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Research Paper

Efficient Desulfurization of 4-Methyldibenzothiophene Using $H_5PV_2Mo_{10}O_{40}$ /TiO_2: A Catalytic Approach

Ana Anggraini[1], Jefri Jefri[2], Komis[2], Aldes Lesbani[2,3*]

[1] *Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Sriwijaya, Ogan Ilir, 30862, Indonesia*
[2] *Master Program of Material Science, Graduate School, Universitas Sriwijaya, Palembang, 30139, Indonesia*
[3] *Research Center of Inorganic Materials and Coordination Complexes, Universitas Sriwijaya, Palembang, 30139, Indonesia*
[*] **Corresponding author**: aldeslesbani@pps.unsri.ac.id

Abstract
Oxidation desulfurization (ODS) is an effective method to remove sulfur compounds from fuels by converting them into more polar oxidized forms that are easier to extract. In this study, $H_5PV_2Mo_{10}O_{40}/TiO_2$ catalysts were synthesized and characterized using FTIR spectroscopy and XRD. The FTIR spectra and Diffractogram XRD confirmed the successful impregnation of polyoxometalate into the TiO_2 matrix, indicated by the appearance of TiO_2 vibrations and diffractions. The performance of the $H_5PV_2Mo_{10}O_{40}/TiO_2$ catalyst in the desulfurization of 4-methyldibenzothiophene (4-MDBT) was studied based on variations in reaction time, catalyst mass, temperature, and amount of H_2O_2. The optimal conditions obtained were a reaction time of 2 hours, a catalyst mass of 0.05 g, a temperature of 60°C, and a volume of H_2O_2 of 5 mL, with a maximum sulfur conversion of 99.0%. These results indicate that $H_5PV_2Mo_{10}O_{40}/TiO_2$ has potential as an efficient ODS catalyst.

Keywords
Polyoxometalates, $H_5PV_2Mo_{10}O_{40}/TiO_2$, Desulfurization, 4-MDBT

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1. INTRODUCTION

Polyoxometalates are metal-oxygen cluster compounds that have very high acidity, even exceeding the acidity of sulfuric acid, and are non-toxic, so they are effectively used as catalysts in various chemical reactions (Artetxe et al., 2022; Breibeck et al., 2022; Hanifah and Amri, 2023; Helmi et al., 2022; Wang and Li, 2024)). Polyoxometalates can act as catalysts in acid-base reactions and oxidation-reduction reactions (Wang and Li, 2024). However, the main drawback of this compound is its high solubility in polar solvents or solvents containing oxygen atoms, such as ether and alcohol, while in nonpolar solvents, polyoxometalates are insoluble (Cui, 2023). The high solubility in polar solvents causes the surface area of polyoxometalates to be low, which may limit their catalytic activity (Shi et al., 2023b).

To overcome this limitation, polyoxometalates in a homogeneous phase need to be modified so that they can be used in a heterogeneous phase, allowing separation and reuse of the catalyst. Tang et al. (2020) reported that Fe-containing polyoxometalates (Fe-POMs) exhibited 10- and 33-times higher activity in persulfate activation compared to single photocatalysis and photoactivated persulfate, respectively, due to improved charge carrier separation and the presence of additional active sites. Similarly, Azmani et al. (2021) demonstrated that cobalt polyoxometalates (Co-POMs) function as water oxidation catalysts (WOCs), but their catalytic activity was significantly enhanced when incorporated into a structured framework, achieving a more stable performance and a lower overpotential compared to their unmodified counterparts. Beyond photocatalysis, Zhang and Fu (2024) investigated the role of polyoxometalates in lignin depolymerization and found that while pure POMs could facilitate oxidation reactions, their efficiency increased substantially when combined with ionic liquids or deep eutectic solvents (DES), leading to a more selective conversion of lignin into value-added compounds. Furthermore, Liu et al. (2023) reported that Anderson-type polyoxometalates could catalyze oxidative desulfurization, yet their efficiency was significantly enhanced when supported on TiO_2, achieving 100% sulfur removal in just 30 minutes under room temperature conditions. These studies collectively demonstrate that modifying polyoxometalates not only improves their catalytic efficiency but also broadens their applicability, making them far superior to their unmodified counterparts across various catalytic applications.

