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Research Paper

Synthesis, Characterization, and Catalytic Performance of $H_3[\alpha\text{-}PMo_{12}O_{40}]$ $.nH_2O{-}TiO_2$ in Cyclohexane Oxidation

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Abstract

This study aims to synthesize and characterize the $H_3[\alpha\text{-}PMo_{12}O_{40}].nH_2O{-}TiO_2$ composite as a heterogeneous catalyst for cyclohexane oxidation using H_2O_2. FT-IR and XRD characterization showed the interaction between POM and TiO_2, with an increase in TiO_2 ratio leading to a decrease in POM crystallinity. Reaction optimization showed that the POM-1.00TiO_2 catalyst gave the highest conversion of 99.95% at 80°C, 2 hours reaction time, 3 mL H_2O_2 volume, and 0.1 g catalyst mass. GC analysis confirmed the formation of cyclohexanol and cyclohexanone, with the optimized conditions resulting in a balanced selectivity of 35.51% for cyclohexanol and 34.69% for cyclohexanone, demonstrating the catalyst's effectiveness in cyclohexane oxidation.

Keywords

$H_3[\alpha\text{-}PMo_{12}O_{40}].nH_2O{-}TiO_2$, TiO2, Cyclohexane, Cyclohexanol, Cyclohexanone

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1. INTRODUCTION

Oxidation reactions are one of the chemical transformations that are widely used in the synthesis of organic compounds (Guangbo et al., 2025; Kar and Milstein, 2022), both in the laboratory and on an industrial scale. To increase the efficiency and selectivity of the reaction, catalysts are often used to lower the activation energy and accelerate the reaction rate (Isahak and Al-Amiery, 2024). In its development, research on catalysts is increasing, especially in finding alternatives that are more stable, environmentally friendly, and reusable. In general, catalysts can be categorized into biocatalyst, homogeneous, and heterogeneous catalysts (Wang et al., 2023; Ward and Copéret, 2023), with heterogeneous catalysts being more widely applied in industry due to their ease of product separation and stability under various reaction conditions (Lang and Li, 2023).

One group of heterogeneous catalysts that has attracted attention is polyoxometalate (POM), which is a metal-oxygen group with catalytic properties in acid-base and redox reactions (Evtushok et al., 2022; Hanif et al., 2019; Hanifah and Amri, 2023; Jiang et al., 2025; Palapa et al., 2021). POM has been widely applied in oxidation reactions due to its stability and ability to increase reaction efficiency (Zhong et al., 2021). However, POM has the disadvantage of difficulty separating from the solution, which hinders their reuse and low selectivity (Samaraj et al., 2021). Modifications can be made through various methods to improve the performance of POM, such as doping (Mirante et al., 2022; Sakthinathan et al., 2023; Zhi et al., 2023), encapsulating (Gao et al., 2023), and impregnation (Tubul-Sterin et al., 2023). The modified POM can be utilized in various applications, including important oxidation reactions in the chemical industry, such as the conversion of cyclohexane to cyclohexanol and cyclohexanone, which are key precursors in the production of nylon-6 and nylon-6,6 (Alnefaie et al., 2022; Huo et al., 2024; Niakan et al., 2024).

The conversion of cyclohexane using POM-based catalysts has been studied using various approaches, including the use of Keggin-type POM (Mouanni et al., 2019), POM supported by other materials (Trakarnpruk and Jatupisarnpong, 2013), and POM with based-metal material (Sativa and Lesbani, 2017). One of the supporting materials that are widely used is titanium oxide (TiO_2) because it has stable, heat-resistant, and non-toxic properties (Chandoliya et al., 2024; Hsu et al., 2024; Idris et al., 2022). The addition of TiO_2 to POM aims to increase the active surface area of the catalyst to increase the efficiency of the oxidation reaction.

