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SYNTHESIS AND CHARACTERIZATION OF V/SIO$_2$ CATALYSTS FOR CATALYTIC OXIDATIVE DESULFURIZATION OF DIBENZOTHIOPHENE USING HYDROGEN PEROXIDE

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ABSTRACT

In the present study, vanadium-supported silica (V/SiO$_2$) catalysts with different nominal vanadium loadings (1, 3, and 5 wt%) were prepared by a two-step procedure involving sol–gel synthesis of the silica support followed by impregnation with ammonium metavanadate (NH_4VO_3). The prepared materials were characterized by Fourier-transform infrared (FT-IR) spectroscopy and N_2 adsorption–desorption analysis. The FT-IR spectrum of V/SiO$_2$ exhibited characteristic absorption bands at approximately 1095 and 798 cm^{-1}, corresponding to vibrational modes of the Si–O–Si framework, together with a band at approximately 948 cm^{-1} associated with vanadyl/vanadium oxide species on the silica surface. The bare SiO$_2$ exhibited a BET surface area of 340 m^2 g^{-1}, a total pore volume of 0.51 cm^3 g^{-1}, and an average pore diameter of 6.8 nm. With increasing vanadium loading, the BET surface area decreased to 328, 230, and 226 m^2 g^{-1} for the 1, 3, and 5 wt% V–SiO$_2$ catalysts, respectively, while the pore volumes decreased to 0.49, 0.43, and 0.42 cm^3 g^{-1}. The corresponding average pore diameters were 6.3, 5.8, and 5.8 nm, respectively. The catalytic performance of the prepared catalysts was evaluated for the oxidative removal of dibenzothiophene (DBT) from a model fuel using H_2O_2 as the oxidizing agent. Among the investigated catalysts, 3 wt% V–SiO$_2$ exhibited the highest catalytic activity. Under the optimum conditions of 60 °C, 60 min, 0.2 g catalyst, and an H_2O_2/DBT molar ratio of 6, a maximum DBT removal of 94.8% was achieved. The optimum catalyst retained 79.4% of its initial activity after four consecutive reaction cycles, indicating good stability and reusability. These results demonstrate the potential of V/SiO$_2$ as an effective heterogeneous catalyst for the oxidative removal of refractory sulfur compounds from model fuels under relatively mild conditions.

KEYWORDS: Oxidative desulfurization; Silicate catalysts; Vanadium; Sulfur compounds; Deep desulfurization.

1. INTRODUCTION

Oil remains one of the world's major energy sources and holds particular importance owing to its high energy density, ease of transport and storage, and versatility. According to the 2025 Statistical Review, oil accounted for nearly 34% of global energy demand in 2024, making it the largest single source of energy worldwide.[1] Beyond its role as an energy source, petroleum is also a major feedstock for the chemical and petrochemical industries, providing hydrocarbons to produce a wide range of materials and consumer products.[2, 3]

Crude oil is a complex mixture of hydrocarbon and non-hydrocarbon compounds whose composition, and physicochemical properties vary considerably depending on its geological origin. Generally, crude oil cannot be used directly for most applications and must undergo a series of refining and upgrading processes to separate its components, convert heavier and less valuable fractions into more valuable products, and remove undesirable components. Petroleum refining involves a range of separation, conversion, and treatment operations through which crude oil is transformed into fuels such as gasoline, diesel, and jet fuel, as well as feedstocks for the petrochemical industry.[4, 5] The increasing demand for

high-quality fuels and increasingly stringent environmental and fuel-quality requirements has driven the development of more efficient refining and upgrading technologies.[6]

Among the undesirable components present in crude oil and petroleum-derived fuels, sulfur-containing compounds are of particular concern. Sulfur occurs in petroleum in a wide variety of chemical forms, including hydrogen sulfide, mercaptans, sulfides, disulfides, thiophenic compounds, and their alkylated derivatives. The concentration and distribution of these compounds vary considerably depending on the origin and composition of the crude oil. In addition to affecting fuel quality, sulfur compounds can contribute to serious operational problems during petroleum processing, particularly corrosion of pipelines, reactors, and other refinery equipment, as well as deactivation or poisoning of catalysts used in refining processes.[7–9]

