Epoxidation of Cyclohexene Catalyzed by Vanadium Complex Supported on Montmorillonite K-10

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Abstract

Epoxides are one of the important precursors for the formation of organic compounds with oxygen as they undergo ring-opening reactions easily to form bifunctional compounds. Homogeneous catalysts have been used in producing epoxide compounds. However, as these types of catalysts require further separation processes as well as corrosive issues, heterogeneous catalysts have received massive attention. In this study, natural clay from montmorillonite K10 (MMT-K10) was modified via the incorporation of VO(acac)$_2$ at various concentrations. The result of XRD indicated that the crystalline nature of incorporated MMT-K10 was maintained with the 001 basal spacing calculated to be 9.95 Å. The AAS suggested that VO(acac)$_2$ complex has been successfully incorporated onto MMT-K10 with the increasing amount of vanadium element as the concentration of vanadium complex solution increased. The catalytic activity of the vanadium complex supported on MMT-K10 showed that 20% of cyclohexene was converted with 70% selectivity to cyclohexene oxide using tert-butyl hydroperoxide (TBHP) as the oxidant.

Disciplinary: Chemistry (Heterogeneous Catalysis).

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1 Introduction

Considerable attention has been paid to vanadium complexes as catalysts because of their good performance. Vanadium complexes as homogeneous catalysts have been utilized in numerous organic reactions such as hydroxylation of alkanes (Sharma et al., 2018), bromination reactions

(Maurya et al., 2015), oxidations of sulfides (Rayati et al., 2017; Nikoorazm et al., 2016), and oxidations alcohols to aldehydes and ketones (Sarmah et al., 2014; Zavahir et al., 2016; Steffensmeier & Nicholas, 2018).

The epoxidation process usually involves strong mineral acids as homogeneous catalysts with peracids as oxidizing agents. Vanadium-based catalysts can also be utilized in the epoxidation of alkenes. Homogeneous catalytic systems based on vanadium complexes are considered to be very efficient catalysts for the epoxidation of various types of alkenes (Vandichel et al., 2012; Dar et al., 2019; Maurya, 2020). It was found the system that consists of $VO(acac)_2$ and TBHP permits highly chemoselective mono-epoxidation of olefinic alcohol like geraniol (Vandichel et al., 2012). Vanadium complex $[VO(hyap)(acac)_2]$ was successfully synthesized and proven to be catalytically active for epoxidation of various alkenes with H_2O_2 as the oxidant (Maurya, 2020).

Incorporation of transition metal complexes on solid porous materials as a catalyst for epoxidation of alkenes has been one of the techniques used in order to minimize hazardous waste exposed to the environment. The epoxidation of styrene has been conducted using VO^{2+} Schiff base complex immobilized onto graphene oxide and it was found that the conversion of styrene was ca. 31.1% with high selectivity to styrene epoxide (96%) (Su et al., 2014). The immobilization of VO^{2+} Schiff base complex onto mesoporous carbon (CMK-3) functionalized with the amino group was also tested for styrene epoxidation with conversion and selectivity to styrene epoxide were 91.8% and 63.7%, respectively (Wang et al., 2016).

Natural aluminosilicates, such as clays and zeolites, are solid acids that are notably considered as favourable competitors to replace liquid acids in catalysis reactions. Among these two catalysts, natural and modified clays have serious attention due to their good catalytic activity and environmentally friendly properties. Particularly for the most common and potentially modified clays in organic synthesis application are the montmorillonite K10 (MMT-K10) and montmorillonite KSF. MMT-K10 has a higher surface area (about 250 m^2g^{-1}) in contrast to that of KSF (10 m^2g^{-1}), making it a more effective and excellent catalyst (Kaur & Kishore, 2012). Apart from high surface area property, it also holds a high cation exchange capacity. Due to these characteristics, clays have been used as support for epoxidation reactions.