In this study, the synthesis and characterization of $H_3[\alpha\text{-}PMo_{12}O_{40}].nH_2O$ supported with TiO_2 as a heterogeneous catalyst for the oxidation of cyclohexane with hydrogen peroxide (H_2O_2) as an environmentally friendly oxidant were carried out. Catalyst characterization was conducted using fourier transform infrared spectrophotometer (FT-IR) and X-ray diffractometer

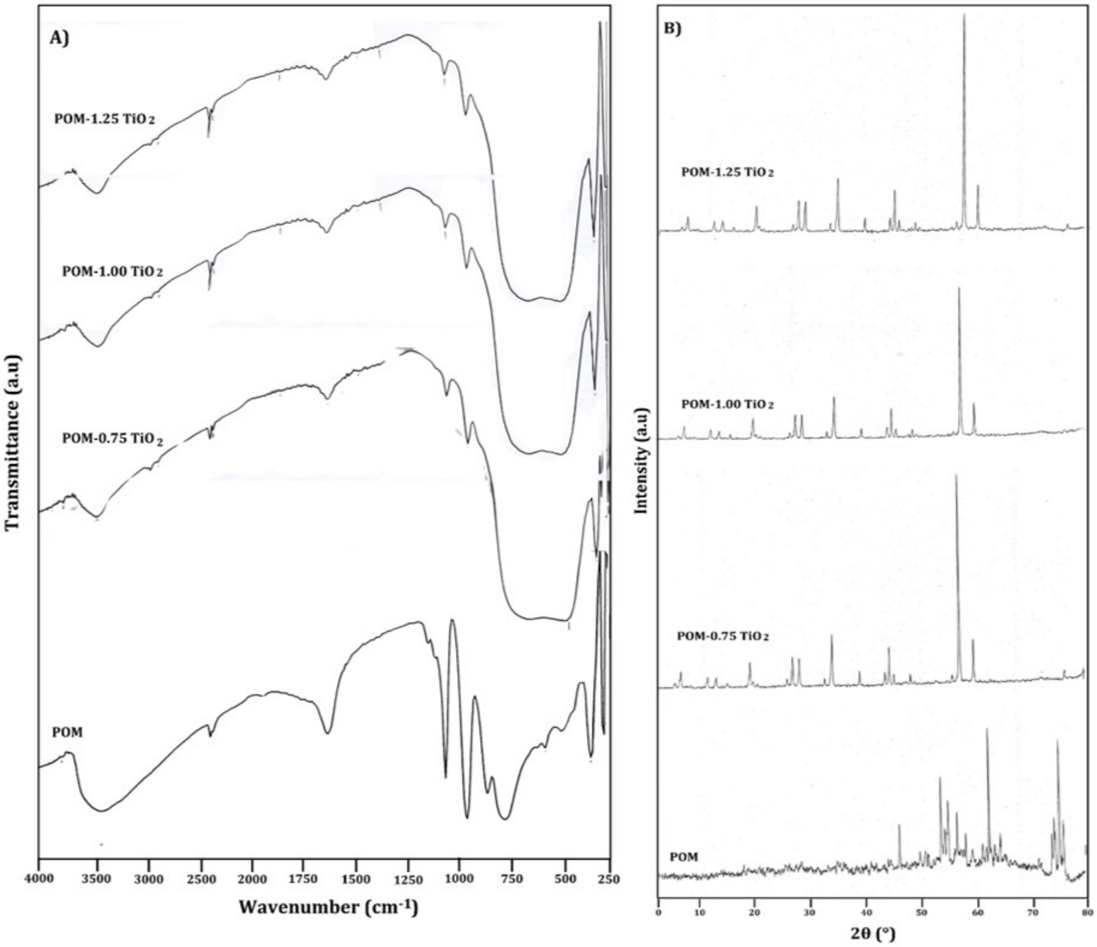

Figure 1. Spectra FT-IR and Diffractogram of POM, POM-0.75TiO_2, POM-1.00TiO_2, and POM-1.25TiO_2

(XRD) to evaluate its structure and catalytic properties, while reaction product analysis was conducted using gas chromatography (GC). This research is expected to produce catalysts with high activity and better selectivity towards cyclohexanol and cyclohexanone.

2. EXPERIMENTAL SECTION

2.1 Materials and Instrumentals

All materials used in this study were sourced from Merck with pro-analyst quality, including sodium molybdate ($Na_2MoO_4.2H_2O$), phosphoric acid (H_3PO_4), perchloric acid ($HClO_4$), diethyl ether ($(C_2H_5)_2O$), water (H_2O), hydrochloric acid (HCl), titanium oxide (TiO_2), ethanol (C_2H_6O), acetone (C_3H_6O), cyclohexane (C_6H_{12}), naphthalene ($C_{10}H_8$), and hydrogen peroxide (H_2O_2).

