 2. The image shows low electron-density regions, attributed to pores, that are uniformly distributed throughout the sample. The average pore size is approximately 2.3 nm. Compared to the parent PANI sample, a slight reduction in pore size is evident after $\text{Cr}_2\text{O}_7^{2-}$ loading, likely resulting from the deposition of dichromate species onto the pore surfaces.

UV-Visible Spectra

The UV-Visible spectra of mesoporous PANI and Cr-PANI (Figure 3) reveal broad absorption bands centered within the regions of 310–460 nm and 660–720 nm for mesoporous PANI. Such absorption behavior is characteristic of nanostructured polyaniline, which commonly exhibits two distinct electronic transitions at around 320–340 nm and 600–660 nm (Laska & Widlarz, 2005). The higher-energy band is attributed to the $\pi \rightarrow \pi^*$ transition of the benzenoid rings, while the lower-energy band corresponds to the transition from benzenoid to quinoid structures. In the case of Cr-PANI, the absorption band associated with the quinoid transition shifts from around 700 nm to a higher wavelength region (red shift) upon incorporation of Cr (VI). This shift can be explained by the formation of a more compact coil structure in the Cr-PANI chains, which reduces the energy gap associated with the quinoid transition, thereby facilitating electronic transitions. In contrast, the $\pi \rightarrow \pi^*$ transition band shows a negligible shift, indicating that Cr (VI) species preferentially interact with the nitrogen atoms of the quinoid segments.