The presence of sulfur-containing compounds in petroleum and petroleum-derived fuels has necessitated the development of efficient desulfurization processes to produce fuels that comply with increasingly stringent sulfur specifications and to minimize their adverse effects on refinery operations and the environment. Hydrodesulfurization (HDS) remains the predominant industrial technology for removing sulfur from petroleum fractions. In this process, sulfur-containing compounds react with hydrogen over heterogeneous catalysts, resulting in the cleavage of C–S bonds and the conversion of organic sulfur mainly into hydrogen sulfide (H_2S), which is subsequently removed from the product stream.[10, 11] HDS is highly effective for the removal of many relatively reactive sulfur compounds and has therefore become an integral part of conventional petroleum refining. However, achieving ultra-deep desulfurization by HDS becomes increasingly difficult because of the severe operating conditions required, including elevated temperatures and hydrogen pressures, together with considerable hydrogen consumption and associated energy and operating costs.[11, 12] Moreover, refractory aromatic sulfur compounds, particularly dibenzothiophene (DBT) and its alkylated derivatives such as 4,6-dimethyldibenzothiophene (4,6-DMDBT), exhibit high resistance toward conventional HDS because of their chemical stability and, in the case of alkyl-substituted derivatives, steric hindrance around the sulfur atom.[11, 13] These limitations have stimulated increasing interest in alternative or complementary desulfurization technologies capable of operating under milder conditions. Among these approaches, oxidative desulfurization (ODS) has emerged as a promising alternative, particularly for the removal of refractory sulfur compounds. In ODS, organosulfur compounds are selectively oxidized to more polar sulfoxides and sulfones using an appropriate oxidant, generally in the presence of a catalyst, after which the oxidized products can be separated from the nonpolar fuel phase by extraction or adsorption.[12, 13] The possibility of achieving effective sulfur removal under relatively mild temperatures and pressures, without the direct use of hydrogen, has made ODS an attractive approach for deep and ultra-deep desulfurization of petroleum-derived fuels.

In view of the aforementioned considerations, the present study aims to develop a silica-supported vanadium oxide (V/SiO_2) catalyst and investigate its potential for the oxidative removal of refractory sulfur compounds from petroleum-derived model fuel. Dibenzothiophene (DBT) was selected as a representative sulfur-containing compound, and H_2O_2 was employed as the oxidizing agent. The catalytic performance of the prepared material was evaluated by monitoring DBT removal under different reaction conditions, including reaction time, temperature, catalyst dosage, and H_2O_2/DBT molar ratio. Overall, this study assesses the potential of the prepared V/SiO_2 material as an effective heterogeneous catalyst for oxidative desulfurization under relatively mild reaction conditions.

1. Experimental Section

1-1- Materials and regents

Tetraethyl orthosilicate (TEOS) was used as the silica precursor for the preparation of the SiO_2 support. Ammonium metavanadate (NH_4VO_3) was employed as the vanadium precursor for the preparation of the V/SiO_2 catalysts, while oxalic acid was used, where required, to facilitate the preparation of the vanadium impregnation solution. Ethanol, hydrochloric acid (HCl), ammonium hydroxide (NH_4OH), and deionized water were used during the sol–gel preparation of the silica support. Dibenzothiophene (DBT) was selected as the model sulfur-containing compound, and n-decane was used as the model fuel solvent. Hydrogen peroxide (H_2O_2) was used as the oxidizing agent in the oxidative desulfurization experiments. All chemicals were of analytical grade and were used as received without further purification.

1-2- Preparation of V/SiO_2 Catalyst

Silica-supported vanadium oxide catalysts (V/SiO_2) were prepared by a two-step procedure involving the synthesis of the silica support by the sol–gel method followed by impregnation with a vanadium precursor.[14–17] Three catalysts with nominal vanadium loadings of 1, 3, and 5 wt% were prepared to investigate the effect of vanadium loading on the catalytic performance.

Step 1: Preparation of the SiO_2 Support

The silica support was synthesized by the sol–gel method using tetraethyl orthosilicate (TEOS) as the silica precursor. Briefly, TEOS was mixed with ethanol and deionized water at an approximate molar ratio of TEOS:EtOH:H_2O = 1:4:4 under continuous magnetic stirring. The hydrolysis of TEOS was initiated under acidic conditions by adjusting the pH of the reaction mixture to approximately 2 using dilute HCl. After

sufficient hydrolysis, the pH of the mixture was gradually increased to approximately 7–8 using dilute NH_4OH to promote condensation and gel formation. The resulting silica gel was aged at room temperature, separated by filtration, and washed repeatedly with deionized water to remove residual soluble species. The obtained material was then dried at 100–120 °C and calcined in air at approximately 500–600 °C for several hours to obtain the silica support.