Characterization instruments used in this study include the Shimadzu Prestige-21 FT-IR spectrophotometer, Shimadzu Lab Type 6000 XRD, and Shimadzu Type 2010 Plus GC.

2.2 Preparation of $H_3[\alpha\text{-}PMo_{12}O_{40}].nH_2O$

The compound $H_3[\alpha\text{-}PMo_{12}O_{40}].nH_2O$ was synthesized from α-$Na_2H(PMo_{12}O_{40})$-nH_2O via acid extraction with diethyl ether. The α-$Na_2H(PMo_{12}O_{40})$-nH_2O salt was obtained by mixing 420 mL of Na_2MoO_4 (2.85 M), 6.8 mL of H_3PO_4 (85%, 14.7 M), and 284 mL of $HClO_4$ (70%, 12 M), stirring to produce a yellow precipitate, then vacuum filtered. Next, 250 g of α-$Na_2H(PMo_{12}O_{40}).nH_2O$ salt was dissolved in 200 mL of water, 50 mL of 12 M HCl was added, and then extracted with 400 mL of diethyl ether using a separatory funnel. The lower fraction containing polyoxometalate was taken, dried by rotary evaporator, and characterized using FT-IR and XRD.

2.3 Preparation of $H_3[\alpha\text{-}PMo_{12}O_{40}].nH_2O-TiO_2$

A total of 0.01 g of TiO_2 was mixed in 10 mL of ethanol, then 1 g of $H_3[\alpha\text{-}PMo_{40}].nH_2O$ was added slowly while stirring at 60°C in a water bath. After cooling, the mixture was filtered, washed with acetone, and vacuum-dried for 24 hours, then stored in a desiccator. A similar procedure was carried out for TiO_2 variations of 0.75 g, 1 g, and 1.25 g (POM-0.75TiO_2, POM-1.00TiO_2, and POM-0.75TiO_2). The synthesized products were characterized and analyzed. The synthesized products were characterized using FT-IR and XRD.

2.4 Oxidation of Cyclohexane

The oxidation of cyclohexane to cyclohexanol and cyclohexanone was carried out using POM, POM-0.75TiO_2, POM-1.00TiO_2, and POM-0.75TiO_2 catalysts, with H_2O_2 as the oxidizing agent. The reaction took place in a 100 mL triple neck flask equipped with a magnetic stirrer. The reaction mixture consisted of 0.038 g (0.021 mmol) catalyst, 2 mL (18.5 mmol) cyclohexane, 3 mL (26.4 mmol) 30 wt% H_2O_2, and 0.0013 g naphthalene. The process took place at 80°C for 2 hours with constant stirring, then analyzed using GC.

Several reaction parameters were varied to optimize the conversion of cyclohexane. The effect of reaction time was studied with variations of 0.5; 1; 1.5; 2; and 2.5 hours to determine the optimum conditions. In addition, the amount of H_2O_2 as an oxidizing agent was varied by 2; 2.5; 3; and 3.5 mL to see its effect on reaction efficiency. Variations in reaction temperature were also carried out at 60°C, 70°C, 80°C, and 85°C to determine the optimal temperature for cyclohexane oxidation. In addition, the effect of catalyst weight on cyclohexane conversion was evaluated by using 0.01 g; 0.05 g; 0.038 g; and 0.1 g of catalyst. The catalytic process is calculated through % conversion, % yield, and % selectivity, which are determined based on GC chromatogram data with the following Equations (1-3).

$$\%\text{Conversion} = \left(\frac{\frac{A}{B} - \frac{A'}{B'}}{\frac{A}{B}} \right) \times 100 \tag{1}$$

Where,

A = Area of cyclohexane before reaction
B = Area of naphthalene before reaction
A' = Area of cyclohexane after reaction
B' = Area of naphthalene after reaction

$$\%\text{Yield} = \frac{\text{Area of Product}}{\text{Area of Product} + \text{Area of Substrate}} \times 100\% \tag{2}$$

$$\%\text{Selectivity} = \frac{\text{Yield}}{\text{Conversion}} \times 100\% \tag{3}$$