One of the main applications of modified polyoxometalate catalysts is in the desulfurization process. Desulfurization is the process of removing sulfur from petroleum to reduce the content of sulfur compounds such as benzothiophene, dibenzothiophene, 4-methyldibenzothiophene, and 4,6-dimethyldibenzothiophen, which can produce harmful emissions to the environment when used as fuel (Aribike et al., 2020; Gooneh-Farahani and Anbia, 2023; Saleh et al., 2018). Various methods have been developed for sulfur removal, including hydrodesulfurization Castro et al. (2024); Coutinho et al. (2024); Navarro Yerga et al. (2022), biodesulfurization Al-Khazaali and Ataei (2023); Gunam et al. (2021); Mamuad and Choi (2023), and oxidative desulfurization (ODS) (Ahmad et al., 2023b; Piscopo et al., 2020; Trisunaryanti et al., 2021; Wu et al., 2025). The ODS method has several advantages over other methods, as it can remove sulfur in the liquid phase and operates at moderate temperatures and pressures (Karimi and Golmohammadi, 2024). Oxidative desulfurization (ODS) has been extensively explored using various catalysts to enhance sulfur removal efficiency from fuels. For instance, a simple yet effective POM-based catalyst, $[C_{16}H_{33}N(CH_3)_3]_3PW_4O_{24}$, has been employed in the oxidation desulfurization of tire pyrolysis oil, successfully reducing sulfur content from 11,033 ppm to 961 ppm with an impressive desulfurization efficiency of 91.28% (Zhang et al., 2023). Similarly, polyoxometalates $H_3[a-PW_{12}O_{40}]$ and $H_4[a-SiW_{12}O_{40}]$ exhibited remarkable catalytic activity in the desulfurization of benzothiophene, achieving up to 99% conversion within 3 hours at 40°C (Lesbani et al., 2015). In addition, amphiphilic hybrid catalysts, modified with tetrabutylammonium bromide (TBAB), 1-butyl-3-methylimidazole bromide (BMIMBr), and octadecyl trimethyl ammonium bromide (ODAB), demonstrated exceptional efficiency in removing dibenzothiophene (DBT) while maintaining catalytic performance over multiple cycles (Zhao et al., 2023). Moreover, layered double hydroxide-modified polyoxometalate (ZnAl-PW) was synthesized and effectively applied for the oxidative desulfurization of dibenzothiophene, reaching a high conversion rate of 99.16%, thereby confirming its potential as a robust heterogeneous catalyst (Ahmad et al., 2022). Collectively, these findings underscore the continuous advancements in ODS catalysis, showcasing the potential of polyoxometalate-based and hybrid catalysts as promising solutions for efficient sulfur removal.

In this study, the synthesis of polyoxometalate type $H_5PV_2Mo_{10}O_{40}-nH_2O$ impregnated with TiO_2 was carried out, where TiO_2 was obtained through the hydrolysis of tetra isopropyl ortho titanate (TIOT). Characterization was performed using FT-IR spectroscopy to evaluate the structural changes and interactions between $H_5PV_2Mo_{10}O_{40}-nH_2O$ with TiO_2 after impregnation. Furthermore, the synthesized catalyst was tested in the desulfurization process of 4-methyldibenzot hiophene (4-MDBT) to assess its effectiveness in reducing sulfur content. The results of this study are expected to provide insight into the potential of polyoxometalate modification as a more efficient heterogeneous catalyst in fuel desulfurization applications.

