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Catalyst Support Strategies: Encapsulation of Polyoxometalate Catalysts in Magnetic Particles in Oxidative Desulphurization

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High sulphur levels in liquid fuels increase sulphur oxide emissions upon combustion, exacerbating environmental and health risks. This makes desulphurization critical in fuel refining processes to reduce sulphur content. Among several methods, oxidative desulphurization (ODS) has become increasingly recognized due to its mild operating conditions and efficiency in the removal of refractory sulphur compounds, such as dibenzothiophene (DBT) and benzothiophene (BT), from fuels. The sulphur conversion efficiency of ODS is influenced by several key parameters: reaction duration, operating temperature, the type and concentration of oxidant, and the nature and dosage of the catalyst. While polyoxometalates (POMs) exhibit excellent catalytic activity for ODS, their application is limited by their homogeneous nature and poor recoverability. To address these challenges, recent advancements have focused on enhancing their performance through heterogeneous support. The encapsulation of POMs in magnetic particles has been shown to increase their structural stability and accessibility to active sites, contributing to sustained catalytic performance. The magnetic properties also contribute to simplifying catalyst recovery and reuse through magnetic separation, eliminating the need for complex and energy-intensive processes. This review highlights the recent advancements in using magnetic particles as heterogeneous support for POM catalysts, emphasizing their role in stabilizing POM catalysts while improving the sustainability of the ODS process.

1. Introduction

The continuous rise in global energy demand, particularly for fossil fuels, necessitates the implementation of cleaner fuel production in the refining industry to mitigate environmental impacts and comply with strict government regulations. In the Philippines (PH), the Department of Energy (DOE) and the Department of Environment and Natural Resources (DENR) developed the Philippine National Standard for Petroleum Products, adopting Euro 4-PH specifications. This restricts sulphur content to 50 ppm in gasoline and automotive diesel fuels. (DOE, 2023). Other regions have implemented more stringent regulations. The European Union (EU) and the United States (US) have a sulphur limit of 10 ppm in fuels as mandated by the European Parliament (Directive 2009/30/EC, 2009) and the US Environmental Protection Agency (EPA) (Tier 3 Gasoline Sulfur Regulations, 2016), respectively.If untreated, high sulphur levels contribute to increased sulphur oxides (SOx) emissions upon combustion, increased risk of acid rain, and potential respiratory health risks (Haruna et al., 2022). Sulphur-containing compounds (SCCs) can cause equipment corrosion, engine deterioration, and deactivation of fuel cell electrodes and catalytic converters (Beshtar et al., 2025).

Desulphurization, the removal of sulphur from fuels, is essential for improving emission control and reducing the environmental effects of sulphur pollutants. Common technologies include hydrodesulphurization (HDS) (Alwan et al., 2024), absorptive desulphurization (ADS) (Alcaraz et al., 2024), and oxidative desulphurization (ODS)

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(Haboc et al., 2024). These processes are vital not only for supplying sulphur for industrial use but also for enabling sulphur-free feedstocks in catalytic processes (Correa et al., 2024).

HDS is the most widely used method on the industrial scale. It effectively removes smaller sulphur compounds such as sulphides, disulphides, and thiols (Zaidi et al., 2023). However, it is unable to target sterically hindered compounds such as benzothiophene (BT), dibenzothiophene (DBT), and 4,6-dimethyl-dibenzothiophene (4,6-DMDBT), leading to conversion inefficiencies and high energy use (Zhang et al., 2023). It requires high operating conditions, typically temperatures ranging from 300 °C to 400 °C and pressures of 20 to 100 atm H_2, leading to high energy consumption and operational cost. Catalyst deactivation due to hydrocarbon buildup and resistance of complex sulphur molecules further limits its effectiveness (Pham et al., 2024).

These limitations drive the interest and need for alternative desulphurization methods that operate in milder conditions and provide greater selectivity. ODS is effective for removing refractory sulphur compounds like BT, DBT, and 4,6-DMDBT under mild conditions by converting them into extractable sulfoxides or sulfones using catalysts and oxidants.

Polyoxometalates (POMs), known for their redox activity, thermal stability, and selectivity, are effective ODS catalysts (Zhang et al., 2023). However, their homogeneous nature complicates recovery and limits reuse. Various immobilization strategies, including magnetic encapsulation, have been developed to address this, enabling easier separation with sustained catalytic performance.