Step 2: Preparation of V/SiO_2 Catalysts

Vanadium species were subsequently introduced onto the calcined silica support using the incipient wetness impregnation method, with ammonium metavanadate (NH_4VO_3) selected as the vanadium precursor. The amount of NH_4VO_3 required for each catalyst was calculated according to the desired nominal vanadium loading. For the preparation of 10 g of SiO_2 support, the amounts of NH_4VO_3 required to obtain nominal vanadium loadings of 1, 3, and 5 wt% were 0.23, 0.69, and 1.15 g, respectively. The precursor amounts were adjusted according to the actual mass of silica used in each preparation.

Prior to impregnation, the silica support was pretreated by heating at approximately 120 °C to remove physically adsorbed moisture. An impregnation solution containing the required amount of NH_4VO_3 was then prepared. Oxalic acid was added to the precursor solution to facilitate dissolution and stabilization of the vanadium precursor, following previously reported procedures for the preparation of silica-supported vanadium oxide catalysts. The total volume of the impregnation solution was adjusted according to the pore volume of the calcined silica support, as determined by N_2 adsorption–desorption analysis, in order to achieve incipient wetness conditions.

1-3- Catalytic Oxidative Desulfurization

The catalytic performance of the prepared V/SiO_2 catalysts was evaluated in the oxidative desulfurization (ODS) of a model oil containing dibenzothiophene (DBT) as a representative refractory sulfur compound. The model fuel was prepared by dissolving a known amount of DBT in n-decane to obtain a defined initial DBT concentration. Hydrogen peroxide (H_2O_2) was used as the oxidizing agent. In a typical experiment, a predetermined amount of the model fuel was mixed with the required amount of H_2O_2 and V/SiO_2 catalyst under continuous stirring at a controlled temperature. To simplify the experimental investigation, the effects of reaction time, reaction temperature, catalyst dosage, and H_2O_2/DBT molar ratio were studied systematically while keeping the remaining experimental conditions constant.

At predetermined reaction times, aliquots of the reaction mixture were withdrawn and separated from the solid catalyst by filtration or centrifugation. The residual concentration of DBT was then determined by UV–Vis spectrophotometry using a previously established calibration curve. The percentage of DBT removal was calculated from the initial and residual DBT concentrations according to the following equation.

$$DBT\ removal(\%) = \frac{C_0 - C_t}{C_0} \times 100$$

Where C_0 and C_t represent the initial DBT concentration and the DBT concentration after reaction time, respectively. The oxidation of DBT proceeded through the formation of dibenzothiophene sulfoxide (DBT–SO) followed by further oxidation to dibenzothiophene sulfone (DBT–SO_2). Since these oxidized sulfur compounds are more polar than DBT, a suitable extraction step was employed to facilitate their separation from the nonpolar model-oil phase prior to quantitative analysis, depending on the selected analytical procedure.

The catalytic performance of the V/SiO_2 catalysts was compared with that of the bare SiO_2 support and a blank reaction performed in the absence of catalyst under otherwise identical conditions. These control experiments were used to distinguish the contribution of the silica support and the oxidant from the catalytic effect of the supported vanadium species. The catalyst exhibiting the highest DBT removal under the selected optimum conditions was subsequently subjected to repeated reaction cycles to evaluate its stability and reusability. The obtained results were compared with previously reported vanadium-containing silica and other supported metal-oxide catalysts investigated for the oxidative conversion of DBT using hydrogen peroxide or related oxidants.[18–21]