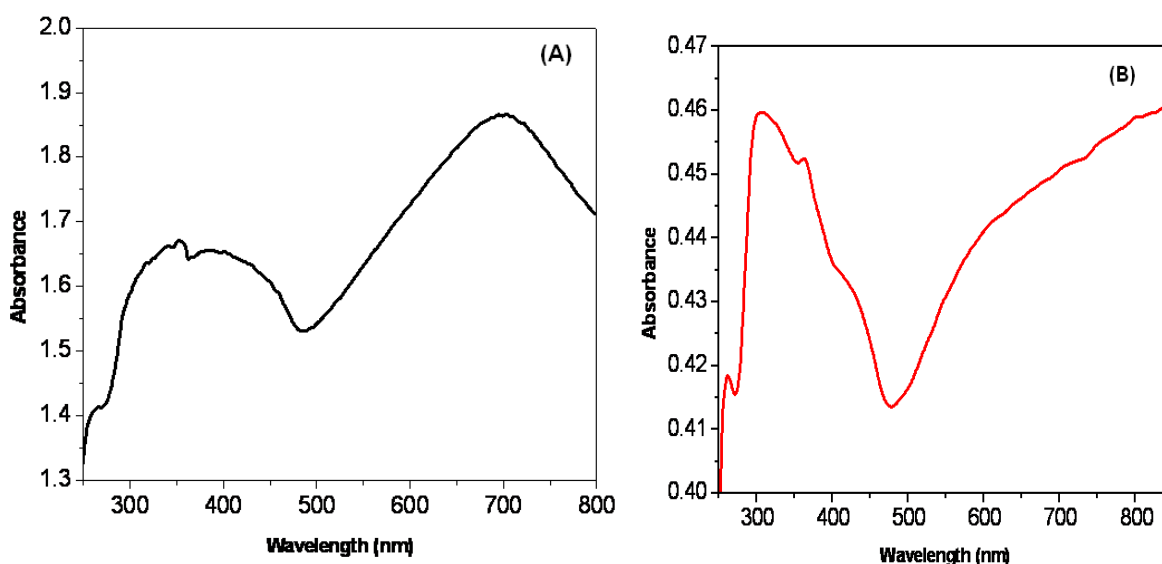


Figure 3: UV-Visible Spectra of (A) PANI and (B) Cr-PANI

Fourier Transform Infrared (FTIR) Spectra

FTIR spectroscopy (Figure 4) further confirmed the interaction between Cr (VI) species and the PANI framework. The FTIR spectrum of PANI exhibits distinct bands at 1578 and 1493 cm^{-1} , attributed to the C=C stretching vibrations of quinoid and benzenoid rings, respectively (Chen *et al.*, 2010). The absorption peak located at 1306 cm^{-1} is associated with the stretching vibration of C-N bonds (Ahn *et al.*, 2011), whereas the band appearing at 1148 cm^{-1} is attributed to the in-plane bending vibration of C-H bonds (Dexmer *et al.*, 2008). In the Cr-PANI composite, the C=C stretching bands exhibit a noticeable blue shift, indicating strong interaction between Cr (VI) and the polymer backbone (Han *et al.*, 2013). Additionally, the C-N stretching band shifts to a lower wavenumber upon Cr (VI) incorporation, suggesting coordination between chromium species and nitrogen atoms in the polymer chain.

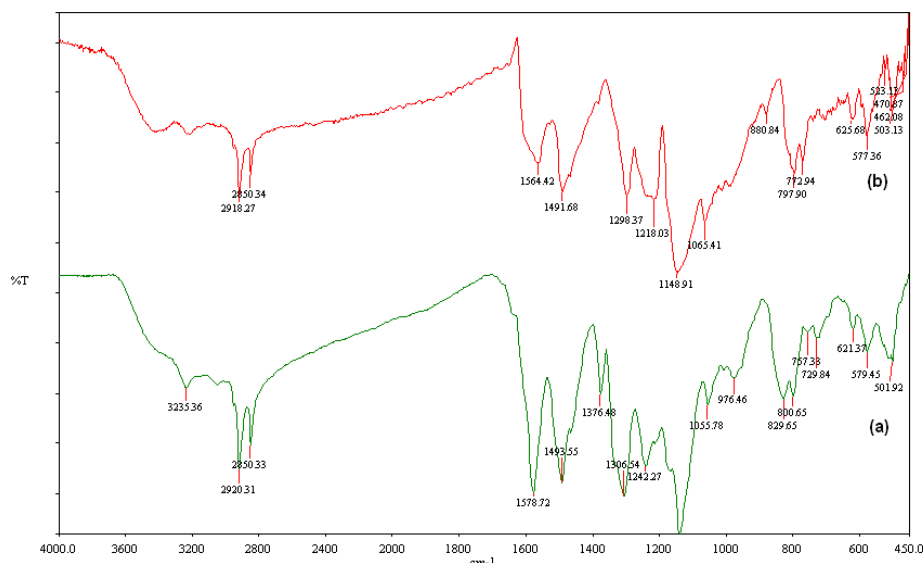


Figure 4: FT-IR Spectra (a) PANI and (b) Cr-PANI

Thermogravimetric (TG) Analysis

Thermal stability of the mesoporous polyaniline materials was evaluated using thermogravimetric analysis (TGA), as shown in Figure 5. The measurements were carried out under a nitrogen atmosphere at a heating rate of $10\text{ }^{\circ}\text{C min}^{-1}$ over the temperature range of 50–600 $^{\circ}\text{C}$. Mesoporous PANI exhibits significant weight loss in the temperature range of 190–475 $^{\circ}\text{C}$, corresponding to the thermal degradation of the polymer backbone.

In comparison, the Cr-PANI nanocomposite displays a similar degradation profile; however, the onset of mass loss shifts to a higher temperature ($\sim 230^{\circ}\text{C}$), indicating enhanced thermal stability. Notably, the thermal stability of Cr-PANI improves by approximately 40–50 $^{\circ}\text{C}$ relative to pristine PANI. This increased stability is attributed to strong interactions between Cr(VI) species (originating from $\text{K}_2\text{Cr}_2\text{O}_7$) and the PANI chains, which restrict the segmental motion of the polymer and delay its thermal decomposition.