3. RESULTS AND DISCUSSION

3.1 Characterization of Materials

Based on the FT-IR spectra in Figure 1(A), typical absorption of POM compounds is seen at wave numbers around 786 cm^{-1} for Mo−Oc−Mo vibrations, 871 cm^{-1} for Mo−Oe−Mo, 964 cm-^{1} for Mo=O, and 1064 cm^{-1} for P−O (Novais et al., 2024; Pascual-Borràs et al., 2024). As the ratio of TiO_2 in the composite material increases (POM-0.75TiO_2 to POM-1.25TiO_2), the intensity of typical POM absorption decreases, especially for the Mo−Oc−Mo and Mo−Oe−Mo bands, while the typical TiO_2 absorption in the 524-671 cm^{-1} region is increasingly dominant (Shi et al., 2023). In samples with higher TiO_2 ratios, the typical POM absorption becomes increasingly difficult to identify, possibly due to the effect of coatings that cover the POM signal. In addition, the absorption at 1620 cm^{-1} indicates bending −OH bound to Mo in the POM structure, while the absorption at 3410 cm^{-1} is related to the −OH vibrations of crystalline water. This suggests that although POM is still present in the composite material, the presence of high amounts of TiO_2 can obscure its characteristic absorption in the FT-IR spectrum.

The XRD analysis in Figure 1(B) shows the XRD patterns of the POM compound $H_3[\alpha\text{-}PMo_{12}O_{40}].nH_2O$ before and after being supported with TiO_2. The pure POM diffractogram displays characteristic peaks at 2θ ranges between 7-12°, 18-22°, and 25-29° (Shi et al., 2023), with the strongest peaks at 7.8°, 20°, and 28.8°, indicating a Keggin structure with high crystallinity. After supporting with TiO_2, the diffraction pattern shows the dominance of typical TiO_2 peaks, especially at 2θ around 25.49°, 47.7°, and 22.0°, which indicates the presence of anatase TiO_2 phase. The disappearance or decrease in intensity of typical POM peaks demonstrates that these compounds have decreased crystallinity due to immobilization by TiO_2. The use of excessive amounts of TiO_2 is likely to cause the polyoxometalate compound to be covered by the TiO_2 matrix so that the diffraction signal from POM is no longer visible. The composite material produced from this immobilization is then utilized as a catalyst in the oxidation reaction of cyclohexane to cyclohexanol and cyclohexanone.

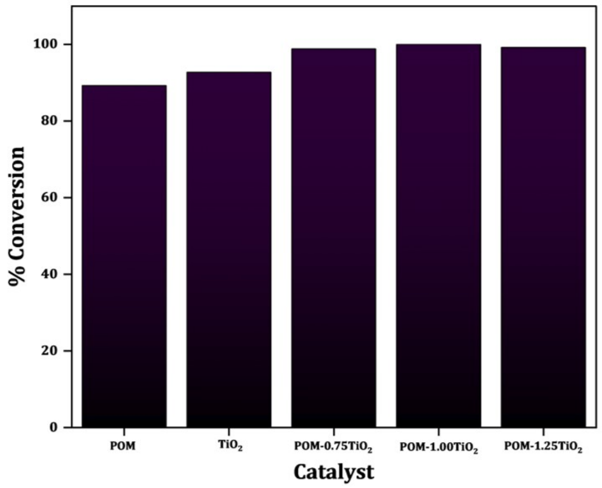

Figure 2. Effect of Different Catalysts of % Conversion Cyclohexane

3.2 Oxidation of Cyclohexane

3.2.1 Catalyst Optimization

Figure 2 shows the percentage conversion of cyclohexane using various POM-based catalysts $H_3[\alpha\text{-}PMo_{12}O_{40}].nH_2O$ bonded with TiO_2 after a 2 hour reaction with H_2O_2 as the oxidizing agent. From the graph, it can be seen that the POM-1.00TiO_2 catalyst has the highest conversion, reaching 99.95%, indicating