1-4- Investigation of the Factors Affecting DBT Removal

The effects of selected experimental parameters on the oxidative conversion of DBT over the V/SiO_2 catalyst were investigated under controlled reaction conditions. The parameters selected for the catalytic study included reaction time, reaction temperature, catalyst dosage, and the H_2O_2/DBT molar ratio. The effect of reaction time was investigated by withdrawing samples at different reaction times while maintaining the other reaction conditions constant. The influence of temperature was evaluated over a selected temperature range to determine its effect on DBT removal. The effect of catalyst dosage was studied by varying the amount of V/SiO_2 while maintaining a constant initial DBT concentration and H_2O_2/DBT molar ratio. Finally, the H_2O_2/DBT molar ratio was varied to determine the amount of oxidant required to achieve efficient DBT oxidation. For each experiment, the residual DBT concentration was determined using the established UV–Vis calibration curve, and the DBT removal percentage was calculated from the initial and residual DBT concentrations. The optimum reaction conditions were selected based on the highest DBT removal while minimizing the required catalyst and oxidant amounts. Blank experiments without

catalyst and control experiments using the unmodified SiO_2 support were performed under identical conditions to distinguish the catalytic contribution of the supported vanadium species from the effects of H_2O_2 and the silica support.

2. RESULTS AND DISCUSSION

3-1- FT-IR

The FT-IR spectrum of the prepared V/SiO_2 catalyst exhibited characteristic absorption bands at approximately 1095, 948, and 798 cm^{-1}. The intense band observed at 1095 cm^{-1} can be assigned to the asymmetric stretching vibration of the Si–O–Si bonds, representing the characteristic siloxane network of the silica support. The band at approximately 798 cm^{-1} is attributed to Si–O–Si vibrational modes associated with the silica framework. In addition, the absorption band observed at around 948 cm^{-1} can be associated with V–O-related vibrations, particularly the stretching vibration of V=O groups in dispersed vanadium oxide species supported on the silica surface. Similar absorption bands in the 920–950 cm^{-1} region have been reported for silica-supported vanadium oxide species and attributed to polymeric vanadyl/vanadate surface species.[22]

The presence of this band, together with the characteristic Si–O–Si bands, supports the successful incorporation of vanadium oxide species onto the silica support. However, FT-IR alone does not allow definitive determination of the oxidation state or dispersion of vanadium species.

3-2- BET Surface Area and Porous Properties

The textural properties of the prepared SiO_2 and $V–SiO_2$ catalysts were evaluated by N_2 adsorption–desorption analysis, and the obtained results are summarized in Table 1. The bare SiO_2 support exhibited a relatively high BET surface area of 340 $m^2\ g^{-1}$, with a total pore volume of 0.51 $cm^3\ g^{-1}$ and a BJH average pore diameter of 6.8 nm. After incorporation of vanadium, a gradual decrease in the textural properties was observed. The BET surface area decreased to 328, 230, and 226 $m^2\ g^{-1}$ for the 1, 3, and 5 wt% $V–SiO_2$ catalysts, respectively. Similarly, the total pore volume decreased from 0.51 $cm^3\ g^{-1}$ for SiO_2 to 0.49, 0.43, and 0.42 $cm^3\ g^{-1}$ with increasing vanadium loading. The BJH pore diameter also decreased from 6.8 nm for the unmodified silica to 6.3 nm for 1% $V–SiO_2$ and 5.8 nm for both 3% and 5% $V–SiO_2$. The observed decrease in surface area and pore volume with increasing vanadium loading may be attributed to the deposition of vanadium oxide species on the silica surface and/or within the pore structure, which can partially block the pores and reduce the accessible surface area. Nevertheless, all prepared materials retained pore diameters within the mesoporous range, indicating that the incorporation of vanadium did not destroy the mesoporous character of the silica support.[23]

Table 1: BET surface area, total pore volume, and BJH average pore diameter of the prepared SiO_2 and V/SiO_2 catalysts.

Sample	BET surface area $S_{BET}/m^2.g^{-1}$	Pore volume $V_{total}/cm^3.g^{-1}$	BJH pore size D_{BJH}/nm
SiO_2	340	0.51	6.8
1% V- SiO_2	328	0.49	6.3
3% V- SiO_2	230	0.43	5.8
5% V- SiO_2	226	0.42	5.8

3-3- Catalytic Performance of V/SiO_2 in Oxidative Desulfurization

The catalytic activity of the prepared V/SiO_2 catalysts was evaluated in the oxidative desulfurization of a DBT-containing model fuel using H_2O_2 as the oxidizing agent. The results obtained under the investigated conditions are summarized in Tables 2–6 and Figures 1-5. The catalytic performance was evaluated based on the percentage of DBT removal, calculated from the difference between the initial and residual DBT concentrations. The blank experiment and the unmodified SiO_2 support showed negligible activity compared with the V/SiO_2 catalysts, indicating that the presence of supported vanadium species plays an important role in the oxidation of DBT. Among the investigated catalysts, the catalyst containing 3 wt% V exhibited the highest catalytic activity, achieving a DBT removal of 87.55% under the optimum reaction conditions.