This review aims to present and discuss recent developments using magnetic-particle support for POM-based catalysts in ODS systems. While previous reviews have broadly analyzed ODS mechanisms, oxidants, or homogeneous catalysts, fewer have explicitly focused on integrating magnetic materials for POM immobilization. This review addresses that gap by highlighting how magnetic encapsulation can enhance POM stability, catalytic efficiency, and reusability, all of which are key aspects for improving the sustainability and practical feasibility of ODS technologies.

The paper is structured as follows: Section 2 presents an overview of ODS; Section 3 discusses POM catalysts in ODS; Section 4 analyzes magnetic encapsulation techniques; and Section 5 concludes and outlines future research directions.

2. Oxidative Desulphurization

ODS offers a milder and more selective approach to desulphurization. First introduced by Heimlich & Wallace (1966) through the oxidation of DBT using hydrogen peroxide-acetic acid solutions, ODS has been extensively studied to improve its efficiency and optimize the ODS process by exploring various oxidants and catalysts. The ODS process can target refractory sulphur compounds (BT, DBT, and 4,6-DMDBT) that are resistant to the reactions employed in the traditional HDS method.

The process proceeds in two steps: (1) the oxidation of sulphur compounds into polar sulfones and sulfoxides, and (2) the removal of the oxidized compounds through methods such as solvent extraction that utilize their difference in polarity with hydrocarbons. A significant advantage of ODS is its mild operating condition requirements, which are performed at ambient pressure and relatively low temperatures below 100 °C, leading to reduced energy consumption, lower operating costs, and improved environmental compatibility.

ODS utilizes oxidants to convert sulphur compounds into extractable forms. Widely adopted oxidants include hydrogen peroxide (H_2O_2) and tert-butyl hydroperoxide (TBHP). H_2O_2 stands out for its strong oxidizing power, cost-effectiveness, wide accessibility, and environmentally friendly nature. It is rich in active oxygen and decomposes into water as its only by-product, eliminating harmful side products and minimizing secondary pollution (Teong et al., 2019).

Catalysts are critical in enhancing oxidant performance in ODS by lowering the activation energy and increasing the reaction rate (Gao et al., 2018). Various catalysts were explored, including metallic oxides (Rivoira et al., 2017), ionic liquids (Wang et al., 2018), photocatalysts (Beshtar et al., 2025), and polyoxometalates (POMs). From this selection, POMs have shown to be the most promising due to their sulphur removal efficiency for model oils and real fuels (Wang et al., 2021) and shown to have an effective performance when used with H_2O_2 as an oxidant (Choi et al., 2016).

Despite the mild conditions of ODS, several challenges limit its application in the industry. The main limitation is the extensive usage of catalysts due to the difficulty of separating and recovering homogeneous catalysts after the reaction, resulting in increased operational costs, chemical usage, and waste produced. The biphasic nature of the ODS reaction also contributes to the lower mass transfer efficiency between the hydrocarbon and aqueous phases, reducing the overall efficiency. These issues highlight the need for the development of heterogeneous catalyst systems that can address these limitations without compromising the catalytic activity.

3. Polyoxometalate Catalysts

Polyoxometalates (POMs) are anionic metal-oxygen clusters that consist of transition metal ions, typically molybdenum (Mo), tungsten (W), or vanadium (V), connected through shared oxygen atoms (Zhang et al., 2021). Their structure is often modified through the substitution of different metal ions or heteroatoms, resulting in the formation of different POM archetypes such as Keggin, Wells–Dawson, Lindqvist, and Anderson–Evans (Anyushin et al., 2020). These are widely used as catalysts in ODS due to their distinctive properties, including high thermal stability, redox activity, and resistance to oxidative decomposition (Li et al., 2018).

POMs offer several advantages in ODS applications, including their strong electrophilic oxygen centers, allowing for the rapid oxidation of sulphur compounds under mild conditions. Their tunable redox potentials and acid-base properties facilitate the matching of the catalytic activity to SCCs in different types of fuel (Deng et al., 2023). This is exhibited in a study by Zhang et al. (2023) wherein a POM-based catalyst $[C_{16}H_{33}N(CH_3)_3]_3PW_4O_{24}$ was synthesized and demonstrated a 91.28 % sulphur removal efficiency within 1 h when tested against model oils containing BT, DBT, and 4,6-DMDBT.