2. EXPERIMENTAL SECTION

2.1 Chemicals and Instrumentals

The chemicals used in this research were sodium bis(2-ethylhexyl) sulfosuccinate ($C_{20}H_{38}NaO_7S$), tetra isopropyl ortho titanate ($Ti[OCH(CH_3)_2]_4$), acetonitrile (CH_3CN), sodium hydrogen phosphate (Na_2HPO_4), cyclohexane (C_6H_{12}), sodium molybdate dihydrate ($NaMoO_4{\cdot}2H_2O$), diethyl ether ($C_4H_{10}O$), sodium vanadate ($NaVO_3$), hydrogen peroxide (H_2O_2), all obtained from MERCK, and aquadest from Bratachem. The instruments used for characterization and measurement are the Shimadzu Prestige-21 FTIR Spectrophotometer, the Shimadzu-6000 X-Ray Diffractometer (XRD), and the Shimadzu 2010 Plus Gas Chromatograph.

2.2 Synthesis of $H_5PV_2Mo_{10}O_{40}-nH_2O$

The polyoxometalate $H_5PV_2Mo_{10}O_{40}-nH_2O$ was synthesized by dissolving 6.1 g of sodium vanadate in 25 mL of warm water (Solution A). A solution of 1.775 g sodium hydrogen phosphate in 25 mL of distilled water was mixed into Solution A, cooled, and then acidified with 1.25 mL sulfuric acid, forming a red solution (Solution B). Next, 30.25 g sodium molybdate dihydrate in 50 mL water was added to Solution B, followed by 21.25 mL sulfuric acid. The mixture was extracted with 20 mL diethyl ether and 20 mL sulfuric acid, and the red bottom layer was collected. The solution was evaporated using a rotary evaporator, recrystallized with water, and dried under a vacuum. The final product was characterized by FTIR and XRD.

2.3 Synthesis of $H_5PV_2Mo_{10}O_{40}/TiO_2$

$H_5PV_2Mo_{10}O_{40}/TiO_2$ was synthesized from 0.5 g of sodium bis(2-ethylhexyl) sulfosuccinate and dissolved with 1 mL of cyclohexane (solution A). The compound $H_5PV_2Mo_{10}O_{40}-nH_2O$ of 0.76 g was dissolved with a small amount of aquadest (solution B). Solution B was added to solution A while stirring for 1 hour. A total of 2 mL of tetra isopropyl ortho titanate (TIOT) was added drop by drop. Stirred with a stirrer and heated at 50°C until a solid was obtained. The light green solid formed was$H_5PV_2Mo_{10}O_{40}/TiO_2$ and dried at room temperature. The $H_5PV_2Mo_{10}O_{40}/TiO_2$ compound was characterized by FTIR and XRD.

2.4 Desulfurization of 4-Methyldibenzothiophene (4-MDBT)

The desulfurization process was carried out on the compound 4-methyldibenzothiophene (4-MDBT). A total of 0.05 g of 4-MDBT compound dissolved in 5 mL of n-hexane was put into the flask, and then 0.05 g of $H_5PV_2Mo_{10}O_{40}/TiO_2$ catalyst was added. Then, 5 mL of acetonitrile and 3 mL of 30% hydrogen peroxide were added to the solution. The solution was stirred with a magnetic stirrer and then heated at 50°C using a bath. The content of 4-MDBT and its oxidation products were monitored periodically using gas chromatography (GC) at reaction time intervals of 0 to 3 hours. The chromatograms obtained were made % conversion to determine how much organosulfur compounds were reduced. The desulfurization process of the 4-MDBT compound was also carried out by determining the optimum reaction time, optimum catalyst weight, optimum temperature, and optimum amount of H_2O_2 on the effect of the desulfurization reaction. The reaction time variation used was 1 hour, 2 hours, and 3 hours. The variation of catalyst weight used 0.05, 0.1, 0.2, and 0.3 g, in the variation of temperature at 40, 50, 60, and 70°C, and in the variation of H_2O_2 as much as 1, 3, 4, and 5 mL.