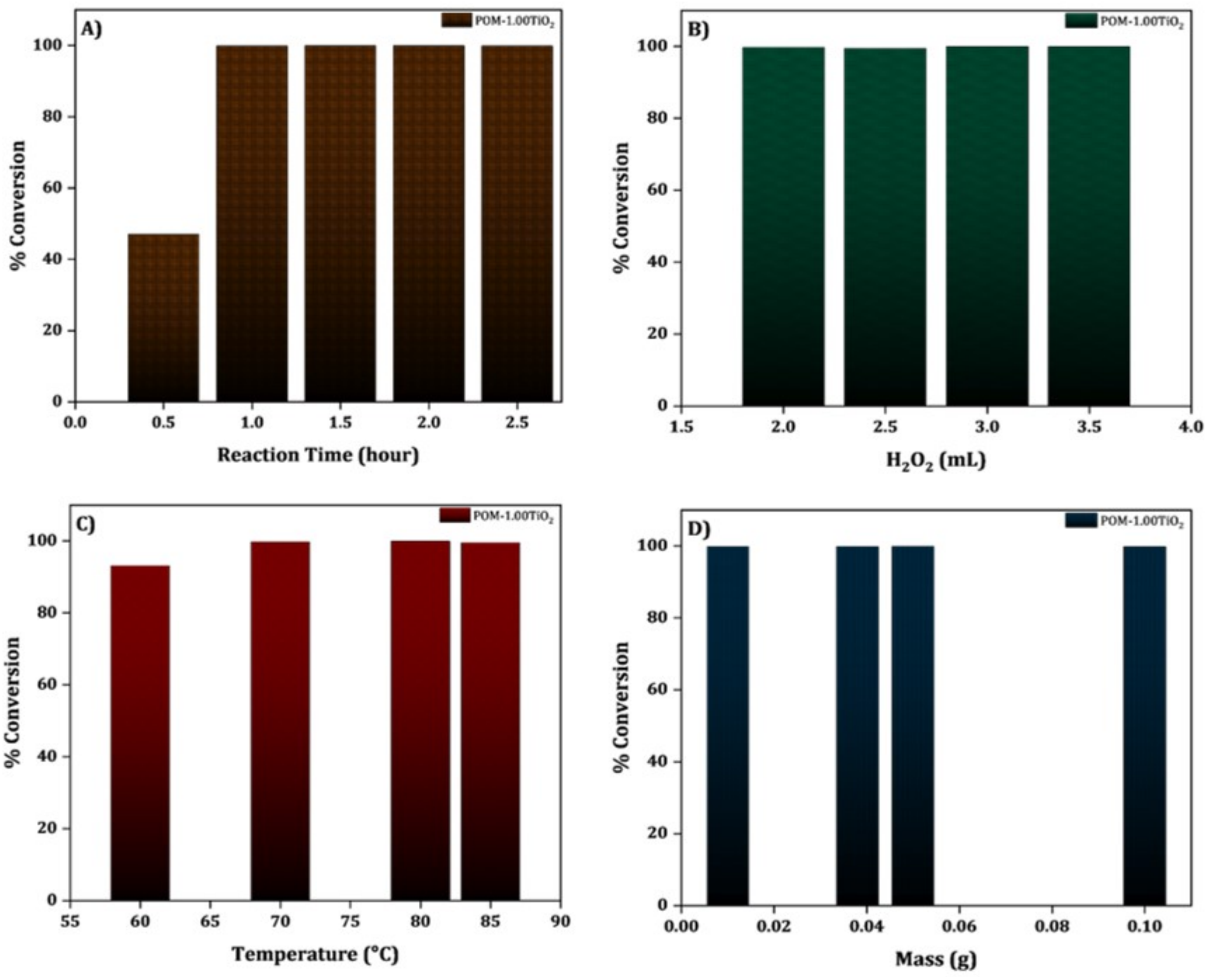

Figure 3. Effect of A) Reaction Time, B) Volume H_2O_2, C) Temperature, and Mass Catalyst POM-1.00TiO$_2$ of % Conversion Cyclohexane

that the combination of POM with TiO$_2$ can improve catalytic activity in the cyclohexane oxidation reaction. The catalyst with a ratio of 1:0.75 also showed a high conversion of 98.79%. The pattern of increasing conversion shows that the addition of TiO$_2$ up to a certain ratio increases the reaction efficiency, but at a higher ratio (1:1.25), there is a slight decrease in conversion to 99.17%, possibly due to the over-immobilization effect of TiO$_2$ which may block the access of reactants to the active site of POM. Compared to single catalysts, either POM or TiO$_2$ alone, the composite catalyst showed higher conversion, indicating that the combination of both materials creates synergy in improving catalytic performance, both through increased stability and changes in surface properties that favor more effective oxidation reactions (Liu et al., 2021).