For example, boehmite modified with 3-(trimethoxysilyl)propylamine or 3-(tri-methoxysilyl)-propyl chloride to support vanadium complex was investigated as heterogeneous catalyst for epoxidation of various alkenes. The high conversion and selectivity values (>90%) in alkenes epoxidation with up to 10 recyclabilities of the used catalyst indicate that this heterogeneous catalyst is an excellent catalyst (Mirzaee et al., 2015, Mirzaee et al., 2019). Oxovanadium(IV) Schiff base complex was immobilized onto montmorillonite for epoxidation of cyclooctene. Maximum conversion of cyclooctene was achieved (up to 90%) with excellent 100% selectivity to cyclooctene oxide using TBHP as an oxidant (Bezaatpour & Sheikh, 2016). Recently, olefins' epoxidation with H_2O_2 was catalyzed by VO^{4+} Schiff base complex entrapped in zeolite-Y (Modi et al., 2018). The reaction condition was optimized and it was revealed that under the

optimized condition VO^{4+} Schiff, base complex was found to be a potential catalyst with up to 90% conversion of olefin. Herein, we report on the vanadium-based catalyst i.e. $VO(acac)_2$ supported on MMT-K10 for epoxidation of cyclohexene under low temperature with tert-butyl hydroperoxide (TBHP) as the oxidant.

2 Method

2.1 Incorporation of Vanadium Complex into MMT-K10 Clay

The method for supporting MMT-K10 metal complex with $VO(acac)_2$ was done based on the previous method (Farias et al., 2011). Prior to the modification step, MMT-K10 was dried at 110°C. Then, 10.0 g of clay was immersed in a $VO(acac)_2$ solution in dry toluene (100 mL) and stirred under N_2 flow for 18 h at 25°C. The product was filtered and washed with a mixture of hot toluene and hot ethanol to remove the unreacted metal complex. Finally, the solid was placed in an oven of 110°C for 5 h. These procedures were repeated to produce K10-V with various concentrations of $VO(acac)_2$ solutions. $VO(acac)_2$ supported on MMT-K10 is designated as K10-V (1-4) with (1-4) indicating the concentrations of vanadium complex i.e. 0.05 (1), 0.1 (2), 0.15 (3) and 0.2 M (4).

2.2 Characterization of Supported MMT-K10 Clay

All catalysts diffractograms were recorded using Bruker AXS Germany D8 Advance, with CuK α radiation (40 kV, 40 mA, wavelength, = 1.5406 Å). The identification of a phase from the diffraction patterns of the powder was based on the position of the lines in terms of 2θ ranging from 2-80°. Atomic absorption spectroscopy (AAS) was utilized to assess the content of vanadium in solid catalysts. 20 ml of 1.0 M hydrochloric acid was mixed with 0.1 g of incorporated clay samples. The solution was stirred for 1 h and centrifuged. This step was repeated a few times until all the vanadium metal leached from MMT-K10. This can be indicated by the reoccurrence of the light grey colour of unmodified MMT-K10. A Micromeritic ASAP 2020 was utilized experimentally to obtain N_2 adsorption-desorption data at −196°C. The isotherm used was the BJH (Barret–Joyner–Halenda) adsorption-desorption branch to evaluate the pore diameters of the materials. The samples were degassed under vacuum at 110°C for 4 h prior to the adsorption experiments.

2.3 Catalytic Epoxidation of Cyclohexene

The epoxidation reaction was conducted in a 50 ml two-neck flask connected to a reflux condenser, and airtight rubber septum for N_2 flow. The temperature was maintained at 60-70°C using the rotamantle. Cyclohexene was chosen as substrate and TBHP as oxidant, in the 1:2 molar ratio of substrate:oxidant (Shen et al., 2017; Behera & Parida, 2013). A 50 mmol of TBHP was added to 25 ml toluene containing either raw MMT-K10 or incorporated MMT-K10 clays as heterogeneous catalysts, whereas 25 mmol cyclohexene was added to 25 ml of the same solvent. The mixture was stirred vigorously and the samples were collected every 30 minutes. The solid catalyst was filtered out from the reaction mixture. To remove the unreacted peroxide, the filtrate flask was cooled in an ice bath (approximate 0°C) and a small amount of $NaHSO_3$ solution with a concentration of 15% w/v was slowly added. The peroxide content was qualitatively observed using test strips (Quantofix

Peroxide 100). The final product was isolated and dried over anhydrous Na_2SO_4 (Farias et al., 2011). Finally, the samples were analyzed using gas chromatography.