Despite these promising results, homogeneous POM catalysts have limitations, including small surface areas, a lower number of active sites, and high solubility in polar solvents, which hinder their separation and reuse (Liu et al., 2020). Their solubility limits easier recovery methods, such as filtration, and instead require more complex processes, such as solvent evaporation and other extraction techniques. In addition to this, homogeneous catalysts are prone to leaching during the reaction, resulting in the contamination of the product and reduced sustainability. These limitations contribute to increased operational costs, environmental impact, and waste generation.

To address these issues, heterogeneous POM catalysts have been developed using solid supports to enhance performance and facilitate recovery (Ng et al., 2024). Supports with high surface areas allow for increased catalytic phase deposition, whereas supports with low surface areas are prone to aggregation and obstruction of diffusion pathways (Guntida et al., 2024).

Various solid supports have been explored, including metal-organic frameworks (MOFs) (Haruna et al., 2023), mesoporous silica (Wu et al., 2024), magnetic particles (Gao et al., 2023), and carbon-based supports (Liu et al., 2025), among others. Among these, magnetic encapsulation is a favoured approach for its ability to facilitate easier catalyst separation and recovery using an external magnetic field, eliminating the need for more complex processes. Its operational simplicity, combined with its high catalytic activity and recoverability, makes it well-suited for ODS systems. Moreover, by overcoming the challenges presented by homogeneous POM catalysts, magnetic encapsulation offers a practical and economically feasible solution for advancing ODS processes.

4. Magnetic Encapsulation

The encapsulation of the catalyst plays a critical role in maintaining the stability of the active phase, directly affecting catalytic performance and durability (Guntida et al., 2024). Several encapsulation techniques are employed to immobilize the POM catalyst within the magnetic support. Table 1 summarizes the common encapsulation methods, including co-precipitation (Zhang et al., 2023), impregnation (Gao et al., 2023), sonication (Kermani et al., 2021), and electrostatic bonding (Liu et al., 2020), along with their descriptions, advantages, and limitations. These encapsulation methods are widely applied in POM-based catalysis for their practicality, reproducibility, and compatibility with a variety of POM structures.

Table 1: Common Encapsulation Methods

Encapsulation Methods	Description	Advantages	Limitations
Co-precipitation	Precipitation of POM and magnetic support to form a composite	Straightforward process and good dispersion of the POM in the magnetic support	Possible aggregation and uneven distribution, leading to decreased catalytic activity
Impregnation	Addition of magnetic support in POM solution, followed by drying	Flexible loading capacity and higher structural stability	Possible leaching and uneven distribution of POM
Sonication	Utilization of ultrasonic waves to ease the dispersion of the POM and magnetic particles	Rapid encapsulation, good dispersion, and reduced risk of agglomeration	Requires combination with other encapsulation methods; may damage POM structure
Electrostatic Bonding	Binding of POMs to magnetic supports through electrostatic interactions	Strong bond interactions and reduced risk of leaching	Decreased stability under harsh reaction conditions; sensitive to changes in pH

Magnetic encapsulation offers several advantages, including enhanced mechanical stability, as the magnetic matrix protects the active sites of the catalyst from thermal and chemical degradation throughout the reaction, without compromising its structure or activity (Liu et al., 2020). This contributes to the sustained performance of the catalyst over multiple consecutive cycles. The recovery is also significantly simplified with an external magnetic field, eliminating the need for complex processes. Aside from minimizing cost, this also reduces energy and resource consumption, making the process more sustainable. Encapsulation also contributes to an increased surface area, which improves its dispersion across the reaction medium and enhances the contact between the catalyst and reactants. This facilitates mass transfer by preventing POM aggregation and promoting uniform dispersion.
Table 2 provides a summary of magnetic POM-based catalysts, highlighting the operating parameters, desulphurization performance, and reusability. Operating parameters include reaction temperature (50-90 °C), time (30-120 min), oxidant-to-sulphur molar ratio (typically 3-6), and catalyst loading (expressed as %wt or g catalyst/g model fuel). Desulphurization efficiency is expressed as percent sulphur conversion, while reusability is evaluated by the number of consecutive catalytic cycles completed before a noticeable decline in performance is observed, indicating the stability and longevity of the catalyst.