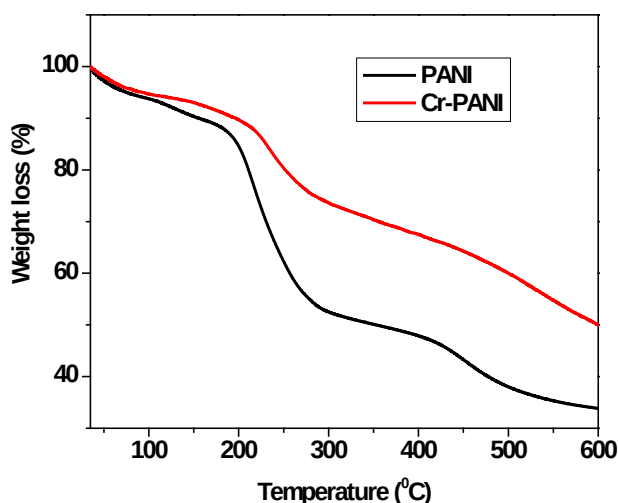


Figure 5: TGA Plots of Mesoporous PANI and Cr-PANI

Overall, the combined XRD, TEM, UV-Vis, FTIR, and TG analyses reveal that while the mesoporous structure and backbone of PANI remain largely intact after Cr(VI) loading, significant electronic and chemical interactions occur between Cr(VI) species and the mesostructure polymer.

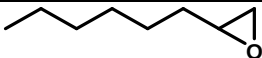
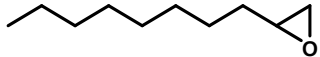
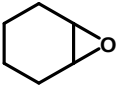
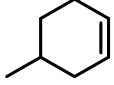
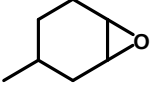
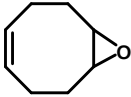
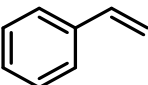
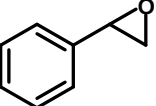
Catalytic Activity

Oxidation of Alkenes Catalyzed by Cr-PANI

The epoxidation activity of Cr-PANI toward various alkenes in the liquid phase was investigated, and the results are presented in Table 1. Alkenes possessing terminal C=C bonds showed high levels of conversion and selectivity (Table 1, entries 1 and 2), yielding only the corresponding epoxide products. In contrast, the oxidation yield decreased progressively with increasing aliphatic chain length in alkenes containing a single terminal double bond, which may be attributed to steric constraints and reduced diffusion efficiency. In contrast, cyclic unsaturated hydrocarbons containing one or more double bonds demonstrated significantly higher reactivity. These substrates were efficiently converted to their corresponding epoxides within 4-6 hours, depending on the nature of the substrate (Table 1, entries 3-5). The enhanced reactivity of cyclic alkenes may be attributed to their higher electron density and favorable interaction with the active sites of the Cr-PANI catalyst. In the Cr-PANI-catalyzed oxidation of cyclohexene, cyclohexene oxide was produced with 97% selectivity after a reaction time of 4 h (Table 1, Entry 3).

In contrast, aromatic alkenes generally require longer reaction durations than carbocyclic alkenes to achieve comparable conversions. Styrene was efficiently transformed into styrene oxide in high yield (Table 1, Entry 6), while substituted styrene also selectively formed the corresponding epoxide products (Table 1, Entries 7 and 8). Similarly, α -methyl styrene predominantly afforded its epoxide as the dominant product (Table 1, Entry 9). However, substrates bearing a double bond at the benzylic position show high reactivity, leading to the formation of a considerable number of by-products during oxidation. Depending on the nature of the substrate, aldehydes and/or ketones are generated as the major side products. Cr-PANI also demonstrates notable catalytic performance in the oxidation of terpenes, where the corresponding epoxides are predominantly formed under similar reaction conditions (Table 1, Entries 10 and 11). In contrast, the oxidation of indene mainly produces the corresponding ketone in high yield (Table 1, Entry 12).