2.4 Identification and Quantification of Reaction Products

The products from the epoxidation reaction were recorded using gas chromatography with a flame ionization detector (GC-FID). GC-FID condition was as follows: column DB-Wax (30 m x 0.25 mm x 0.5μm), interface temperature (230°C), the temperature of the detector (250°C), the temperature of column (150°C (hold for 1 min) to 245°C at 30°C/min (hold for 20 min)) and helium as the carrier gas. Gas chromatography-mass spectrometry (GC-MS) Shimadzu, QP5050A equipped with a Carbowax column was used to identify the products. The condition of column temperature was similar to GC-FID. The calculation of conversion was done based on the percentage molar of cyclohexene (Equation 1) (Jiang et al., 2009). The conversion (Equation (1)) and selectivity (Equation (2)) were calculated based on the results obtained from gas chromatography.

$$\text{Conversion of cyclohexene (\%)} = \frac{\text{initial mol} - \text{final mol}}{\text{final mol}} \times 100 \qquad (1).$$

$$\text{Selectivity of cyclohexene oxide (\%)} = \frac{\text{GC peak area of cyclohexane oxide}}{\text{GC peak area of all products}} \times 100 \qquad (2).$$

3 Result and Discussion

3.1 Characterization of Supported MMT-K10 Clay

Figure 1 shows the diffractogram for raw MMT-K10 and K10-V samples. The XRD patterns revealed that the raw MMT K10 and K10-V samples had crystalline nature observed from the peak intensities and sharpness (Muthuvel et al., 2012). From the diffractogram pattern of the raw MMT-K10, a layered structure with a basal spacing (d_{001}) was calculated to be ca. 9.95 Å.
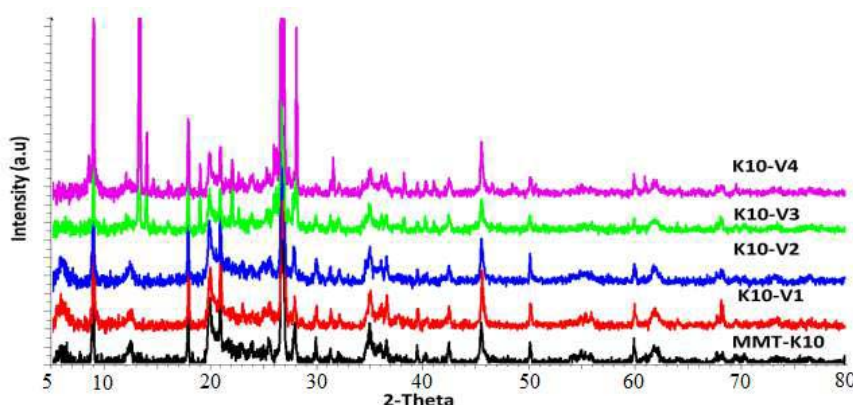

Figure 1: XRD diffractogram of raw and supported MMT-K10.

After modification of MMT-K10 with vanadium complex, the XRD profiles showed that the layered structure was unchanged and the d_{001} was calculated to be 9.89 Å. The similarities between the XRD patterns and d_{001} values of unmodified and modified MMT-K10 indicated that no change occurred in the lattice parameter during the complex supported on MMT-K10. The MMT-K10 structure was also found to remain unchanged after the immobilization with manganese salen

complex (Bahramian et al., 2006), cobalt salophen complex (Jiang et al., 2009), and molybdenum acetylacetonate complex (Farias et al., 2011; Harun et al., 2018).

Table 1 shows the vanadium content incorporated in MMT-K10 analysed by the AAS method. Generally, the amount of vanadium detected gradually increased as the concentration of the metal complex increased. The less amount of vanadium complex incorporated in the MMT-K10 structure could be due to the loosely bound ligands from the metal complex. It was reported that $VO(acac)_2$ mainly bonded to the silica surface by a hydrogen bond between the pseudo-π-system of the acetylacetonate ring and the silanols from the clay surface (Baltes et al., 1998).