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3. RESULTS AND DISCUSSION

3.1 Characterization of Materials

3.1.1 FTIR

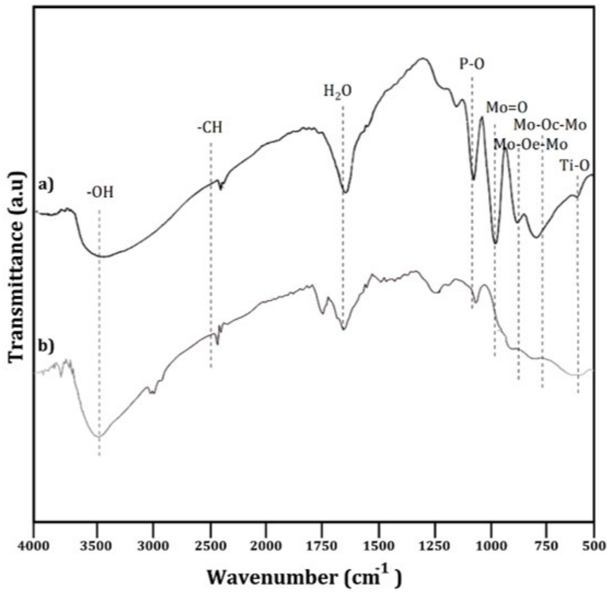

Figure 1. Spectra FTIR of a) $H_5PV_2Mo_{10}O_{40}-nH_2O$ and b) $H_5PV_2Mo_{10}O_{40}/TiO_2$

This FT-IR spectra image (Figure 1) shows a comparison between two materials, namely (a) $H_5PV_2Mo_{10}O_{40}-nH_2O$ and (b) $H_5PV_2Mo_{10}O_{40}/TiO_2$. In spectrum (a), the main peaks of polyoxometalate vibrations are seen at 1056.9 cm^{-1} for P-O vibrations, 964.4 cm^{-1} for Mo=O, 864.1 cm^{-1} for Mo−Oe−Mo, and 779.2 cm^{-1} for Mo−Oc−Mo, which correspond to the characteristics of $H_5PV_2Mo_{10}O_{40}-nH_2O$ compounds (Huo et al., 2021; Tiwari et al., 2020; Zhai et al., 2021). In addition, the peak around 3410.2 cm^{-1} shows the O−H vibration of adsorbed water, while the C−H vibration appears around 2337.2 cm^{-1}. The peak at 1625 cm^{-1} indicates the presence of H_2O bound in the compound structure.

After the impregnation process with TiO_2 (spectrum b), there are changes in several wave numbers, especially with the appearance of O−Ti−O vibrations around 1049.2 cm^{-1} and Ti−O vibrational peaks at around (Huo et al., 2021; Mallick et al., 2024; Shi et al., 2023a) 530.4 cm^{-1}, which indicates the success of the impregnation. The characteristic peaks of polyoxometalates are still visible, such as Mo=O at 964.4 cm^{-1} and Mo−Oe−Mo at 864.1 cm^{-1}, but there is a slight shift in Mo−Oc−Mo, which changes from 779.2 cm^{-1} to 786.9 cm^{-1}. These changes confirmed that the compound $H_5PV_2Mo_{10}O_{40}$ was successfully embedded in the TiO_2 matrix, which could improve the stability and catalytic performance of the material.

3.1.2 XRD

XRD analysis was performed to observe the crystalline structure of the polyoxometalate compound $H_5PV_2Mo_{10}O_{40}-nH_2O$ before and after impregnation on TiO_2. In Figure 2a, the XRD diffraction pattern shows major peaks at 2θ angles of about 8-10°, 21-29°, and 33-37°, which are typical of this compound (Zhai et al., 2021). The peaks with the highest intensity appear at 8°, 9°, and 28°, indicating high crystallinity. The presence of peaks at 2θ below 10° confirms that this compound has an ordered structure and dominant crystallinity.