Table 2: Summary of Literature on Magnetic Particles-Encapsulated POM Catalysts

Reference	Catalyst	Oxidant	Operating Parameters	Maximum Sulphur Conversion	ODS Cycles
Dou et al. (2022)	HPW@mSiO_2/GO-HDA	H_2O_2	60 °C, 120 min, 6 O/S, 3 %wt catalyst, 120 min	99.21 % (DBT, 4,6-DMDBT, BT) 97.22 % (real oil sample)	5
Gao et al. (2023)	$(Bu_4N)_3PW_4$@Fe_2O_3/C	H_2O_2	70 °C, 60 min, 3 O/S 70 °C, 30 min, 3 O/S 70 °C, 50 min, 3 O/S 70 °C, 50 min, 3 O/S	97.9 % (BT) 99.9 % (DBT) 99.9 % (4-MDBT) 99.6 % (4,6-DMDBT)	8
Gooneh-Farahani & Anbia (2024)	Fe_3O_4@CTS@PMoW	H_2O_2	60 °C, 120 min, 5 O/S	99 % (DBT, C, BT)	5
	Fe_3O_4@CTS@HPWV	H_2O_2	60 °C, 120 min, 5 O/S	97 % (DBT, 4,6-DMDBT, BT)	5
Kermani et al. (2021)	MSN/IL-{Mo_{132}}	H_2O_2	62.5 °C, 30 min, 20.5 O/S, 0.0065 g cat/g MF	99.97 % (DBT)	6
Liu et al. (2020)	Fe_3O_4@CS@PMo_{12}	H_2O_2	60 °C, 0.1 g catalyst, 60 min	99.7 %	5
Nejati et al. (2022)	Co_4-POM@APTES-(Fe_3O_4/GO)	H_2O_2	50 °C, 30 min, 30 %wt catalyst, 4 O/S	100 % (DBT)	5
Zhou et al. (2015)	MoO_3/Fe_3O_4	TBHP	90 °C, 3 h, 4 O/S	99.5 %	10

The studies utilized different experimental setups and catalysts, resulting in variations in optimized operating conditions. This diversity reflects the varying encapsulation strategies used to immobilize the POMs. The high reusability of heterogeneous catalysts is evident, with most catalysts maintaining consistent performance across 5 to 10 cycles. Under optimal conditions, magnetic POM catalysts consistently achieve high sulphur conversion rates, often exceeding 97 %, demonstrating their effectiveness and durability in ODS.
Although magnetic encapsulation shows great promise in ODS applications, it presents several limitations. Agglomeration of magnetic particles can reduce dispersion, block active sites, and eventually lead to a decline in catalytic efficiency (Gutierrez et al., 2024). Moreover, the chosen encapsulation method may not form a uniform coating or distribution over the surface of the POM catalyst, resulting in an inconsistent particle size distribution and uneven catalyst loading. These issues emphasize the need for careful design and comprehensive characterization of the catalyst support to ensure its stability and performance.

5. Conclusion

The encapsulation of POM catalysts within magnetic particles is a promising strategy to overcome the limitations of conventional homogeneous catalysts in ODS processes. Although POM catalysts are effective in sulphur removal in fuels, their recovery and reuse remain critical limitations to their applicability in large-scale industrial systems. Immobilizing POMs onto magnetic supports converts them into a heterogeneous catalyst, significantly improving structural stability and making catalyst recovery easier through magnetic separation. Aside from extending their operational life, this strategy also contributes to waste reduction and lower operational costs, which are important considerations in the development of a cleaner and more sustainable fuel refining process. Moreover, the ease of separation makes it a strong prospect for integration into continuous or semi-continuous desulphurization systems. However, there are still limitations to be addressed. Current studies often evaluate catalyst performance using model fuels rather than real fuels with complex composition and characteristics. The testing of the catalyst under real-fuel conditions and large-scale operations will be critical to evaluate its feasibility for industrial applications. Future studies may also focus on optimizing encapsulation techniques. Hybrid support systems combining magnetic particles with other supports, such as MOFs and mesoporous silica, may be explored to further enhance catalyst performance. Moreover, the long-term environmental impacts of magnetic catalyst systems must be investigated to ensure regulatory compliance. Addressing these limitations is critical for advancing magnetically encapsulation POMs as a commercially viable and sustainable green desulphurization technology.

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