Table 1: Epoxidation of Alkenes with Cr-PANI Catalyst Using 30% H₂O₂

Entry	Substrate	Time (h)	Conversion (%) ^a	Product	Selectivity (%) ^a
1		12	76		100
2		12	69		100
3		4	92		97
4		4	94		95
5		5	82		93
6		8	89		95

7		8	90		94
8		8	78		90
9		8	82		89
10		4	48		67
11		3	56		72
12		3	86		86

Reaction conditions: catalyst (0.02 g), alkene (5 mmol), water (10 mL), 30% aqueous H_2O_2 (10 mmol), reaction temperature 60°C . ^a Conversion and selectivity were determined by GC analysis.

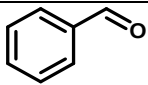
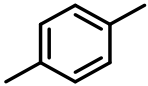
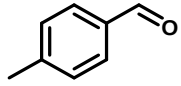
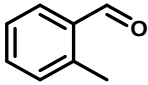
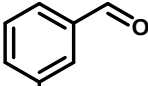
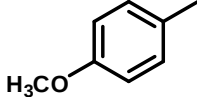
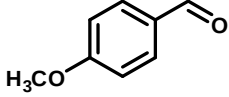
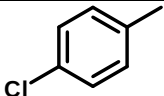
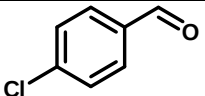
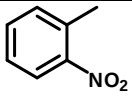
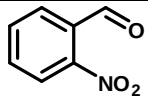
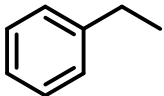
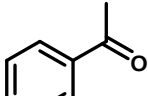
Oxidation of Alkanes Catalyzed by Cr-PANI

The Cr-PANI catalyst was also investigated for the oxidation of alkyl benzenes using H_2O_2 at 70°C . As shown in Table 2, it effectively promotes the oxidation of various alkyl benzenes bearing electron-donating groups, producing the corresponding aldehydes with good yields and high selectivity. For toluene derivatives bearing electron-donating groups, the reaction proceeded at a significantly faster rate than that observed for unsubstituted toluene.

With reactions reaching near completion within 4 hours (Table 2, entries 2–4). Similarly, *p*-methoxy toluene, which contains a strongly electron-donating methoxy group, exhibited a high reaction rate and conversion. However, it showed relatively lower selectivity (75%) toward 4-methoxybenzaldehyde (Table 2, entry 5), possibly due to competing side reactions under the reaction conditions. The high reactivity of *p*-methoxy toluene can be attributed to the electron-donating effect of the methoxy group, which activates the benzylic position toward oxidation. As a result, besides the formation of 4-methoxybenzaldehyde, some over-oxidized products, such as carboxylic acids, were also observed. In the case of ethylbenzene, oxidation occurred exclusively at the benzylic C–H bond, leaving the methyl group intact, yielding acetophenone in high yield (94%) and selectivity (96%) (Table 2, Entry 8).

In contrast, toluene derivatives bearing electron-withdrawing groups exhibited lower reactivity compared to those with electron-donating substituents (Table 2, entries 6 and 7). This diminished reactivity can be ascribed to the reduced electron density on the aromatic ring, which makes it less susceptible to oxidation. As an example, *p*-chlorotoluene was oxidized to 4-chlorobenzaldehyde with 85% selectivity (Table 2, Entry 6). The strongly electron-withdrawing nitro group in *o*-nitrotoluene further decreased reactivity, giving only 34% yield of 2-nitrobenzaldehyde with 91% selectivity after 6 hours (Table 2, Entry 7).

Table 2: Oxidation of Various Alkanes with Cr-PANI Catalyst Using 30% H₂O₂

Entry	Substrate	Time (h)	Conversion (%) ^a	Product	Selectivity (%) ^a
1		6	79		84
2		4	87		90
3		4	92		86
4		4	81		92
5		4	86		75
6		6	46		85
7		6	37		91
8		4	94		96

Reaction condition: catalyst (0.02 g), substrate (5 mmol), H₂O₂ (10 mmol), water 10 mL, temperature (70 °C). ^a Conversion and selectivity were determined by GC analysis.