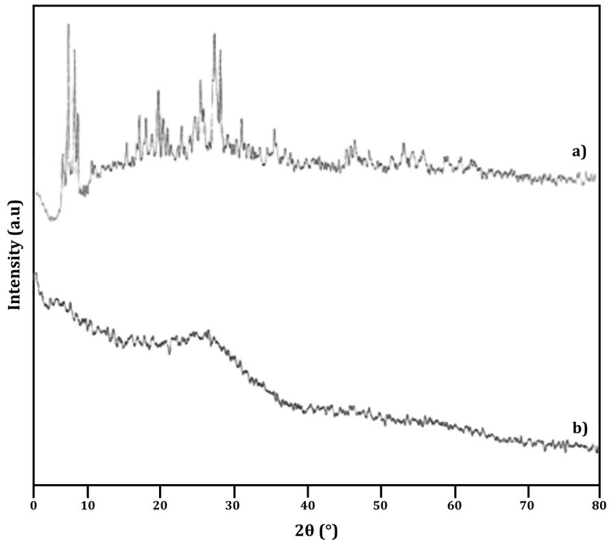

Figure 2. Diffractogram of a) $H_5PV_2Mo_{10}O_{40}-nH_2O$ and b) $H_5PV_2Mo_{10}O_{40}/TiO_2$

In Figure 2b, after the $H_5PV_2Mo_{10}O_{40}-nH_2O$ compound is impregnated on TiO_2, the XRD diffraction pattern shows a widening of the peaks and a decrease in intensity, which indicates that the crystallinity of the compound decreases and becomes more amorphous. This is likely due to the synthesis process in microemulsion media, which causes structural changes. In addition, the presence of a broadened TiO_2 diffraction peak at 2θ = 25.49° indicates the interaction between TiO_2 and $H_5PV_2Mo_{10}O_{40}$, which may affect its catalytic properties.

3.2 Desulfurization of 4-Methyldibenzothiophene (4-MDBT)

Oxidative desulfurization (ODS) is one of the effective methods in removing sulfur compounds from fuels, mainly through oxidation reactions that convert sulfur compounds into more polar forms so that they can be easily extracted (Figure 3). In this study, $H_5PV_2Mo_{10}O_{40}/TiO_2$ was used to improve the oxidation efficiency of 4-MDBT. To analyze the effect of various operational parameters on the reaction efficiency, the experimental data were visualized in the form of bar charts (Figure 3a-d), which show the conversion rate of 4-MDBT based on variations in reaction time, catalyst mass, temperature, and total H_2O_2.