The effect of reaction time indicated that DBT removal increased with increasing reaction time up to 60 min, after which the conversion showed a slight increase. This behavior may be attributed to the progressive oxidation of DBT by the active vanadium species in the presence of H_2O_2. Increasing the reaction temperature from 30 to 60 °C resulted in an increase in DBT removal, suggesting that temperature influences the rate of the oxidation process. Similarly, increasing the catalyst dosage from 0.05 to 0.2 g resulted in an increase in DBT removal, which can be associated with the availability of a greater number of active sites. The effect of the H_2O_2/DBT molar ratio showed that increasing the oxidant amount improved DBT removal up to an optimum ratio of 6, whereas further addition of H_2O_2 produced no significant improvement in the removal efficiency.

Table 2: Effect of vanadium loading on the catalytic activity

Catalyst	V loading (wt%)	DBT removal (%)
SiO_2	0	0
V/SiO_2-1	1	37.15
V/SiO_2-3	3	79.71
V/SiO_2-5	5	75.58

Table 3: Effect of reaction time.

Time (min)	DBT removal (%)
15	23.58
30	57.45
60	84.51
90	86.09
120	86.79

Table 4: Effect of temperature.

Temperature (°C)	DBT removal (%)
30	42.01
40	59.15
50	73.81
60	87.56

Table 5: Effect of catalyst dosage.

Catalyst dosage (g)	DBT removal (%)
0.05	33.17
0.10	51.14
0.20	82.54
0.30	78.21

Table 6: Effect of H_2O_2/DBT molar ratio.

H_2O_2/DBT molar ratio	DBT removal (%)
1	8.57
2	29.1
4	70.58
6	84.54
8	84.99

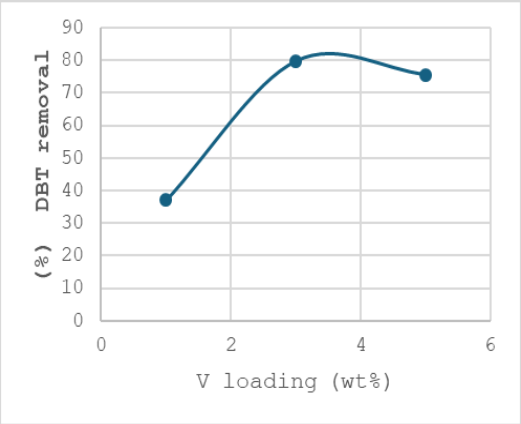

Figure 1: Effect of vanadium loading on the catalytic activity.

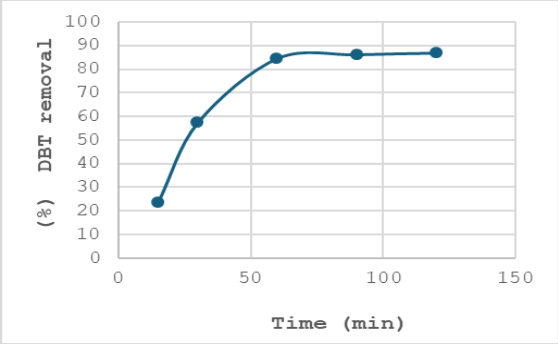

Figure 2: Effect of reaction time.

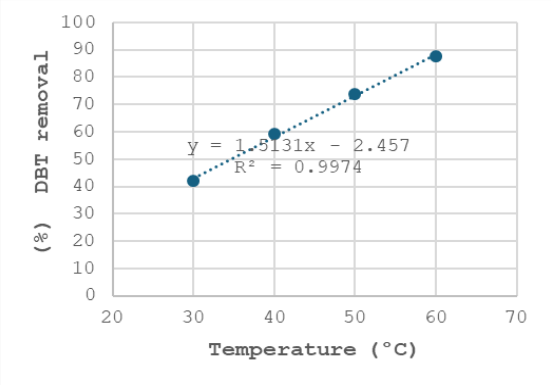

Figure 3: Effect of temperature.