Heterogeneity Test

To examine possible chromium leaching during the process, styrene oxidation was selected as a model reaction under optimized conditions. After 3 hours, the reaction was stopped, and the filtrate was collected and further treated under the same conditions for an additional 3 hours. The lack of any further increase in conversion indicated the absence of active catalyst species in the solution phase. The stability of the Cr-PANI catalyst was further evaluated using UV-visible spectroscopy. Following the first reaction run, the spectrum of the reaction mixture showed no distinct absorption features attributable to chromium species, indicating that metal leaching during oxidation was minimal. These findings confirm that chromium species are strongly immobilized on the polyaniline surface, supporting the heterogeneous character of the catalyst. They further show that Cr-PANI remains stable and can be recycled through several oxidation cycles with no significant decrease in activity.

Reusability of the Catalyst

A major benefit of heterogeneous catalysis is the ease with which the catalyst can be recovered and reused. Because the catalyst is insoluble under the reaction conditions, it can be readily separated by simple filtration and then washed. The reusability of the heterogeneous Cr-PANI catalyst was investigated in the oxidation of styrene and ethyl benzene (Figure 6). After each run, the catalyst was collected by filtration, rinsed successively with ethyl acetate and acetone, and finally dried under reduced pressure at 60°C. The recovered catalyst was then used in subsequent runs with the addition of fresh substrates under the optimum reaction conditions. As shown in Figure 6, the recycled catalyst exhibited no significant loss of activity, indicating that it is stable and can be regenerated for repeated use over an extended period.

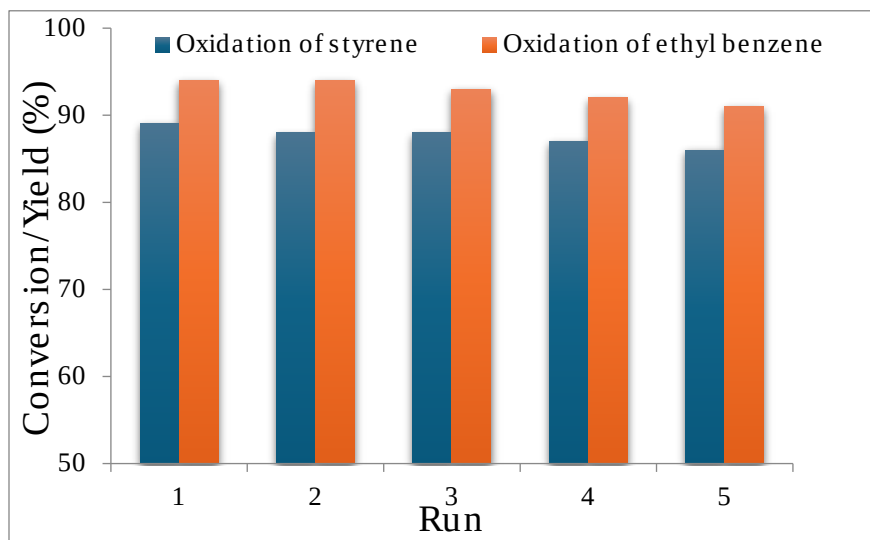


Figure 6: Reusability Test for the Oxidation of Styrene and Ethyl Benzene Using Cr- PANI

Conclusion

Mesoporous Cr-PANI was prepared via immobilization of chromate anions on the polyaniline surface. The resulting heterogeneous catalyst shows high catalytic performance in the oxidation of alkenes and alkanes, selectively giving epoxides from alkenes, while alkanes are converted into the corresponding aldehydes or ketones. A key advantage of this catalyst is its stability and recyclability under the reaction conditions. Recycling experiments show no significant loss of activity, and leaching tests confirm that the active sites remain bound to the support, indicating the reaction proceeds primarily via a heterogeneous mechanism. The catalyst can be reused multiple times without appreciable decrease in performance. The high catalytic efficiency of this chromium-grafted mesoporous polymer suggests promising potential for future applications of related mesoporous organic polymers.

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