3.2.2 Reaction Time Optimization

Based on Figure 3A and the data in Table 1, the cyclohexane conversion increases as the reaction time increases, with the optimum time at 2 hours. At a reaction time of 0.5 hour, the conversion is still very low, and the yield of cyclohexanone is only 0.01% with a selectivity of 0.02%. After 1 hour of reaction, the conversion increased significantly with cyclohexanol and cyclohexanone yields reaching 27.68% and 29.48%, respectively,

Table 1. Effect of Reaction Time to % Yield and % Selectivity of Oxidation Cyclohexane

Time Reaction (hour)	% Yield		% Selectivity	
	Cyclohexanol	Cyclohexanone	Cyclohexanol	Cyclohexanone
0	-	-	-	-
0.5	-	0.01	0.02	-
1	27.68	29.48	27.71	29.51
1.5	42.81	-	42.83	-
2	35.49	34.67	35.51	34.69
2.5	52.53	-	52.59	-

with almost the same selectivity. At 1.5 hours, cyclohexanol had the highest increase in yield and selectivity (42.81% and 42.83%), while cyclohexanone was not detected. At 2 hours, the yield and selectivity of cyclohexanol and cyclohexanone were almost balanced at around 35.49% and 34.67%, indicating optimum conditions. However, at 2.5 hours, the conversion was no longer effective, with cyclohexanol reaching 52.53% but with no contribution from cyclohexanone, indicating saturation of the catalyst's active site (Peng et al., 2020). This indicates that reactions longer than 2 hours do not provide significant improvements in conversion and selectivity.

Table 2. Effect of Volume H_2O_2 to % Yield and % Selectivity of Oxidation Cyclohexane

Volume H_2O_2 (mL)	% Yield		% Selectivity	
	Cyclohexanol	Cyclohexanone	Cyclohexanol	Cyclohexanone
2.0	33.76	33.57	33.84	33.65
2.5	20.67	14.86	20.79	14.95
3.0	35.49	34.67	35.51	34.69
3.5	28.37	62.73	28.40	62.80

3.2.3 Volume of H_2O_2 Optimization

Figure 3B and Table 2 show the effect of varying the amount of H_2O_2 on the conversion of cyclohexane and the distribution of reaction yields. At 2 mL of H_2O_2, the yield and selectivity of cyclohexanol and cyclohexanone were relatively balanced, about 33.76% and 33.57%, respectively. When the amount of H_2O_2 was increased to 2.5 mL, there was a significant decrease in the yield and selectivity of cyclohexanone, indicating that this amount of oxidant was insufficient for optimal conversion. Maximum conversion was achieved when 3 mL of H_2O_2 was used, resulting in a cyclohexanol yield of 35.51% and cyclohexanone of 34.69%. However, the addition of H_2O_2 up to 3.5 mL caused the conversion to decrease, but instead drastically increased the yield and selectivity of cyclohexanone to 62.73%. This indicates that excessive amounts of H_2O_2 tend to oxidize cyclohexanol to cyclohexanone further (Pan et al., 2021). Thus, the use of 3 mL of H_2O_2 is the optimal condition to achieve the desired balance of conversion and product distribution.

3.2.4 Temperature Optimization

Temperature variation in the cyclohexane oxidation reaction using $H_3[\alpha\text{-}PMo_{12}O_{40}].nH_2O-TiO_2$ catalyst has a significant effect on the conversion and product selectivity. Based on Figure 3C and Table 3, increasing the reaction temperature led to an increase in cyclohexane conversion, which reached the highest value of 99.95% at 80°C, with cyclohexanol and cyclohexanone selectivity of 35.51% and 34.69%, respectively. However, at 85°C, there is a decrease in conversion and selectivity due to excessive H_2O_2 decomposition, so the oxidation process does not take place optimally (Pan et al., 2021). Therefore, 80°C is considered the best condition for this reaction, as it provides a balance between high conversion and maximum product selectivity.