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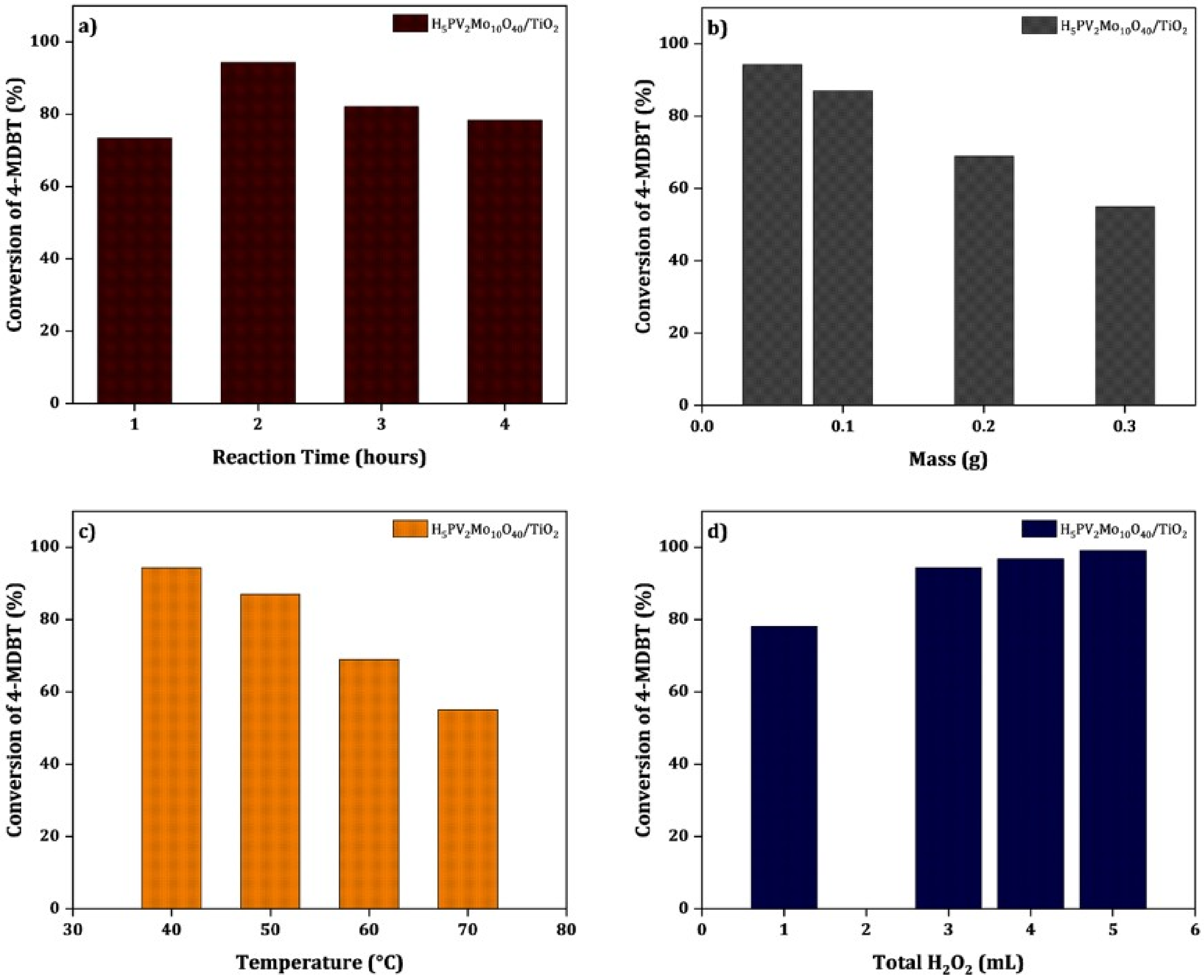

Figure 3. Desulfurization of 4-MDBT Using $H_5PV_2Mo_{10}O_{40}/TiO_2$ Catalyst with Variation a) Reaction Time, b) Mass of Catalyst, c) Temperature, and d) Total H_2O_2

The first parameter observed was the reaction time, which directly affects the conversion rate of 4-MDBT (Figure 3b). The results showed that the conversion increased from 73.4% at 1 hour to 94.3% at 2 hours, indicating that there was an increase in the number of interactions between the 4-MDBT molecules and the active oxidative species formed from H_2O_2 with the help of the catalyst (Ahmad et al., 2023a). However, after 2 hours, the conversion decreased to 82.0% at 3 hours and 78.3% at 4 hours. This decrease is likely due to the decomposition of H_2O_2 due to prolonged heating, which generates excess amounts of hydroxyl radicals ($\cdot$OH) that can cause catalyst degradation or secondary oxidation products that inhibit further conversion.

Catalyst mass also plays an important role in the effectiveness of the desulfurization reaction (Figure 3c). The results show that the conversion increases with the addition of catalyst until it peaks at 0.05 g catalyst weight with 94.3% conversion. However, the further addition of catalyst led to a decrease in efficiency, with the conversion dropping to 55.0% at 0.3g. This can be explained by the aggregation effect of the catalyst at higher concentrations, leading to reduced active surface area available for reaction (Amri et al., 2024). In addition, too high a catalyst concentration can lead to excessive adsorption of reactant molecules on its surface, which hinders the diffusion of the oxidizer to the active sites and reduces the effectiveness of the reaction (Xu et al., 2024).