Table 1: Concentration of vanadium (μg/g) in K10-V

Sample	Vanadium (μg/g)
MMT-K10	n.d.
K10-V1	378.29 ± 12.2
K10-V2	321.09 ± 20.1
K10-V3	1161.1 ± 40.6
K10-V4	1397.0 ± 43.5

The large surface areas measured from N_2 adsorption/desorption isotherms of raw and supported clay samples indicated that the size of the pores was in the mesopores range. The average pore diameter was found to be between 6.26 and 9.41 nm. The incorporation of vanadium complex on the MMT-K10 structure also determined the N_2 adsorption capability. The incorporation of MMT-K10 clay with vanadium complex reduced its surface area and pore volume significantly (Table 2). K10-V proved less N_2 uptake (lower BET surface area and pore volume) in comparison with raw MMT-K10, and further less N_2 uptake at higher concentrations of modified clay (from K10-V1 to K10-V4). This change can be attributed to the functionalization of MMT-K10 with $VO(acac)_2$ moieties (Wang et al., 2016). A significant reduction of the BET surface areas and the pore volumes of the supported K10-V samples was due to the presence of bulky vanadium complex form inside the pore during the introduction of this complex on the walls of MMT-K10 (Joseph et al., 2004).

Table 2: Surface properties of MMT-K10 and K10-V samples.

Catalyst	BET Surface Area (m^2/g)	BJH Pore Volume (cm^3/g)
MMT-K10	223.25	0.38
K10-V1	171.27	0.32
K10-V2	162.65	0.30
K10-V3	46.44	0.13
K10-V4	37.67	0.10

3.2 Catalytic Epoxidation of Cyclohexene

The catalytic activity of K10-V(1-4) catalysts was experimentally tested for epoxidation of cyclohexene with TBHP, in the 1:2 molar ratio of substrate:oxidant. The main products in the epoxidation of cyclohexene analyzed by GCMS were found to be cyclohexene oxide, 1,2-cyclohexane diol, 2-cyclohexenol, and 2-cyclohexenone, while the main by-products were cyclohexene 3-(tert-butyl) peroxide and 5-hexyn-3-ol (Figure 2).
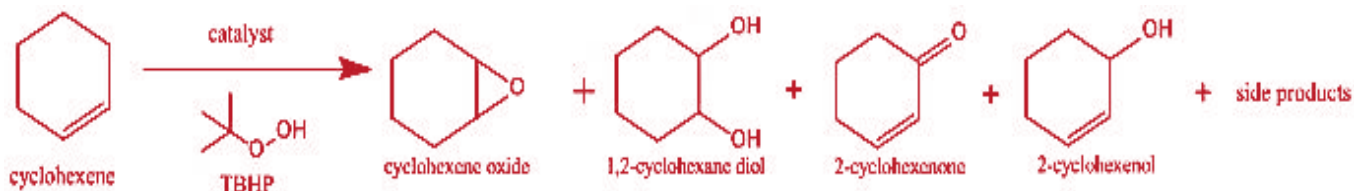
Figure 2: Chemical reaction of cyclohexene with TBHP with the presence of a catalyst.

The percentages of conversion and selectivity from epoxidation reaction of cyclohexene using raw and incorporated MMT-K10 catalysts with TBHP as oxidant were plotted in Figures 3 and 4, respectively. Among the examined catalyst, raw MMT-K10 showed a strikingly low catalytic activity with the maximum percentage of cyclohexene conversion of ca. 17% while none of it converted into cyclohexene oxide. Notably, all of the K10-V catalysts were active for the epoxidation of cyclohexene with the maximum conversion of ca. 20% and cyclohexene oxide selectivity of 70%. The activity of different K10-V catalysts decreased in the following order of K10-V2 (20%) > K10-V3 > K10-V1 > K10-V4 (14%). The selectivity of cyclohexene oxide showed a different decreasing trend: K10-V4 (70%) > K10-V1 (50%) > K10-V3 (46.5%) > K10-V2 (20%). At a high conversion of cyclohexene (20%), the selectivity of epoxide formation was about 20%.
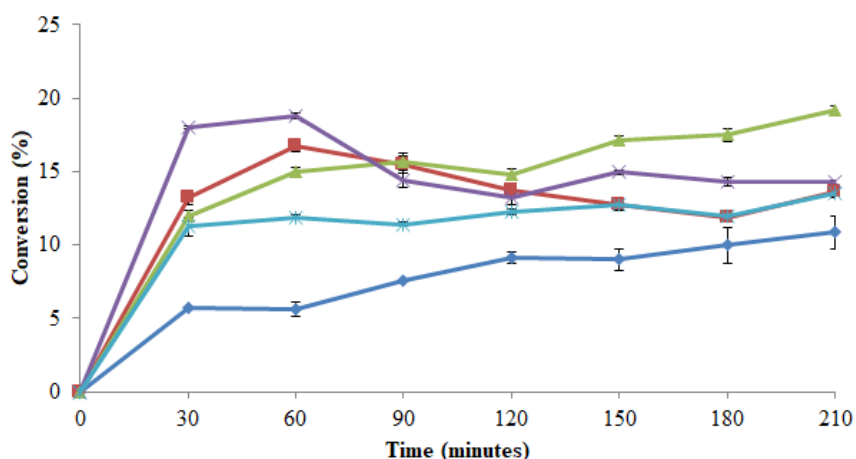