Table 3. Effect of Temperature to % Yield and % Selectivity of Oxidation Cyclohexane

Temperature (°C)	% Yield		% Selectivity	
	Cyclohexanol	Cyclohexanone	Cyclohexanol	Cyclohexanone
60	0.02	0.16	0.02	0.17
70	9.27	12.87	9.29	12.89
80	35.49	34.67	35.51	34.69
85	31.63	7.21	31.77	7.24

3.2.5 Mass Optimization

The effect of catalyst weight variation on the cyclohexane oxidation process using $H_3[\alpha\text{-}PMo_{12}O_{40}].nH_2O-TiO_2$ is shown in Figure 3D and Table 4. Increasing the amount of catalyst contributes to the increase of active surface area, thus increasing the possibility of collision between reactants and catalyst active sites (Mouanni et al., 2018). The results showed that a catalyst weight of 0.1 g resulted in the highest conversion, 99.83%, with selectivity to cyclohexanol and cyclohexanone of 36.47% and 49.63%, respectively. Although increasing the amount of catalyst generally increases the reaction efficiency, there is a certain limit where the excess catalyst is not always directly proportional to the increase in product, possibly due to diffusion factors and active site saturation.

Table 4. Effect of Mass Catalyst to % Yield and % Selectivity of Oxidation Cyclohexane

Mass Catalyst (g)	% Yield		% Selectivity	
	Cyclohexanol	Cyclohexanone	Cyclohexanol	Cyclohexanone
0.010	1.58	3.37	1.63	3.48
0.038	35.49	34.67	35.51	34.69
0.050	40.56	38.74	40.59	38.76
0.100	36.41	49.54	36.47	49.63

3.3 Product Identification

GC analysis (Figure 4) identified peaks for cyclohexanol at 5.11 minutes and cyclohexanone at 5.20 minutes, confirming their formation during the oxidation process. The presence of additional peaks in the chromatogram indicates incomplete conversion of cyclohexane, leading to the formation of byproducts. Notably, 2-pentanone was detected at 4.63 minutes, butanoic acid at 6.78 minutes, butane at 7.18 minutes, and cyclopentanone at 9.69 minutes. These byproducts suggest side reactions occurring during the oxidation process, potentially influenced by reaction conditions and catalyst properties.

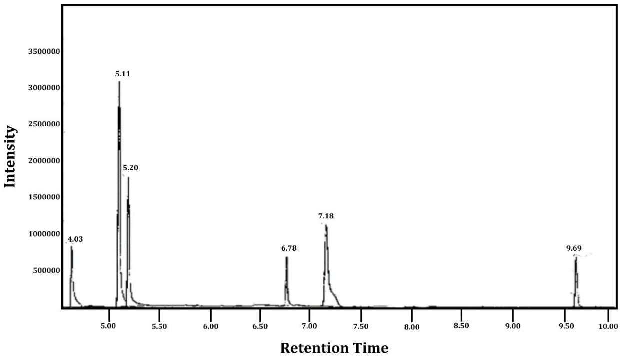

Figure 4. Chromatogram After Oxidation Process

4. CONCLUSIONS

Based on the FT-IR and XRD characterization results, the $H_3[\alpha\text{-}PMo_{12}O_{40}].nH_2O-TiO_2$ composite shows the interaction between POM and TiO_2, with an increase in the TiO_2 ratio causing a decrease in the crystallinity of POM. Optimization of the cyclohexane oxidation reaction showed that the POM-1.00TiO_2 catalyst gave the highest conversion of 99.95% at 80°C, 2 hours reaction time, 3 mL H_2O_2 volume, and 0.1 g catalyst mass. The combination of POM and TiO_2 increases the catalytic activity synergistically, but too high a ratio of TiO_2 can reduce reaction efficiency due to the closure of the POM active site. GC analysis further confirmed the presence of cyclohexanol and cyclohexanone as major oxidation products, indicating the catalyst's potential for effective cyclohexane oxidation.

5. ACKNOWLEDGEMENT

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