Furthermore, the reaction temperature has a significant effect on the conversion of 4-MDBT (Figure 3d), because higher temperatures generally increase the kinetic energy of molecules and accelerate the reaction. The experimental results showed that the conversion increased from 90.6% at 40°C to a maximum of 99.0% at 60°C, indicating that increasing the temperature increases the catalyst activity and oxidation rate. However, at 70°C,

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Table 1. Comparison of Dibenzothiophene Desulfurization with Another Study

Catalyst	Time	Catalyst Dosage (g)	Temperature (°C)	Total H_2O_2 (mL)	% Conversion	References
ZnO@Ni-Al	30 min	0.25	50	2.5	99	(Ahmad et al., 2023a)
ZnO	120 min	0.1	60	0.1	71.0	(Trisunaryanti et al., 2021)
Activated Carbon	120 min	0.1	60	0.1	91.2	(Trisunaryanti et al., 2021)
ZnO-AC	120 min	0.1	60	0.1	93.8	(Trisunaryanti et al., 2021)
$(NH_4)_6Mo_7O_{24}\cdot 4H_2O$	3 h	-	50	-	89.2	(Shao et al., 2014)
MgAl-LDH	40 min	0.1	50	-	95.4	(Amri et al., 2024)
$H_5PV_2Mo_{10}O_{40}/TiO_2$	2 h	0.05	60	5	99.0	This Study

there was a significant decrease in conversion to 71.4%. This decrease is likely due to the thermal decomposition of H_2O_2 at high temperatures, which reduces the number of active species that play a role in oxidation (Wu et al., 2025). In addition, high temperatures can also cause structural changes in the catalyst that impact its stability and effectiveness in the desulfurization process (Zhao et al., 2023).

The last parameter observed was the amount of H_2O_2 as an oxidizer in the reaction (Figure 3d). Increasing the amount of H_2O_2 from 1 mL to 5 mL showed an increase in conversion from around 80% to a maximum of 99.0%, indicating that a sufficient amount of oxidizer is essential in driving the oxidation reaction optimally (Xu et al., 2024). However, if the amount of H_2O_2 exceeds a certain limit, there may be excessive degradation of oxidation products or even the formation of excess radical species that can cause side reactions that hinder the desulfurization process (Xu et al., 2024).

Based on these results, it can be concluded that the optimal parameters for desulfurization of 4-MDBT using $H_5PV_2Mo_{10}O_{40}/TiO_2$ catalyst are reaction time of 2 hours, catalyst weight of 0.05 g, temperature of 60°C, and amount of H_2O_2 of 5 mL. These results indicate the effectiveness of the catalyst in increasing the desulfurization efficiency through an oxidative mechanism controlled by these parameters. Further research is needed to understand the interaction between these parameters as well as to evaluate the long-term stability of the catalyst. A comparison of desulfurization dibenzothiophene against other studies is shown in Table 1.

4. CONCLUSIONS

$H_5PV_2Mo_{10}O_{40}-nH_2O$ compound impregnated with tetra isopropyl ortho titanate has been successfully synthesized, as evidenced through FTIR and XRD characterization showing typical vibrations and diffractions of polyoxometalate and titanium dioxide groups. The $H_5PV_2Mo_{10}O_{40}/TiO_2$ catalyst showed high efficiency in the desulfurization process of 4-MDBT, with conversion reaching 99% under optimal conditions, namely reaction time of 2 hours, catalyst usage of 0.05 g, maximum temperature of 60°C, and H_2O_2 volume of 5 mL. The successful synthesis and effectiveness of this catalyst show its potential in more efficient and environmentally friendly desulfurization applications.

5. ACKNOWLEDGEMENT

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