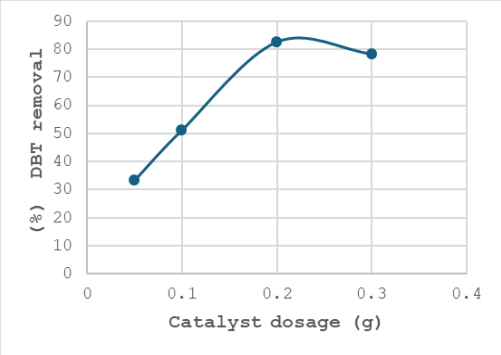

Figure 4: Effect of catalyst dosage.

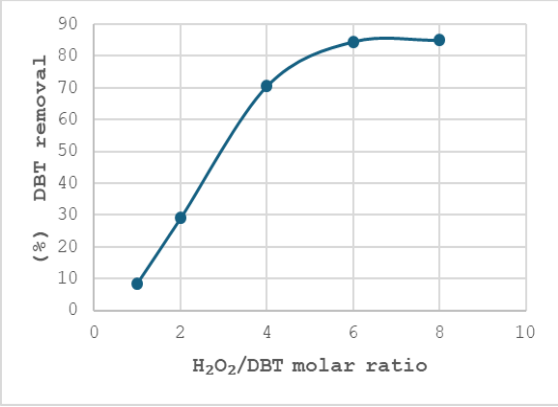

Figure 5: Effect of H_2O_2/DBT molar ratio.

Under the optimum conditions of 60 °C, 60 min, 0.2 g, and H_2O_2/DBT molar ratio of 6, the highest DBT removal of 94.8% was obtained. The improved catalytic

performance of V/SiO_2 can be attributed to the ability of dispersed vanadium oxide species to activate H_2O_2 and promote the oxidation of DBT to more polar oxidized sulfur species, mainly DBT sulfoxide and DBT sulfone. The catalytic activity and stability of the optimum catalyst were further evaluated through repeated reaction cycles Table 7. The catalyst retained 79.4% of its initial activity after 4 consecutive cycles, indicating good reusability. Overall, the results demonstrate the potential of the prepared V/SiO_2 material as a heterogeneous catalyst for oxidative desulfurization.

Table 7: DBT removal efficiency and catalytic activity retention of V/SiO_2 over four consecutive cycles.

Cycle	DBT removal (%)	Activity retention (%)
1	94.8	100
2	87.01	91.7
3	81.4	85.8
4	75.3	79.4

3. CONCLUSION

V/SiO_2 catalysts with nominal vanadium loadings of 1, 3, and 5 wt% were successfully prepared by sol–gel synthesis of the silica support followed by impregnation with NH_4VO_3. FT-IR analysis confirmed the characteristic silica framework through the absorption bands at 1095 and 798 cm^{-1}, while the band at approximately 948 cm^{-1} was associated with vanadyl/vanadium oxide species supported on the silica surface. The BET results showed that the pristine SiO_2 possessed a surface area of 340 $m^2\ g^{-1}$, a pore volume of 0.51 $cm^3\ g^{-1}$, and an average pore diameter of 6.8 nm. Following vanadium incorporation, the surface area decreased to 328, 230, and 226 $m^2\ g^{-1}$ for 1, 3, and 5 wt% V–SiO_2, respectively, accompanied by decreases in pore volume to 0.49, 0.43, and 0.42 $cm^3\ g^{-1}$ and in average pore diameter to 6.3, 5.8, and 5.8 nm. Despite these changes, the prepared materials retained their mesoporous characteristics.

The prepared catalysts exhibited catalytic activity toward the oxidative removal of DBT using H_2O_2. Among the investigated materials, 3 wt% V/SiO_2 showed the highest catalytic performance. Under the optimum reaction conditions of 60 °C, 60 min, 0.2 g catalyst, and an H_2O_2/DBT molar ratio of 6, a maximum DBT removal of 94.8% was obtained. The enhanced catalytic activity is attributed to the presence of dispersed vanadium oxide species capable of promoting the oxidation of DBT in the presence of H_2O_2. After four consecutive reaction cycles, the catalyst retained 79.4% of its initial activity, indicating good stability and reusability. Overall, the results confirm the potential of V/SiO_2 as a simple and effective heterogeneous catalyst for the oxidative removal of refractory sulfur compounds under relatively mild conditions.

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