Figure 3: Conversion of cyclohexene using ♦: raw MMT-K10; ■: K10-V1; ▲: K10-V2; ×: K10-V3 and ж: K10-V4 as catalyst.
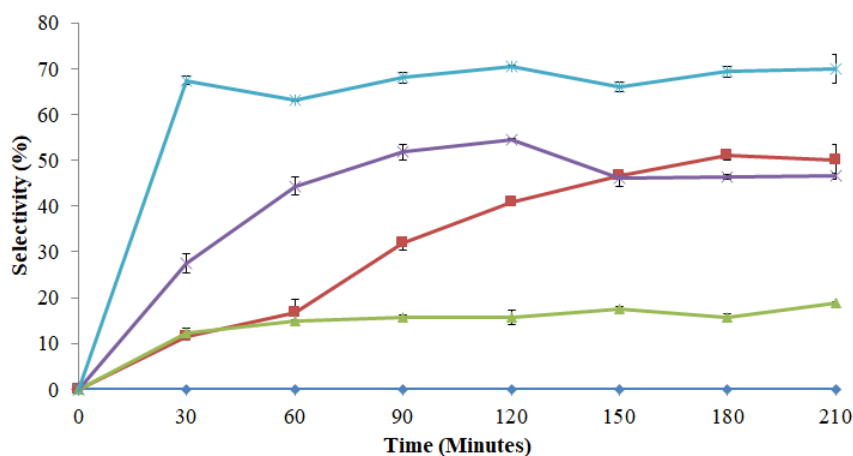

Figure 4: Selectivity of cyclohexene oxide using ♦: raw MMT-K10; ■: K10-V1; ▲: K10-V2; ×: K10-V3 and ж: K10-V4 as a catalyst.

The low catalytic activity could be due to the catalyst deactivation during the epoxidation process (Morales-delaRosa et al., 2015). As can be seen from Figure 3, the trend of catalytic activity for all tested catalysts was found to increase for the first 30–60 minutes of reaction time. However, the conversion of cyclohexene has remained constant even after 210 minutes of reaction time.

The presence of a metal complex was significant for epoxidation reactions of alkenes due to the Lewis acidity of the catalyst. Vanadium ion plays a major role to withdraw electrons from the tert-butylperoxo, t-BuOO$^-$ which increases the electrophilic nature of the peroxidic oxygens. This makes nucleophiles such as olefins easily attack the peroxidic oxygens to form epoxides (Gnecco et al., 2004). Thus, efficient catalysts should be strong Lewis acids and weak oxidants with a high oxidation state. A weak oxidant is required to reduce competing oxidation of an electron of the t-BuOO$^-$ ligand which could lead to hemolytic decomposition of t-BuOOH (Behera & Parida, 2013).

4 Conclusion

In this work, we demonstrated the synthesis and characterization of heterogeneous catalyst that has been prepared by incorporation of VO(acac)$_2$ onto the MMT-K10 clay matrix. The XRD, AAS, surface area, and pore volume data show that the vanadyl complex is physically supported throughout the matrix without changing the crystallinity of the MMT-K10 structure. Catalytic tests showed that the increased VO(acac)$_2$ amount on MMT-K10 has improved the selectivity of cyclohexene oxide to 70% with TBHP as the oxygen source.

5 Availability of Data And Material

Data can be made available by contacting the corresponding author.

5 Acknowledgement

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